

THE MICROSTRUCTURE OF UK MUDROCKS

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By

STEPHEN WILKINSON

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Department of Civil and Environmental Engineering
Imperial College London

ABSTRACT

The microstructures of Jurassic and Cretaceous mudrocks reflect their environment of deposition and the processes which occurred during their burial. One measurement of mudrock microstructure is particle long axis orientation. Preferred particle orientations are measured by analysis of SEM images of broken mudrock surfaces. Strong preferred particle orientations are observed in unweathered material with high quantities of illite. They are observed across a range of burial depths within Cretaceous and Jurassic materials. No preferred orientation is observed where a material is weathered, or where large quantities of silt are present. Moderate preferred orientations are observed where materials are carbonate rich such as with the Blue Lias, or are shallowly buried such as with the London Clay. Strong preferred particle orientations formed through particle rearrangement are created at greater burial depths than the London Clay samples (200m) and at shallower depths than the Gault Clay samples (500m).

Microstructures form in response to each event in a mudrocks history. The final structure of a mudrock is dependent on all of the events in its history. The relative effect of each event is dependent on its intensity of its application to the mudrock. Each event has the possibility of destroying structures formed by previous events. For example burial microstructures often replace depositional structures. Particles will align by rearrangement as much as possible during initial consolidation. Further alignment can occur by the process of pressure solution at a much greater depth. Fluids rich in chemical ions may deposit cements in the pores of mudrocks as they flow through them. The growth of these cements can make further alignment of clay mineral particles impossible.

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The Lias Clay samples were taken from Junction 13 of the M5. The samples were taken as part of a site investigation, and samples were donated to the project. These samples were granted to us by Ian Duncan, Rob Bailey and Francesca Ryan of Interoute and Rob Ewens of Geotechnical Engineering

Stratigraphic information

Much of the geological information, which forms one of the foundations to this work, was obtained from the British Geological Survey. Helen Reeves, Peter Hobbs and Lee Jones were of great assistance in this endeavour. Many of the samples obtained for this work could not be dated using macrofossils, the microfossils in the Gault and Kimmeridge Clays were identified by Ian Wilkinson of the BGS, while the microfossils of the Lias Clay were identified by Philip Copestake from Merlin Energy Resources Ltd. The mineralogical analysis of all of the samples was carried out by Jenny Huggett of Petroclays.

Imaging and image analysis

All information on the various available imaging techniques came from members of the Society of Electron Microscope Technology. I am indebted to David McCarthy the secretary of the Society for his help in identifying the appropriate expertise. My early introduction to the scanning electron microscope was under the instruction of Tony Brain from the London Centre for Ultrastructural Imaging, and Dean Edwards of Carl Zeiss

A large number of imaging techniques were tested using the Oxford Clay. The following people are thanked for their introduction to and assistance with various imaging techniques: Martin Spitaler (Imperial College) - confocal microscope; Richard Sweeney (Imperial College) - x-ray goniometer. Eyal Miller carried out an investigation into the application of the atomic force microscope to the Oxford Clay; Alex Ball and Lauren Howard (Natural History Museum) - x-ray ultramicroscope; Javier Cuadros (Natural History Museum) - particle orientation analysis using x-ray diffraction; and Brian Kyte (Alicona) - infinite focus optical microscope apparatus.

The majority of the imaging for this project was carried out at the University of Glasgow using devices which are maintained to the highest standard by Peter Chung. The quality of the images obtained is a result of Peter's hard work organising the best conditions for sample preparation and imaging

The majority of the 3D processing was undertaken using AxioVision macros for this program were written by Paul Sheppard of Imaging Associates. Without these macros the advanced image processing would not have been possible.

Draft improvement

Early drafts of this thesis were read by Jennie Wilkinson and Benjamin Gardner their effort and the suggestions they have put forward have greatly improved the presentation of this thesis.

Funding

This project was only possible because of funding from EPSRC

CHAPTER 1

INTRODUCTION

Mudrocks are the most common global rock type, making up about 50% of the sedimentary rocks in the geological record (Boggs, 2005), and approximately 50% of the rocks outcropping in the southern UK are mudrocks (Figure 3.10). Because of their relative weakness, they are situated in valleys surrounded by higher ground of more resistant limestones and sandstones (Lane et al., 2008). Many cities are built on mudrocks, the UK's transport links rely on them as foundations, and they are quarried for bricks and cement. Old mudrock quarries are often used as landfill sites. Mudrocks are responsible for many major landslides, and their fluid flow properties are important for both hydrogeology and petroleum industries. Thus the correct analysis of behaviour of mudrocks is essential for ground engineering in the UK and across the world.

1.1 Overview

In soil mechanics the microstructure of a mudrock is used to explain unexpected macroscopic behaviour of a mudrock during laboratory investigations (Cotecchia, 1996). The difference in behaviour between undisturbed and reconstituted samples is attributed to changes in particle arrangement or cementation of the mudrock. The relative contributions of these two effects cannot be clearly identified without the use of direct observation of the structure of the mudrock. A successful project has been recently carried out to investigate the relationship between laboratory behaviour and microstructure of the London Clay in the UK (Gasparre, 2005; Gasparre et al., 2007b; Nishimura, 2005; Nishimura et al., 2007). This thesis is part of a project to expand on this work. This thesis covers several of the older mudrocks in the UK, specifically focusing on the Jurassic Lias, Oxford and Kimmeridge Clays and the Cretaceous Gault Clay. These materials are of particular interest because they fall on the boundary between weak rocks and strong soils (Oliveira, 1993). The main emphasis of this thesis is the measurement of mudrock microstructure using image analysis with the aim of relating this to each mudrocks geological history. Two other researchers worked on further aspects of this project. Their work includes the characterisation of the laboratory behaviour of the mudrocks (Brosse, 2011; Hosseini-Kamal, 2011).

1.2 Objectives/Scope

Mudrocks are some of the most poorly understood geotechnical materials due to their variability and small grain size. They are composed of a great number of different minerals, which can be combined in a variety of arrangements. The specific structure a mudrock attains from the possible structures is a result of its geological history. This project analyses the history, microstructure and engineering behaviour of mudrocks in order to study the links between these three factors. This thesis focuses on the geological history and measurement of mudrock microstructure.

The original aims of the project as a whole were:

- Quantify the effects of structure on the mudrocks' engineering properties, particularly their anisotropy;
- Determine explicitly their three-dimensional (3-D) microstructure, using environmental scanning electron microscopy (ESEM), X-ray computed tomography (X-ray CT), 3-D imaging, and mechanical and chemical destructuring;
- Develop micro-mechanical structural models for the clays;
- Generate a general unified framework linking together post-depositional geological processes, micro-mechanical structural models, and mechanical properties (including anisotropy).

The measurement of the engineering properties of these mudrocks was carried out by Bross (2011) and Hossenei-Kamal (2011) and is not presented here. The objectives of this thesis are to:

1. Identify sites to investigate several mudrocks older than the London Clay, focusing mainly on Jurassic age mudrocks;
2. Image the microstructure of these mudrocks using environmental scanning electron microscopy (ESEM), X-ray computed tomography (X-ray CT), and 3-D imaging;
3. Quantify the microstructure of each of these mudrocks;
4. Explore links between post-depositional geological processes and micro-mechanical behaviour.

During the initial stages of the project the use of X-ray CT was discounted due to insufficient resolution to identify individual mudrock particles. Several other imaging methods were also tested (Appendix C). The focused ion beam showed promise, however its cost was beyond the resources of this project. Thus 3D imaging was confined to surface elevation models taken using stereo imaging in the ESEM. This limited the quantification of structure to measurements of preferred particle orientation. The work was further constrained by limited access to appropriate samples for testing/imaging, resulting in a delay while samples were obtained. Because of these constraints the objectives had to be modified. Although indications of mudrock behaviour are identified from images, full models of material behaviour could not be created without the full 3-D data.

A mudrock's laboratory behaviour is caused by the arrangement of its particles, and this arrangement is determined by the depositional and burial history of the mudrock. By measuring mudrocks' structure it is possible both to see the types of structures which occur in geological environments and the engineering response which each structure produces. Mudrock microstructure is a connection between descriptive geological histories, and quantitative engineering properties. If microstructure can be quantified then laboratory behaviour can be predicted based on simple geological observations. Such predictions have the potential to greatly aid engineering design. The key aspect identified in this work is preferred particle orientation which correlates with material anisotropy. This correlation becomes important where bedding dips into a slope or cutting for example.

1.2.1 Site selection

It was assumed that finding construction sites with deep excavations within Cretaceous and Jurassic mudrocks would be a simple matter. This would probably have been the case if an economic recession had not affected the UK at the very beginning of the project. Locating high quality sampling sites turned out to be extremely difficult. It was fortunate that samples of the Oxford Clay could be obtained early in the project.

When it became obvious that sites were not going to be forthcoming, locations in the Gault and Kimmeridge Clays were selected for independent sampling. Early access to High Cross for block sampling allowed samples of Gault Clay to be obtained. These samples contrasted well with the Oxford Clay, as images of joint surfaces (which were

very common in the Gault Clay due to desiccation) had no preferred particle orientation. These two completely different mudrocks allowed the perfecting of the method for measurement of particle orientation. The Kimmeridge Clay took much longer to obtain due to difficulties getting details from the Land Registry about a large area. After the initial analysis of the Gault Clay further samples of deeper non desiccated material were required.

Borehole samples of Gault and Kimmeridge Clay were obtained two years and three months after the start of the project. Borehole samples from the Lias Clay became available shortly afterwards. Boreholes are particularly difficult for the analysis of high magnification structure, because they cut across stratigraphy. Laboratory tested material a metre away from the imaging horizon may not have the same structure as the imaged horizon. Thus multiple imaging samples were required from different depths in each mudrock to allow characterisation. Even so, given the number of mudrocks and the unavoidable delays in obtaining the samples, only a limited number of depths in each mudrock could be imaged. The change in character of each mudrock with depth is therefore uncertain. The observed changes can, and have, where possible, been linked to changes in the geological logs of each mudrock. The late retrieval and use of boreholes for most of the mudrocks created serious scheduling problems for the imaging side of the project.

1.3 *Thesis organisation*

This thesis is arranged into four parts and thirteen chapters. The first part contains chapters 2-4, the second part contains chapters 5-9, the third part contains chapters 10-12. Chapters 13 and 14 conclude the thesis.

The first part (chapters 2-4) is a literature review, summarising the current state of the science in the geological and geotechnical understanding of mudrocks. Chapter two defines mudrocks, chapter three discusses the geological variations in the history of a mudrock prior to sampling and the lateral and stratigraphic limits of the image data presented in the third part chapter four defines structure and microstructure and their development,

The second part (chapters 5-8) contains all data obtained relating to each of the sampled mudrocks, including a brief summary of the literature, and a discussion of the deposition and burial history, details of the work carried out on the sampling sites, mineralogy, microfossils, and sample descriptions. Chapters five, six, seven and eight contain data on the Oxford, Gault, Kimmeridge and Lias Clay respectively.

The third part (chapters 9-11) contains the results from the imaging and image analysis of all the mudrocks. The methods for imaging and the analysis of images are presented in chapter nine. Chapter ten contains the qualitative descriptions of all of the mudrocks and the London Clay. Chapter eleven describes the quantification of the preferred alignment observed in some of the mudrocks.

The fourth part (chapters 12-13) of this thesis summarises the results of these investigations. Chapter twelve discusses the results of the quantification of structure and how they relate to the geological history of the mudrocks. Chapter thirteen contains the conclusions and recommendations for future work.

Finally, all supporting material is presented in a number of Appendices. Appendix A is a glossary of terms used throughout the thesis. Appendix B contains the site investigation methods used on all sites and the borehole logs for the Gault and Kimmeridge Clay sites. Appendix C is an introduction to the imaging techniques on mudrocks tested during the research project. Appendix D is an introduction to the electronic supplement (CD bound into the thesis) which contains all of the image analysis data.

CHAPTER 2

MUDROCK

This chapter describes mudrocks, their mineralogy and their environments of deposition. Mudrocks are complex systems due to their variability, which stems from their definition based on grain size, and from the large number of ways in which geological systems can satisfy this simple criterion.

2.1 Definition

Many varying definitions exist for the classification of fine grained sedimentary rocks (Figure 2.1). Throughout this work a mudrock is considered to be a material which contains greater than 50% silt and/or clay (particles $<63\mu\text{m}$) (ISO 14689-1, 2003). The term has no connotations as to the mudrocks' breaking characteristics (fissile/massive) (Ingram, 1953), nor to its mechanical strength (very soft soil-very strong rock) (Folk, 1974). Mudrock is therefore used as a general term for all fine grained geological materials.

Several other widely used classification systems for mudrocks exist (Figure 2.1). There is a wide agreement for the use of the term shale to describe mudrocks which break easily along layers. Some of the mudrocks studied in this project have a high anisotropy, however they could not quite be described as fissile and so could not be called shale. Although the suffix “-stone” is used to denote massive or non fissile material (Figure 2.1), it has been suggested more recently that mudstone should be reserved for all lithified fine grained material. Unlithified material being known as “mud” (Potter et al., 2005). This allows parity between mudstone and other sedimentary rock descriptors such as sandstone, limestone and dolostone. Some of the mudrocks described in this work have partial cementation, however they are not completely lithified therefore are not mudstones, *sensu stricto*. In engineering the key factors which are used to subdivide mudrocks are grain size, durability and anisotropy (Figure 2.2).

Sedimentary rock containing more than 50% silt and/or clay			
	No connotations as to breaking characteristics	Massive	Fissile
No connotations as to relative amounts of silt & clay	Mudrock	Mudstone	Mudshale
Silt > Clay	Siltrock	Siltstone	Siltshale
Clay > Silt	Clayrock	Claystone	Clayshale

(Ingram, 1953)

Sedimentary rock containing more than 50% silt and/or clay			
Grain size of mud fraction	Soft	Indurated, Nonfissile	Indurated, Fissile
Over 2/3 Silt	Silt	Siltstone	Silt - Shale
Subequal Silt And Clay	Mud	Mudstone	Mud - Shale
Over 2/3 Clay	Clay	Claystone	Clay - Shale

(Folk, 1974)

Classification of mudrocks			
Ideal Size Definition	Field Criteria	Fissile Mudrock	Nonfissile Mudrock
> 2/3 Silt	Abundant silt visible with hand lens	Silt - Shale	Siltstone
1/3 < Silt < 2/3	Feels gritty when chewed	Mud - Shale	Mudstone
> 2/3 Clay	Feels smooth when chewed	Clay - Shale	Claystone

(Blatt et al., 1972)

Percentage Clay Size Constituents			0 - 32	33 - 65	66 - 100
Field Adjective			Gritty	Loamy	Fat or Slick
Indurated	Beds	Greater Than 10mm	Bedded Siltstone	Mudstone	Claystone
	Laminae	Less Than 10mm	Laminated Siltstone	Mudshale	Clayshale

Modified from (Potter et al., 1980)

Figure 2.1 Four different widely used classification systems for mudrocks. Upper left (Ingram, 1953), upper right (Blatt et al., 1972), lower left (Folk, 1974), lower right (Potter et al., 1980). From (Potter et al., 2005).

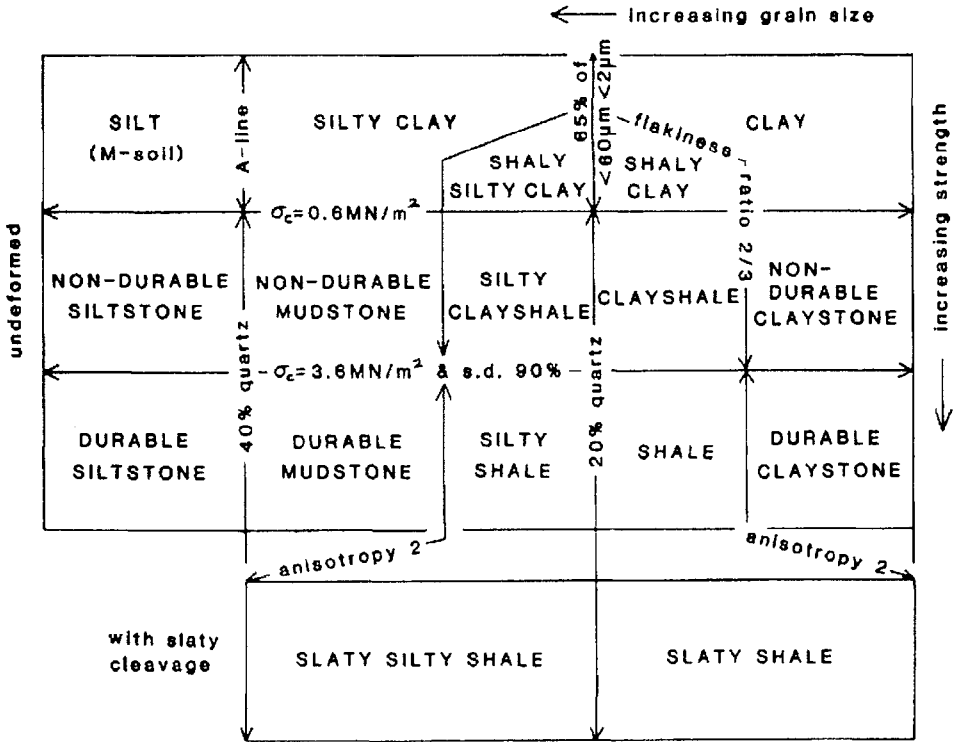


Figure 2.2 Mudrock classification commonly used in engineering (Grainger, 1984).

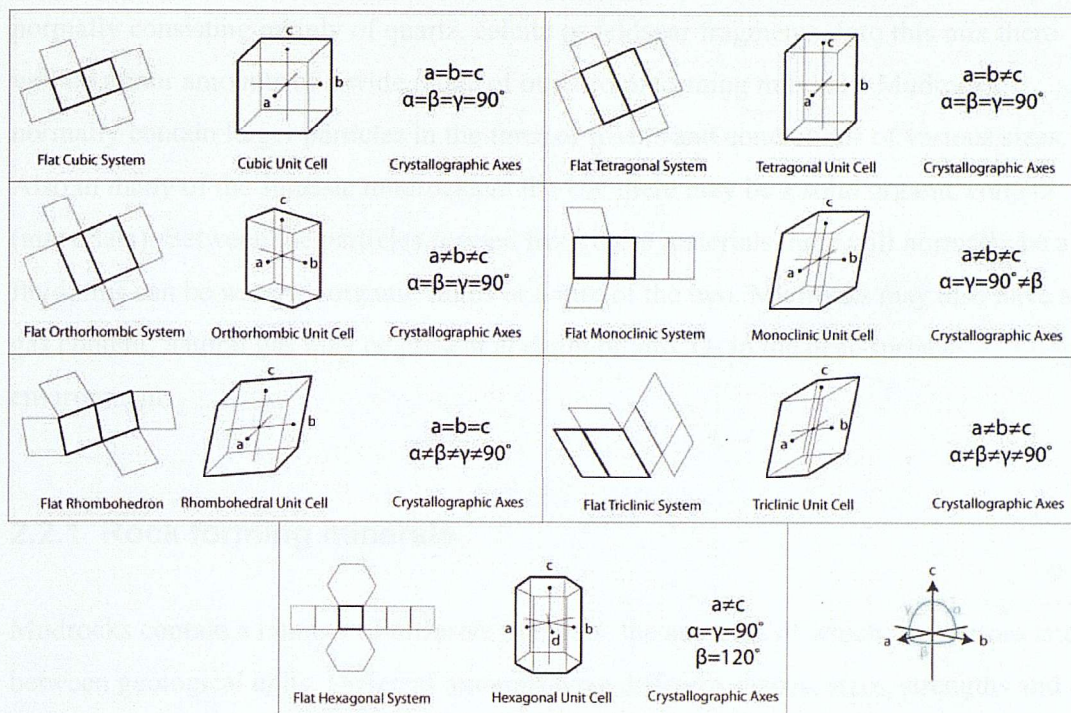


Figure 2.3 Seven crystal unit cell shapes axes a , b , c , d angles between axes α , β , γ . Based on (Whittaker, 1981)

There are many different, but similar definitions for the term “clay” (Appendix A). In the context of grain size the prefix “clay-” is used to describe a rock unit which is predominantly made up of particles $<2\mu\text{m}$ in size. The prefix “silt-” is used to describe a rock unit which is predominantly made up of particles between $2\mu\text{m}$ and $63\mu\text{m}$ in size. The silt content of geological materials tends to vary both laterally as the geological environment of deposition varies and vertically as the environment changes with time, so some acceptance of variability is required within the naming system. For examples the Gault Clay Formation becomes increasingly sandy towards the West of the UK (Jukes Browne, 1900). Thus all the predominantly fine grained materials described in this project are referred to by the general all embracing term mudrock to avoid confusion.

2.2 Composition

As mudrocks are only defined as mudrocks on the basis of the size of their particles there is nothing in their definition to suggest what material they are made of. Using this definition alone they could be composed of any non silicate material, giving a vast range of possible properties and behaviours. In reality mudrocks are composed of a varying quantity of clay minerals (Section 2.2.2) with varying amounts of silt, the latter

normally consisting mainly of quartz, calcite or feldspar fragments. Into this mix there will be minor amounts of a wide range of other rock forming minerals. Mudrocks normally contain larger particles in the form of fossils and concretions of various sizes. Also in many of the Jurassic mudrocks in the UK there may be a solid organic content (macerals). Between the particles formed from these materials there will normally be a fluid, this can be water or organic fluids or a mix of the two. Mudrocks may also have a gas content; natural gas may be present at depth or air/CO₂ in the near-surface environment.

2.2.1 Rock forming minerals

Mudrocks contain a number of different minerals, the amounts of which vary across and between geological units. Different minerals have different shapes, sizes, strengths and stiffnesses (Foster and De, 1971). Thus the variation in quantities of different minerals within a mudrock will greatly affect its engineering properties. In mudrocks which contain a large variety of minerals the different crystal structures can affect how stress is transferred through the system and thus how such systems can be modelled. The shape and structure of each mineral is a result of its chemical composition. Major rock forming minerals can be partitioned, according to their chemistry, into: silicates, carbonates, oxides, sulphides and sulphates. Several rarer chemical classes exist such as the halides which are not found as commonly as rock forming minerals (Press and Siever, 2001). Sulphates are compounds of the sulphate anion (SO₄²⁻) such as gypsum (CaSO₄ · 2H₂O) or anhydrite (CaSO₄). Sulphides are compounds of the sulphide anion (S²⁻) such as pyrite (FeS₂). Oxides are compounds of oxygen and a metallic cation, such as hematite (Fe₂O₃). Carbonates are compounds of the carbonate anion (CO₃²⁻) such as calcite (CaCO₃) or dolomite (CaMg(CO₃)₂). Silicates are compounds of oxygen (O) and silicon (Si) and are the most abundant minerals in the Earth's crust

Crystallographic structures

All minerals form crystalline shapes. The structure of a crystal is defined by its unit cell, this is the smallest three dimensional shape which repeats throughout the crystal. There are seven possible unit cell shapes: Orthorhombic, tetragonal, cubic, monoclinic, triclinic, hexagonal, trigonal (Figure 2.3). Unit cells can be put together to create the final crystal shape (Whittaker, 1981). The strength and stiffness of a crystal is dependent on its shape which in turn is controlled by the geometry of its unit cell.

Silicates

Silicon and oxygen can be combined in a number of ways producing a vast array of mineral groups. The basic building block of silicate minerals is the SiO_4 tetrahedron (Figure 2.4) which has four oxygen atoms forming a triangular based pyramid around a central silicon atom. The arrangement of silica tetrahedra is strongly related to the strength and stiffness of the resulting crystals. The structures formed by silicate tetrahedra determine the planes of weakness within the silicate mineral (the major rock forming minerals). If a mineralogical weakness is aligned in multiple mineral grains then it becomes a weakness (anisotropy) of the rock as a whole.

Silica tetrahedra may be isolated from each other (neosilicates or island silicates) such as the olivines ($(\text{Fe,Mg})_2\text{SiO}_4$). They can be paired (sorosilicates or group silicates) such as the epidotes ($\text{Ca}_2\text{Al}_2(\text{Fe}^{3+},\text{Al})(\text{SiO}_4)(\text{Si}_2\text{O}_7)\text{O}(\text{OH})$). They can form a ring of 6 tetrahedra (cyclosilicates or ring silicates) such as the beryls ($\text{Be}_3\text{Al}_2(\text{SiO}_3)_6$).

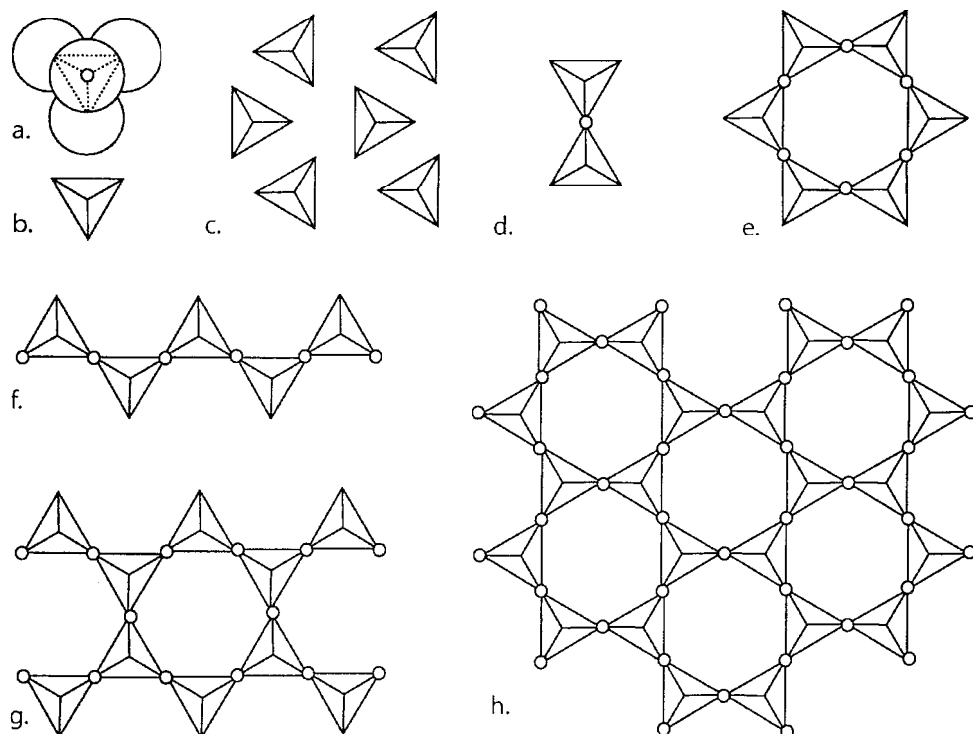


Figure 2.4 The structural arrangement of silicate minerals. a) The SiO_4 tetrahedron, ions approximately to scale. b) a diagrammatic representation of a. c) isolated tetrahedra. d) a pair of tetrahedra sharing one oxygen. e) a ring of tetrahedra. f) a single chain of tetrahedra. g) a double chain of tetrahedra. h) a single sheet of tetrahedra redrawn from (Duff, 2002)

They can form either single or double chains (inosilicates or chain silicates) such as the single chain pyroxenes, *e.g.*, diopside ($\text{MgCaSi}_2\text{O}_6$) or the double chain amphiboles, *e.g.*, tremolite ($\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$). They can form layers (phylosilicates or sheet silicates) such as micas, *e.g.*, biotite $\text{K}(\text{Mg,Fe})_3\text{AlSi}_3\text{O}_{10}(\text{F,OH})_2$. Finally they can form three dimensional structured lattices (tectosilicates or framework silicates) such as quartz SiO_2 (Duff, 2002; Gribble and Hall, 1992) (Figure 2.4). The phylosilicates are very important for the study of mudrocks because they form the clay minerals. As clay minerals have a layered structure, mudrocks containing large amounts of clay minerals will have a clear propensity towards anisotropy in their mechanical behaviour.

2.2.2 Clay minerals

Clay minerals are normally the major constituents of mudrocks. They are phylosilicates, formed from tetrahedral silicate layers joined together by octahedral layers of gibbsite (Al-OH) or brucite (Mg, Fe - OH). The clay minerals produced by the combination of these layers are classified by the ratio of tetrahedral to octahedral layers in the basic units of the material (Figure 2.5). The basic types of clay mineral have silica:gibbsite/brucite ratios of 1:1, 2:1, and 2:2. 1:1 minerals are groups of one octahedral and one tetrahedral layer, separated by an interlayer space which is normally filled with water and various ions (Figure 2.5). The ions in the interlayer space commonly form a double layer of charged ions close to the clay mineral surface (Figure 2.6) Kaolin and halloysite are good examples of this. Both of these are chemically similar, the main difference is the layers in halloysite are normally less ordered and separated by water molecules. The 2:1 group of minerals contain two tetrahedral layers for every octahedral layer. Pyrophyllite, smectite, vermiculite and illite are all very similar the difference between them is the ions present in their interlayer space. The main example of a 2:2 mineral is chlorite. Chlorite is similar in form to a 2:1 mineral except that instead of having ions in the inter layer space chlorite has another octahedral layer. Thus it has a ratio of one tetrahedral to one octahedral layer, it is assigned 2:2 to distinguish it from the mechanically different 1:1 materials. Chlorite although similar in structure to the clay minerals forms macroscopic crystals, due to its size it is not considered a true clay mineral (Gribble and Hall, 1992).

Mineral	Width (w) (μm)	Thickness	Plasticity Index (%)	Activity	Cation exchange capacity (meq/g)
Quartz sand	2000 - 60	$\sim w$	-	-	-
Kaolinite	1.1 - 0.4	$\sim 0.2w$	~ 40	~ 0.4	0.03-0.06
Illite	2.0 - 0.2	$\sim 0.05w$	~ 90	~ 0.9	0.1-0.4
Calcium Montmorillonite	1.0 - 0.01	$\sim 0.01w$	~ 150	~ 1.5	0.8-1.5

Table 2-1 Typical properties for clay minerals in comparison to quartz. Note that values are estimated from samples with >90% of each mineral. Samples of clay minerals are never pure (Bergaya et al., 2006).

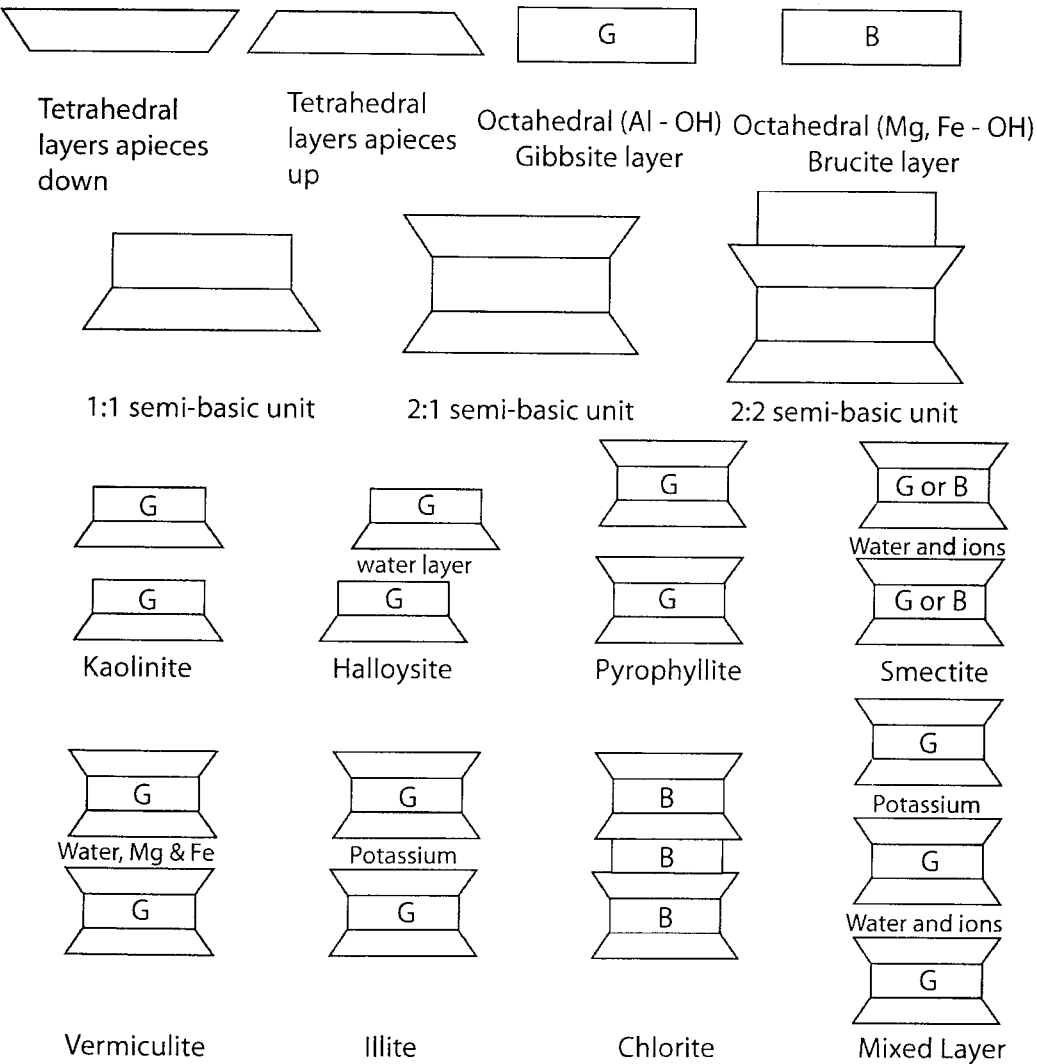


Figure 2.5 Major clay minerals and there composition and construction (Gribble and Hall, 1992)

One complexity with 2:1 mineral is the inter layer ions can change from layer to layer forming mixed layer materials (Figure 2.7). This makes individual clay particles potentially incredibly complex objects.

Clay mineral morphology

Early images of clay minerals identified their variety of mineral particle shapes and sizes (Bates and Comer, 1954). However in mudrocks individual clay mineral particles are rarely observed, therefore morphology is not normally used for mineral identification. Kaolin is an exception due to its regular hexagonal shape. Clay mineral particle sizes are hard to approximate due to the manner in which they form. However there are some data on crystal sizes in the literature. Typically a kaolin particle varies from 0.4-1.1 μm in diameter and 45-100nm in thickness (Palomino et al., 2008). A typical particle of montmorillonite has a diameter of 0.35 μm and a thickness of 1.25nm (Nadeau, 1985). Illite typically has a diameter of 2 μm and a thickness of 10nm (Nadeau, 1985). In terms of plasticity, activity and cation exchange capacity, montmorillonite has the highest, illite is intermediate and kaolin has the lowest (Table 2-1).

Clay minerals are layered materials, their dimensions are dependent on the number of layers present in the particle. The layers are held together by weak electromagnetic (Van der Waals) forces making the particle highly anisotropic (Altaner and Ylagan, 1997). The layers in clay mineral particles can be of different minerals forming interlayered materials, classically illite and smectite combine in this fashion (Figure 2.7).

2.3 Particles, internal and external forces

All forces governing particle behaviour and interaction are electrical in character (Bennett and Hulbert, 1986). In decreasing order of strength they are: Born repulsion forces, covalent bonds, electrostatic interactions, hydrogen bonds, and van der Waals forces.

Born repulsion is the force opposing the compression of matter without limit. It is the force which resists atoms moving increasingly close together. It acts over a distance of several angstroms and usually acts in all directions. Born repulsion is responsible for both normal and frictional forces between particles. The covalent bond is an attractive

force between two atoms resulting from the sharing of electrons; it acts over the distance between two atomic centres and is strongly directional. In most sand grade materials these two forces control almost all interactions. In clay minerals these two forces explain the behaviour between atoms within layers.

Electrostatic interactions which include ionic bonds form as a result of the attraction of opposite charges and the repulsion of like charges. These forces act in all directions and over large distances, reducing in strength with distance. Hydrogen bonds form between hydrogen atoms and other elements which are relatively rich in electrons. Hydrogen bonds are directional, attractive, and they can form when the hydrogen atom is covalently bonded to a small non metallic atom (oxygen, fluorine, nitrogen). Van der Waals forces are attractive and form due to an electrical dipole in atoms or molecules. These dipoles may be permanent, or induced in the case of London forces. They are the weakest forces affecting clay minerals and apply over intermediate distances in all directions.

The interlayer space of clay particles are governed by electrostatic interactions, hydrogen bonds and Van der Waals forces. Electrostatic interactions are of great importance for the study of clay minerals (Bai and Smart, 1997), the overall strength of these forces vary with the orientation of clay particles to each other. The arrangement of clay particles to each other is governed by electrical interactions, which may be disrupted or enhanced by the surface fluid chemistry of the particles (Palomino et al., 2008). Normally electrostatic forces result in repulsion between clay mineral surfaces, the opposite charges on the ends of the platy particles create a loose attraction, resulting in the open structures observed in many newly deposited marine muds (Bolt, 1956). The addition of cations into the solution can result in the formation of a more open structure as the positive charges repel the clay mineral surfaces. Whereas in some mudrock systems the effect of these forces may be limited by the presence of organic matter, which if it covers the particles it allows them to be deposited with a high level of preferred particle alignment (Bennett et al., 1989).

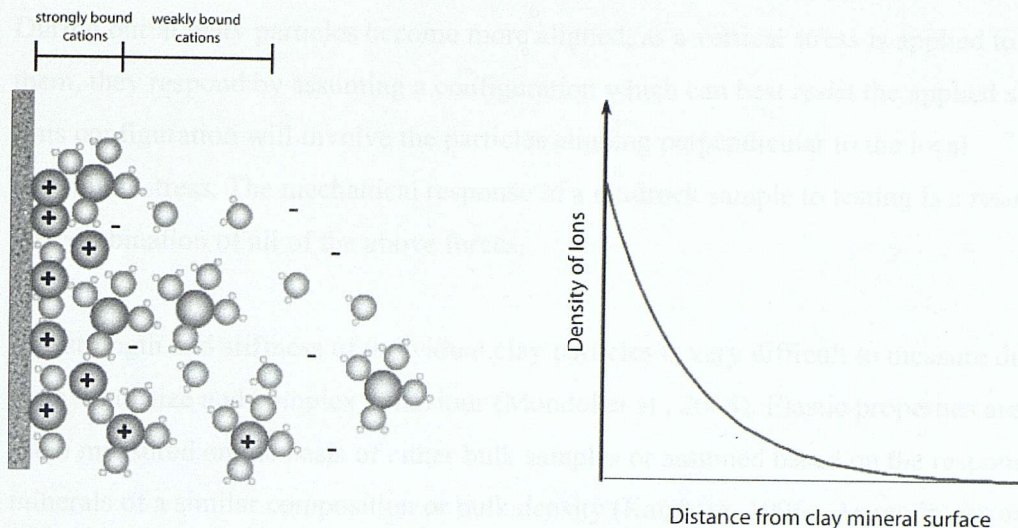


Figure 2.6 Due to the attraction of charged particles to the clay mineral surface a diffuse double layer is formed next to the particle surface.

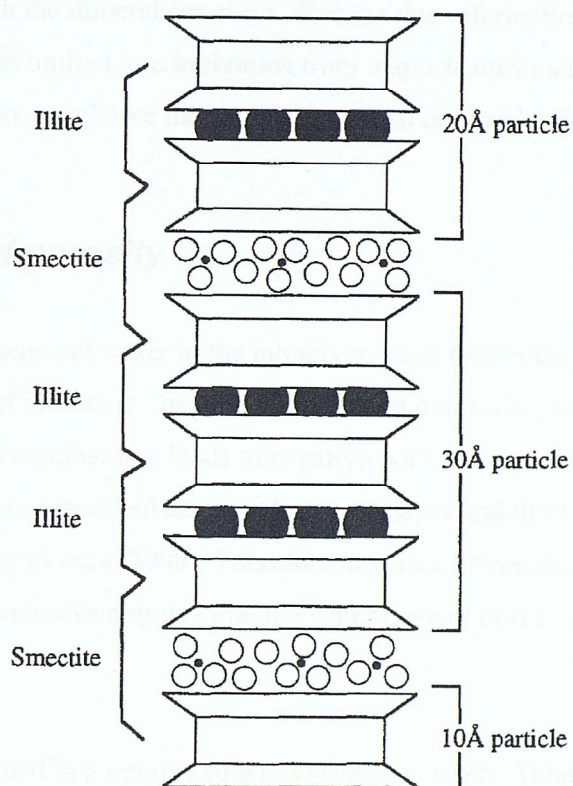


Figure 2.7 Structure of interlayered clay particles from (Altaner and Ylagan, 1997) the identification of these interlayers based on particle thickness was suggested by MacEwan et al., (1961)

During burial platy particles become more aligned, as a vertical stress is applied to them, they respond by assuming a configuration which can best resist the applied stress. This configuration will involve the particles aligning perpendicular to the local maximum stress. The mechanical response of a mudrock sample to testing is a result of the combination of all of the above forces.

The strength and stiffness of individual clay particles is very difficult to measure due to their small size and complex behaviour (Mondol et al., 2008). Elastic properties are often measured on the basis of either bulk samples or assumed based on the response of minerals of a similar composition or bulk density (Katahara, 1996). As mudrocks are usually modelled as continuous media, their particle mechanics have received little attention. The properties of individual particles are required for the study of the stress propagation through the mineral structure. Without this information the study of mudrock structure is limited to correlations from major features such as particle preferred orientation, or relative measures of material order/disorder.

2.4 *Fluid and porosity*

The ubiquitous presence of water in the interlayer space makes the water content a structural element of mudrocks. In partially saturated mudrocks, water forms menisci which pull particles together and leads to negative pore water pressures. The shape of the menisci formed is dependent on particle sizes, shapes and their arrangement in relation to each other (Vesga, 2008). Therefore any measurements of the structure and microstructure of mudrocks requires that the water content of the sample to remain unchanged.

Porosity can be defined in a number of ways (Pearson, 1999). Total porosity is the total void space of a material, effective porosity is the void space which is not entirely enclosed, transport porosity is the void space through which a fluid can flow and geochemical porosity is the void space in which a certain reaction can occur. The pore size distribution of mudrocks shows a wide range of void sizes, from 0.1 m cracks down to nm inter clay particle space. Ignoring outliers, mudrocks often display a binomial (or polynomial) distribution of pore sizes, measured by porosimetry (Li and Zhang, 2009). These distributions often centre on pore sizes of about 10 micron and 0.1 micron. These two sizes can be thought of as inter-particle and inter-aggregate spaces. The void space

of mudrocks can be estimated based on an understanding of particle interactions. If the structure of the solid portion of a material is anisotropic the fluid flow properties of a material will normally also be anisotropic (Peters and Maltman, 1999).

Fluids flowing through mudrocks often carry dissolved ions. Or the mudrocks pore fluid can be enriched in ions through pressure solution. Dissolved material can be deposited in the mudrocks pores as cement. Calcite, gypsum and clay minerals are common cements found in mudrocks.

2.5 Geological variation

The causes of geological variation in mudrocks are discussed later (Chapter 3). However as these variations are an important part of mudrock characteristics they are discussed briefly here. Mudrocks are most widely deposited in a variety of marine environments, from deep marine as distal turbidites and deep water fans, as mud flows and slides on the continental slope, and as widespread mud deposition on continental shelves (Merriman, 2005). They are also deposited in deltas, lagoons, lakes, river beds, and flood plains. They are deposited by ice as boulder clays and by wind as loess. This range of environments produces a variety of grading curves, structure, mineralogy and colour.

The clay size constituent of the majority of mudrocks is normally derived from the chemical weathering of surface rocks (Potter et al., 2005). Clay size material may also be derived from volcanic ash (Jeans et al., 1982, 2000) and as glacial rock flour (Wentworth, 1922). The silt grade material can be derived from any geological process (physical, chemical or biological) which results in the breakdown of larger particles. Although the majority of particles in mudrocks are fine grained, the grain size distribution of a mudrock varies with the energy of the environment of deposition, and the grain size distribution of the sediment source (Wignall, 1989). The amount of silt within a mudrock can change with the stratigraphic horizon being observed as the energy of the depositional environment changes. The quantity of silt within a sample increases the sample's shear strength (Bohlke and Bennett, 1980). Silt particles also tend to disrupt the alignment of platy particles; clay minerals have been observed splayed around silt particles aligning perpendicular to silt grains (Dewhurst et al., 1998). They have also been observed coating the surface of particles, known as clothed grains

(Collins and McGown, 1974). The majority of the silt and sand grains in mudrocks are composed of quartz, with a minor quantity of feldspar grains. Calcite is also often a minor component. A large proportion of calcite is in the form of biogenic particles, such as fossil shells. Pyrite commonly forms a small percentage of mudrocks usually with a framboidal or euhedral habit (Belin and Kenig, 1994). Pyrite nodules have been identified as crack nucleation points in mudrocks (Fouché et al., 2004). Mudrocks can contain very large, even metre scale particles, either cementstones or concretions. In Jurassic clays plesiosaurs and ichthyosaurs remains have been found inside single metre scale concretions (Howe et al., 1981). These concretions greatly disrupt the fabric of the mudrock surrounding them (Lash and Blood, 2004). Layers of the mudrock often appear to drape over the concretions. Fossils are not deposited by the same sorting processes that apply to the rest of the mudrock and so tend to be much coarser grains than their surroundings. Sometimes fossils are found accumulated in one layer of the mudrock, known as a shell bed. These beds sometimes mark the extinction of species, and are thought to represent short periods of no deposition.

This variability of material in mudrocks produces a variety of behaviour which makes the engineering behaviour of mudrocks difficult to predict without intensive laboratory testing. However an in-depth study of the environments of deposition of mudrocks and the micro- and macrostructures these produce may allow predictions of engineering properties to be attempted.

2.6 *Material behaviour*

Taking all of these components (Sections 2.2 - 2.5) into account, mudrocks can be incredibly complex materials. Their particle size distribution varies through nine orders of magnitude, from smectite with a thickness of 1.25nm (Section 2.2.2) up to large metre scale concretions (Section 2.5). Their void distribution varies through several orders of magnitude from the inter clay layer space to the size of open joints. The inter-particle forces in mudrocks are normal contact and frictional forces, electrostatic forces, pore water surface tensional forces, and cementation between particles (Section 2.4). They can be deposited in a variety of environments and their particles can be arranged in a multitude of geometries depending on depositional and burial processes which form the particle scale structures. This variability of material produces variability of behaviour and this makes mudrocks one of the most interesting geo-materials to study.

CHAPTER 3

GEOLOGICAL VARIATIONS

3.1 *Introduction*

To allow for comparisons between this work and previously undertaken studies at Imperial College the geological units studied were to be similar in character to the London Clay with the only difference being the age of the mudrock. Due to the inherent variability of geological units it was not possible completely to fulfil this requirement. The fact that the mudrocks under investigation are all continental shelf deposited materials of a similar grain size indicates that their depositional environments were similar. However local and regional geological variations will cause differences between the mudrocks.

A named geological unit is normally a collection of varying beds which have been grouped together based on similar visual properties usually defined by historical convention. The unit may change laterally and still remain distinct from the surrounding geological units and so may still retain its name across regions displaying a range of engineering behaviours. The same can be said about variations in a geological material with depth. A pattern of repeating beds of different lithological character may be combined as one geological unit if it is distinct from the surrounding material. Thus, a named geological division is rarely, if ever, an engineering geological division.

In this project samples were taken from single locations, in each geological unit studied, across a very limited range of depths. The results from this project can be used to compare general trends between the specific samples studied. They cannot be used as examples of how the mudrocks may behave in other locations across the UK. In this chapter the major variations in geological history experienced across the UK since the Jurassic are described. For the sake of simplicity it is assumed that the mudrock is in the same orientation as when it was deposited when discussing lateral and vertical variability.

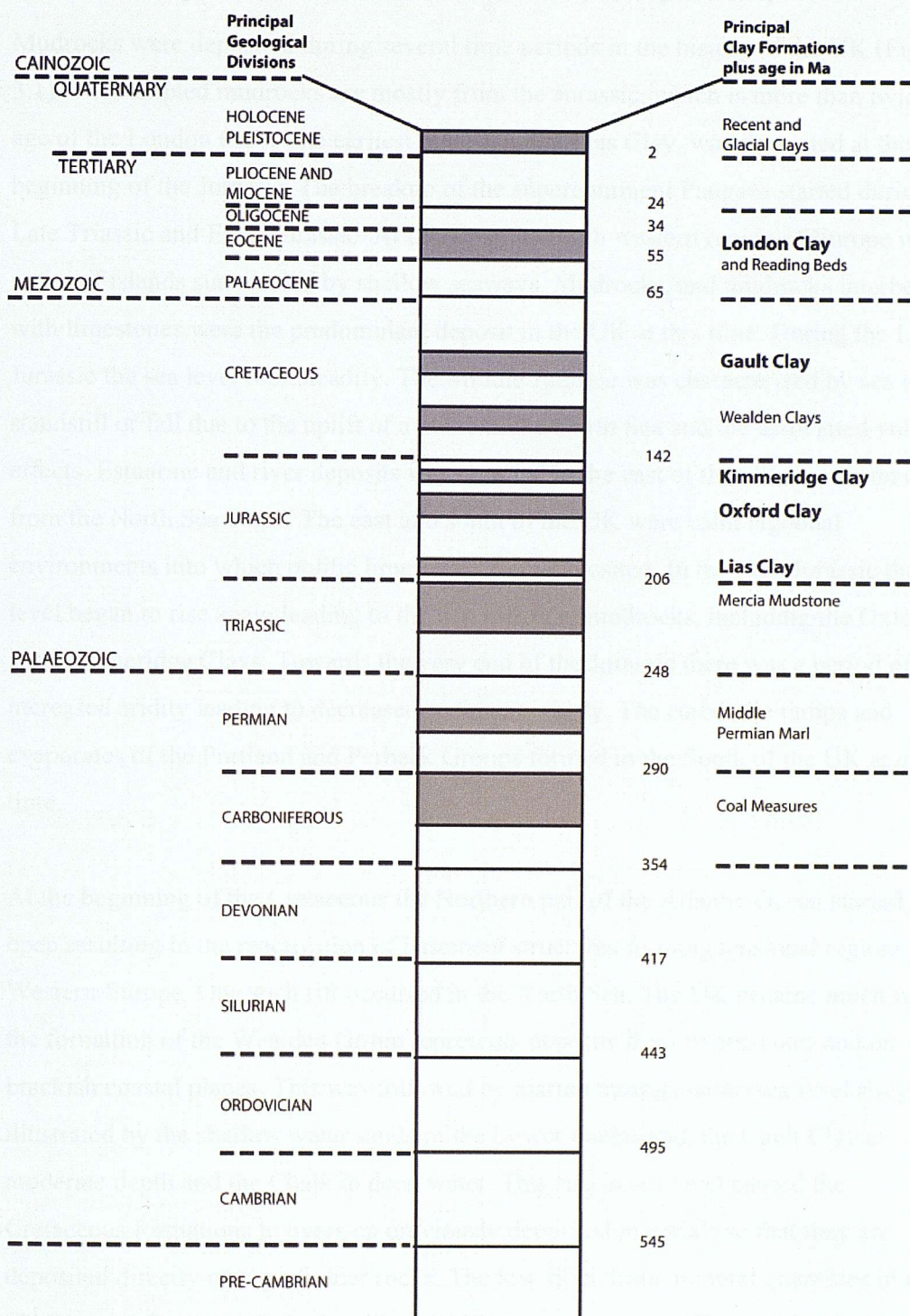


Figure 3.1 Major mudrock deposits and their distribution through geological time (Gradstein and Ogg, 1996). Mudrocks studied during this project are shown in bold. Mudrocks older than the Carboniferous are omitted from the original. Modified from (Reeves et al., 2006).

3.1.1 History of the Southern UK (Jurassic to present)

Mudrocks were deposited during several time periods in the history of the UK (Figure 3.1). The sampled mudrocks are mostly from the Jurassic, which is more than twice the age of the London Clay. The earliest mudrock, the Lias Clay, was deposited at the very beginning of the Jurassic. The breakup of the supercontinent Pangaea started during the Late Triassic and Early Jurassic. At this time the north western region of Europe was a series of islands surrounded by shallow seaways. Mudrocks, and mudrocks interbedded with limestones were the predominant deposit in the UK at this time. During the Lower Jurassic the sea level rose steadily. The Middle Jurassic was characterised by sea level standstill or fall due to the uplift of a dome in the North Sea and the associated volcanic effects. Estuarine and river deposits were formed in the east of the UK due to run off from the North Sea dome. The east and south of the UK were calm lagoonal environments into which oolitic limestones were deposited. In the Late Jurassic the sea level began to rise again leading to the deposition of mudrocks, including the Oxford and Kimmeridge Clays. Towards the very end of the Jurassic there was a period of increased aridity leading to decreased sediment supply. The carbonate ramps and evaporates of the Portland and Perbeck Groups formed in the South of the UK at this time.

At the beginning of the Cretaceous the Northern part of the Atlantic Ocean started to open resulting in the reactivation of basement structures forming tensional regions in Western Europe. One such rift occurred in the North Sea. The UK became much wetter, the formation of the Wealden Group represents deposits from rivers, lakes and on brackish coastal planes. This was followed by marine transgression (sea level rise) illustrated by the shallow water sands of the Lower Greensand, the Gault Clay at moderate depth and the Chalk in deep water. This rise in sea level caused the Cretaceous formations to overstep previously deposited materials so that they are deposited directly on top of older rocks. The low siliciclastic mineral quantities in the Chalk result from a period of aridity (resulting in reduced runoff), combined with the distancing of landmasses from depocentres due to the marine transgression.

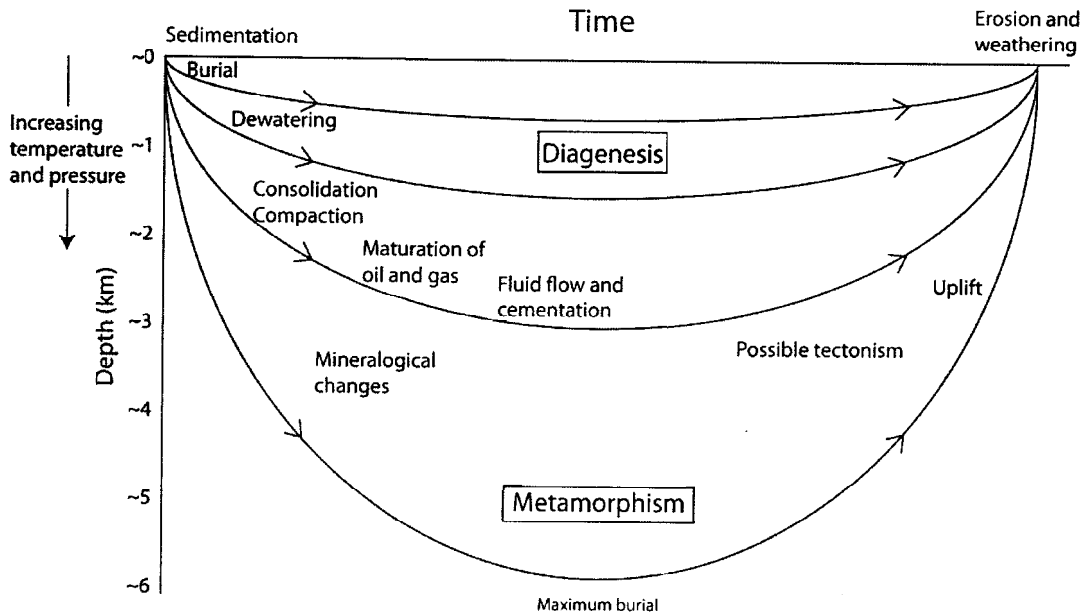


Figure 3.2 General model of basin evolution, the geotechnical cycle (after Chandler, 2000). Lines indicate burial paths of different depths. Other paths with rapid burial and slow uplift, or gradual burial followed by rapid uplift can also occur. (See Section 3.2.4).

The opening of the North Atlantic between the UK and Greenland related to a new mantle plume caused the formation of the Tertiary Igneous Province and initiated the uplift of the UK during the Early Tertiary. The convergence of Africa with Europe also occurred during the Late Cretaceous and early Tertiary, resulting in the formation of a compressional regime by the reactivation of older structures in the southern basins of the UK. Material was deposited within small sea inlets in the south of the UK. These deposits (including the London Clay) were formed from runoff into the London and Hampshire basins from the erosion of the surrounding Chalk and Jurassic mudrock dominated landmasses.

During the Quaternary, global temperatures reduced resulting in several glaciations centred on the north of the UK. This caused the removal of much of the weathered cover, the formation of boulder clay beneath the glaciers and windblown loess in the periglacial region, and the uplift of the southern UK due to the loading of ice on to the north. Meltwaters from the glaciers deposited outwash gravels across much of the southern UK.

3.2 General model of basin history

The geological history of a material causes it to form a structure. Geological history can be divided into depositional, burial and exhumation histories (Chandler, 2000). Burial and exhumation histories may be combined as diagenetic history, or in cases of deep burial metamorphic history (Figure 3.2). The depositional history of a material is linked to its depositional setting or environment. The burial history relies on measurements of paleo-temperature/pressure, or a reconstruction of the thickness of materials which have been removed. A reconstruction of the burial curve of a mudrock is created based on limited information. Small scale or short duration aberrations in the burial curve of a material may have occurred without leaving any evidence, only a general burial curve recording the major events can be constructed. For the behaviour of a material to be interpreted the effects of each event in a material's history must be known (Brevik, 2005).

3.2.1 Deposition

Clay rich fine grained sediments are deposited in a wide variety of settings, from continental to abyssal plain. The depositional mechanisms operating in each of these environments create a variety of structures in the deposited material. The Gault, Kimmeridge, Oxford and Lias Clays were all deposited in shallow marine basins in the north western corner of the quiet Tethys Sea. The mechanisms discussed here are appropriate for this sort of environment.

The most common mechanism for deposition in a low energy environment is dispersed settling out of the water column (Section 4.3), forming a mud blanket (Allen and Allen, 2002). In saline environments this sort of deposition creates a very open, high porosity clay particle structure (cardhouse or honeycombe structure, see Section 4.3), where particles aggregate with edge to face associations (Moon, 1972). The charged ions of the saline pore fluid act to keep the structure open. In the absence of salts, or in brackish water, or where the clay particles are coated with a substance (such as organic material) so that the salt cannot act, clay minerals can, by dispersion, form a more aligned structure (dispersed structure) (Collins and McGown, 1974; Ingram, 1953).

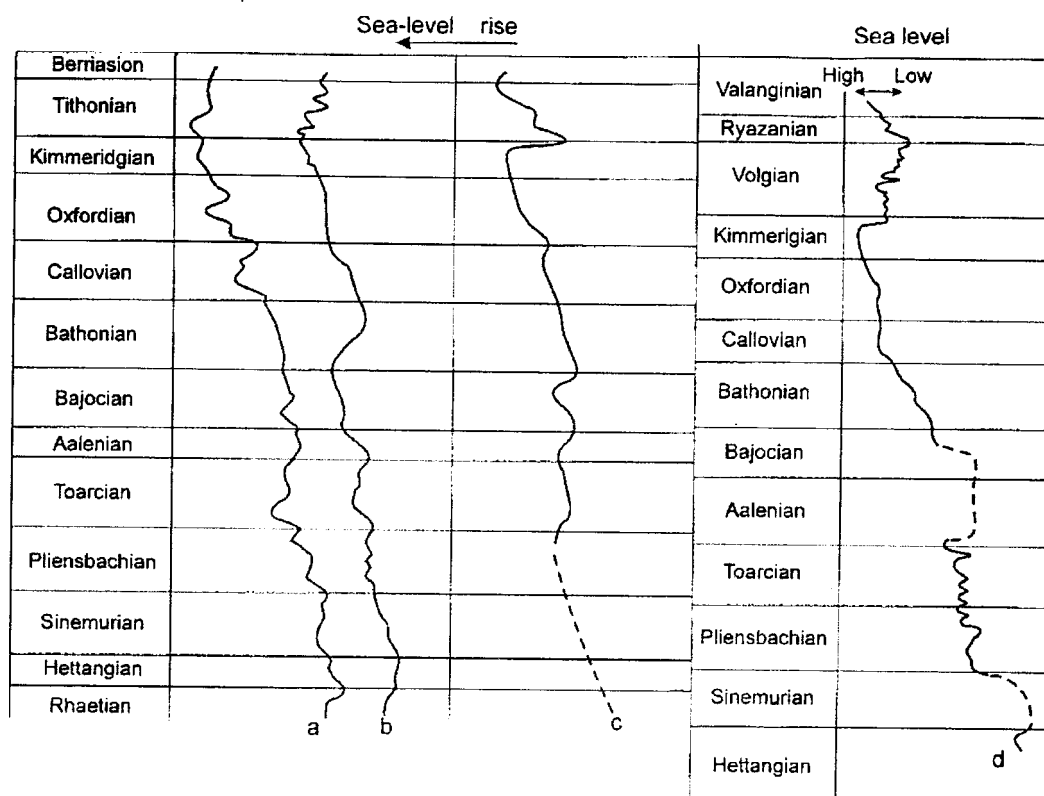


Figure 3.3 A number of different sea level curves for the Jurassic after (Hallam, 2001). Global sea level curves (a & b) for the Himalayas (c) and for east Greenland (d). All show a general trend of sea level rise.

Clay minerals can also be deposited in high energy environments. When the amount of sediment in the water column reaches the maximum that environment can transport grains can be dropped out of the turbulent water column individually (turbostatic structure).

The formation of larger particles by the amalgamation of clay mineral flocs can allow mudrocks to be deposited in high energy environments. The aggregated particles (colloids), become too large and the material is deposited as migrating floccule ripples (quasi dispersed structure) in the same way as sands in the high energy environment (Macquaker and Bohacs, 2007; Schieber et al., 2007). The formation of colloids may also explain the bookhouse structure also common in marine (salt-rich) environments (Collins and McGown, 1974).

The energy level during the deposition of Jurassic mudrocks is difficult to gauge accurately. Each mudrock is deposited over a time scale of several million years, over which the predominant environment will have been low energy with calm seas, however there will also have been periods of violent storms (Wignall, 1989). A single storm or

turbidity current can deposit in a few days what calm conditions deposit in many years. On balance it is likely that many processes are responsible for the deposition of these mudrocks and the processes which change depending on the position the stratigraphic sequence (Macquaker and Curtis, 1989). The rate of sedimentation is likely to be episodic, its extent is controlled by sediment type quantity and the sorting processes which occurred during transportation (resulting in different sediments being deposited on the edges and in the centre of basins). These processes give rise to a variation in composition and sedimentation rate which in turn affect the geothermal gradient in the material.

3.2.2 Sea level variation

The most important control on basin sedimentation changes is sea level variation. Global sea level changes are notoriously hard to estimate (Hallam, 1981). It is very difficult to separate changes seen at a local level from the local environmental regime. Local subsidence and uplift makes the estimation of global effects very difficult. Local or relative changes in sea level are easier to infer from the rock mass (Immenhauser, 2005). The specific depth at which a mudrock is deposited can be inferred by sedimentary structures and fossil assemblages present within the material (Immenhauser, 2009). Deposits formed during relative sea level rise tend to be condensed (Hallam, 1999). Relative sea level fall is inferred from clearly eroded sequences. The relative changes in sea level for the UK also changes from basin to basin; this is especially due to tectonic effects (Hesselbo, 2008). Sea level determines sedimentary structures, and to a great extent the grain size (mineralogy) of the material being deposited. Sea level in general rose throughout the Jurassic (Figure 3.3). The scale of the global sea level rise is suggested to be greater than 150m (Hallam, 1981). Relative sea level rise is also thought to have occurred during the deposition of the Gault Clay (Cretaceous).

3.2.3 Material stratigraphy

The stratigraphic location of a sample can be defined either by the name of the rock which it is a part of (lithostratigraphy), based on the material's fossil content (biostratigraphy), or using absolute time from radiometric dates (Rudwick, 1988). In

this work lithostratigraphic details are given for each mudrock, biostratigraphic data are provided where this information was available.

The stratigraphic changes observed include: variations in grain size, especially quartz silt content, variations in organic content, and the presence of carbonate and/or fossiliferous horizons. Mudrock composition may change within metres to a limestone band (Blue Lias Formation). The mineralogy may change rapidly from kaolin rich to illite/smectite rich (White Stone Band Kimmeridge Clay). The organic content is confined to specific layers so changes from organic rich to organic poor are not uncommon (Upper Kimmeridge Clay). These vertical changes are obvious in the field and easily described and defined. Material properties are also affected by more subtle changes which may not be evident on observation of material colour. The deposition of a material is not constant in all locations at all times. The rate of deposition is dependent on the tectonic movements, volcanic eruption rates, rainfall, weathering and transportation processes. It is common in mudrocks to observe nodule bands or shell death beds (Oxford Clay), these semi-planar features tend to be areas where the faunal communities change, there will be species which become extinct at the plane and others which appear at the plane (Raup and Crick, 1982). These features are known as sequence breaks. If there is no deposition for an extended period of time then a hard ground forms. These features may mark a change in material properties as well as a gap in time (Zorina et al., 2008).

3.2.4 Burial

Changes to material with depth

As geological materials are buried they undergo a process of diagenesis. These are all of the processes a materials undergoes during burial where the pressures and temperatures are not enough for metamorphism (Bucher and Frey, 2002). Perhaps the most important changes to the material with depth for this project are: the general observation of a decrease in porosity of materials with both age and depth of burial (Borst, 1982; Mondol et al., 2007), and the progressive production of preferred orientation with depth (Bowles et al., 1969) as these are key components of the current investigation. These early stages of compaction are of course influenced by the material's initial water content, organic content, temperature and the ions present in the pore water, (Meade,

1966). The burial process however involves further changes to mudrocks beyond compaction, there are many diagenetic changes which mudrocks undergo (Figure 3.6).

The most important mineralogical change to a mudrocks in terms of preferred particle orientation is the change from smectite to illite. Many mechanisms have been proposed for this transition (Bethke, 1986; Bethke et al., 1986; Drits et al., 1997; Velde and Vasseur, 1992). An analysis of numerous basins shows that no single mechanism can explain all the observations. It is suggested that multiple mechanisms may occur (Elliott and Matisoff, 1996). The rate of change from smectite to illite is dependent on pressure and temperature, water content (Whitney, 1990), and the concentrations of ions within the pore water (Huang et al., 1993). The change to illite has the side effect of creating a system with much greater order or alignment of layers (Ahn and Peacor, 1986). In some cases perfect crystals are formed (Lee et al., 1985). Such order greatly increases the anisotropy of the material as a whole.

At fairly low temperatures (<40°C) and shallow depths (<1km) pyrite and carbonate cements can form (Curtis, 1985; Emery et al., 1988). The conversion of smectite to illite releases calcium which can form cements. In more deeply buried materials the amount of calcite tends to decrease with increasing depth (Hower et al., 1976). As does the amount of potassium feldspar, which is absent by 3,700m, chlorite forms in samples deeper than 2,500m and kaolinite decreases rapidly below 3,400m (Bjørlykke, 1998). The reactions causing these changes are known as chemical compaction.

The organic matter within materials reaches maturity as oil and then gas with increasing depth and can migrate away from the mudrock system (Scotchman, 1991b). The decrease in porosity with depth is one of the drivers of the expulsion of fluids (Bjørlykke, 1999; Bjørlykke and Høeg, 1997). During burial, concretions start to grow, they can reach considerable size (~30cm) without deep burial (<500m) (Hugget, 1994). Concretions tend to be found in the carbonate rich areas of materials (Scotchman, 1991a), suggesting that the dissolution and reprecipitation of carbonate is occurring at these shallow depths.

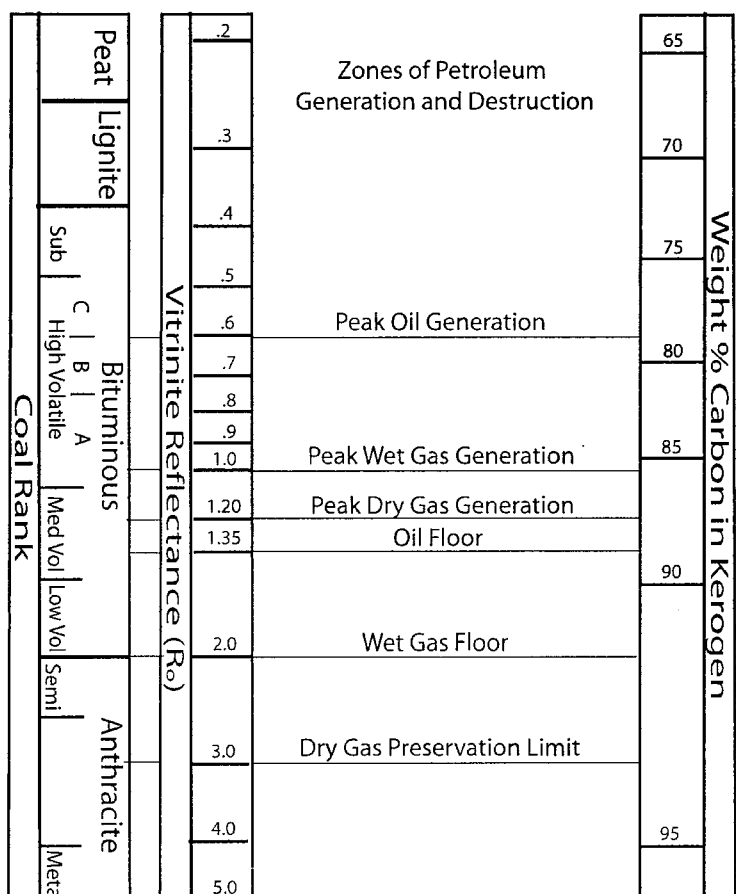


Figure 3.4 A correlation of coal grades and vitrinite reflectance with petroleum production zones (based on Dow, 1977)

Depth/Pressure	Temperature	Both
Stratigraphy	Organic maturity	Mineralogical changes e.g. smectite→illite
Sonic velocity	Coal grades	
Consolidation testing	Vitrinite reflectance	Illite crystallinity
	Spore colour	
	Conodont colour	
	Apatite fission track analysis (AFTA)	

Table 3-1 Some of the most important methods used for the identification of maximum depth of burial

Estimating maximum burial depth

Traditionally geologists calculated the depth of burial of a geological stratum by estimating the thickness of material which could have been eroded from above the unit (Jackson, 1972; Jackson and Fookes, 1974; Scotchman, 1987). Petroleum geoscientists who are interested in the organic material within the rocks tend to use the changes which occur to the material and its organic content as it is buried. These changes are often related to the temperature which the rock has been elevated to; this can be correlated to the depth of burial via the geothermal gradient. Some temperature related methods associated with the maturation of organic material are: the different grades of coal (Dow, 1977), vitrinite reflectance (Allen and Allen, 2002) darkening of spore (Yule et al., 1999), conodont colours (Burnett, 1987) and the maturation of oil and gas (Ainsworth et al., 1990; Scotchman, 1994). The major methods used for identifying depth of burial have been summarised (Table 3-1).

Coal goes through the series of changes as it is buried. The different states it goes through, referred to as grades, are peat, lignite, bituminous and finally to anthracite (Dow, 1977). This series is formed from various chemical changes in the material which have been related to the burial history of the coal (Figure 3.4).

Organic matter held within sediments can be broadly classified into two subsets: bitumen and kerogen. Bitumen is the part which is soluble in organic solvents and kerogen is the part which is not soluble in organic solvents (Dow, 1977). Kerogen comprises a number of different macerals. Macerals are composed of organic material; they are like minerals. They are different from minerals in that they do not have clearly defined crystal boundaries, one maceral is vitrinite. An interesting property of vitrinite is its reflectance: the ratio of incoming light onto the sample to the light leaving the sample, (similar to the albedo which is the reflectance where the sun is used as the original light source). The reflectance of vitrinite can be related to the temperature which the material has reached during its history (Figure 3.4) by an empirical relationship (Allen and Allen, 2002; Baird, 1986).

Index	Colour Alteration	Temperature Range (°C)
1	Pale yellow	<50-80
1.5	Very pale brown	50-90
2	Brown	60-140
3	Increasingly dark brown	110-200
4		190 – 300
5	Black	300-350

Figure 3.5 Conodont colour alteration index, based on the changes in conodont colour with increasing temperature (Dean, 1992)

Spore colour is a measure of temperature based on the increasing opacity of palynomorphs (organic microfossil; fossilised pollen is commonly used). A history of its use for the study of depth of burial can be found in the literature (Yule et al., 1999). Conodonts are eel-like creatures. The only part of them that is well preserved in the UK is their feeding apparatus (Sweet and Donoghue, 2001). They are known to change colour with temperature from pale yellow at around 50°C to black at around 350°C (Figure 3.5).

In all locations across the North of the UK these methods, based on temperature consistently predict a greater maximum depth of burial than would be expected from a purely stratigraphic extrapolation (Scott and Colter, 1987). Because of the large errors associated with these empirical methods the elevated temperatures were thought of as anomalies by stratigraphers. They gained more importance when apatite fission track analysis also suggested burial to a great depth (Green, 1986, 1989).

A fission track is produced when a radiogenic isotope decays. In the 1960s it was discovered that the number of fission tracks in a sample was proportional to the sample's age (Dickin, 2002) (*i.e.*, the older the sample, the more radioactive particles will have decayed and so the more fission tracks there will be in the sample). However it was also shown that the number of fission tracks reduces due to a process called annealing or healing. This may be caused by a number of different processes, such as electron bombardment, however the principal cause is elevated temperature. At a certain temperature depending on the material the fission tracks will start to shorten; this may begin at a very low temperature (40°C). Above this temperature the rate of annealing will increase, up to a certain temperature at which all fission tracks will have been removed (125°C) (Dickin, 2002). By comparing fission track lengths in a borehole the

temperature history of levels in the borehole can be reconstructed. The analytical precision of AFTA (Apatite Fission Track Analysis) is given as $\pm 10^{\circ}\text{C}$ (Bray et al., 1992); this is much greater than other paleotemperature measures.

The main problem with methods based on temperature is that there is no strict relationship between temperature and burial depth. In order to relate the two the surface temperature and geothermal gradient at a given time in the past must be assumed. A common assumption is that the basal heat flow is the same throughout history and so the geothermal gradient will remain constant, this is unlikely to be correct. In the past the UK has been situated much closer to spreading centres where hot mantle rises close to the surface, higher heat flow during the past is quite likely (Hendry, 2002). Additionally during geological time the UK has been moving slowly northwards, so the surface temperature will have been greater during the past (Brown, 1991; Holliday, 1993). Also geothermal gradients do not have to be linear as is often assumed (Bray et al., 1992). The thermal conductivity of the ground varies with the material and the depth to which it is buried. An over-pressured shale at 4km depth may have a thermal conductivity of approximately $1\text{Wm}^{-1}\text{K}^{-1}$ whereas a sandstone at 4km would have a much greater thermal conductivity of $4\text{Wm}^{-1}\text{K}^{-1}$ (Holliday, 1999). The geothermal gradient of the UK during the past has been constrained by combining apatite fission track and vitrinite reflectance data. This predicts a similar palaeo-geothermal gradient as today, corroborating the assumed linear geothermal gradient (Green, 2005). For this work a thermal gradient the same as today's $30^{\circ}\text{C}/\text{km}$ is used (Brown, 1991). The effects of this assumption cannot be overstated. If the thermal gradient doubles the paleo-depth halves, and the reverse is also true. The estimates presented here (Figure 3.11) are only true, if the thermal gradient used is correct (McCulloch, 1994).

Due to the uncertainties in working out the past geothermal gradient of an area, a method not related to temperature was required. The main physical property that changes with depth of burial other than the temperature of the material is its porosity. This is often measured using geophysical down-borehole instruments such as density or sonic probes.

Sonic velocity was commonly used in offshore environments to estimate burial history for the purpose of petroleum exploration (Bulat and Stoker, 1987). In this case the error bars given for the method are $\pm 1000\text{ft}$ (305m). The large uncertainties in the method

represent the many different assumptions which are required for an estimate. It is assumed that the porosity of the material is related only to compaction, that the material will follow a normal compaction trend irrespective of the materials involved and all compaction which occurs is assumed to be irreversible (inelastic) (Issler, 1992). These assumptions also lead to a substantial amount of noise in the data. Even given these large error bars, depth of burial estimates on the basis of sonic velocity profiles agree with AFTA burial profiles for the East Midlands Shelf (Hillis, 1993) and the Irish Sea (Ware and Turner, 2002). A discussion of work on the Midlands Platform suggests that the burial depth from sonic velocity may be too deep due to: 1. the normal compression trend used was incorrect, 2. the natural variability of the chalk makes its depth to sonic velocity profile hard to establish and 3. Incomplete sequences at the top of the profile are ignored causing an increase in the velocity profile due to averaging (Smith et al., 1994). Even given the disadvantages of this method it has been used in conjunction with apatite fission tracks and vitrinite reflectance to constrain the possible historic geothermal gradients across the UK (Green, 2005).

The consensus amongst the geological community is that the deeper thermal measurement of depth of burial is broadly correct, and alterations are being made to palaeogeographic models to reflect this (Brenchley and Rawson, 2006; Green and Duddy, 2006).

3.2.5 Tectonic effects

Areas which have experienced a tectonic environment will have a different structure to those areas which have not experienced such a history. For simplicity it was decided to avoid tectonised areas of the UK during this project. In fault zones the burial stresses which formed the original material have been influenced by shear stresses. The effect on the preferred orientation of platy particles is to align parallel to the direction of shear (Cetin and Söylemez, 2004; Dewhurst et al., 1996a). With lateral displacements of the shear zone, both extensional and compressional features can arise. The extensional features can be present as large voids in the material, completely changing its permeability (Oda et al., 2004). High energy shears can form preferred alignment through recrystallisation in areas where the temperature is not high enough for such an effect (Herwegh et al., 2008).

3.2.6 Uplift

Uplift is the process by which deeply buried materials are returned to the earth's surface. It can be thought of as the combination of exhumation, the return of the material from depth to the geoid and surface uplift, the further elevation of a material to heights above the geoid (mountain building) (England and Molnar, 1990). Uplift may be tectonic in origin, due to the collision of continents, magmatic in origin in the case of under plating or in the case of isostatic uplift it may be caused by the erosion or removal of a weight of material from above the uplifting rocks. All three of these mechanisms have been put forward to explain the apparent rapid uplift experienced by the UK during the Cenozoic. The first is that plate boundary forces from the Alpine collision were translated far from the collision front causing uplift of the UK (Ziegler et al., 1995). This applies more to the south of the UK than the North. Another is permanent under-plating of the lower crust relating to the Iceland plume during the Palaeocene (Hillis et al., 2008; Jones et al., 2002). The uplift from this process is centred to the North West of the UK and diminishes towards the South East. Finally any surface uplift which results in subaerial exposure causes erosion. Erosion results in the removal of weight from the material causing uplift as the material seek isostatic equilibrium. Thus uplift by erosion may continue long after the event which caused the original uplift. During the glaciations of the UK many of the mudrock units of the UK were incised as much as 200m deeper than the limestone units surrounding them. It is likely that this incision will have resulted in a small amount of uplift across the UK (Blundell and Waltham, 2009). Observations of river terraces across the southern UK have shown that the most recent phases of uplift have been pulsed, matching the glacials and interglacials of the last ice age (Westaway, 2010). This sort of uplift could have been produced by slow emplacement of ice allowing enough time for the asthenosphere to compensate by viscous flow, followed by rapid removal requiring an isostatic response (Blundell and Waltham, 2009). All of these mechanisms possibly applied to different locations of the UK during the Cenozoic. This makes long-term rates of uplift difficult to approximate.

The estimation of the magnitude of uplift is possible through the dating and measurement of the current height of fluvial and marine terraces (Bridgland, 2010). These terraces record past heights of the coast and rivers. As the UK is uplifted these terraces are left as remnants of the river system. This record can only be used for the

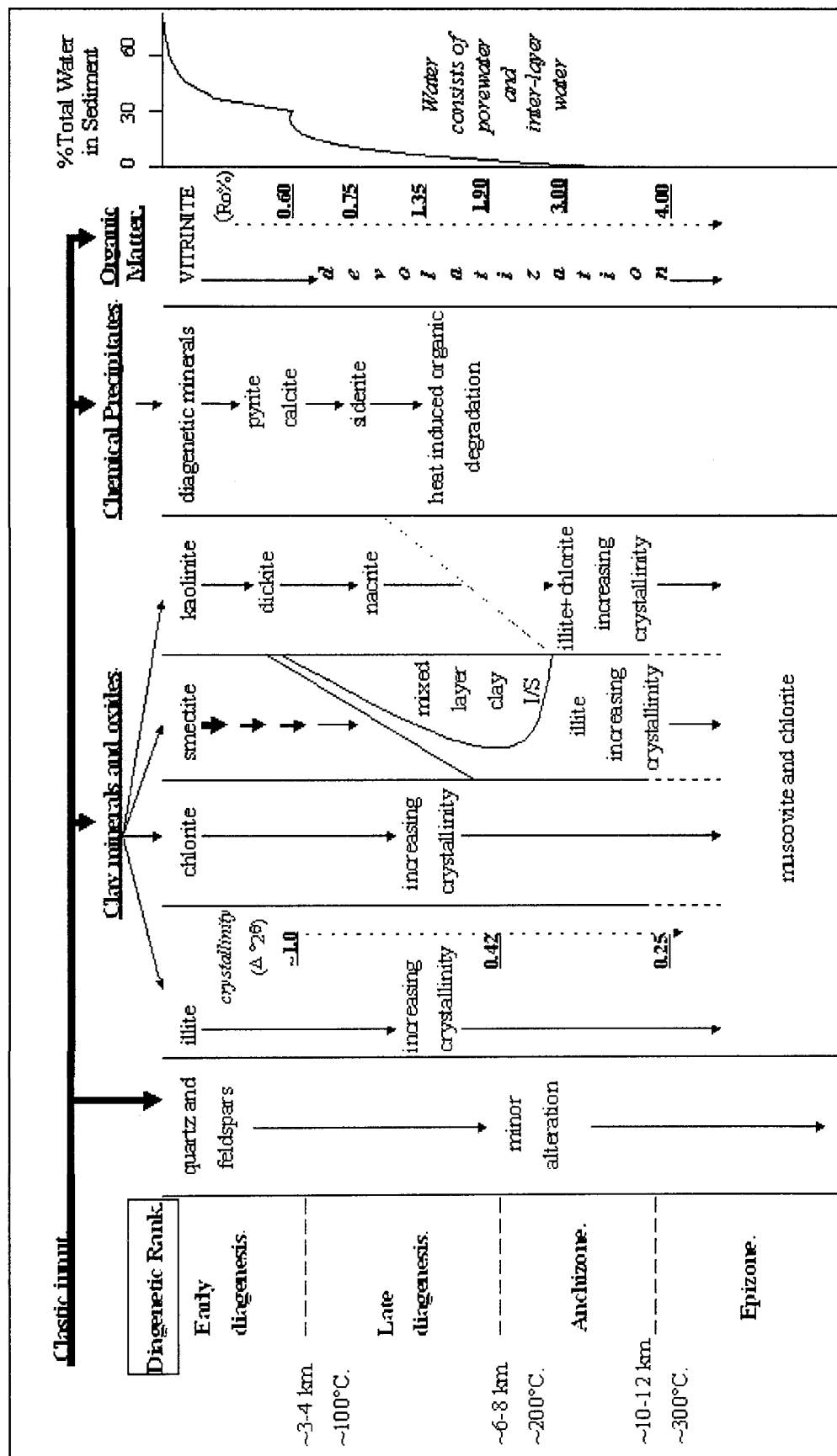
most recent past (~3Ma) as beyond this point in time much of the terrace evidence required to draw conclusion about uplift amount has been removed.

3.2.7 Near surface (recent) processes

Weathering

Weathering occurs by physical, chemical or biological means. Physical and biological weathering causes the disaggregation of a material the eventual effect of which is the break up of material structure. The initial effect of physical weathering may be to enhance the physical fissility of a mudrock, as it breaks along its planes of weakness (Ingram, 1953). Once the material has broken apart and the blocks of the material have been reoriented the structure present is caused more by the weathering than the original structure. Chemical weathering not only removes the structure of a material it also can change the mineralogy (Cafaro and Cotecchia, 2001).

Weathering types can be divided on the basis of the processes which are occurring (*e.g.*, biological weathering may occur through: tree roots, bacterial growth, worms, moles, and rabbits). Each process is different and has a different extent/depth of effect on the original material, multiple processes can be present in any location. So the weathering profile of a material is a complex zone. All weathering effects occur at the surface, with depth individual processes cease to be applied to a material, forming a gradational zone of weathering. The deepest weathering effects such as cryoturbation and valley bulge can affect a material, which appears unweathered, to great depths. The differences between several mechanical and chemical process are presented in the literature (Chandler, 1972). Each weathering process imposes a weathered microstructure onto mudrocks. They extend to a different depth below ground to the extent which it has progressed through the material (Wetzel and Einsele, 1991). Sometimes the advance of one weathering process aids the advance of another. For example in the Gault Clay the advance of tree roots caused the desiccation of the near surface samples. This caused the opening of vertical cracks in the material allowing the advance of chemical weathering along those cracks to a lower level than the chemical weathering had previously reached through the rest of the material.



Cryoturbation

During the Quaternary the earth went through a series of glaciations. The effect of these cold periods and the traversing of glaciers across the UK are to greatly disturb the material across which it advances. Due to the extent of frozen ground to great depths, cryoturbation “of all recent processes” has the possibility to affect the soil to great depth (~50m). For the study of mudrocks these effects are slightly mitigated by two factors, the first mudrocks form the valleys of the UK along which the rivers run. These rivers have the effect of raising the temperature of the ground locally reducing the depth of effect of frozen ground. Also there is evidence from river terraces that the mudrock valleys have been incised by the glacial outwash rivers since they were last frozen (Bridgland, 2010). So although the effects of cryoturbation can be extensive they may be limited at our sampling locations. It was not possible in this project to sample from great depth so it must be accepted that the soil may have been frozen in the past.

Root action

Tree roots have the effect of both breaking up and desiccating the soil, relative to the surrounding environment. Intense dewatering creates desiccation polygons which are represented as regular vertical joints in the ground. The tree roots grow along these joints introducing further biological weathering. Measurements of tree root depth in mudrocks suggest that are likely to extend to between 3 and 4m as a maximum (Crow, 2005). It is possible that the vertical joints within the Gault Clay would have encouraged a deeper system. It is known that in the post glacial period, prior to the migration of man to the British Isles, much of the land surface was covered by forests (Hollingworth et al., 1949). The effects of these trees on the soils are unknown; it is thought that they may have caused the desiccation of the soil, such as is observed at our Gault Clay Site. However as trees would have covered the entire UK it is also possible that they will have slowed down the runoff process and limited evaporation resulting in a more moist soil depending on the local setting.

3.3 Basins

Basins are regions of prolonged subsidence and consequently, areas of sedimentary accumulation (Allen and Allen, 2002). Due to the different sediment sources and variable processes occurring, the deposits in each basin will vary. The main differences are a result of subsidence rate (material thickness) and tectonic affects: relative sea level

change (grain size variability) and change in orientation. Intrabasin geography is also important; laboratory results measuring the effects of structure have shown differences in mudrock structure between the centre and at the edges of a basin (Cotecchia et al., 2007).

3.3.1 Palaeogeography

Palaeogeography is literally the shape of the land in the past. The continents which we know today were very different in earlier geological periods. Many areas of land today were at one point in the past below sea level (Figure 3.7). During the Jurassic the UK was located between 40°N and 50 °N, experiencing a much warmer climate than today. At this time the UK was a series of islands between which were open seaways, into which the mudrocks presented here were deposited. The locations of these islands and seaways changed with time through the Jurassic, thus, the configuration is different during the deposition of each of the materials studied (Figure 3.8). However the overall depositional environment of open seaways did not change.

The mineralogy of the mudrocks was dependent on the material being eroded from the surrounding islands. To the north of the UK during the Jurassic kaolin was the dominant clay mineral deposited, while to the south illite was more common (Dera et al., 2009). Within the region occupied by the UK the clay mineralogy contains a mix between the two indicating that the material deposited was probably sourced from a wide area.

The configuration of the landmasses around the depocentre strongly affects both the materials which are being eroded and the transport path taken to the site of deposition. Bedrock geology heterogeneity within small geographic areas means that changes in the position of land masses changes the geological material being eroded. The length of the transport path affects the amount of mineralogical degradation the material experiences and the grain size of a material is usually reduced along a transport length.

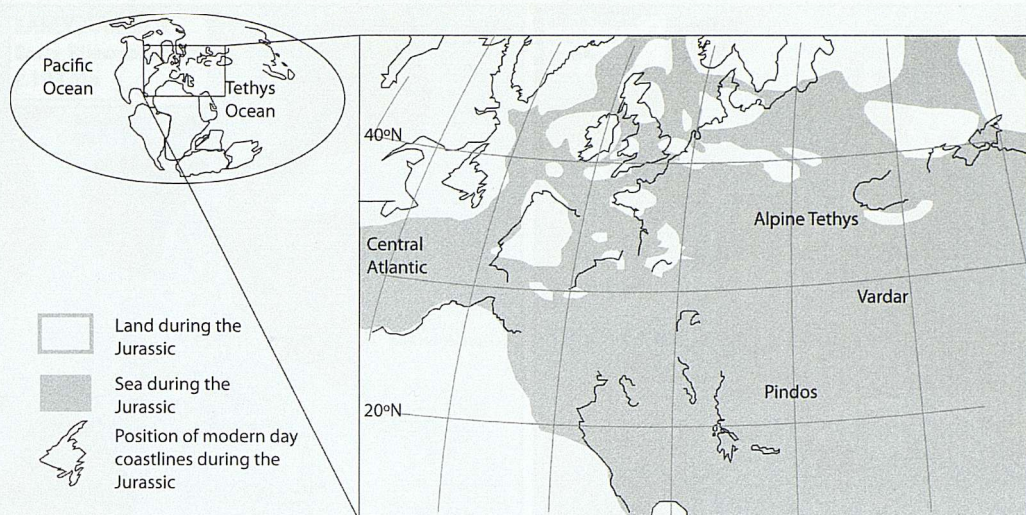


Figure 3.7 Palaeogeography of the region around the UK during the Late Jurassic. The UK was located in the North West corner of the Tethys Ocean, the sedimentary environment was within seaways confined between a number of islands. Modified from (Rais *et al.*, 2007).

The London Clay was deposited in a slightly different environment to the Jurassic mudrocks (Figure 3.9). During the Tertiary the previously open seaways were in the process of closing up as the UK was being compressed from the south. The London Clay was like the other mudrocks deposited in the shallow marine environment, however it is not affected so much by rhythms (*e.g.*, limestone bands/shell beds), due to its more enclosed depositional environment. Recent arguments about depth of burial has resulted in the production of new palaeogeographic maps for the Late Cretaceous (Cope, 2006b), showing a more open sea environment. The palaeogeographic maps are subject to change as more data become available.

Different locations may have experienced different palaeo-water depths, sometimes littoral facies mark the location of landmasses relative to the area of deposition. However with many of the Jurassic mudrocks such near shore materials are not preserved, making the interpretation of land mass locations more difficult (Taylor, 1995)

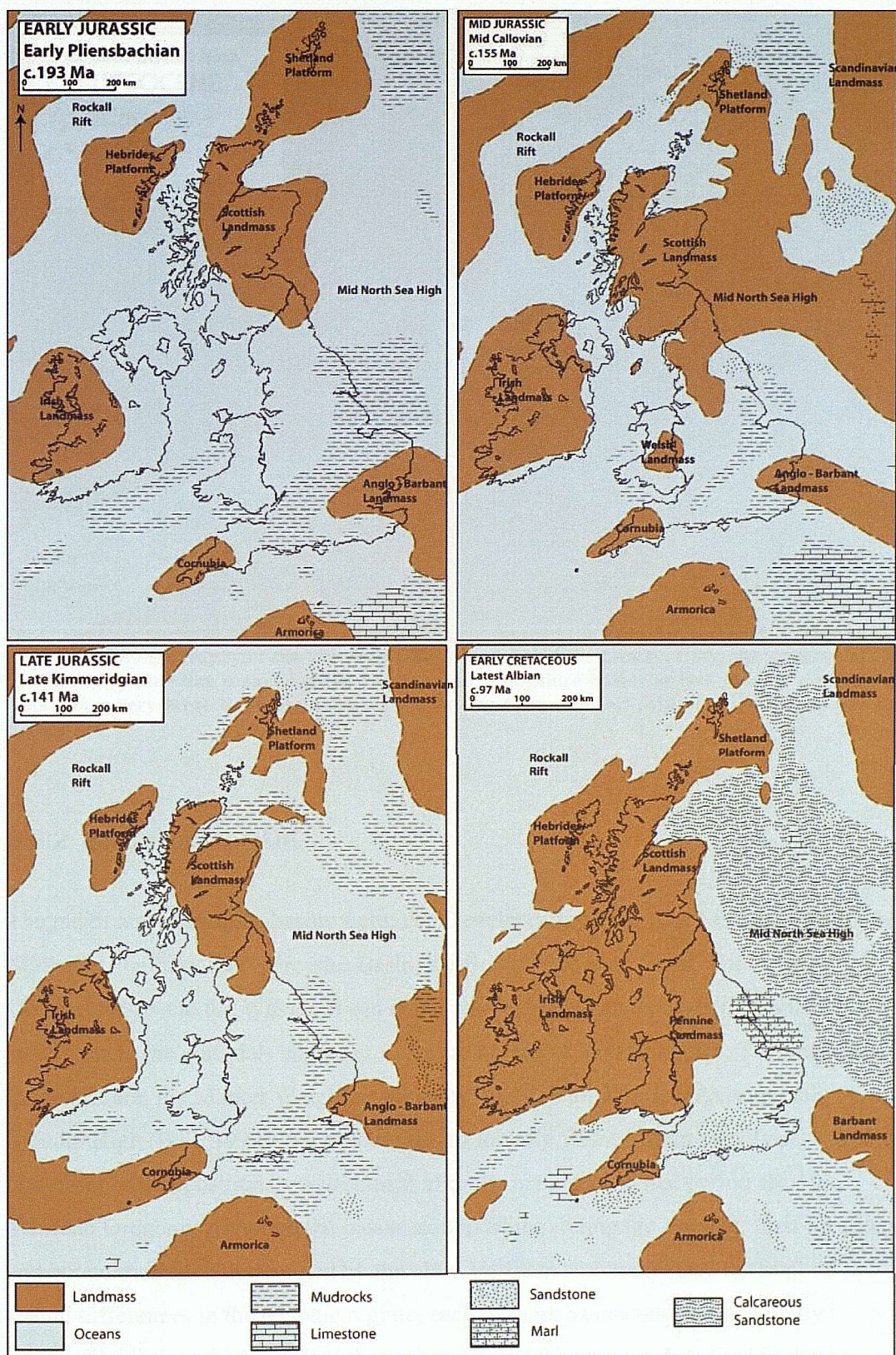


Figure 3.8 The palaeogeography of the UK during the deposition of the (a) Lower Lias Clay (b) Oxford Clay (c) Kimmeridge Clay & (d) Gault Clay. The outline of the UK during the present can be seen on each picture. Brown indicates land, light blue indicates water. All of the clays were deposited in fairly shallow water sheltered between a number of small islands (Bradshaw *et al.*, 1992; Hancock and Rawson, 1992).

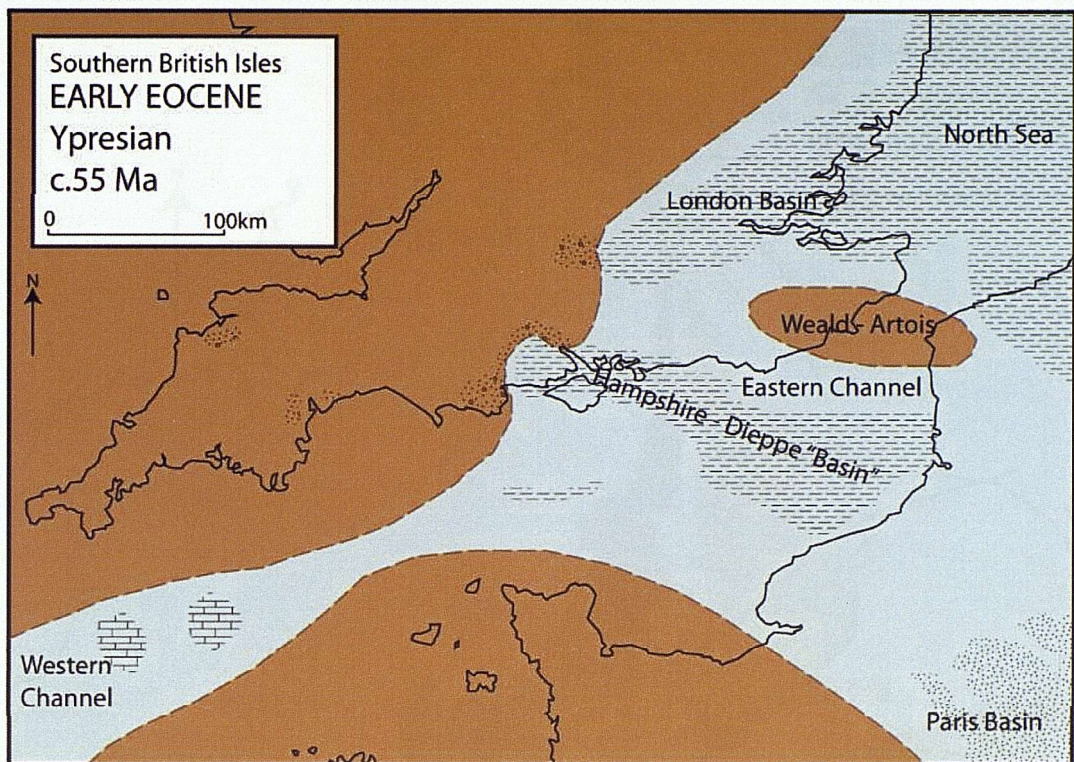


Figure 3.9 The geography of the UK during the deposition of the London Clay, the presence of the Weald-Artois anticline separating the London and Hampshire basins at this point is questioned. This was a very large basin extending to the east as far as Poland (Murray, 1992). For key see Figure 3.8.

3.3.2 Basins of the UK

The major active Jurassic basins were: the Cleveland Basin the East Midlands Shelf, the Midlands Platform, the Worcester Basin, the Bristol-Radstock Shelf, The Bristol Channel Basin and the Wessex Basin (Figure 3.10). The boundaries between these basins where the materials character alters are known as structural axes. The main axes in the UK are, the Market Weighton Structure, the Vale of Moreton Axis and the Mendip High (Bell, 1994). All of these basins initially formed during the Triassic as the UK resumed a depositional environment after the mainly erosional period after the Variscan Orogeny. Although the basins started filling during the Triassic, their locations and boundaries were determined by the older Variscan structures. Due to temporal and spatial differences in the tectonic regime, each of these basins underwent slightly different histories which affected the rock types and thicknesses deposited in them (Sellwood and Sladen, 1981).

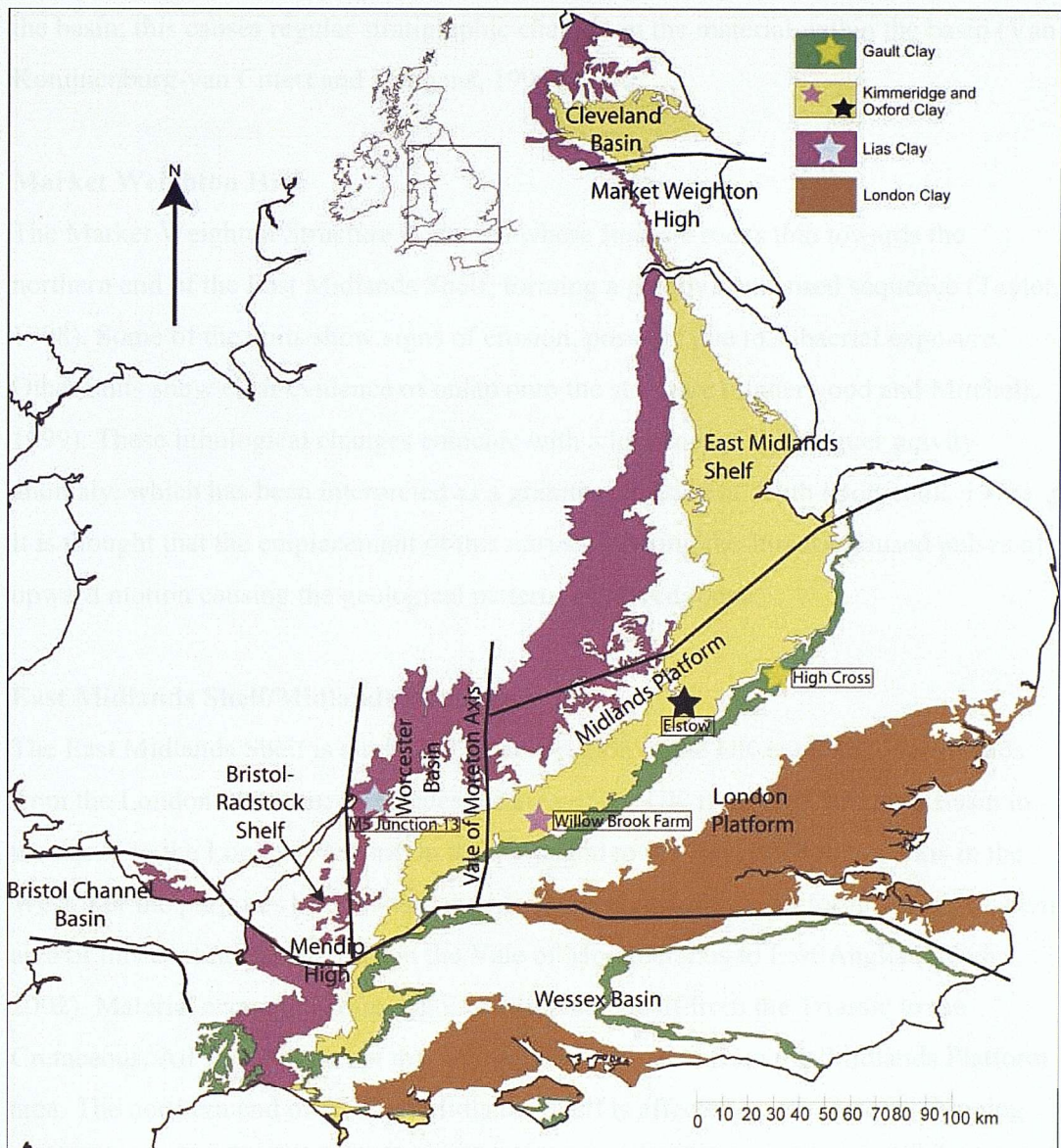


Figure 3.10 The basins of the UK, geology after (British Geological Survey, 2008) UK basins after (Cox *et al.*, 1999). Stars indicate the sampling locations for each of the mudrocks.

Cleveland Basin

The Cleveland basin located in the northeast of the UK, bounded to the south by the Market Weighton High, the northernmost boundary of which is the Howardian – Flamborough fault belt. The Cleveland Basin accumulated sediments from the Triassic to the Cretaceous. The Howardian – Flamborough fault belt marks the northern most boundary of the relatively stable East Midlands Shelf. The Cleveland Basin is a small extensional basin, formed due to differential movement across eastern England (Rawson and Wright, 1995). The movements of both the Market Weighton Structure and Howardian – Flamborough fault belt caused sudden changes in the water depth across

the basin; this causes regular stratigraphic changes in the material within the basin (Van Konijnenburg-van Cittert and Morgans, 1999).

Market Weighton High

The Market Weighton Structure is an area where Jurassic rocks thin towards the northern end of the East Midlands Shelf, forming a greatly condensed sequence (Taylor, 1998). Some of the units show signs of erosion, possibly due to subaerial exposure. Other units show clear evidence of onlap onto the structure (Underwood and Mitchell, 1999). These lithological changes coincide with a local negative Bouguer gravity anomaly, which has been interpreted as a granitic intrusion at depth (Bott et al., 1978). It is thought that the emplacement of this intrusion during the Jurassic caused pulses of upward motion causing the geological patterns observed today.

East Midlands Shelf/Midlands Platform

The East Midlands Shelf is a relatively stable region of the UK extending northwards from the London Platform; it includes the area of the UK from the Cleveland Basin in the north, to the London Platform in the south and to the Vale of Moreton Axis in the West. For the purposes of this work the Midlands Platform is used for the south western area of this continuous shelf, from the Vale of Moreton Axis to East Anglia (Hendry, 2002). Material accumulated on the East Midlands Shelf from the Triassic to the Cretaceous. All samples except that of the Lias Clay came from this Midlands Platform area. The northern end of the East Midlands Shelf is affected by the material thinning caused by the Market Weighton Structure (Gaunt et al., 1992). The Midlands Platform is free from the influences of this structure.

Vale of Moreton Axis

The Vale of Moreton Axis forms the boundary between the stable London platform and the Worcester Basin. The thickness of deposits within the Worcester Basin is greater than that on the London Platform. It is thought that the valley was a previous route for the Thames in more recent times, and the inversion of the Worcester Basin has produced an offset in the Cotswold (Middle Jurassic) Hills (Lane et al., 2008; Watts et al., 2005). The movement of the fault along the Vale of Moreton controlled the deposition within the Worcester Basin mainly from the Permian through to the Early Jurassic. It is thought that the Vale of Moreton axis becomes less important into the later Jurassic

Worcester Basin

The Worcester basin is located to the west of the London Platform, and to the north of the Wessex Basin. Its boundaries are the Vale of Moreton to the East, the Malvern Fault to the west and the Mendip High to the south. Sedimentary deposition initiated in the Worcester Basin during the Triassic. It contains a very thick (up to 2500m) sequence of Triassic sediments (Chadwick, 1985). Sediment accumulation ceased during the Cretaceous. It is thought that the accommodation space for this material formed due to crustal extension. The magnitude of this process had greatly decreased by the start of the Jurassic and had ceased towards the end of the Jurassic. During the Late Jurassic, materials deposited in this basin were of a similar thickness to that on the Midlands Platform (Green, 1992).

Bristol Radstock Shelf/ Bristol Channel Basin

The Bristol Radstock Shelf and the Bristol Channel Basin are located in the Western UK. Both of these areas of the UK are considered to be marginal facies, very different in presentation to the materials across the rest of the UK. The Bristol Radstock shelf contains a condensed sequence of the materials in the nearby Worcester Basin (Cox et al., 1999). The materials in the Bristol Channel Basin are more similar in character to those in the Wessex Basin, however they are a marginal facies of these materials. For example the Blue Lias within the Bristol Channel Basin contains more limestone than within the Wessex Basin (Nemčok et al., 1995). The basin contains Triassic and Jurassic materials, Cretaceous materials may have been deposited, however none remain, the inversion of the basin during the Tertiary will have led to substantial erosion

Mendip High

The Mendip High marks the boundary between the Worcester Basin to the north and Wessex Basin to the South. The boundary is the Variscan tectonic front. The Mendip Hills are described as four *en-echelon* periclinal folds of Palaeozoic rocks (Wall and Jenkyns, 2004). The most well known material of the Mendips is the Carboniferous Limestone. These periclinal folds represent the northern effects of the Variscan compression. Many of the early Variscan structures were later reactivated up to the Alpine Compression, this means the material to the south in the Wessex Basin has been affected by tectonic deformation, whereas the materials to the north have experienced much less of these effects.

Wessex Basin

The Wessex Basin is located across the south of the UK to the south of both the London Platform and the Worcester Basin. The basin contains many east-west trending faults, it has a much more complex structure compared to the rest of the UK. These faults were originally formed during the Variscan compression, from the Permian to Cretaceous, material was deposited in the Wessex Basin and the basin underwent extension reusing many of the Variscan faults. During the Alpine collision the basin was inverted also reusing the Variscan structures (Taylor et al., 2001). The basin structure has been imaged using deep seismic measurements (Chadwick et al., 1983). Three major fault zones can be identified, these align with the Vale of Pewsey, the Vale of Wardour and the Portland –Wight Fault (Chadwick, 1986). The Wessex Basin contains one of the thickest sequences of onshore Jurassic material (Cope, 2006a).

Basins can be subdivided or change name with geological time. For example during the Cretaceous the Wessex Basin is subdivided into two regions, the Wessex Basin to the West and the Wealden Basin to the East. The Wealden Basin suffered enhanced deposition during the Cretaceous compared to what occurred further west (Allen, 1981; Hopson et al., 2001). The Wealden Basin was inverted during the Tertiary reversing the structures present (Chadwick and Kirby, 1982).

The London Platform/London Basin

The London Platform is located in the south eastern UK, it is surrounded by the east Midlands Shelf to the north, the Worcester Basin to the west and the Wessex Basin to the south. It is an area of prolonged stability against which many Jurassic rocks onlap, it is thought to have been a landmass for much of the Jurassic (Brenchley and Rawson, 2006). During the Cretaceous rises in sea level did submerge the London platform. And deposition started to occur. Thus the platform turned into a basin into which the London Clay was deposited. The London platform/Basin is a good example of how basins change with time. Deposition continued in the London Basin during the Tertiary. The London Basin was an isolated offshoot of the southern part of the North Sea.

One of the aims of this work was to isolate material age as the subject of study, and compare each of the older mudrocks with the London Clay. The above discussion shows the extent of the variability of the different basins. The open seaways which

dominated the environments of the Jurassic and Cretaceous are different to the isolated environment of the London Clay. There are some similarities in the environment of deposition. They are all shallow continental shelf environments into which mudrocks are being deposited. However the processes occurring within the environment, the mineralogy, the climate, silt contents and the organic content of the mudrocks are all different. Thus age is not a factor which can be separated out from the others.

3.4 *Reconstruction of material history*

The reconstruction of the depositional histories of each of the mudrocks studied here will be discussed in future chapters. The reconstruction of the burial and uplift histories of our materials requires the correlation of multiple techniques in time-depth space. The most complete burial and uplift histories can be obtained from apatite fission track analysis (AFTA). However the 10° (333m) error bounds are quite large (Figure 3.11). This initial burial history of most materials can be obtained by adding up the stratigraphic thicknesses of materials which would have been above them. This takes into account all materials currently present, however cannot account for materials which were present and have been subsequently eroded. For the East Midlands Platform the most recent material which was certainly deposited above our sites was the Chalk. AFTA suggests that the materials across much of the UK were buried to a much greater depth the material under which it was buried subsequently being removed. The very recent uplift history of these materials can be estimated based on marine and river terrace heights the one data point used here is a surface 135m high from 3.1Ma.

The burial histories used (Jackson and Fookes, 1974) are based on estimates for the Oxford Clay in Bedfordshire. For the Lias Clay samples these data are augmented by material thickness data for the Worcester Basin (Brenchley and Rawson, 2006; Cox et al., 1999). However due to the scale and variability in geological thicknesses within the Worcester Basin these data are most uncertain. The AFTA data (Green et al., 2001) are based on the closest data to our sites which in many cases is a long distance away. The data for the Midlands Platform can be used with some confidence. The data for the Lias Clay was taken from further to the north as no data are available within the Worcester Basin. It is certain that the depth of burial is a little deeper than that of the Midlands Platform for the early part of the Jurassic, coming more into line with the thicknesses

deposited in the Midlands. However this depth is not likely to be wildly different (Carter et al., 1995).

The uplift data point (Westaway, 2010) is taken from South Devon, the record of uplift is more extensive in the south of the UK than in the north as the north was heavily eroded during the glaciations. The south of the UK was not covered with ice and thus older river terraces have survived. This site is a long way from where our samples have come from and so may not be completely representative of the uplift at the site. However it is a good estimate of the magnitude of uplift that the site would have experienced.

The burial and uplift trajectories which can be estimated from the stratigraphic evidence broadly agree with that produced by AFTA suggesting that the great depths of burial produced by this method may indeed be accurate.

3.5 Summary

There are many possible ways in which the wide variety of geological environments can satisfy the simple definition of mudrock based on grain size. Mudrocks may therefore exhibit a wide variety of structures on deposition. The variability of mudrocks and their histories limits the extent to which the data presented in this thesis can be applied to other locations and/or stratigraphic horizons. The structure of the mudrock evolves as the environment of the material changes. One of the most important changes geological materials experience is burial and uplift. The burial and uplift histories of each mudrock can be estimated using stratigraphy, river terraces and palaeothermal measurements, the best estimates of this history for each of the mudrock is presented here (Figure 3.11).

The detailed variations in geological history cannot be observed in a material's microstructure. The most important factor from the burial and uplift histories for comparison between materials is the maximum burial depth.

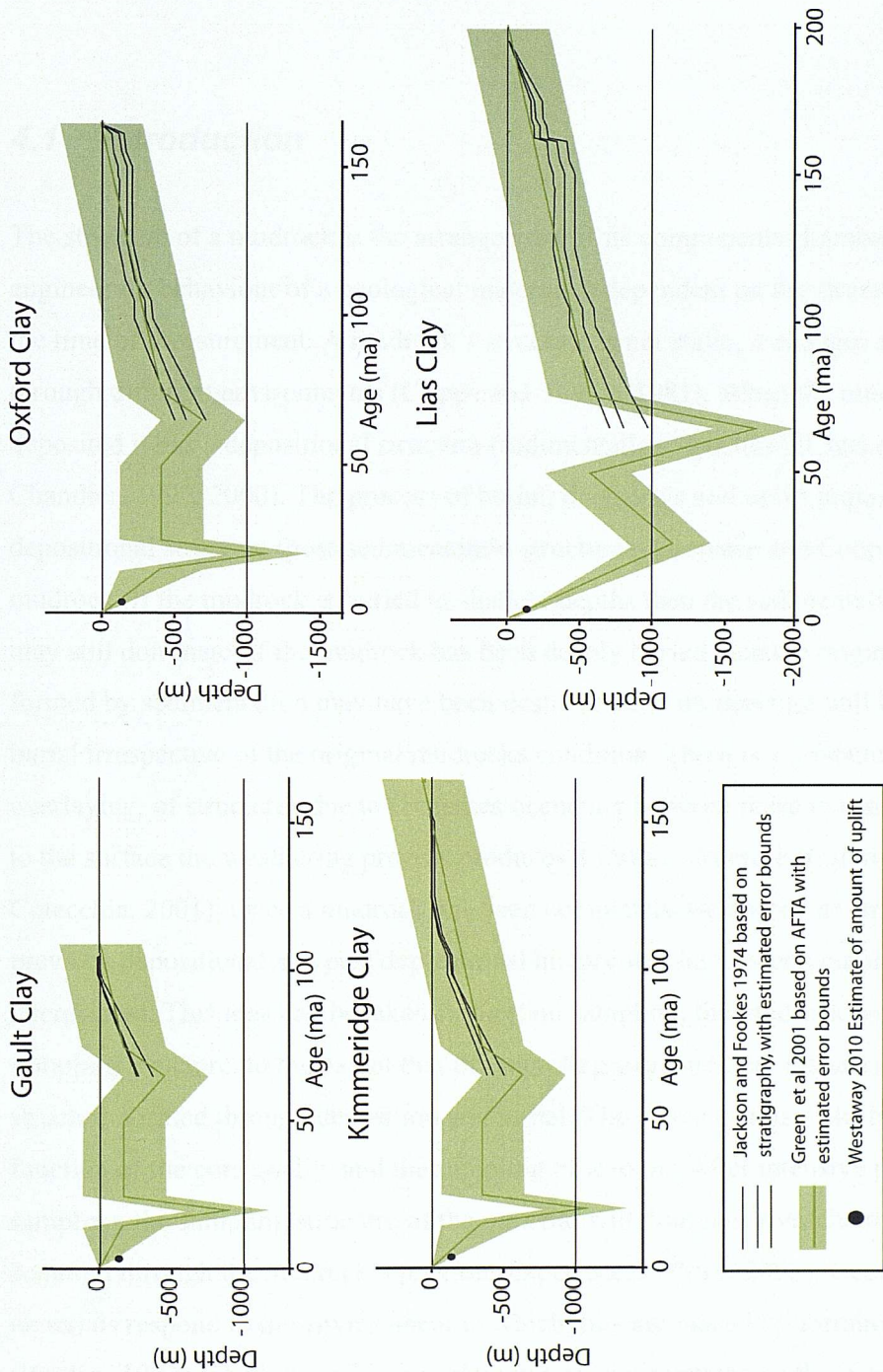


Figure 3.11 Estimates of past depth of burial in depth-time space for the Gault, Kimmeridge, Oxford and Lias Clay sites. The present day is at the top left of all graphs at (0,0). In green is the full burial curve based on apatite fission track analysis using the closest reliable data available for each sampling location based on (Green et al., 2001). The black lines are measures of the probable stratigraphic amount of material which is likely to have been present above each sampling site during burial based on materials which are present on the earth's surface today (Brenchley and Rawson, 2006; Cox et al., 1999; Jackson, 1972; Jackson and Fookes, 1974). Finally the dot in the top left is an indication of uplift magnitudes based on river terraces which can be measured today (Westaway, 2010).

CHAPTER 4

STRUCTURE & MICROSTRUCTURE, ANISOTROPY & PREFERRED PARTICLE ORIENTATION

4.1 *Introduction*

The structure of a mudrock is the arrangement of its components (Lambe, 1958). The engineering behaviour of a geological material is dependent on the structure present at the time of measurement. A mudrock's structure is not static, it changes as it evolves through different environments (Cripps and Taylor, 1981). When the mudrock is first deposited it has a depositional structure (sedimentation structure) (Cotecchia and Chandler, 1997, 2000). The process of burial, diagenesis and uplift imposes a post-depositional structure (post-sedimentation structure) (Gasparre and Coop, 2008) on the mudrock. If the mudrock is buried to shallow depths then the sedimentation structure may still dominate. If the mudrock has been deeply buried then the original structure formed by sedimentation may have been destroyed and its structure will be solely due to burial irrespective of the original mudrocks condition. There is a gradation, or overlaying, of structures due to processes occurring between these two states. On return to the surface the weathering process produces a further structure (Cafaro and Cotecchia, 2001). Once a mudrock has been completely weathered its structure from its previous depositional and post depositional history will have been completely overprinted. This idea can be taken further; on sampling, the mudrock will acquire a sampling structure, to the extent that the sampling structure may disturb (overprint) the structure formed through deposition and burial. The extent of this disturbance is a function of the core quality and the sampling procedure. After intensive poor quality sampling, the sampling structure of the material will dominate over the structures acquired through the mudrock's previous experiences (Price, 2008). Geological materials respond to the environment in which they are placed by forming structure (Barden, 1972). The longer they are placed in an environment, or the more intensively the environment is applied to them, the more pervasive the structure becomes. Thus, the structure of a mudrock at the time of measurement is dependent on its all of the events it has experienced. Therefore the insitu behaviour of a mudrock will be primarily dependent on its geological history (Chandler, 2000; Fookes, 1997)

4.2 Definition

In the laboratory the structure of geological materials is measured as the difference in response to testing between an intact, undisturbed sample and a reconstituted sample of the same material. The term structure is often subdivided into the arrangement of the particles (fabric) and non frictional inter-particle contacts (cementation, or bonding) (Foster and De, 1971; Mitchell and Soga, 2005). Fabric and structure are synonymous in unbonded materials, these are mainly recently deposited or near surface samples (Sivakumar et al., 2002).

The term microstructure is used to describe the physical arrangement of particles measured through observation (normally by imaging at the microscale). A given mudrock may contain a wide variety of geological structures across a range of scales including: discontinuities (*e.g.*, bedding, joints, faults, and desiccation cracks), orientation of particles, aggregations of particles, fossils, sedimentary structures (*e.g.*, varves, cross bedding, ripple marks, flame structures, desiccation cracks), and variations in grading/sorting (Van Loon, 2009). These heterogeneities give rise to a range in the testing behaviour of mudrocks. In order to define the variability of the fabric of a mudrock the structures present in a given geological horizon must be located and their uniformity throughout that horizon must be quantified. The majority of mudrock fabrics which have been identified are repetitive, pervasive, ordered arrangements of particles. Local differences to this overall fabric such as nodules or discontinuities do occur, and may indeed be important in terms of the mudrocks behaviour. However these macroscale heterogeneities are not important in terms of the overall measured micro-fabric of the mudrock.

Cements are mineral growths which often form after deposition during diagenesis. Bonding is the measurable effect of certain types of cement. The cement must be extensive enough to be a load bearing element of the mudrock system. Common cementing minerals include quartz, calcite, clay minerals, gypsum, halite and iron oxide. The properties of cementing minerals may vary greatly from those of the mudrock grains. Generally the presence of cement allows a mudrock to sustain a void ratio to a higher stress than a reconstituted sample (Leroueil and Vaughan, 1990). However there is no reason why a natural structure cannot cause a mudrock to attain a given void ratio

at a lower stress than the reconstituted material. The effect has been observed in scaly clays due to a large number of shear surfaces within the material (Vannucchi et al., 2003).

A mudrock system may contain a structure at one scale, however at other scales the mudrock may be dominated by a different structure (Potter et al., 2005). The quantification of the order of a mudrock must include a recognition of the disordered elements, and the scales at which order/disorder dominate. Likewise the bonding observed in a mudrock may not be uniform. Between any two given particles there may be strong cement or there may be a void. The extent of cement in a mudrock will be dependent on its geological history in particular, the duration and extent of any diagenetic process. However the existence of bonding in a given location cannot be predicted.

Anisotropy is a variation in the properties of a material with direction. The most important cause of this is a preferred particle orientation which can be measured through imaging.

Several terms are used to describe clay minerals and clay mineral associations. The most common are: particle, chain, floc and domain (Bennett et al., 1981). Particle is used for a clearly defined unit of material (Figure 4.1). This is different to how it is used in Figure 2.7 to describe separated regions of the same mineralogy. A chain is a series of particles linked face to face or edge to edge in a linear arrangement (Figure 4.1), it is common in depositional fabrics but is not observed in consolidated materials. A floc is a well defined series of particles which contains voids within its structure (Figure 4.1). A domain is a stack of near perfectly arranged particles (Figure 4.1), this is commonly observed in kaolin (Bohor and Hughes, 1971), however it is rare in other minerals. In deeply buried mudrocks aggregates of clay minerals particles are common. Aggregates are defined as a common association of particles which are distinct from the surrounding material (this is a general term which could also be used to describe a floc or a domain).

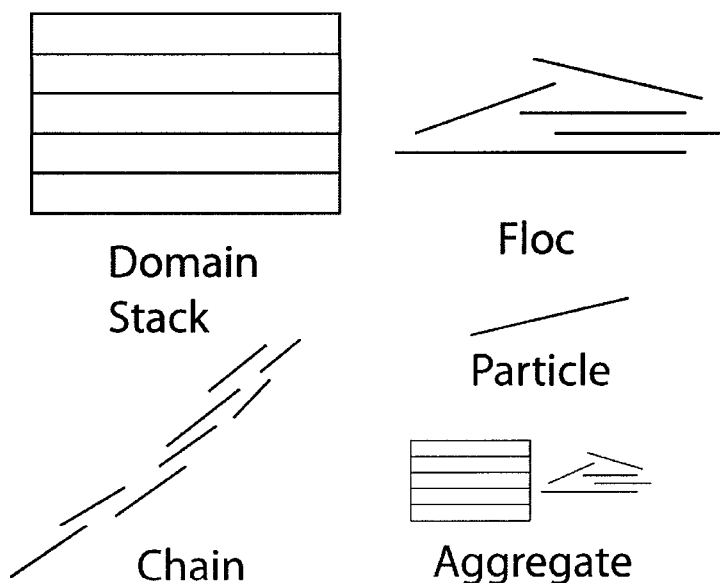


Figure 4.1 Arrangements of clay particles

4.3 *Microstructure: a historic review*

To date, most investigations of the microstructures of mudrocks focus on the arrangement of the platy particles; these are the most obviously identified particles in any small scale mudrock image. They are also the particles from which the strongest indications of preferred particle orientation can be measured. The earliest ideas about the structure of mudrocks, theorised that the particles were small enough that molecular forces might cause them to adhere to each other (Sloane and Kell, 1966). This led to the suggestion of the honeycomb fabric (Figure 4.4). Observation of mudrock particles was very difficult before the invention of the electron microscope, and even with early microscopes, details of individual particles remained hard to unravel. Early models of the microstructure of mudrocks were based on simple observations of materials and their properties. Diagrams of open structures on initial deposition were produced. It was assumed that these were then compressed during loading/burial to form stronger particle alignment (Figure 4.2). Other hypothesised structures were presented showing all of the geometrical arrangements that may form from the platy particles in a fluid suspension just prior to deposition (Figure 4.3). Some of these structures have been subsequently observed (Barden and Sides, 1971).

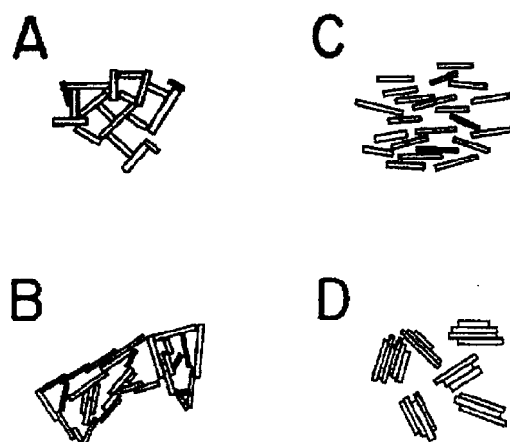


Figure 4.2 Very early ideas about the development of preferred orientation with depth. A and B card house and book house respectively (Lambe, 1958) which would then compact to C or D preferentially orientated and orientated into domains respectively from (Meade, 1966)

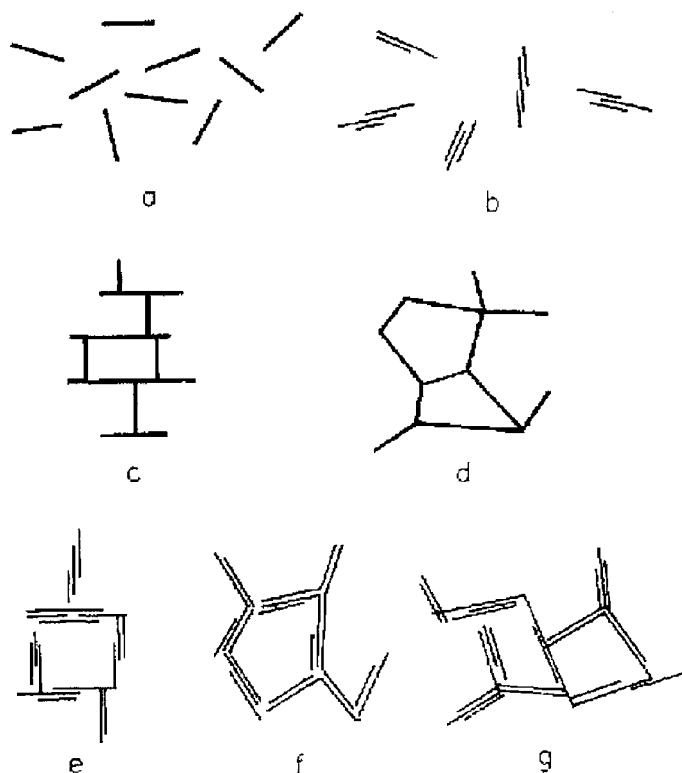


Figure 4.3 Theoretical concepts developed on the basis of colloid suspension chemistry showing the sorts of structures which might be observed in clay mineral particles prior to or during the very early stages of deposition. A, dispersed and deflocculated. B, aggregated but deflocculated. C, Edge Face flocculated but dispersed. D, Edge Edge flocculated but dispersed. E, Edge Face flocculated and aggregated. F, Edge Edge flocculated and aggregated. G, Edge Edge and Edge Face flocculated and aggregated From (Moon, 1972) originally from (Van Olphen, 1963).

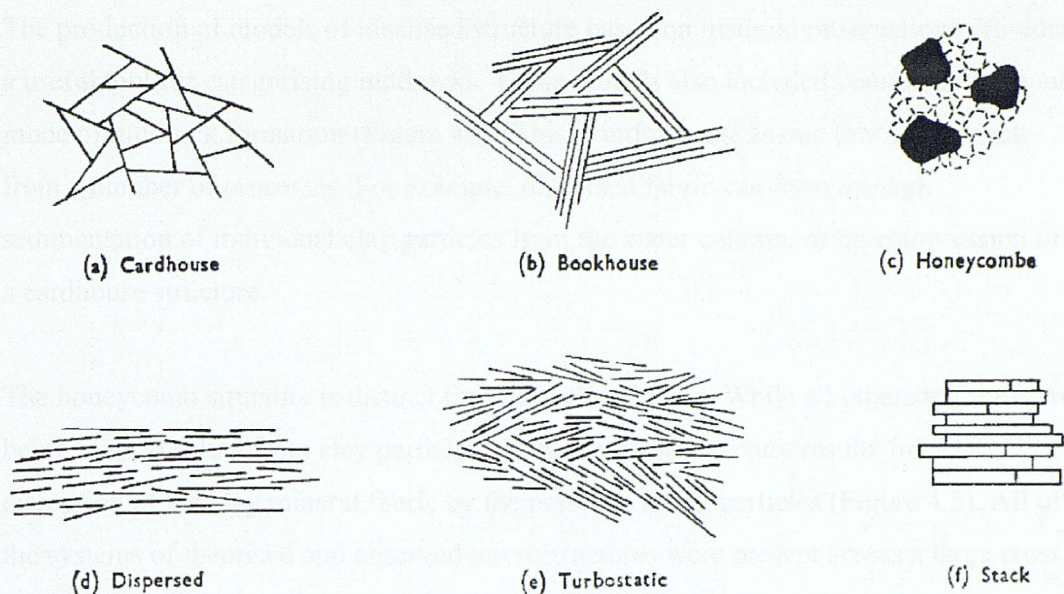


Figure 4.4 a, & b, flocculated structures. c, based on (Casagrande, 1932) d, closely packed face to face structure. e, turbostatic more realistic than dispersed (Aylmore and Quirk, 1960) from (Barden and Sides, 1971). They state that actually structure of three dimensional so is often harder to identify than these indicate.

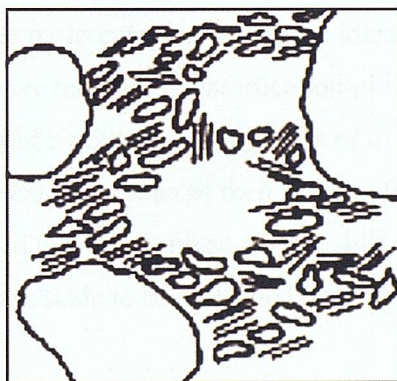


Figure 4.5 Clay binding larger silt particles and preventing relative movement (Beroya et al., 2009). Similar to honeycomb structure (Casagrande, 1932).

The production of models of idealised structure based on material observations provided a useful tool for categorising mudrocks. These models also included connotations about mode of mudrock formation (Figure 4.4). This is unfortunate as one fabric can result from a number of processes. For example, dispersed fabric can form through sedimentation of individual clay particles from the water column, or by compression of a cardhouse structure.

The honeycomb structure is distinct from the other fabrics. While all other structures are being formed solely from clay particles, the honeycomb structure results from the disruption of the clay mineral fabric by the presence of silt particles (Figure 4.5). All of the systems of theorised and observed microstructures were present across a large cross section of the literature. Collins and McGown (1974) collated and summarised a wide variety of both conceptual and observed clay microstructures. The microstructures shown in this paper are still accepted and used for illustrative purposes today (Mitchell and Soga, 2005). They include the structures formed from clay minerals alone (Figure 4.6), and the structures formed from mixtures of clay minerals and silt particles (Figure 4.7). Collins and McGown (1974) identified that the structures they were showing were of early idealised mudrocks, many of which are formed by specific processes. They also recognised that mudrocks are more complex than these idealised models. They put forward a clear, comprehensive method of classification of mudrock fabrics which, unfortunately, was not adopted by others. Descriptions of the complexities of mudrock structure used in addition to categorisation of their fabric, allowed structures which had not previously been discussed to be recognised (Figure 4.8). This allowed the variable density of mudrock at the pore scale to be identified and explained.

Most of the observed fabrics only apply to newly deposited mudrocks, and many of the structures which have been documented are those known to produce specific geomaterials behaviour, for example sensitivity, collapse and expansiveness (Collins and McGown, 1974). These microstructures tend to only represent idealised structures resulting from mudrock depositional environment. These are grouped into end members to form a wide spectrum of microstructures which do not allow analysis of pervasiveness or the level of order of the microstructure in 'real' rocks.

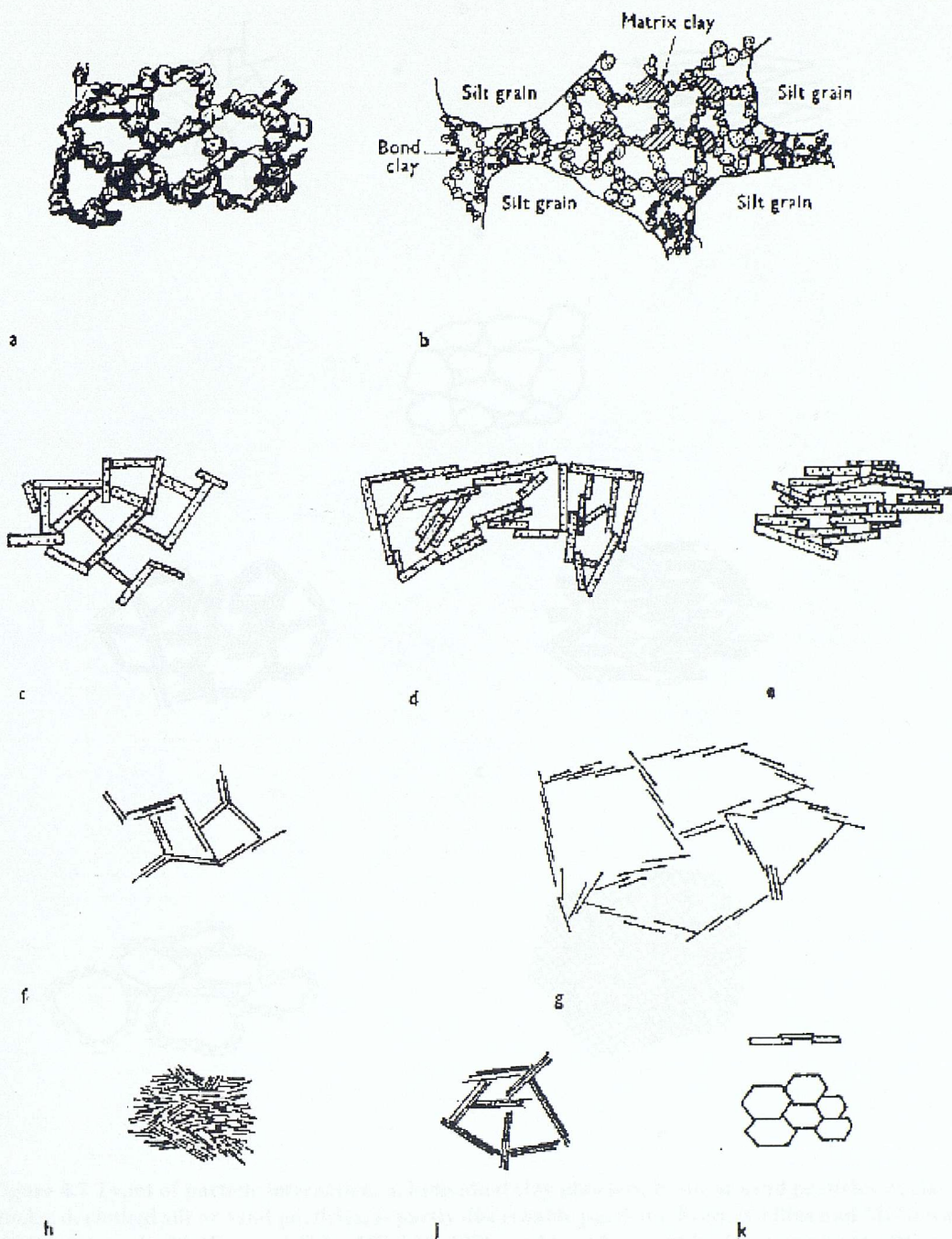


Figure 4.6 Summary of hypothesised and observed mudrock fabrics (Collins and McGown, 1974). (a) Honeycomb arrangement, (Terzaghi, 1925); (b) structure of undisturbed marine clay, (Casagrande, 1932) see Figure 4.4 c; (c) non-salt flocculated arrangement, (Lambe, 1958) ;(d) salt-flocculated arrangement, (Lambe, 1958) ;(e) dispersed arrangement, (Lambe, 1958) see Figure 4.2; (f) EE and FE flocculated and aggregated arrangement, (Van Olphen, 1963) see (Figure 4.3) g; (g) stair step cardhouse arrangement, (O'Brien, 1971); (h) turbostatic arrangement, after (Barden and Sides, 1971) see Figure 4.4 e; (j) bookhouse arrangement, after (Barden and Sides, 1971) see Figure 4.4 b; (k) particle intergrowth in domains, (Bowles, 1968; Smart, 1969);

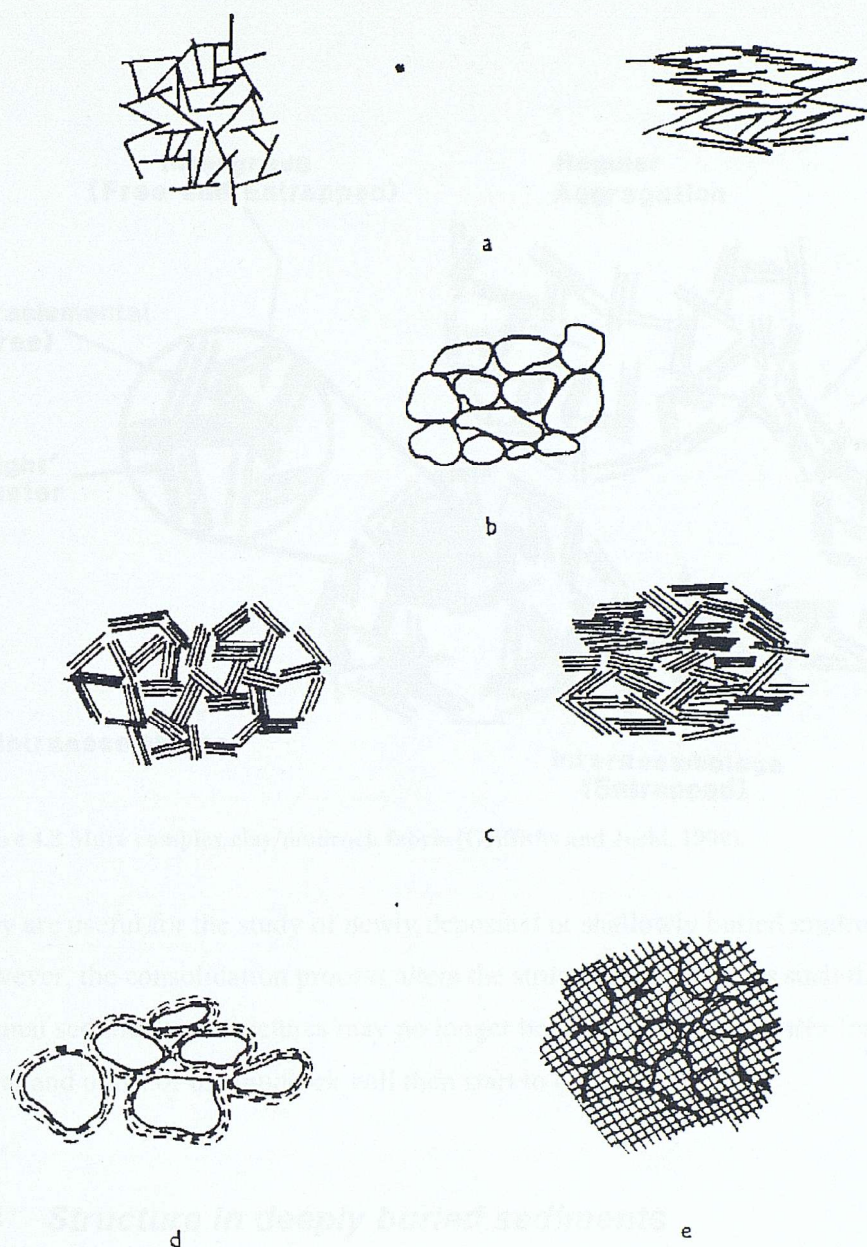


Figure 4.7 Types of particle interaction, a. individual clay platelets, b. silt or sand particles, c. clay stacks, d. clothed silt or sand particles, e. partly discernable particles. From (Collins and McGown, 1974) later used with (Figure 4.6) in (Mitchell, 1976) and is still present in the most recent edition (Mitchell and Soga, 2005).

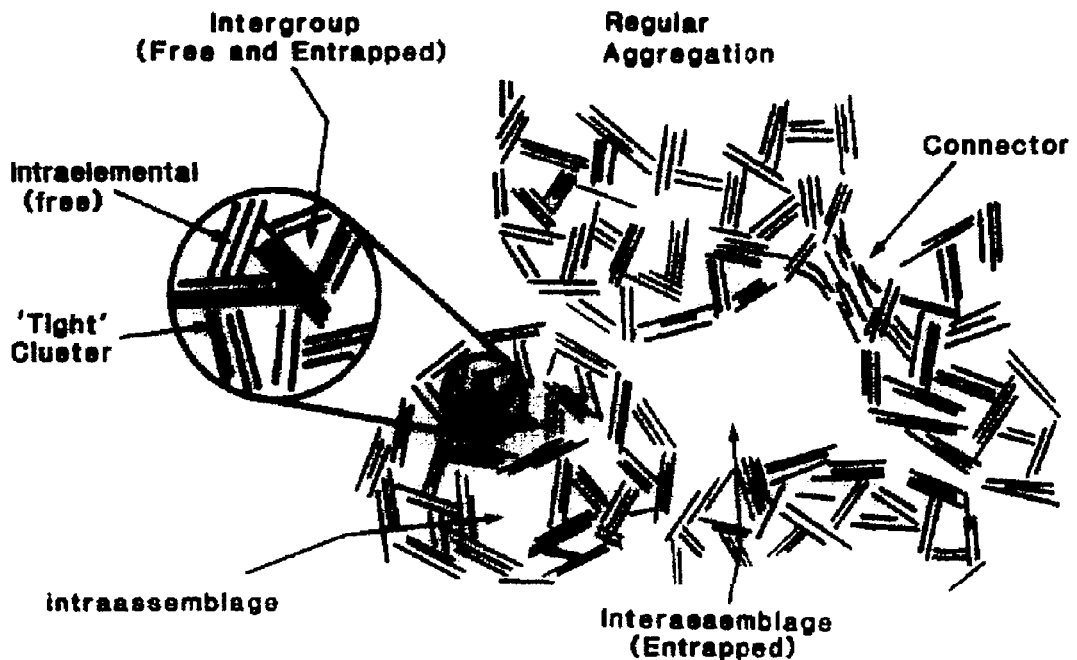


Figure 4.8 More complex clay/mudrock fabric (Griffiths and Joshi, 1990).

They are useful for the study of newly deposited or shallowly buried mudrocks. However, the consolidation process alters the structure of mudrocks such that the original sedimentary structures may no longer be identifiable. Structures formed by the burial and uplift of the mudrock will then start to dominate.

4.4 Structure in deeply buried sediments

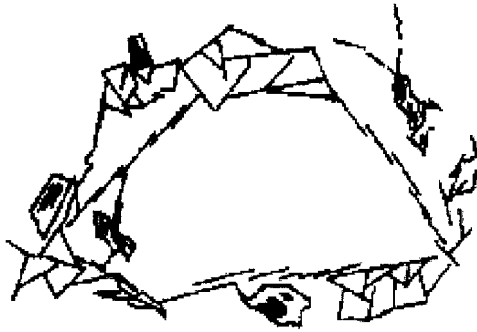
Some of the earliest descriptions of UK mudrocks identify the importance of burial processes on the development of structure in sedimentary rocks (Sorby, 1908). The initial effect of burial (or compaction) is the reduction in the amount of void space (Jones, 1944; Skempton, 1969; Skempton and Jones, 1944). This reduction occurs initially by the reorganisation of particles due to the weight of the overlying material. Particle reorganisation has been observed in several materials by compaction experiments applying load in a laboratory (Djéran-Maigre et al., 1998). The effects of compaction are dependent on the mineralogy of the sample. They are also dependent on the chemistry of the pore water. Experiments with saline pore water show a more open structure under consolidation than the same material with distilled water (Engelhardt

and Gaida, 1963). Initial models of post-depositional structural changes in mudrocks involve taking the initial structures and reducing their void ratios (Figure 4.9). Bennett *et. al.* (1981) specifically state that most models of mudrock structure during compaction are specific to an illite-smectite rich material, the mineralogy of the mudrock therefore determines its behaviour with burial.

Processes observed at shallow depths become of lesser importance as depth of burial increases. For example, pore water pressure resists the reduction in void space of materials. When a mudrock is buried to great depth (2-3km) pore water becomes entrapped and the stresses experienced by the mudrock increases towards lithostatic stresses (Goult, 1998). Consolidation processes at the depth where this happens occur less due to particle reorganisation (the particles have “locked up”) and more due to pressure solution and other diagenetic processes (Oertel, 1983). The major effects of diagenesis in mudrocks are, the conversion of smectite, kaolinite and chlorite (in the presence of K-feldspar) to illite, and the formation of cements (Gutierrez *et al.*, 2008). The growth of cements due to dissolution and reprecipitation tends not to eradicate pores or pore interconnectivity. The closing of pores produces areas of higher pore pressure, which causes dissolution (Katsube and Williamson, 1994). Diagenetic processes are discussed further in the next chapter (Chapter 3).

Mudrocks which have been returned to the surface after being at a considerable depth tend to be either fissile (break along parallel planes) or massive. The difference in the material which causes this variation in behaviour is compositional (Ingram, 1953). The presence of organic matter also plays an important role, increasing the tendency towards parallel arrangement of clay particles. In rocks or well cemented materials the presence and orientation of microcracks (another component of microstructure) are important elements for the study of the mechanics of failure (Xia *et al.*, 2008).

In summary the sequence of changes a mudrock will undergo with depth are: mechanical rearrangement, dewatering, mechanical deformation, and finally recrystallization (Reeves *et al.*, 2006). The extent to which these processes have occurred in a mudrock is related to the depth it has been buried to, the time which it has spent there and the fluid flow through the system. The structure of deeply buried mudrocks is commonly thought to be measurable as preferred orientation.



'SINGLE PLATE LIKE'
PARTICLES AND CHAINS

VERY HIGH VOID RATIO

> 3.0



DOMAIN PARTICLES
AND CHAINS

HIGH VOID RATIO

> 2.5



MEDIUM TO HIGH
VOID RATIO

$\sim 1.5-2.5$



LOW VOID RATIO

< 1.5



VERY LOW VOID RATIO

< 1.2

Figure 4.9 Modes of clay fabric proposed for smectite/illite rich materials from (Bennett et al., 1981). The recognition of the importance of mineralogy to mudrock fabric.

It is true that with depth platy particles of a mudrock become more aligned. However depth of burial is not the only parameter defining preferred orientation. Mineralogy, pore water chemistry and composition (including organic matter) are all important parameters in this respect.

4.5 Laboratory analysis

As previously discussed, mudrock structure can be studied from two different directions. The majority of this thesis covers the imaging of the physical particles. The other method of studying structure is by studying its effects using geotechnical laboratory tests. The laboratory responses of the mudrocks studied here are contained in companion theses discussing the lab-based aspects of this project (Brosse, 2011; Hosseini-Kamal, 2011). At the time of writing these works were not complete. The results contained in these theses are based on index, oedometer, triaxial and hollow cylinder tests. The importance of mudrock fabric was selected as the topic of the twelfth Rankine Lecture (Rowe, 1972). Rowe (1972) discusses the identification of structure in the field and then associates it with laboratory behaviour. The effects of structure are observed in the laboratory as the difference in behaviour of a structured material from a datum (Amorosi and Rampello, 2007; Low et al., 2008). The datum most often used is a sample reconstituted from the original material. The deviation of the intact material behaviour from that of the reconstituted material is caused by the arrangement of the constituent particles and their bonding, (Leroueil and Vaughan, 1990). This does not mean the reconstituted sample has no structure. In the reconstituted sample, the structure formed throughout the material's history, is replaced by the structure formed by the reconstituting process, *i.e.*, consolidation from a slurry (Bennett et al., 1981). Therefore, the structure of the reconstituted sample differs from that formed in situ, effects of compaction and other processes are removed. As the reconstituted material has been used as a datum for structure for many years, it is thought of as having no structure, or as being a de-structured material (Baudet and Stallebrass, 2004).

The classical understanding of structure is the property of a material which enables it to sustain a void ratio to a higher vertical compressive stress than it would be able to attain in its reconstituted form. Recent work has differentiated effects of fabric and bonding on geotechnical laboratory testing results (Baudet and Stallebrass, 2004). A material with fabric, on compression, can sustain a void ratio greater than that of the reconstituted

sample to very high pressure. A bonded sample however will break down to the same behaviour as the reconstituted sample above a given vertical stress, once the bonds have been broken. It is difficult to relate the particle arrangement and cementation of the material directly to its laboratory behaviour without computer modelling. Not all the cement will break during testing, however clearly different processes are being observed.

Deeply buried and highly cemented mudrocks start yielding before they reach high stress levels. They do not tend to cross the compression line for reconstituted samples. Their structure may show as a stiffer response to testing (Gasparre and Coop, 2008). Even this response is not remarkable; some researchers suggest that a better measure of the structure of a material may be seen in shearing tests (Gasparre et al., 2007a).

As well as structure, fabric, and bonding, one of the key measurements of the structure of mudrocks is anisotropy. Anisotropy is a measure of different properties in different directions. Commonly strength and stiffness anisotropy are measured. This should correlate well with preferred particle orientation, which is one of the few measurements of microstructure which can be easily made. Anisotropy is normally measured using the hollow cylinder testing apparatus (Nishimura et al., 2007). Anisotropy of strength of a material as a whole can also be estimated in a triaxial apparatus by using several samples tested as different orientations (Attom and Al-Akhras, 2008). Other measurements of anisotropy include, shear wave velocity (Sarout et al., 2007), permeability, and the alignment of voids and minerals. Shear wave velocity correlates with pore and mineral anisotropy and rock textural features such as bedding. Seismic CPT testing has been used as part of this project to validate the laboratory testing (Brosse, 2011; Hosseini-Kamal, 2011). In fact, all the different anisotropies of a material are interrelated. By assuming this link between preferred particle orientation, permeability anisotropy and seismic anisotropy, particle preferred orientation can be estimated in the field by seismic surveying (Bates and Phillips, 2000). Although such a correlation is known to exist, its extent has not been fully tested. Another anisotropic property of mudrocks is their thermal conductivity (Midttømme and Roaldset, 1999). This has also been correlated to preferred particle orientation.

A further effect is the increase in stiffness of samples the longer they remain in storage (Santagata and Kang, 2007; Shibuya, 2000). This effect is known as aging. It is thought

that aging involves the formation of structure. The effect has been observed in Norwegian quick clays (Rosenqvist, 1953). However the actual processes and effects at the micro-structural level have not been observed. It has also been suggested that the effects of geological time on a material may mirror the aging effects observed in the laboratory. However, this is unlikely as the processes which occur to a sample in storage (*e.g.*, desiccation) are likely to be different to those occurring in the field. Across geological time the environments in which a material experiences will change and so will the rates of the processes.

4.6 Analysis of material fabrics

Structure measured in the laboratory is quantified as a numerical difference, whereas a particle arrangement is not readily quantifiable (one of the objectives of this research is to address this issue. A large difference from the datum is thought of as a large effect of structure, and a small difference a small effect. In contrast mudrock observations may show many different microstructures all of which are highly ordered, with each producing a different laboratory result relative to the reconstituted (also regular) microstructure. For this reason the quantification of microstructure is not a simple process. Therefore the main aim of analysing the microstructure of a mudrock is to find the factors which control its overall behaviour, and use this combined understanding of all these factors to predict how a mudrock will behave on loading.

4.6.1 Sands

The study of the microstructure of sands is much further advanced than that of mudrocks. Even though they are vastly different materials they are a useful starting point to consider the methods used in mudrocks. The best imaging method for use on sands is Computed Tomography (CT) as this produces a full 3D image of the arrangement of the material. Unfortunately CT does not have the resolution capability to apply it to mudrocks.

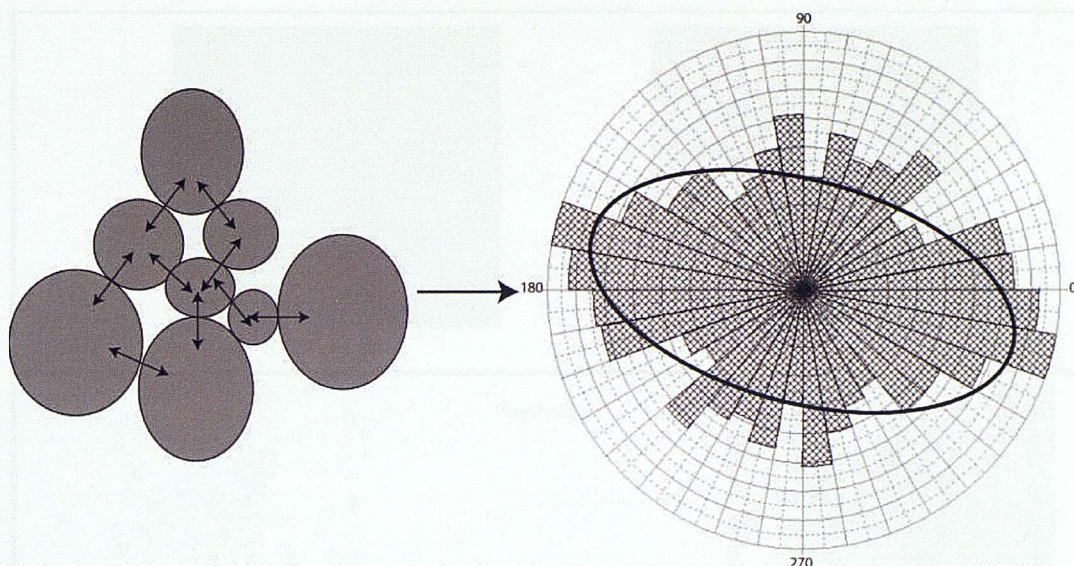


Figure 4.10 Fabric tensor where the summation of contacts between particle can be summarised as an ellipsoid

Possibly the most important measurement of the structure of a sandstone is the fabric tensor (Mehrabadi et al., 1982; Oda and Nakayama, 1989). The fabric tensor is commonly defined as the distribution of orientations of particle-particle contacts throughout a material (Figure 4.10). If each contact is considered as two opposing unit vectors which are orientated perpendicular to the tangent of the point of contact between particles, then the distribution of these unit vectors can be displayed as an ellipsoid (Woodcock, 1977). Any ellipsoid can be described by a tensor, in this case the fabric tensor (Chang et al., 2003). Such a tensor can also be used to describe the distribution of particle long axis orientations in mudrocks.

Another measure of structure used in sands is the contact number, which is the average number of contacts per particle across the material (Oda et al., 1982). In mudrocks contacts between particles are rarely observed clearly, and because of the large size distribution of particles, the contact number is difficult to measure. A similar problem occurs when considering the fabric tensor in mudrocks, as the contacts are not normally observed, their orientations cannot be measured. Although the orientation of contacts cannot be measured the orientation of particles can be.

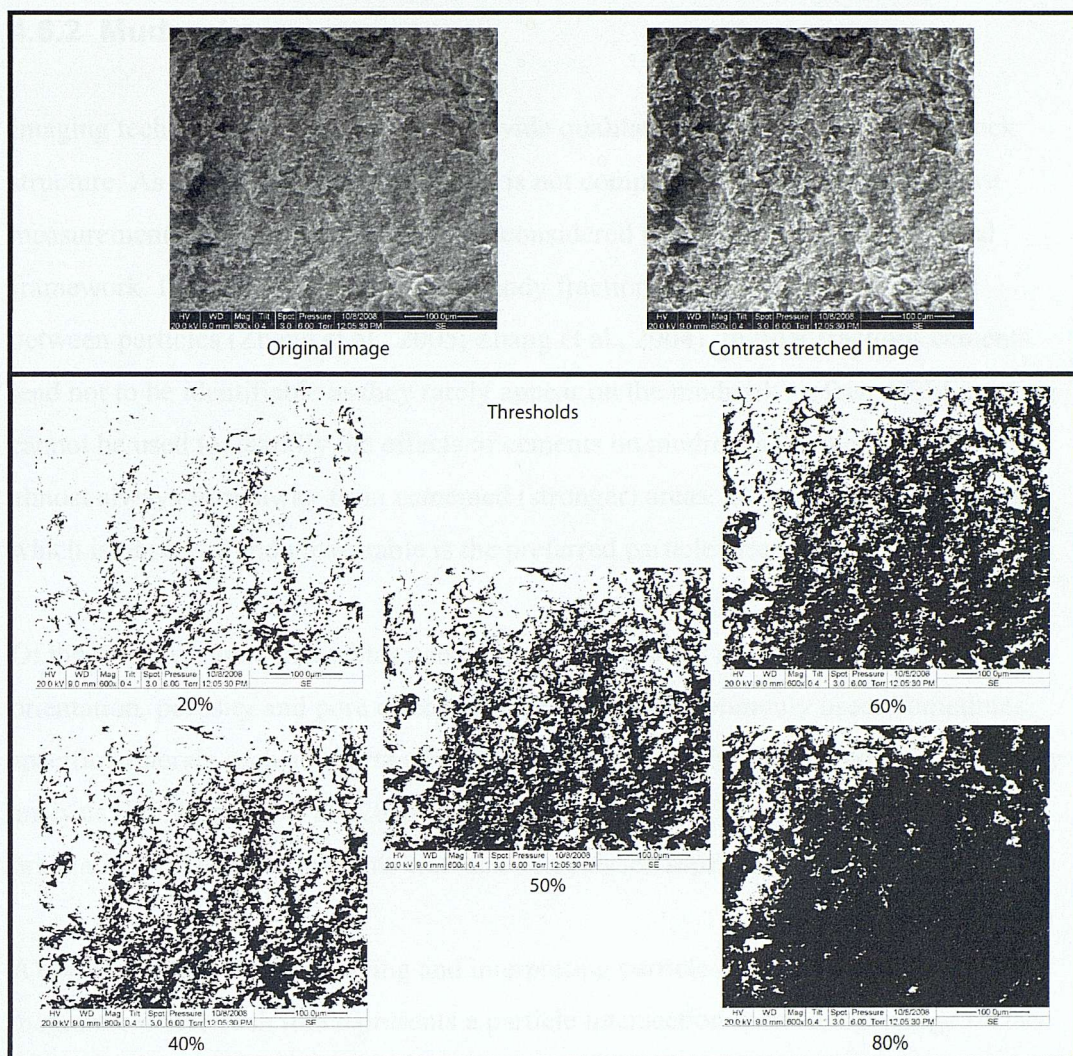


Figure 4.11 The threshold is the most important parameter during automated image analysis. The threshold is the grey level below which the image becomes white and above which becomes black in the resulting binary image. It defines the parts of the image to be measured. Small changes in the threshold can have a big effect on the measurements taken.

During the study of 2D images of sands, binary images (black and white) are often used to differentiate the particles from the void space (Yamamuro et al., 2008). Once particles have been identified then the contacts between particles can be found. From this the stress transfer through the particles on loading can be deduced. In the study of sands there is also a strong interest in the preferred orientation of sand particles (Yang et al., 2008). Once all of the properties of particles and the bonding between particles have been measured computer modelling of the material can be undertaken (Hsieh et al., 2008). The comparison of results from a computer model and those from a laboratory work is the ultimate goal in validating soil constitutive models.

4.6.2 Mudrocks

Imaging techniques most commonly provide qualitative measurements of mudrock structure. As the description of structure is not completely numerical, quantitative measurements of structure must only be considered inside a descriptive structural framework. In coarser grained silty or sandy fractions cements can be identified between particles (Zhang et al., 2005; Zhang et al., 2004). In finer fractions cements tend not to be identifiable as they rarely appear on the mudrock surface. SEM imaging cannot be used to quantify the effects of cements on mudrocks. Broken surfaces will almost always form away from cemented (stronger) areas. The main part of the fabric which is observed and measurable is the preferred particle orientations.

Of the few measurements which can be made of mudrock structure particle preferred orientation, porosity and pore orientations are the most commonly used. Sometimes specific minerals such as quartz or calcite are preferentially selected during SEM x-ray analysis (Klinkenberg et al., 2009; Williams et al., 2001). The structure/preferred orientation of these components can then be analysed separately.

A common method of displaying and interpreting particle orientations, is by line diagrams, where each line represents a particle intersection with the 2D image surface (Bates and Phillips, 2000; Engelhardt and Gaida, 1963; Foster and De, 1971; Nygård et al., 2004; Vasseur et al., 1995). This is a good method for getting an overview of the preferred orientations across the entire image. However with a large number of images, identifying individual particles is a very time consuming process. Automated identification of particles can be achieved using binary images (Whitney and Velde, 1993). This type of analysis is now accepted as a standard image analysis technique for clays (Mitchell and Soga, 2005; Reeves et al., 2006). One of the difficulties in using binary images is the selection of the grey level threshold (Figure 4.11) (Tovey and Hounslow, 1995). This problem is enhanced when using SEM images as the initial contrast of the image is greatly dependent on the user's preference. Some have suggested that this problem may be overcome by including an overlap region, such that particles and voids are identified, with a margin of error between the images known as a contact zone (Hu et al., 2005). Unfortunately, in most mudrocks there is no unique grey level that could be selected such that only particles or only voids are enhanced. For this

reason the binary image gives an overall picture of the fabric of the mudrock, it does not partition the mudrock into particles and voids.

The orientations of particles across the entire image can be displayed using histograms (Nygård et al., 2004) and rose diagrams (Tchalenko, 1968). It is common practice to divide histograms and rose diagrams into thirty six 10° sections. Some are divided into eight 45° sections (Worden et al., 2005). Where few measurements are taken statistical tests can be used to highlight any preferred orientations (Gipson, 1965). Where 3D data on orientation is available it is often plotted on a stereographic projection, allowing cluster analysis (Herwegh et al., 2008; Hounslow, 1985). Another method of measuring preferred orientation selects the highest peaks of material or the highest grey levels using a thinning process. The thinning process decreases the number of pixels progressively from all sides until a single line remains which is interpreted as particle peaks, indicating the material structure (Martinez-Nistal et al., 1999). This technique highlights the orientation of particles that can be measured on the basis of upper grey levels in an image. It assumes that the majority of particles will be flat and platy, which means it, is confined to illite/smectite (platy clay minerals) rich deposits. This technique is able to separate two particles which touch then diverge; which cannot be easily identified using binary images.

Instead of studying the solid parts of the structure of mudrocks, some workers study the porosity of the material (Tovey and Hounslow, 1995; Whitney and Velde, 1993). Most of this work on pore structure uses binary images to identify the porosity of a mudrock. Some alternative methods such as fractal analysis are also used to study porosity (Vallejo, 1996),

Surfaces of individual particles are often measured using roughness parameters. These have been applied to some surfaces made up of multiple particles; however they were not originally designed for this function. Recently surface topographic analyses have used gradient maps of the surface (*e.g.*, Fouché et al., 2004), in this case in relation to small strain movements.

4.7 Summary

One of the aims of this work was to create a predictive tool to connect the microstructure of a mudrock to its laboratory behaviour. The only measurement which can be made of the microstructure of mudrocks with the most commonly used imaging methods is preferred particle orientation. This relates well with the laboratory measurements of anisotropy. However this does not allow a full prediction of the strength and stiffness behaviour of a mudrock as is possible in sands. To study the preferred particle orientation of mudrocks requires the analysis of a large number of images and so it is best carried out using computerized image analysis. This is done by selecting a threshold, forming a binary image and taking measurements from this image. The exact method used in this work is described later (Section 9.3). To predict the effects of structure that would be seen in laboratory testing, the mechanisms of stress transfer through the mudrock microstructure must be understood. This cannot be achieved without the creation of full 3D block models of mudrocks at the particle scale; this was not possible under the constraints of this project. Further aspects of mudrock structure could be quantified during future work (Section 13.1).

CHAPTER 5

THE OXFORD CLAY AT ELSTOW

The Oxford Clay was the first mudrock to be identified for sampling. Indeed it was the only suitable site which was made available as a result of initial enquiries. The Oxford Clay is a Late Jurassic mudrock which can be traced across the UK from Yorkshire to Dorset. Similar deposits are also present in continental Europe. It is a well known anisotropic mudrock and as such was a perfect material with which to initiate an investigation into mudrock structure. The samples were obtained from Elstow in Bedfordshire.

5.1 *Geological setting*

The literature focused on the Oxford Clay, can be divided into several areas of study: stratigraphy, depositional environment, mineralogy and chemistry, engineering behaviour, and studies of preferred particle orientation (Table 5-1).

Stratigraphy

These papers (Table 5-1) are concerned with the proper division of the Oxford Clay into concise units with clear boundaries (Figure 5.1). The Oxford Clay is divided up into three main units, the Peterborough, Stewartby and Weymouth Members (Cox et al., 1992). The Oxford Clay Samples were taken from the Peterborough Member, which is more quartz and organic rich than the other two members which have a greater proportion of calcium carbonate (Norry et al., 1994). This greater amount of organic material has provided a commercial advantage to the brick making industry (Oti et al., 2009). There are a great number of excavations into the Peterborough Member to source materials for brick making. These excavations (areas of surface exposure) allowed the Peterborough Member to be much more widely studied than the other two Members. Geological materials can also be subdivided by changes in their fossil assemblage. Thirteen ammonite subzones have been identified within the Oxford Clay. Based on the fossil evidence the samples come from the Jason zone. The isopach map for the Oxford Clay (Figure 5.2) shows the onlap onto the London Platform, and a depositional gap across the Market Weighton Structure.

Area of Study	References
Stratigraphy	Early identification of Oxford Clay as a separate unit, and division into ammonite zones. (Neaverson, 1925; Woodward, 1895) Sequence stratigraphy across the UK (Callomon, 1968) Biostratigraphic correlations across the UK (Cope et al., 1980a) Stratigraphic thickness across the UK (Sellwood and Sladen, 1981) Field guide to fossils (Martill and Hudson, 1991) Officially defined as a formation with three members (Cox et al., 1992) BGS standard field reference on the Oxford Clay (Berridge et al., 1999) Historic review of stratigraphy including locations of exposures (Cox and Sumbler, 2002)
Depositional Environment	Early ecology of the environment (Duff, 1975; Hudson and Palframan, 1968) Identification of sedimentary breaks (Raup and Crick, 1982) Vertebrate fauna (Martill, 1988) Processes occurring in the depositional environment (Hudson and Martill, 1991) Special edition of the Journal of the Geological Society focusing on the environment of deposition (Anderson et al., 1994; Belin and Kenig, 1994; Hudson, 1994; Hudson and Martill, 1994; Kenig et al., 1994; Macquaker, 1994; Martill et al., 1994; Norry et al., 1994). Lateral small scale variability recognised, including bioturbation (Macquaker and Howell, 1999). Identifies a volcanogenic origin to the upper part of the Oxford Clay (Jeans et al., 2000) Uses carbon isotopes to interpret flooding events (Pearce et al., 2005) Possibilities as a hydrocarbon source rock (Swann and Gray, 2007)
Mineralogy & chemical analysis	Mineralogy of specific locations (Dunham et al., 2001; Mitchell, 1993). Early analysis of the Peterborough member (Jones, 1995; Norry et al., 1994) Origins of minerals (Gaucher et al., 2004) Changes in the clay mineral assemblage (Jeans, 2006b) Mineralogy using SEM/EDS and XRF (Clayton and Pearce, 2007)
Engineering Behaviour	Basic properties (Skempton and Jones, 1944) Investigations local to the site at Elstow (Hird and Pierpoint, 1997; Ove Arup and Partners, 2007; Parry, 1972; Rudrum, 1990) Depth of burial (Hudson, 1978; Jackson, 1972; Jackson and Fookes, 1974) Failure by movement along near horizontal planes (Burland et al., 1977) Anisotropy in weathered Oxford Clay (Russell et al., 1978) Identifies pyrite nodules as controls over behaviour (Fouché et al., 2004) Correlation of various anisotropic properties (David et al., 2007) Triaxial test anisotropy (Sarout et al., 2007) Engineering behaviour of Oxford Clay Bricks (Oti et al., 2009)
Preferred particle orientation study	Preferred particle orientation and imposed magnetic fabric (Hounslow, 1985) Investigation of anisotropy through seismic surveying (Bates and Phillips, 2000)

Table 5-1 summary of the literature on the Oxford Clay, this table is not a complete list of all the available literature; it only includes the sources consulted by the author.

Series	Stage/Substage		Zone	Subzone	Group	Formation	Member
Upper Jurassic	Oxfordian	LOWER	Cordatum	Bukowskii	Ancholme	Oxford Clay	Weymouth
			Mariae	Praecordatum			
				Scarburgense			
Middle Jurassic	Callovian	UPPER	Lamberti	Lamberti			Stewartby
				Henrici			
			Athleta	Spinosum			
				Proniae			
				Phaeinum			
				Phaeinum			
		MIDDLE	Coronatum	Grossouvrei		Kellaways Beds	Peterborough
				Obductum			
			Jason	Jason			
				Medea			
		LOWER	Calloviense	Enodatum			Kellaways Sand
				Calloviense			
			Koenigi	Galilaei			
				Curtilobus			
				Gowerianus			
				Gowerianus			
			Herveyi	Kamptus			Kellaways Clay

Figure 5.1 A stratigraphy of the Oxford Clay (Cox and Sumblar, 2002)

Along the Midlands Platform the isopachs are parallel, showing a possible thinning towards the north east of the platform. This pattern reflects the onlap onto the London Platform. It also suggests that the Midlands platform has experienced only simple tectonic effects as previously suggested, making it perfect for sampling for this investigation.

Depositional environment

During the Late Jurassic the UK experienced a general rise in sea level. One of the first products of this was the Oxford Clay “mud blanket” (Woodcock and Strachan, 2002). It was deposited in an open seaway in a continental shelf environment. This wide shelf was able to support a diverse fauna (Martill et al., 1994), resulting in a regular and well preserved macro-fossil presence within the mudrock structure, and some heavily bioturbated horizons (Macquaker and Howell, 1999). Other horizons remain undisturbed suggesting periodic anoxia, allowing the preservation of organic material. Although the clay minerals which make up the Upper Oxford Clay may have a volcanic origin (Jeans et al., 2000), the Peterborough Member is more likely to have a detrital origin.

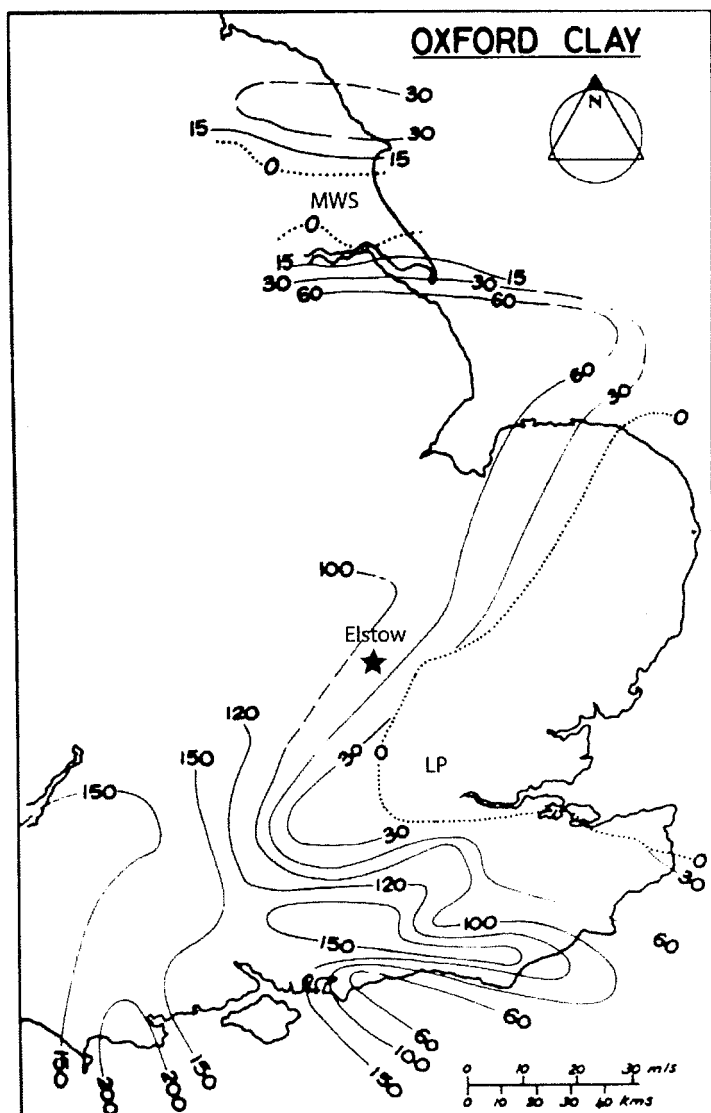


Figure 5.2 Isopachs for the Oxford Clay (m) from (Sellwood and Sladen, 1981). Showing the London Platform (LP) the Market Weighton Structure (MWS) and the sampling site (Elstow).

Mineralogy and chemical analysis

The mineralogy of the Peterborough Member of the Oxford Clay is relatively stable across multiple brick pits across the UK (Norry et al., 1994). Generally illite is the richest mineral within the Oxford Clay, making up just less than a third of the mudrock. Quartz is the next most abundant, forming between a fifth and a quarter of the mudrock. These two minerals make up approximately half of the mudrock. Of the remainder there is approximately a tenth of calcite (Norry et al., 1994), normally of biogenic origin (Gaucher et al., 2004). A tenth kaolin, and a further tenth chlorite originating from both detrital deposition and the post depositional alteration of biotite (Gaucher et al., 2004).

Source	Site	Sampling Method	Lab tests	c' peak (kPa)	Φ' peak (Deg)	Φ' r (Deg)	S_u (kPa)
(Parry, 1972)	Stewartby Lake, 8 km south of Bedford	U-100 tube sampling	CD-TC (V); D-DSB (V,H)	25	28-29	-	95-165
(Jackson and Fookes, 1974)	Stewartby	Blocks	Oedo D-DSB (V,H)	59-89	29-30	15-16	-
(Burland et al., 1977)	Witlesey	BH- core drilling & blocks	CU & CD-TC (V,H); D-DSB (V)	-	27-28	13	50-1200
(Crabb and Atkinson, 1991)	M1, junction 13 14 km SW of Bedford	Thin walled tube	TC	-	25	-	-
(Rudrum, 1990)	Elstow, 5 km South of Bedford	Rotary coring & blocks	UU & CU-TC (V,H); CD-TC (V); Oedo	6	26	-	20-66
(Pierpoint, 1996)	Elstow & Kempston brick pit	Block samples	CD-TC & TE (V); Oedo	-	21-32	-	-
(Cripps and Taylor, 1981)	Liquid Limit (%)	Clay fraction (%)	Plasticity Index (%)	Φ' peak (weathered)	Φ' peak (unweathered)	Φ' residual	
Middle Oxford	58-76	35-70	31-40	21.5-28°	23-40°	13-19°	
Lower Oxford	45-75	30-70	28-50	17.4°	-	12°	

Table 5-2 Example engineering properties for the Oxford Clay from (Hosseini-Kamal, 2011).

Various feldspars are present making up just less than a tenth of the mudrock. The final tenth of the mudrock is made up of a number of minor minerals chiefly pyrite and gypsum.

Engineering behaviour

The behaviour of the Oxford Clay has been studied on several sites across the UK (Table 5-2). The major, regularly identified feature in the literature is the materials anisotropy (Burland et al., 1977; David et al., 2007; Russell et al., 1978). This appears to be the defining characteristic of the Oxford Clay. Another factor is the presence of relatively hard particles such as pyrite. These are thought to act as nucleating sites for micro crack propagation (Fouché et al., 2004).

Preferred particle orientation

Considering the clearly anisotropic nature of the Oxford Clay and the relative ease of access to material in the brick pits it is not surprising that it has been the focus of

particle orientation studies (Bates and Phillips, 2000; Hounslow, 1985). Both of these identify the Oxford Clay's strong preferred particle orientation.

5.2 *Reconstruction of material history*

The depositional environment of the Oxford Clay was an open seaway, on the continental shelf in the North Western corner of the Tethys Ocean. Due to the deepening of sea level just prior to the deposition of the Oxford Clay it is thought to be predominantly formed through settlement out of the water column (Peterborough Member), with increased sediment input from volcanic material in the latter part of the deposit. As outlined in Chapter 3, the burial history of each of the mudrocks studied can be reconstructed from the combination of both stratigraphic and palaeothermal methods. The amount of burial experienced by the Oxford Clay has been of particular interest to the geological community as it is an oil shale. Early estimates for maximum burial suggest a maximum depth of burial of 500m for the Oxford Clay at Stewartby based on stratigraphic thickness estimates (Jackson, 1972; Jackson and Fookes, 1974). It has been suggested that the preservation of deposited aragonite requires a maximum burial of 500m (Brenchley and Rawson, 2006; Hudson, 1978). However based on apatite fission track analysis and other palaeothermal measurements a depth of around 1130m is suggested (Figure 3.11), based on a palaeothermal gradient of 30°C/km. Others using a different thermal gradient, and other stratigraphic considerations have suggested palaeo-depths as high as 1450-1500m (Brenchley and Rawson, 2006; Cope, 1994). Although the maximum depth of burial suggested by apatite fission track analysis is disputed the burial curves which it can produce, give insight into the burial and exhumation events each material experiences. The Oxford Clay underwent approximately 6-7m/My of burial for its first 95 million years. Much of the detail of the curve cannot be measured, there will be minor periods of rapid burial, followed by erosion, and times of non deposition followed by more rapid burial. However, the burial trajectory remains the same. After this initial depositional period there was a time (40My) of neutral deposition. Any material which was deposited during this time was of limited thickness and was removed before the end of this period. Approximately 25Ma apatite fission track analysis suggests a period of rapid burial of 120m/My for 5My. For the following 5My an uplift rate of 120m/My is suggested such that all of the deposited

material during the first 5My is removed. For the remaining 15My the Oxford Clay was uplifted to the surface at a rate of approximately 30m/My (Figure 3.11).

The effects on the microstructure of the Oxford Clay of this burial curve could only be measured if other samples of the Oxford Clay from the same stratigraphic horizon with different burial histories could be measured. The initial deposition of the Oxford Clay by settlement in the presence of organic matter will have resulted in an aligned material (Section 2.3). The further burial of the mudrock will have resulted in the enhancement of this alignment which is today observed as anisotropy.

5.3 Sample site details

The sampling site for the Oxford Clay is located near Elstow Storage Depot to the south of Bedford (Figure 5.3). Ove Arup and Partners kindly organised access to the site and allowed the extraction of samples for testing under the supervision of Lindsay Barnard and Stewart Jarvis. The site is an excavation of 10m depth from which material has been removed to build up the ground level in the surrounding area for a house building project. This excavation is expected to become a pond on completion of the construction works. Due to the size of the excavation, and the size of the overall site, it was possible for uninterrupted access to be arranged to the sampling location for a full week. This level of access allowed the careful extraction of seven block samples. The location of the top of block OC1 was (E 505,211.625 N 245,653.532 H 20.911) measured using the on site GPS surveying system. The excavation from which the samples were taken was ~210m in length ~120m in width and ~10m in depth. The side of the excavation showed a profile of alluvial gravels ~2m weathered Oxford Clay ~2m with the slightly darker unweathered Oxford Clay below. No small-scale features were visible in the sides of the excavation as it had been graded (Figure 5.7). Pond 1 was acting as the overflow for a local brook, so had recently been pumped out prior to the site visit.

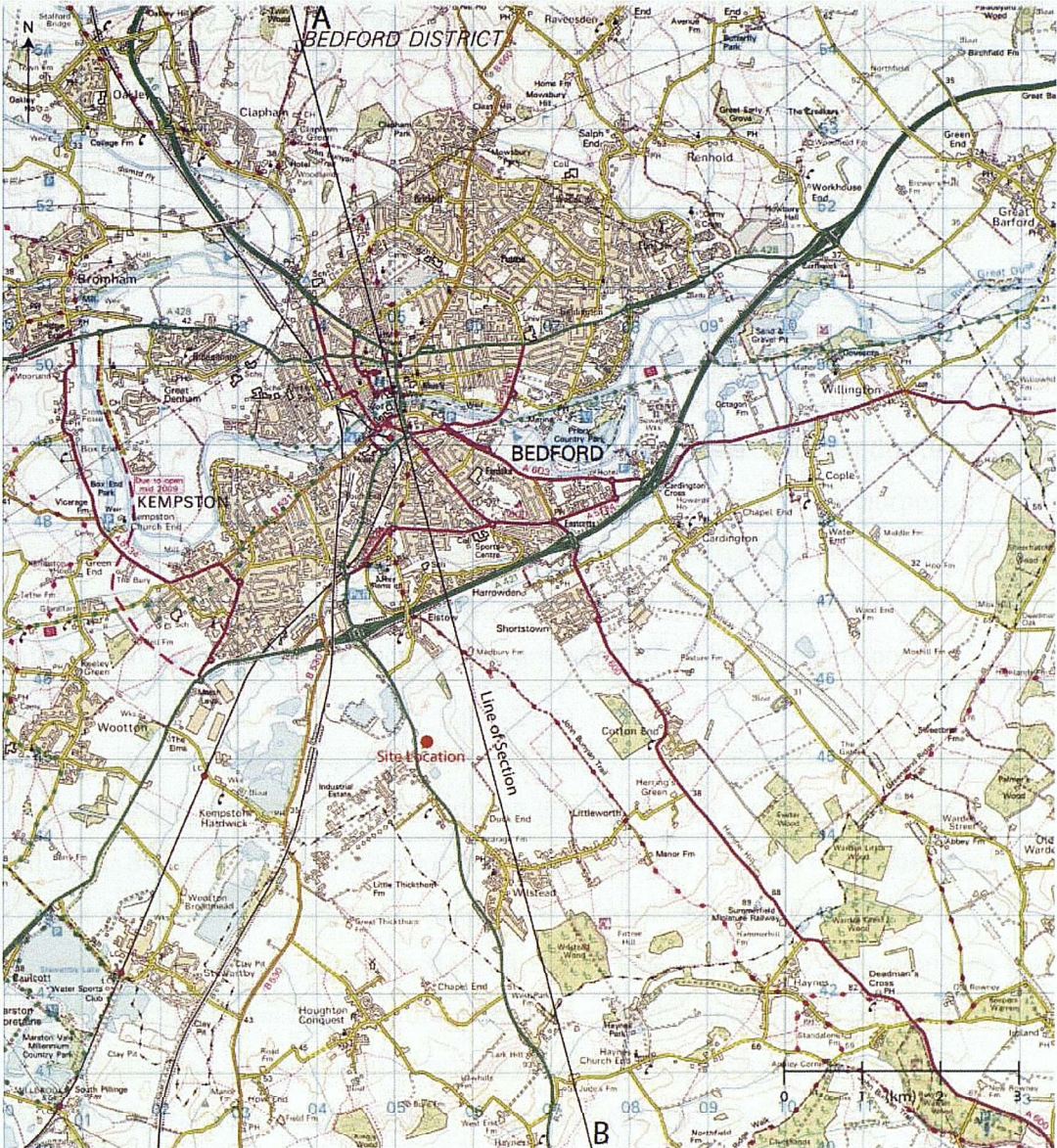


Figure 5.3 Site location: South of Bedford and Elstow, near Elstow storage depot. Black line is line of cross-section (Ordnance Survey, 1963)

At the larger scale, the site is located in a wide open valley within which runs one of the small tributaries to the Great Ouse River (Figure 5.4). The valley gravels and alluvium lying on top of the Oxford Clay suggest that this tributary was much larger in the past. The southern shoulder of this valley is formed from the younger Lower Greensand. Older rocks are visible where the river has been able to cut down to their level. The overall picture is of a series of bedded rocks dipping at a low angle ($2-3^{\circ}$) to the south east (Figure 5.5).

This supports the idea that the Oxford Clay samples have experienced a relatively simple burial and uplift history with limited lateral loading from tectonic action. The section shows the Kellaways Beds as just below the site, they are in fact 15m below

ground level. The samples were taken from 10-11m below ground level. So the samples are from 4-5m above the base of the lowest Member (Peterborough Member) of the Oxford Clay. Large areas of the sides of the valley are covered by Boulder Clay, deposited during the last glaciations. The valley has been carved out below this level by the glacial outwash.

Due to the current construction project there was a substantial amount of site investigation data available (Figure 5.6). As the location is at the very northern part of the site, borehole and CPT data becomes more prevalent towards the south. Also close to the site is a well known trial excavation and site investigation for the purpose of assessing the suitability of sites for the construction of underground repositories for low level nuclear waste (Hird and Pierpoint, 1997; Rudrum, 1990). The presence of this information made this site perfect for the purpose of laboratory comparisons.

The standard block sampling procedure was followed (Appendix B). As the sampling was undertaken in the base of a pre-existing excavation only a shallow 1m trench was required. Seven of the blocks were easily excavated from the sides of the initial trench (Figure 5.8). Some samples broke along planes of weakness during their excavation. Several vertical joints were observed where blocks failed while they were being cut out (Figure 5.9). In general the joints observed were rarer than the fissures present in younger materials (*e.g.*, London Clay), they were also more prevalent in terms of size, often forming large scale near vertical planar surfaces. The joint spacing is assumed to be on the order of a metre, in one case a spacing of 30cm was observed, however the next joint in the set was elusive. From the observed joints the dominant sets were near vertical north-south and east-west trending (Figure 5.10). However as few joints were observed the true major directions and spacings cannot be identified with more accuracy. The Oxford Clay also showed a general and prevalent horizontal weakness. During the excavation of the blocks the tendency for the mudrock to be easily split along the horizontal was observed. The insertion of a spade a short way into the mudrock followed by levering often split open a wide surface sometimes covered in shells suggesting that these are depositional surfaces. Within the excavation several fossils were recovered. Some of these such as the belemnites (Figure 5.11) are made of much harder material than the surrounding mudrock.

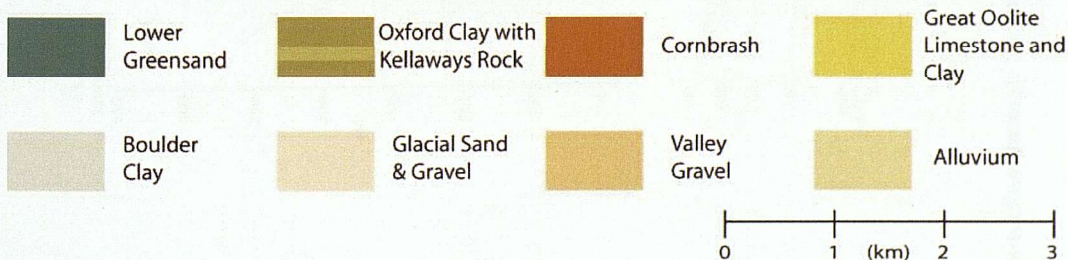


Figure 5.4 The geology of the Elstow area (British Geological Survey, 1900). Black line is line of cross-section.

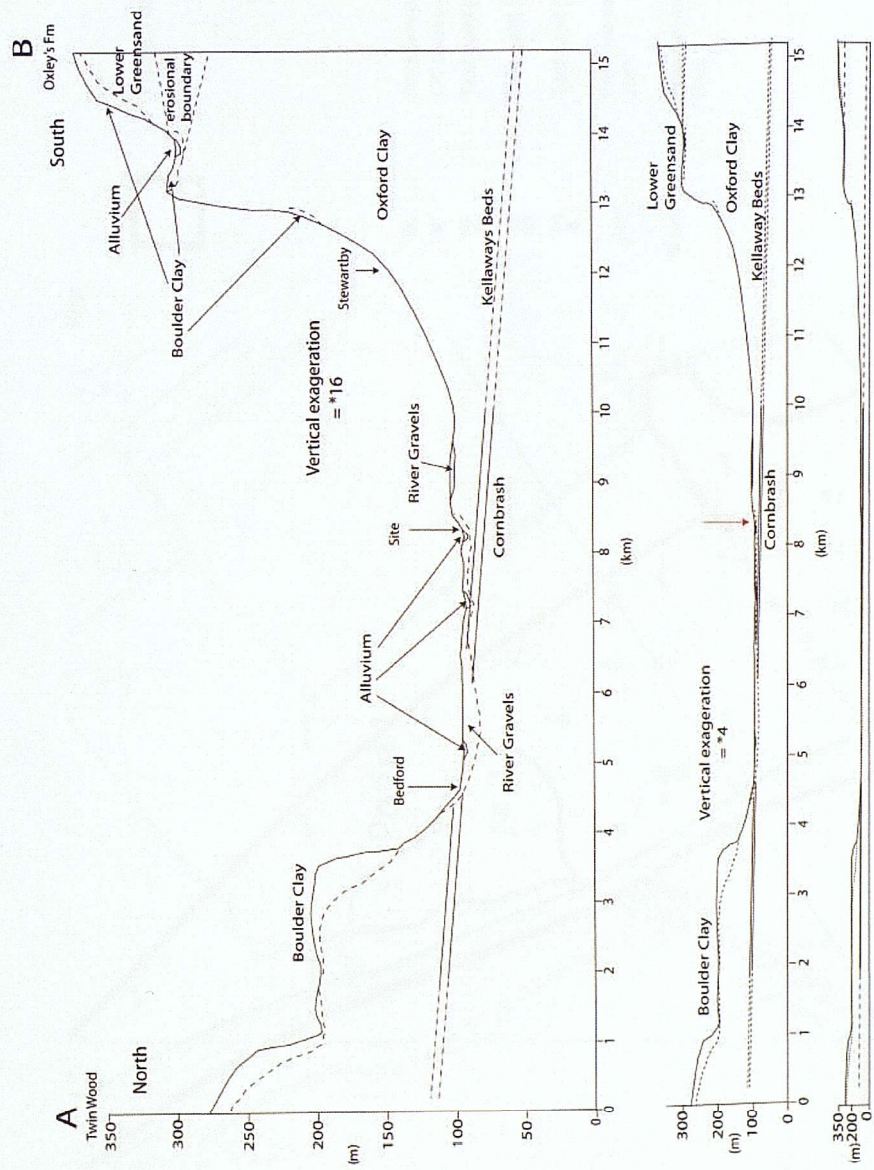


Figure 5.5 Cross-section through the site, the site is located within a river valley. Samples are located close to the base of the Peterborough Member of the Oxford Clay.

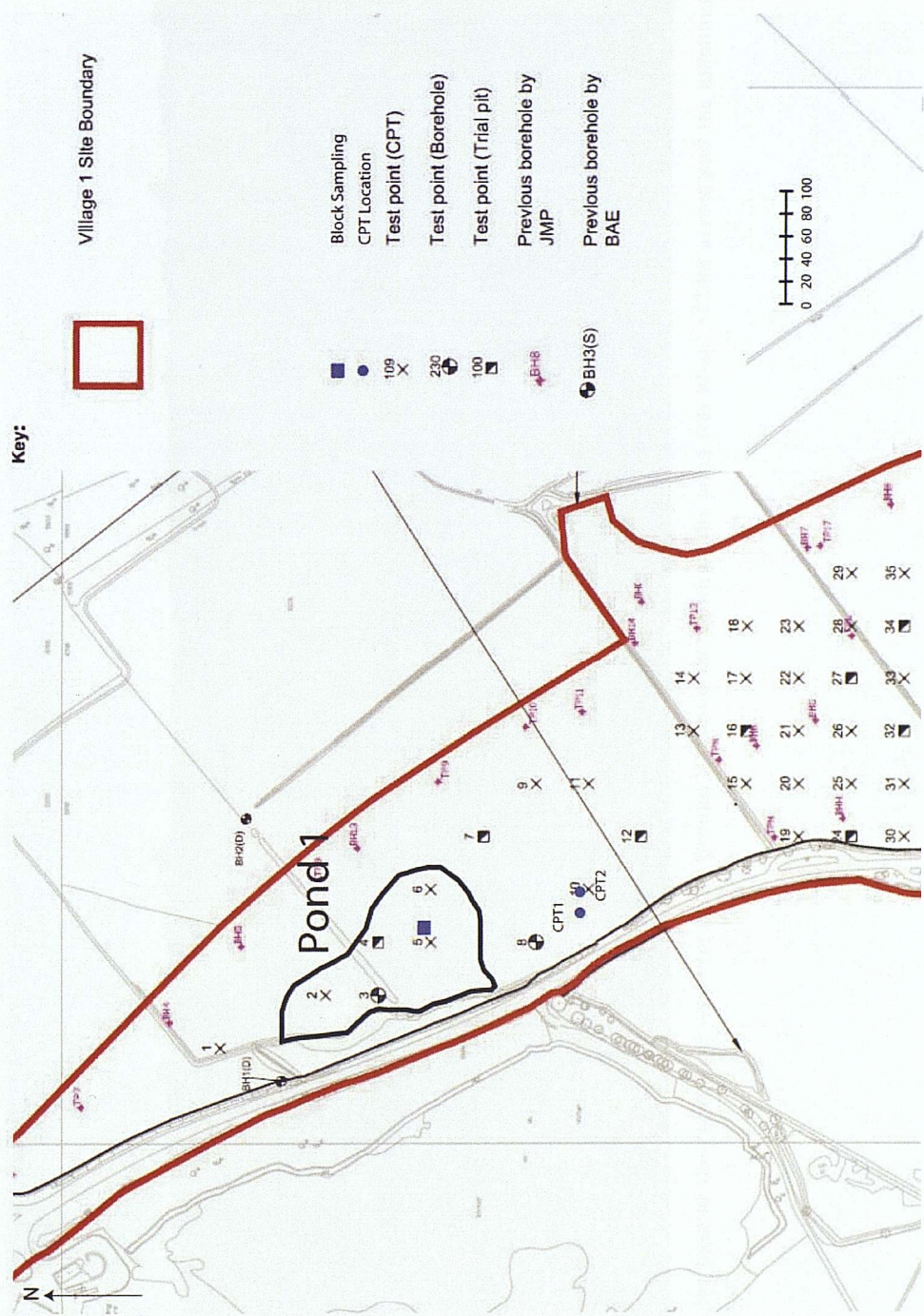


Figure 5.6 Plan view of the excavation at Elstow, the samples located within Pond 1, modified from (Ove Arup and Partners, 2007)



Figure 5.7 Site photo, the base of the excavation was ~9m below ground level and the width of the pond was about ~120m across and the maximum length of the pond being ~210m. Facing east.

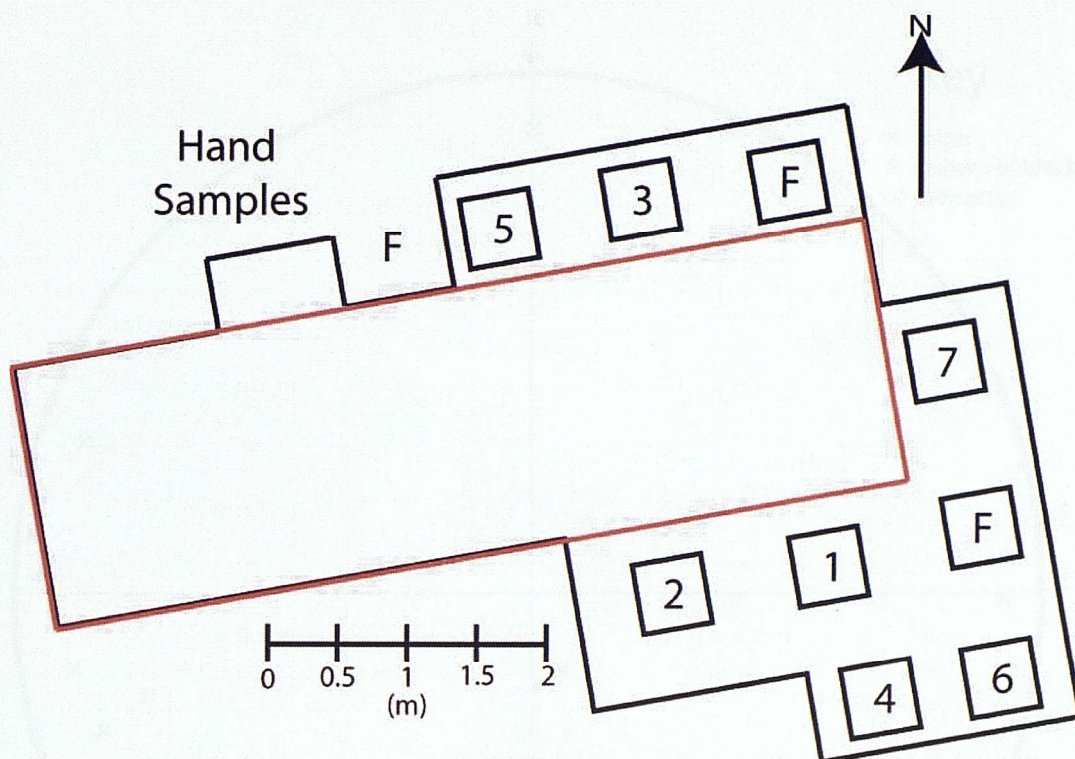


Figure 5.8 Diagram of the location of the recovered block samples in relation to each other. F is where the block failed before its excavation was completed. The red rectangle is the approximate line of the original trench. 0.5 m areas are shown for each block, actual block size is approximately 0.3m

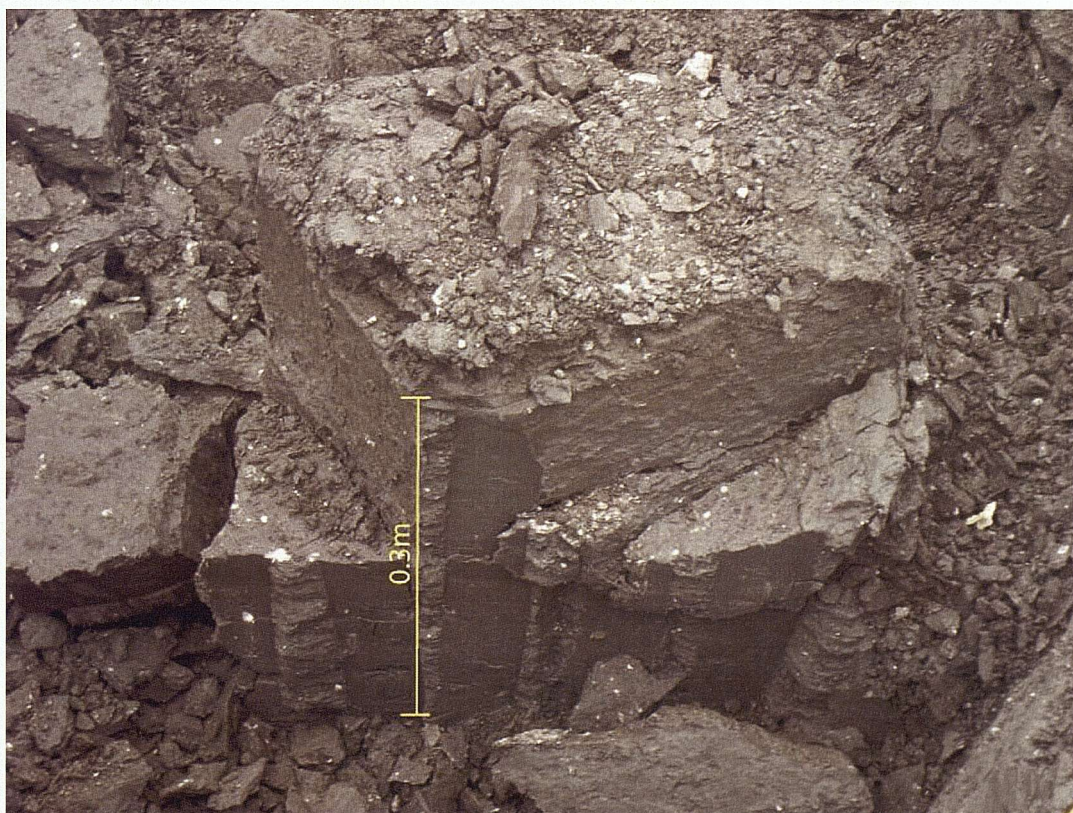


Figure 5.9 Two joints running through a block sample of the Oxford Clay. Scale approximate

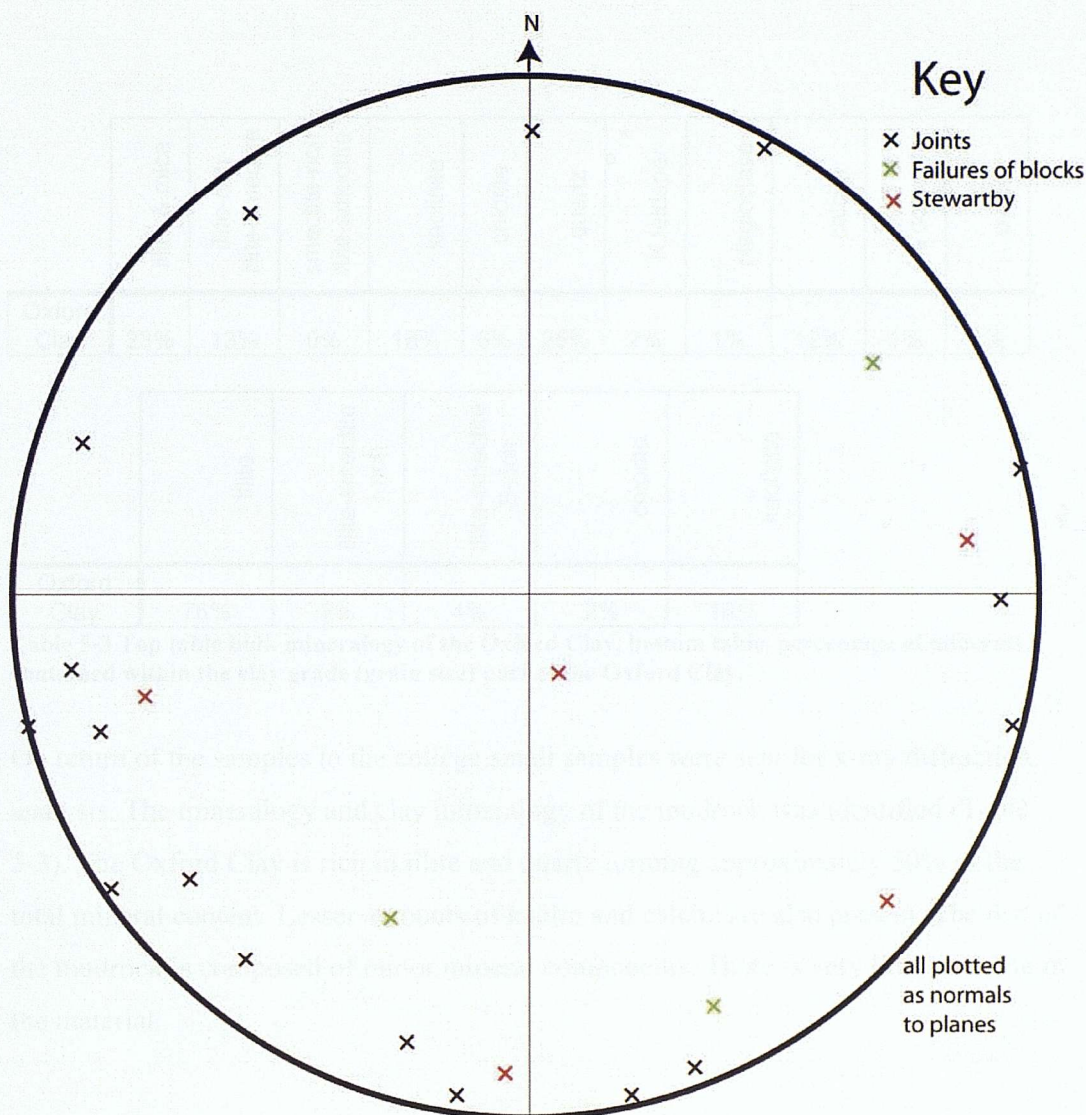


Figure 5.10 A lower hemispherical stereographic projection showing the joint data for the Oxford clay south of Bedford. East-west and north-south directions appear to be prominent.

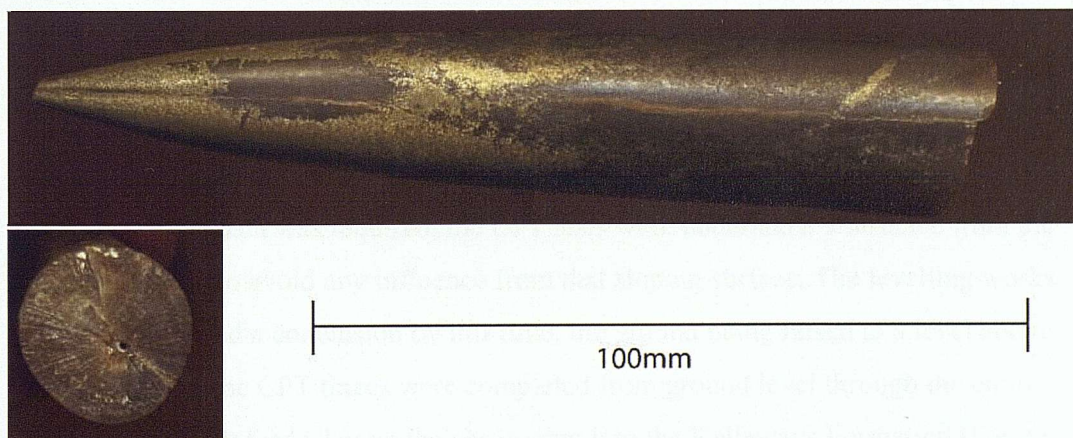


Figure 5.11 *Cylindroteuthis puzosiana*, an example of one of the belemnites found within the Oxford Clay excavation. These hard fossils have a completely different structure compared to the surrounding mudrock

	illite & mica	illite-rich illite-smectite	smectite-rich illite-smectite	kaolinite	chlorite	quartz	K feldspar	plagioclase	calcite	Fe dolomite	pyrite
Oxford Clay	23%	13%	0%	16%	6%	25%	2%	1%	12%	1%	2%

	illite	illite-smectite I-rich	illite-smectite S-rich	chlorite	kaolinite
Oxford Clay	76%	2%	4%	2%	16%

Table 5-3 Top table bulk mineralogy of the Oxford Clay, bottom table, percentage of minerals contained within the clay grade (grain size) part of the Oxford Clay.

On return of the samples to the college small samples were sent for x-ray diffraction analysis. The mineralogy and clay mineralogy of the mudrock was identified (Table 5-3). The Oxford Clay is rich in illite and quartz forming approximately 50% of the total mineral content. Lesser amounts of kaolin and calcite are also present. The rest of the mudrock is composed of minor mineral components. There is very little smectite in the material.

5.4 CPT investigation

The CPT investigation was carried out using the standard method (Appendix B). The locations of the CPT traces were CPT1 (TL 05223, 45517) and CPT2 (TL 05245 45511) (Figure 5.6). The CPT sites are some distance from the original sampling site. By August 2008 the pumps had been turned off and the initial sampling site was flooded. Because seismic data was required, the CPT tests were undertaken a distance from the pond excavation to avoid any influence from that sloping surface. The levelling works on site had reached a conclusion by this time, the ground being raised to a level above the flood plain. The CPT traces were completed from ground level through the entire thickness of the Oxford Clay on the site, toeing into the Kellaways Formation (Figure 5.12). The thickness of the Oxford Clay sequence on site is about ten metres, much less than the entire sequence ~150m (Figure 5.5). The upper portion of the sequence is the compacted layer of material placed on top of the sequence to raise the level of the

ground. This is clearly clay based on the difference between cone end resistance (q_c) and sleeve friction (f_s). The top of this section shows enhanced strength due to desiccation. Below the compacted clay is an area of similar q_c and f_s which indicates a silty material, this is probably a layer of alluvium. Below this the strength of the material increases, indicating the entry into the weathered portion of the bedrock. The boundary between weathered and unweathered Oxford Clay has been placed where the f_s reached the approximate average of that experienced below in the bulk of the material, this boundary may vary slightly from this level.

The Oxford Clay displays lots of variation through its sequence (*i.e.*, the CPT trace has a lot of spikes). The areas of somewhat higher q_c and f_s than the average are shell beds and pyrite bands, also observed by the author at Stewartby (Figure 5.14). The CPT shows that the sampling horizon is representative of the clay part of the sequence on site.

5.5 Comparison sites

Two sites were visited in the local area of the sampling site, with the aim of observing the lateral and vertical variations in the material. Both Stewartby Quarry (TL 0135 4332) and the A421 site investigation (TL 0250 4590) were visited during September 2007.

5.5.1 Stewartby brick quarry

Stewartby Quarry has been the site of several geological and geotechnical investigations in the past, for example (Hudson et al., 2001; Jackson and Fookes, 1974; Parry, 1972). Because of this it is a very important site of scientific interest. Robert Donnelly from Hanson UK kindly granted access to the site under the supervision of Henry Godfrey.

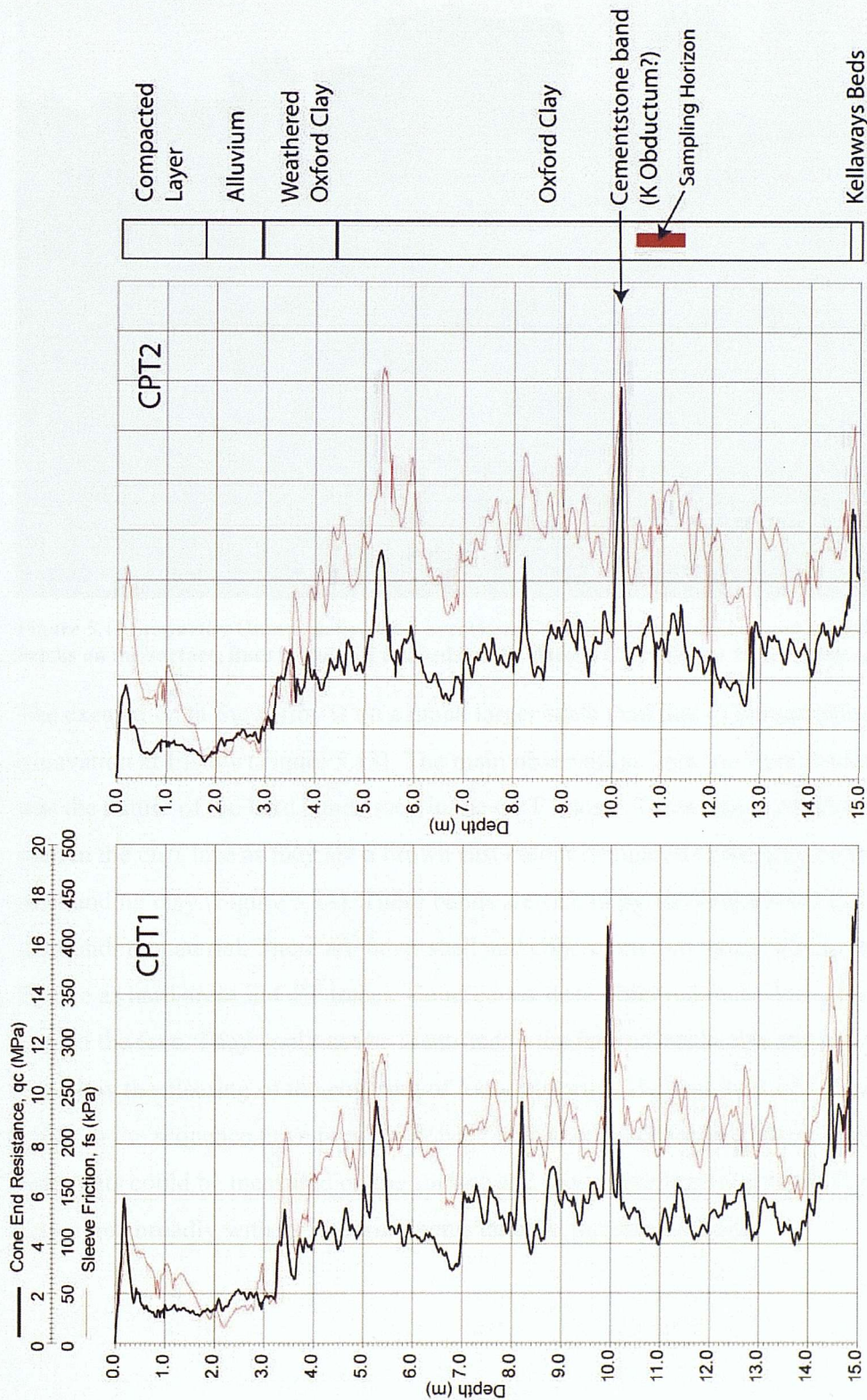


Figure 5.12 CPT traces from the Oxford Clay site for locations see map (Figure 5.6). Geology based on (Ove Arup and Partners, 2007)

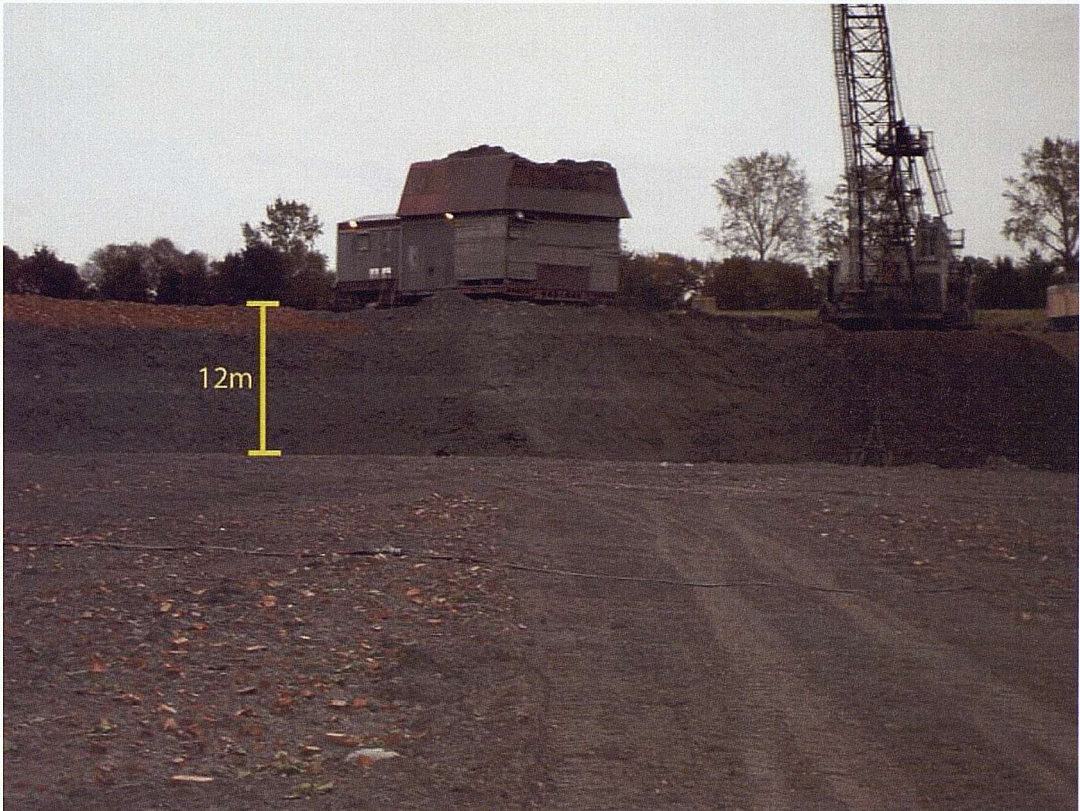


Figure 5.13 Stewartby Quarry active face, overview; Grey areas Oxford Clay, red areas loose bricks on the surface, lines in the cliff are beds in the Oxford Clay (Figure 5.14) Facing south west.

The excavation at Stewartby is on a much larger scale than that of the sampling excavation at Elstow (Figure 5.13). The main observation from the visit to Stewartby was the nature of the hard bands seen in the CPT traces. These bands could be clearly seen in the cliff face as they are a brown rust colour compared to the grey of the surrounding clay (Figure 5.14). These bands are rich in pyrite compared to the surrounding material. There are other shell and concretion rich bands which would behave as hard areas in CPT traces. Concretions were observed in the loose material around the face. They could not be identified in the face as site health and safety would not allow the clearing of the covering of loose material. The locations of the various bands in the sequence have previously been identified (Jackson and Fookes, 1974). A few joints could be measured on the surface and these have been recorded (Figure 5.10), they agree broadly with the measurements taken at the sampling site.



Figure 5.14 The quarry face at Stewartby, the red bands in the image probably mark pyrite rich layers within the Oxford Clay. There are also other shell/concretion rich bands which cannot be picked out in this image. Image taken facing south west.

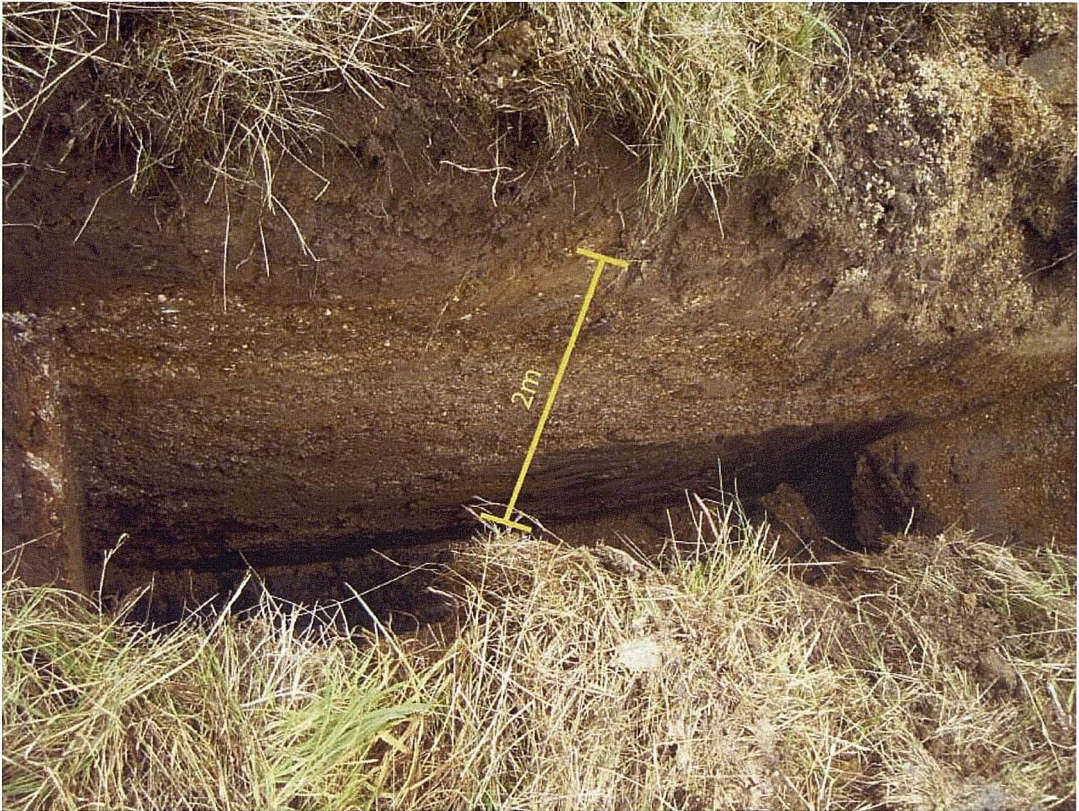


Figure 5.15 Trial pit from the A421 investigation, showing irregular depth to the top of the Oxford Clay.

5.5.2 A421 site investigation

The main investigations going on at the A421 investigation were trial pits. These provided a perfect opportunity to study the contact between the recent river deposits and the Oxford Clay. The contact in the trial pits was irregular, possibly channelled (Figure 5.15). Water was flowing into the trial pit from the deepest parts of the river deposits. This sort of channelling in this material is an artefact of the mechanism of deposition, just as the structures observed in the Oxford Clay are artefacts of its depositional environment. This irregular surface and perched water table will have resulted in varying depth of weathering effects within the Oxford Clay. For this reason it is fortunate that a deep excavation was available.

5.6 Overall material behaviour

There is a clear theme throughout this brief study of the Oxford Clay of anisotropy. It is recognised in the literature, it was observed in the field and the layered structure of the mudrock was seen during the CPT investigation. It is expected that both the study of microstructure and the laboratory studies will observe this anisotropy. Another important aspect of the Oxford Clay is its elevated organics content. This property is its most commercially important aspect. It has been suggested as a casual factor for the mudrocks anisotropy (Bennett and Hulbert, 1986). The organic content of the Oxford Clay is ~6%. The effects of a small organic content on engineering behaviour are not known separately from other effects.

Nodules, hard bands and concretions have been observed throughout the Oxford Clay. The engineering effects of harder zones within a mudrock are dependent on their geometry. Hard bands aligned to bedding will enhance a mudrock's anisotropy. Hard nodules and concretions will compress less than the mudrock around them such that local normal stresses will be concentrated through the nodules and local shear stresses will concentrate on the nodule mudrock boundary, and local tensile stresses can form on the boundary. By this process microcrack initiation can be caused by nodules in the mudrock.

CHAPTER 6

THE GAULT CLAY AT HIGH CROSS (MADINGLEY)

The Gault Clay outcrops from Dorset to The Wash. It is present on the Isle of White and across the Weald in the South East of the UK. It is known in the South of the UK for its swelling behaviour (causing subsidence, heave and landslides). This behaviour is less expressed on the Midlands Platform. The Gault Clay investigations include two sampling campaigns. Due to its extensive sampling, 558 scanning electron microscope images were taken from 11 imaging samples from within the Gault Clay (Section 10.1.2). The first sampling campaign was for block samples, the second for borehole samples. Both sets of samples were taken from High Cross located at junction 13 of the M11 on the outskirts of Cambridge. As boreholes cross-cut stratigraphy multiple samples were required to study the mudrock's variability. Other samples were taken from the boreholes at an equivalent depth to that of the block samples.

6.1 Geological setting

The literature reviewed on the Gault Clay can be divided up based on subject into: stratigraphy, depositional environment, mineralogy/chemistry and engineering behaviour. During the study of the Gault Clay interest was raised into the nodules of the Cambridge Greensand and the effects on the mudrock of the large number of trees on the site.

Stratigraphy

The literature on the stratigraphy of the Gault Clay is relatively extensive, and includes several accepted and used systems many of which cannot be extrapolated across the entire UK. The first system was a series of bed numbers (I – XI) ascribed to units in the cliff at Copt Point in Folkestone (De Rance, 1868). This system is later inverted such that the beds are numbered from the bottom of the cliff instead of from the top (Price, 1874). The bed XI which made up more than half of the sequence in the early classifications was later divided into three parts to complete the system of 13 beds (Jukes Browne, 1900).

Subject	References
Stratigraphy	Initial division of the Gault into beds at Copt Point Folkestone (De Rance, 1868; Price, 1874) Early lithostratigraphic correlation (Jukes Browne, 1900) Early compendium of fossils (Spath, 1923-1943) Biostratigraphic correlation through the Weald (Casey, 1961; Gale et al., 1996; Owen, 1963, 1971) Lithostratigraphy of the Weald (Hopson et al., 2001) Major correlation for cretaceous (Rawson et al., 1978) BGS Gault Clay description (Chatwin, 1961; Kent et al., 1980; Sumbler, 1996) Stratigraphy of the Gault Clay in East Anglia (Gallois and Morter, 1982) Stratigraphy in the Western Gault Clay (Woods and Bristow, 1995; Woods et al., 2001) Fossils from Leighton Buzzard to Buckinghamshire (Bristow, 2003) Compilation of all of the different stratigraphic systems for the Gault Clay across England (Wilkinson, 2006)
Depositional Environment	Smectite rich mineral assemblage possibly of volcanic origin (Jeans et al., 1982) Depositional environments for phosphatic nodules (Knight, 1999) Records of marine transgression (Woodcock and Strachan, 2002) Causes of carbonate deposition (Pomar and Hallock, 2008)
Mineralogy & chemical analysis	Changes in Clay mineralogy across the UK (Jeans, 1978) Mineralogy in landslip in Dorset (Bloodworth, 1990) Mineralogy at Arlesey and Klondyke Farm (Prior et al., 1993) Clay mineralogy changes across the UK throughout the Cretaceous (Jeans, 2006a)
Engineering Behaviour	Joint Orientations in the Gault Clay (Fookes and Denness, 1969) Contains some laboratory data (Burland, 1990) BGS formation study (Forster et al., 1995) BGS shrink swell project (Jones et al., 1996) Correlation across multiple projects (Marsh and Greenwood, 1995) Site investigations in Gault Clay: Selborne cutting stability experiment (Barton and Cooper, 1998; Cooper et al., 1998) Dunton Green (Clarke and Wroth, 1984; Garrett and Barnes, 1984) Folkestone Warren (Hutchinson, 1969; Hutchinson et al., 1980) Castle Hill (Varley et al., 1996) The M25 (Newman, 1985) M4 Landslide (Skempton, 1985) Slope instability near Thame (Forster, 1991) Ely-Ouse Essex water tunnel (Samuels, 1975) Wrotham Heath & Tetsworth (Weeks, 1969) Near the Site: Lion Yard (Lings et al., 1991) and Bin Brook (Parry, 1988) The site, High Cross (Madingley) (Bond et al., 1990; Butcher and Lord, 1993; Butcher and Powell, 1995; Clegg, 1981; Coop, 1987; Coop and Wroth, 1989; Dasari and Bolton, 1998; Fugro Consulting Geotechnical Engineers, 1979; Houlsby and Withers, 1988; Lings et al., 2000; Marsland and Powell, 1990)
Cambridge Greensand	Early description of the Cambridge Greensand (Jukes Browne, 1875) Coprolite mining (Berridge, 2004; Ford and O'Connor, 2002; Grove, 1976; O'Connor, 2001) Cause/depositional conditions (Jarvis, 1992)
Effects of Vegetation	Changes in flora and fauna with ice retreat (Franks, 1960; Hollingworth et al., 1949) Shrink and swell caused by trees (Driscoll, 1983) Depth of influence of tree roots for different species of tree (Crow, 2005)

Table 6-1 summary of the literature on the Gault Clay, this table is not a complete list of all the available literature; it only includes the sources consulted by the author.

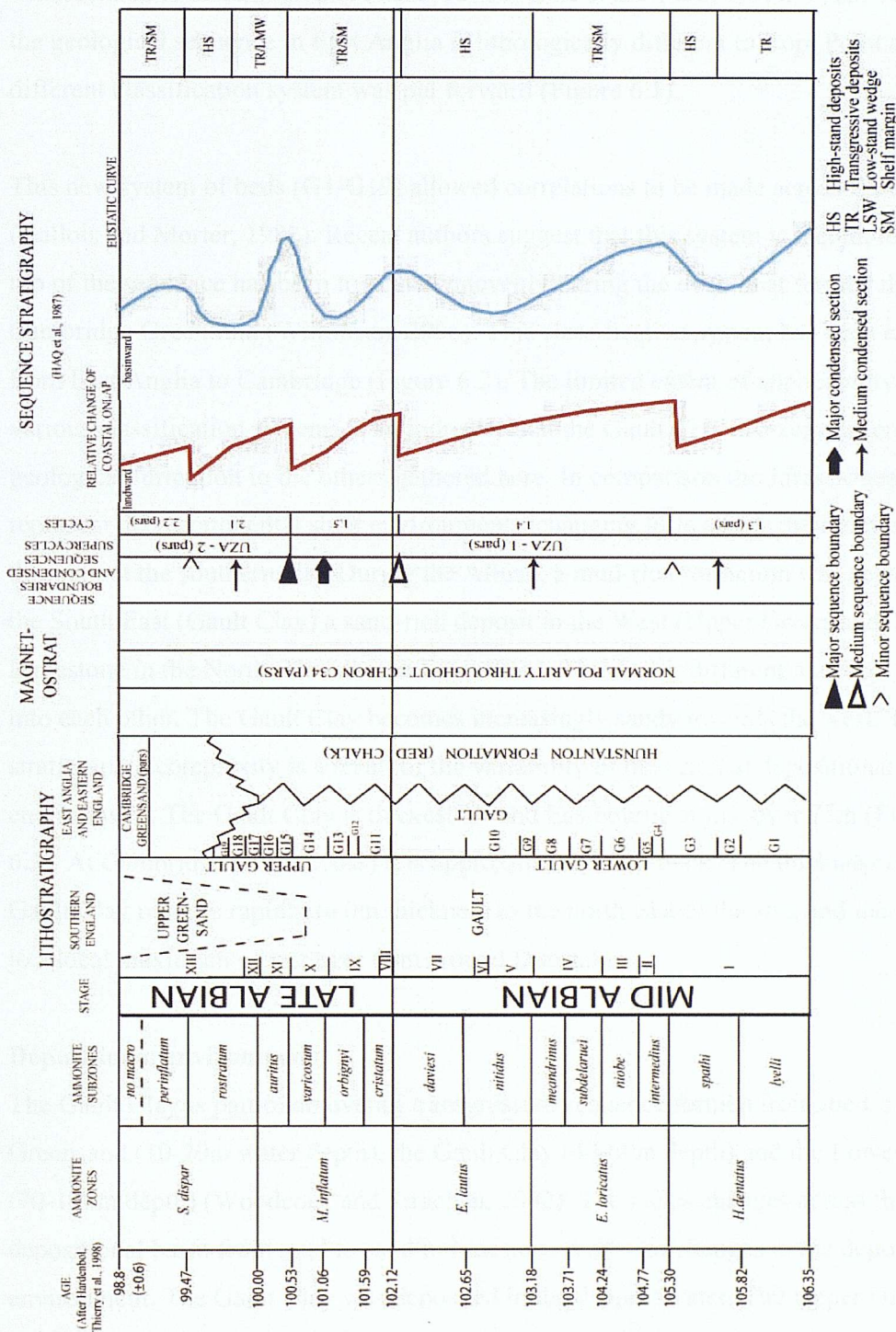


Figure 6.1 Summary of the stratigraphy of the Gault Clay, based on (Wilkinson, 2006) the ages used on the left of the original figure are based on an old time scale where the ammonite zones were defined as 0.53My long. This assumption is not present in the most recent time scale (Gradstein et al., 2004). End Albian 99.6 Ma, +/- 0.9 Mid-Late Albian boundary 106.4Ma, base of the Mid Albian 108.8Ma. The old dates are retained in the figure for comparison.

This system can be used across Southern England (Figure 6.1). Because Folkestone is a condensed sequence compared to most of the Weald, lithostratigraphic correlation is challenging. Because of this limitation the system of bed numbers can only be used when related to biostratigraphy (Casey, 1961; Gale et al., 1996; Owen, 1963, 1971). As the geological sequence in East Anglia is lithologically different to Copt Point a different classification system was put forward (Figure 6.1).

This new system of beds (G1-G19) allowed correlations to be made across East Anglia (Gallois and Morter, 1982). Recent authors suggest that this system is incomplete as the top of the sequence has been truncated unevenly during the event that formed the Cambridge Greensand (Wilkinson, 2006). This classification system has been extended from East Anglia to Cambridge (Figure 6.2). The limited extent of applicability of the various classification systems is an indicator that the Gault Clay is a very different geological formation to the others gathered here. In comparison the Jurassic seas represent deep continental shelf environments, changing little across their exposure throughout the southern UK. During the Albion, a mud-rich formation was deposited in the South East (Gault Clay) a sand-rich deposit in the West (Upper Greensand) and a Limestone in the North (Hunstanton Formation). These very different materials grade into each other. The Gault Clay becomes increasingly sandy towards the west. The stratigraphic complexity is a result of the variability of the original depositional environment. The Gault Clay is thickest around Eastbourne at just over 75m (Figure 6.3). At Cambridge (High Cross) it is approximately 45m thick. The thickness of the Gault Clay reduces rapidly to 0m thickness to the north east of the site, and increasing to a local maximum of just over 60m around Dunstable.

Depositional environment

The Gault Clay is part of an overall transgressive sequence formed from the Lower Greensand (10-20m water depth), the Gault Clay (40-60m depth) and the Lower Chalk (70-100m depth) (Woodcock and Strachan, 2002). The facies changes across the depositional basin from mud to sand to limestone represent changes in the depositional environment. The Gault Clay was deposited in the deepest water. The Upper Greensand was deposited in shallower water near to a sand rich sediment source. The Hunstanton formation was deposited on a relatively shallow platform with limited sediment input. Clearly the water depth was variable across the UK.

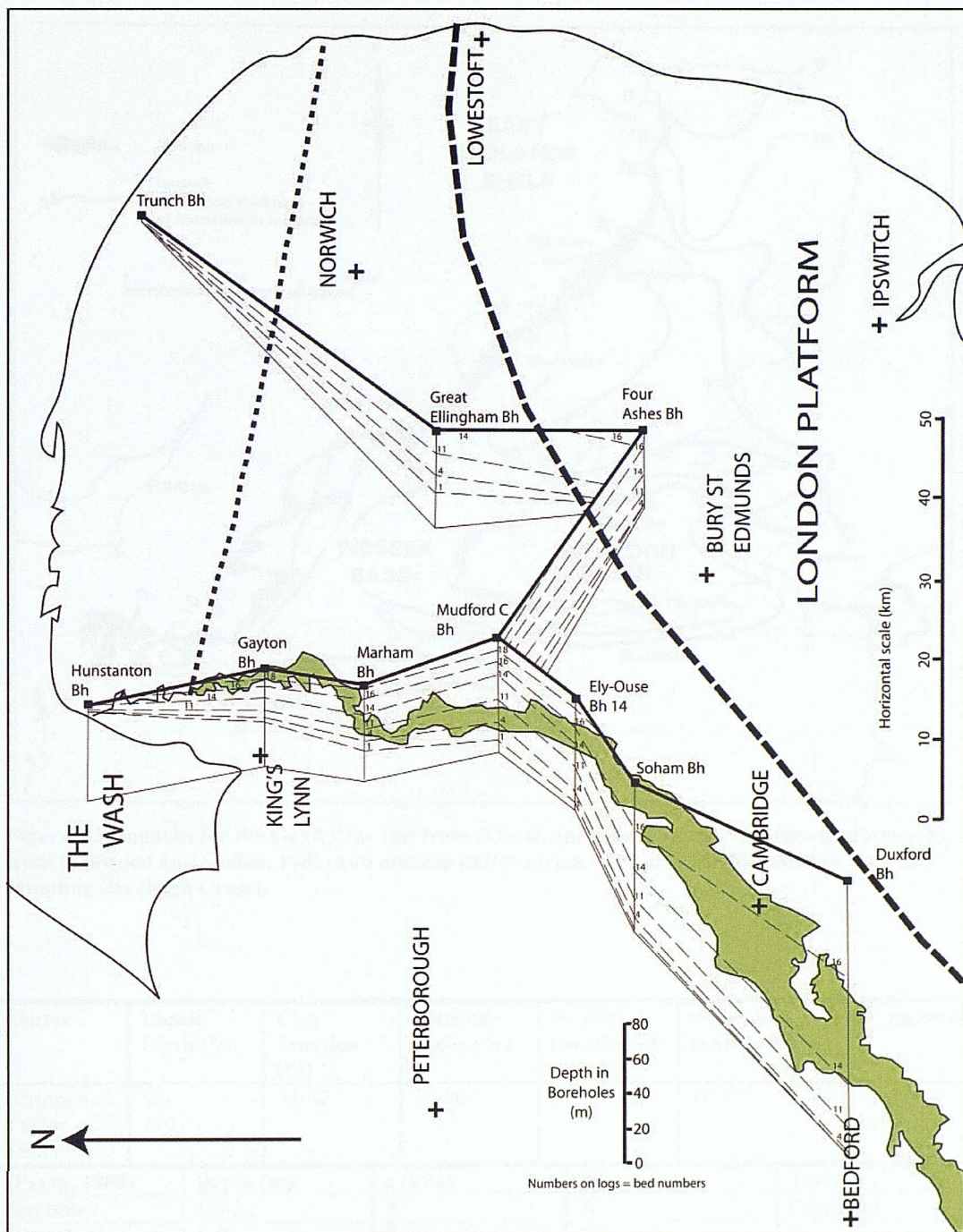


Figure 6.2 Outcrop pattern of the Gault Clay in East Anglia (Green), and thicknesses of the bed units. Redrawn from (Gallois and Morter, 1982)

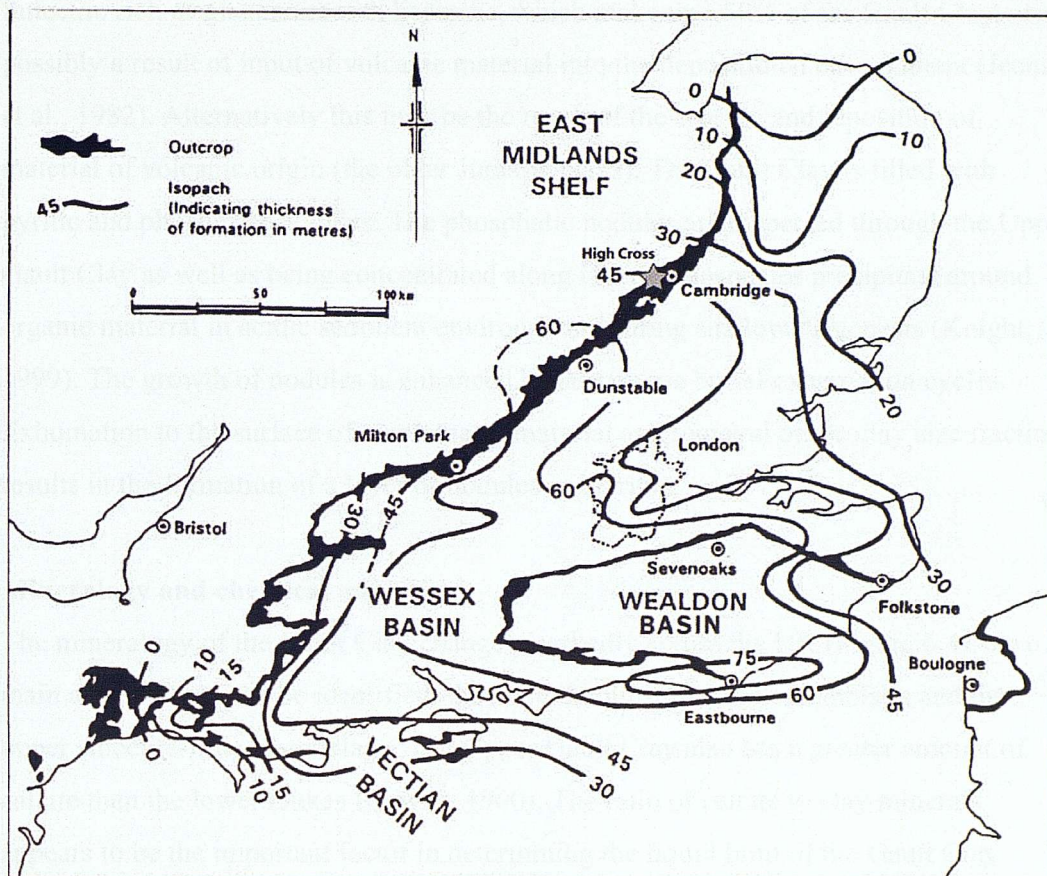


Figure 6.3 Isopachs for the Gault Clay (m) from (Marsh and Greenwood, 1995) based on isopachs from (Sellwood and Sladen, 1981) and outcrop pattern from (Owen, 1971). Modified to show the sampling site (High Cross).

Source	Liquid Limit (%)	Clay Fraction (%)	Plasticity Index (%)	Φ' peak (weathered)	Φ' peak (unweathered)	Φ' residual
(Cripps and Taylor, 1981)	60-120	38-62	27-80	-	19-53°	12- 19°
(Parry, 1988)	Depth (m)	c (kPa)	Φ'	Tests by		
Bin Brook	1.6-2.2	5	28	Fugro Ltd.		
High Cross	1.67	8	24	Fugro Ltd		
		20	22			
High Cross	1-3	20	21.5-24	(Clegg, 1981)		
Source	Moisture Content %	Plasticity Index (%)	Bulk Density Mg/m ³	Dry Density Mg/m ³	Shear Strength Su (kPa)	
(Reeves et al., 2006)	23-42	22-80	1.89-2.13	1.46-1.62	56-1280	

Table 6-2 Example engineering properties of the Gault Clay. The properties of the Gault Clay vary greatly across the UK.

Smectite rich or glauconite rich horizons, which make up ~50% of the Gault Clay, are possibly a result of input of volcanic material into the depositional environment (Jeans et al., 1982). Alternatively this may be the result of the erosion and reposition of material of volcanic origin (the older Jurassic clays). The Gault Clay is filled with pyritic and phosphatic nodules. The phosphatic nodules are dispersed through the Upper Gault Clay as well as being concentrated along layers. Phosphates precipitate around organic material in acidic sediment environments during shallow diagenesis (Knight, 1999). The growth of nodules is enhanced by successive burial/exhumation cycles. Exhumation to the surface of a volume of material and removal of the clay size fraction results in the formation of a layer of nodules as detritus.

Mineralogy and chemical analysis

The mineralogy of the Gault Clay changes markedly across the UK (Figure 6.4). Two main assemblages can be identified: the lower kaolin-illite rich assemblage and the upper smectite-illite assemblage. The Upper Gault Clay also has a greater amount of calcite than the lower (Jukes Browne, 1900). The ratio of calcite to clay minerals appears to be the important factor in determining the liquid limit of the Gault Clay (Marsh and Greenwood, 1995). This ratio is possibly the determining factor for the Gault Clay's engineering properties.

Engineering behaviour

The behaviour of the Gault Clay has been studied on several sites across the UK (Table 6-2). The papers on the behaviour of the Gault Clay are populated by a great number of case histories. The majority of the investigations are concerned in some way with slope stability: Dunton Green (retaining wall), Folkestone Warren (landslide), Castle Hill (Landslip), and M4 (Landslide) (Table 6-1). This relatively high number of stability issues result from the amount of smectite in the Gault Clay (Jones et al., 1996). The most smectite rich part of the formation can be found in the south of the UK (Figure 6.4). So it is unsurprising that the majority of the investigations are located here. Another reason why the Gault Clay is unstable is the presence of the relatively hard Chalk formation lying stratigraphically above the Gault Clay. This caused much of the Gault Clay outcrop to be on sloping ground and thus more susceptible to slope instability.

In the vicinity of the High Cross sampling site for the Gault Clay, there are a great number of well reported engineering investigations. Bin Brook (Parry, 1988) is a slope stability case history. The problem originates within the upper weathered layers of the mudrock which are smectite rich. This is similar to the experiences with the Gault Clay in the South of the UK. Lion Yard (Lings et al., 1991), is a successful construction project, involving a diaphragm wall. The Gault Clay at Lion Yard behaves differently to the Gault Clay at Dunton Green, the wall construction at Lion Yard resulted in a significant drop in lateral stress. This is probably a result of the very different mineral distributions on these sites.

Overall the Gault Clay at High Cross is anisotropic (Butcher and Powell, 1995), however not as anisotropic as the Oxford Clay. Due to its smectite content it is susceptible to slope instabilities, this is not so pronounced near to High Cross due to limited topography. Also due to the high calcite, low quartz contents and limited cementation, the Gault Clay has a much lower strength than the Jurassic mudrocks.

The Cambridge Greensand

The Cambridge Greensand, which contains phosphatic nodules, is an important local marker for the top of Gault Clay (or base of Chalk). Two sets of nodules were recorded in the block sampling excavation for the Gault Clay. There were buff coloured phosphatic nodules approximately 10-20mm in size (Figure 6.5) and yellowish brown sandy nodules approximately 30-40mm in size (Figure 6.6). Locally black phosphatic nodules accumulated in low areas of the weathered surface of the Gault Clay. The nodules are contained within a thin <1m layer of glauconitic sand known as the Cambridge Greensand. These nodules (known locally at the time as coprolites) were being excavated as a commercial product in 1875, for use as a fertiliser. Early workers suggest that the nodules become buff coloured (as at the site) above the Cambridge Greensand, or in the base of the Grey Chalk (Jukes Browne, 1875). No fossils were available within the excavation at the site; concerns were raised that the samples could be from the Grey Chalk (this is a mudrock). This concern was resolved by a visit to Barrington Quarry and microfossil evidence.

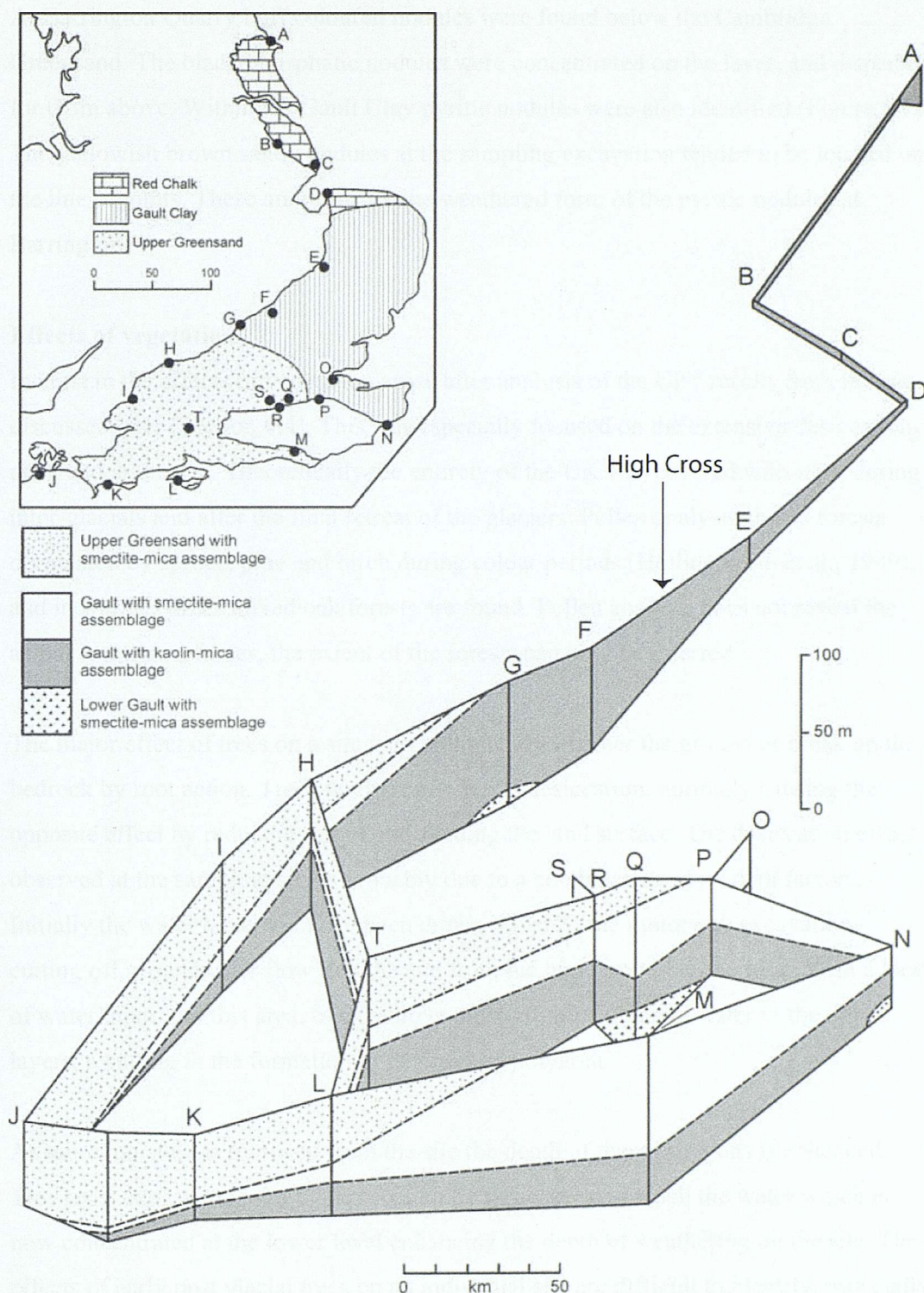


Figure 6.4 Fence diagram showing the mineralogical changes within the Gault Clay and Upper Greensand across the UK. Showing the diachronous transition between these two materials, as well as where the subcrop where each of the Albian formations dominates, from (Jeans, 1978, 2006a). Previous work suggested that the smectite mica assemblage might be indicative of a volcanic origin (Jeans et al., 1982)

At Barrington Quarry buff coloured nodules were found below the Cambridge Greensand. The black phosphatic nodules were concentrated on the layer, and dispersed for 0.5m above. Within the Gault Clay pyritic nodules were also identified (Figure 6.7). The yellowish brown sandy nodules at the sampling excavation tended to be located on the line of joints. These are probably the weathered form of the pyritic nodules at Barrington.

Effects of vegetation

Interest in the effects of vegetation arose after analysis of the CPT results from the site, discussed later (Section 6.4). This was especially focused on the extensive desiccation observed at the site. Theoretically the entirety of the UK was covered with trees during inter-glacials and after the final retreat of the glaciers. Pollen analysis shows forests dominated by spruce, pine and birch during colder periods (Hollingworth et al., 1949), and in warmer times mixed oak forests are found. Pollen analysis does not reveal the actual locations of trees, the extent of the forests can only be inferred.

The major effect of trees on a site is to biologically weather the ground or break up the bedrock by root action. Trees rarely result in soil desiccation, normally causing the opposite effect by reducing runoff and shading the land surface. The desiccation effect observed at the sampling site is probably due to a combination of modern factors. Initially the water table will have been drawn down by the motorway excavation, cutting off groundwater flow into the site from the high ground to the west. With a lack of water input into this area, trees remove much of the remaining water in the upper layers, resulting in the formation of desiccation polygons.

As more moisture is removed from the site the depth of these polygons is enhanced. Tree roots exploit the joint planes created by desiccation to reach the water which is now concentrated at the lower level enhancing the depth of weathering on the site. The effects of early post glacial trees on an individual site are difficult to identify, especially in modern woodland. Root depths for most trees tend not to exceed 4-5m (Crow, 2005). As the material in which these roots are present is biologically weathered it is likely that the erosion process which removed the trees removed the soil which the trees affected. This is especially likely for the mudrocks of the UK which are located in valleys most of which are the result of post glacial processes.



Figure 6.5 Phosphatic nodules from the block sampling excavation in the Gault Clay at High Cross,

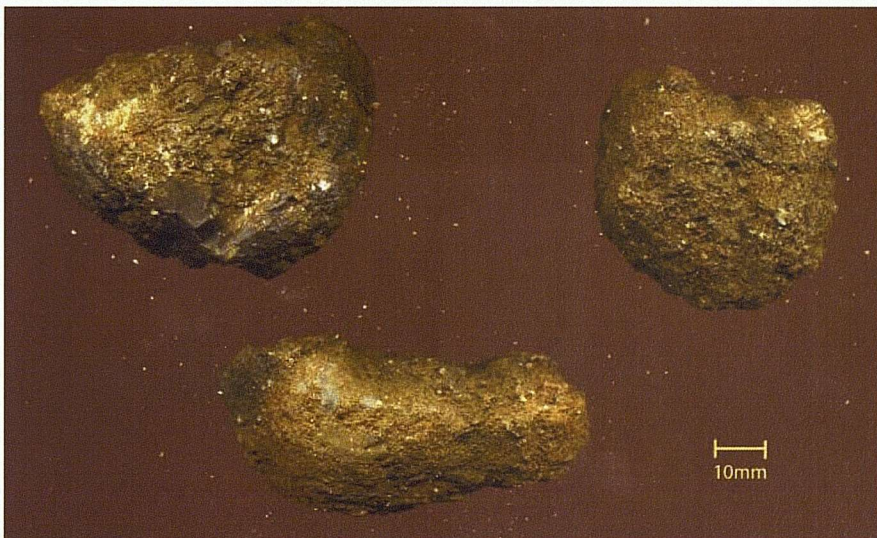


Figure 6.6 Weathered pyritic nodules from the block sampling excavation in the Gault Clay at High Cross



Figure 6.7 Pyrite nodules from the Gault Clay at Barrington.

6.2 *Reconstruction of material history*

Similarly to the Jurassic Mudrocks the Gault Clay was deposited in an open seaway between islands in the north western corner of a much smaller Tethys Ocean than was present during the Jurassic. The rise in global sea levels caused by the opening of the Southern Atlantic slowly caused the drowning of many of the smaller islands creating a more open environment. The major sediment sources for the East Midlands Shelf and Midlands Platform was the London Platform from which silt, phosphates and grey mud was sourced, and the shallow Red Chalk reef near Hunstanton where the red mud is sourced (Figure 6.8). The alignment of phosphatic nodules on layers indicates periods of either limited deposition or erosion (regression). The rest of the sequence represents normal deposition in a gradually deepening environment (Gallois and Morter, 1982). Eroded horizons may have experienced a greater amount of burrowing/bioturbation than the majority of the mudrock. The burial curve for the Gault Clay (Section 3.4) is based on the same data used to produce the Oxford Clay curve. The events in the mudrock's history will be the same as those for the Oxford Clay. However at the time of deposition of the Gault Clay the Oxford Clay had already been buried ~250m. Thus irrespective of the maximum burial of the Gault Clay it must be buried ~250m less than the Oxford Clay. From the available stratigraphy the Gault Clay was buried to at least 300m. From correlated apatite fission track analysis it was buried to a depth of 870m following the same event history as the Oxford Clay (Section 5.2).

6.3 *Sample site details*

The Gault Clay was sampled at High Cross, known in the literature as Madingley (Figure 6.9). The site is owned by Cambridge University. Access to the site was kindly organised by Malcolm Bolton and Brian Lees, on site works being supervised by the Clerk of Works Andrew Merrick. The site is located close to the centre of the Gault Clay outcrop in this area (Figure 6.10). The bedding at this location dips ~1-2° to the South East. The site is located between two chalk outliers which are on hills covered by Quaternary materials (Glacial Till). Stratigraphically the samples are taken from the top of the Gault Clay sequence. The site is within an old river valley, however there are no old river deposits at the location (Figure 6.11).

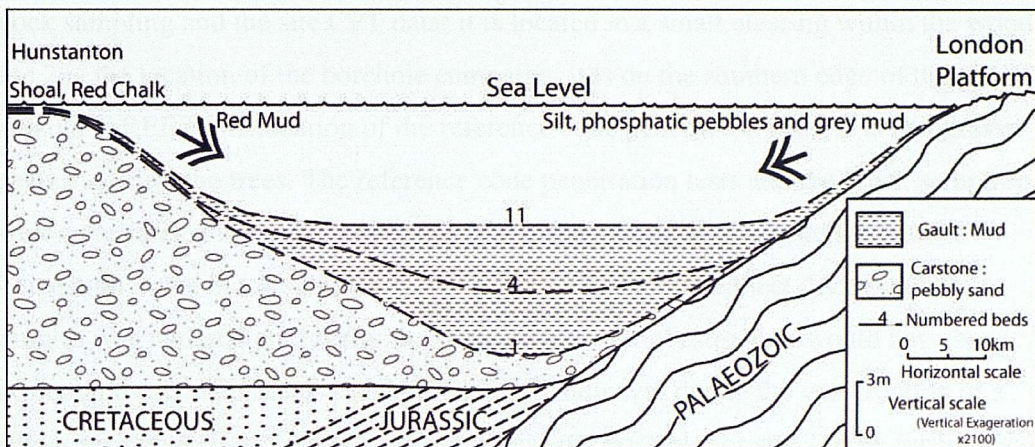


Figure 6.8 The environment of deposition of the Gault Clay modified from (Gallois and Morter, 1982).

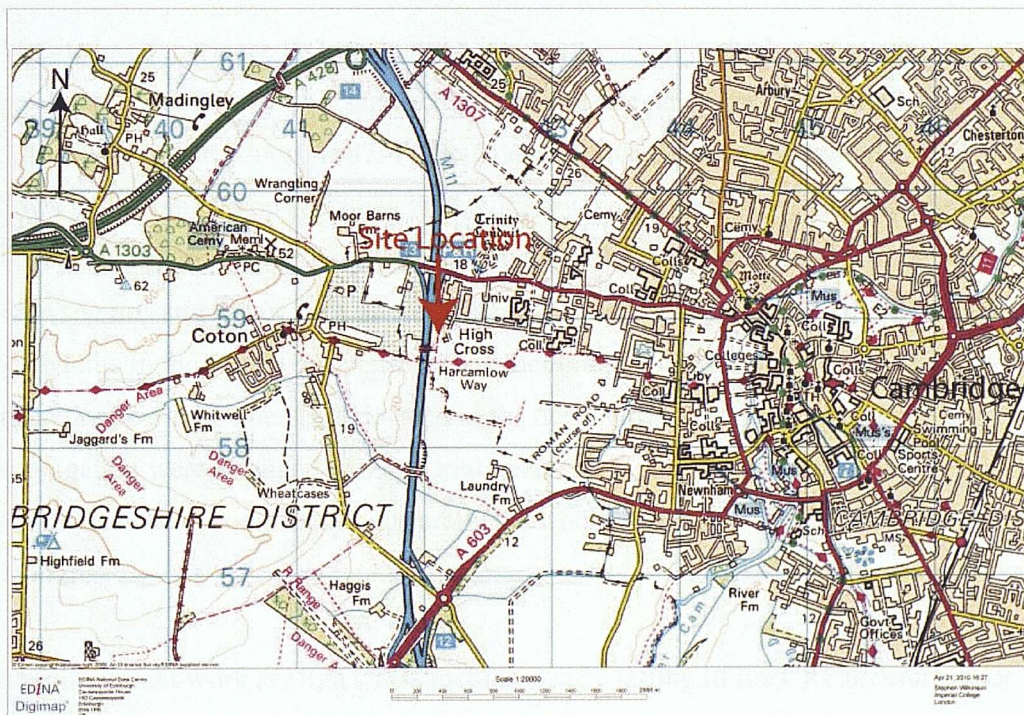


Figure 6.9 Map of Cambridge showing the site location, High Cross. The area generally has very limited range of topography. A small hill of boulder clay is present to the West of the site (Ordnance Survey, 2008).

The site can be divided into three main areas (Figure 6.12). Site 1 is the location of the block sampling and the site CPT data; it is located in a small clearing within the wood. Site 2 is the location of the borehole campaign, it is on the southern edge of the car park. CPT REF is the location of the reference cone penetration tests; it is in a grassy area away from the trees. The reference cone penetration tests and the block sampling work were carried out at the same time. On discovery of the high suctions due to desiccation in the block samples. A further investigation to collect deep borehole samples was undertaken. Ideally the second borehole investigation would have been located in the same place as the REF CPT site, however due to the construction of a badger sanctuary, this was not possible. It was not possible for site 2 to be further west or south due to roosting birds. It was also not possible for the site to be located further to the east due to a high pressure gas line. The locations of the CPT profiles, boreholes and all other features on the site were measured by hand held GPS accurate to 4m (Figure 6.13). Also marked is the location of major previous site works.

This site has experienced a long history of site investigation work, as it was an important location for soils research. Thus the old site plans are quite complex (Figure 6.14). The site data (Coop, 1987; Fugro Consulting Geotechnical Engineers, 1979), allows good comparisons between the laboratory work and how the mudrock behaves on the site. This fulfils the available site investigation data prerequisite for the project. Some site investigations, such as Clegg's model pile tests (Figure 6.14), could not be located due to the density of the trees. Another old pile test location (Figure 6.13) with a different pile arrangement was located to the north of the site. The six piles in this investigation were arranged within a triangular pit (Figure 6.15). These piles are very old and have been completely overrun by the wood; they could not be related to any literature.

Other important work at High Cross includes: early testing of the CPT pressuremeter (Houlsby and Withers, 1988), and self boring pressuremeter at High Cross and at Lion Yard in the centre of Cambridge (Marsland and Powell, 1990), testing several different shear wave velocity measurement techniques (Butcher and Powell, 1995). Comparisons have been made between stiffness measurements made in the laboratory and in the field (Dasari and Bolton, 1998) and most recently measurements of mudrock anisotropy (Lings et al., 2000)

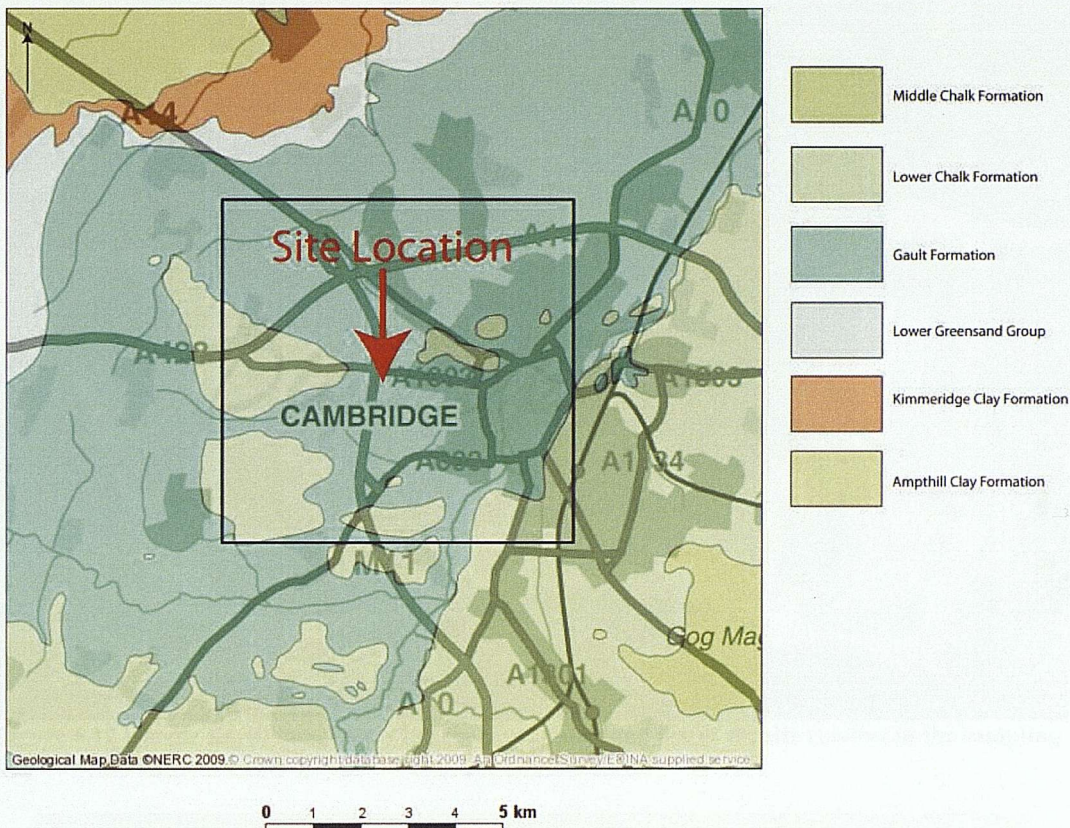


Figure 6.10 Solid geology around High Cross. Modified from (British Geological Survey, 2009a). Square indicates location of superficial geology map (Figure 6.11).

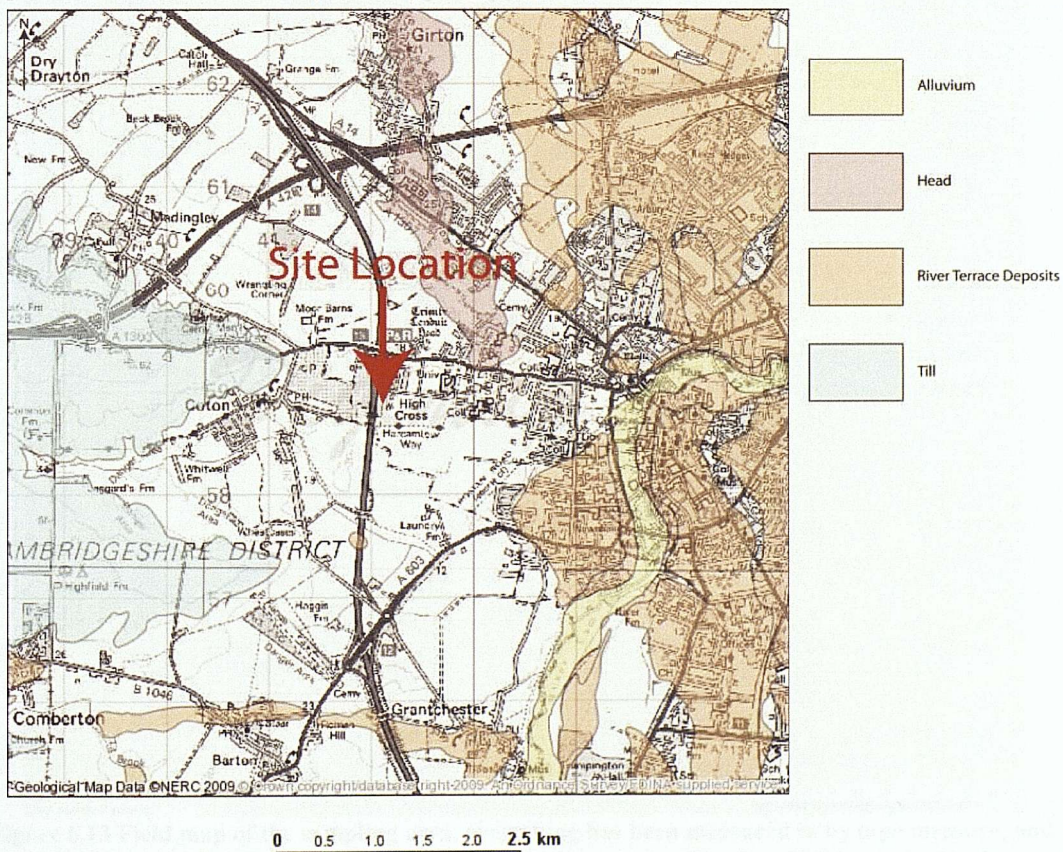


Figure 6.11 Drift cover around High Cross (British Geological Survey, 2009d)



Figure 6.12 Google Earth image showing the layout and land use of the site relative to the sampling localities (Google Inc., 2010).

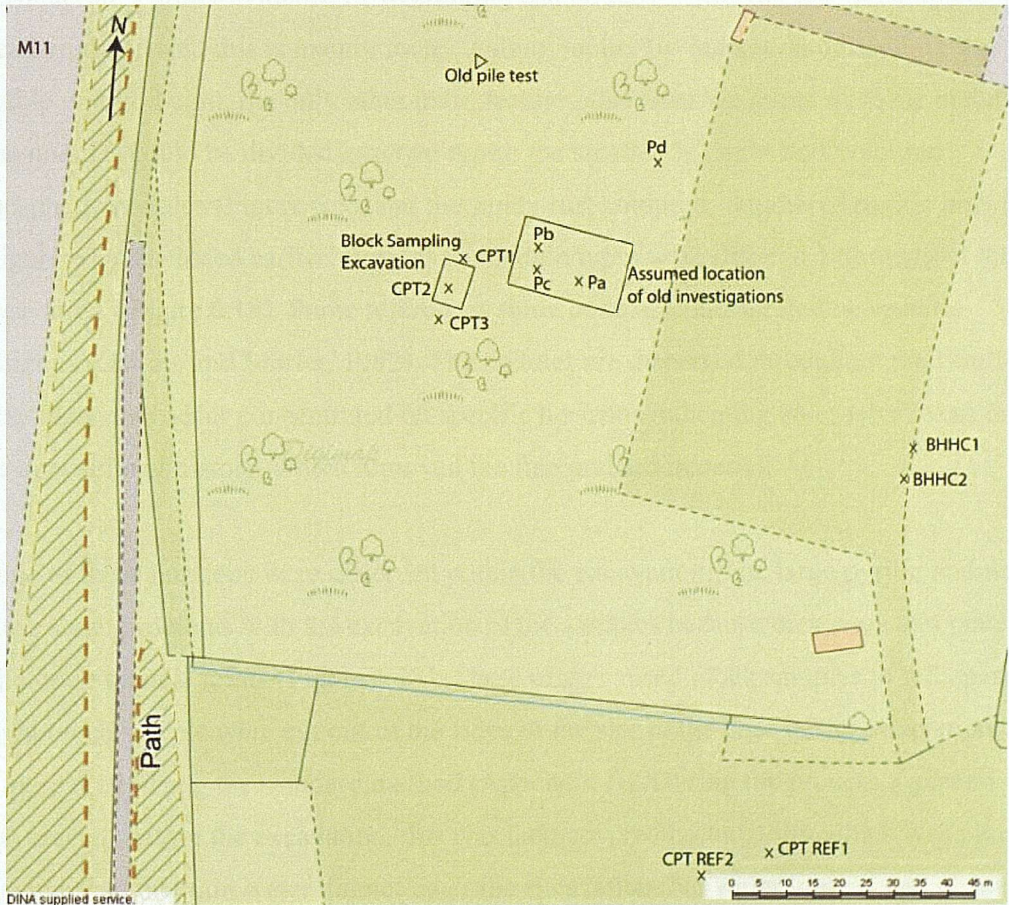


Figure 6.13 Field map of the sampling area, everything has been measured in by tape measure, and hand held GPS. Pa, Pb, Pc, and Pd are pre-existing piezometers. The five CPT tests, the block sampling investigation and the two boreholes are from this investigation. The old pile test is from an unknown historic work. A piezometer was installed in BH HC1 (Ordnance Survey, 2007).

Near to the site small slope instabilities have been examined at Bin Brook (Parry, 1988). In the centre of Cambridge, Lion Yard was extensively studied during construction and papers often link this site to High Cross (Butcher and Lord, 1993; Lings et al., 1991).

Block sampling

Site1 (Figure 6.12) was identified as a good location for an excavation for block samples. To allow sampling, a 3m deep benched trench was excavated in stages (Figure 6.16). An initial 1m deep wide flat bottomed excavation was dug out with a tracked excavator. The centre of this excavation was subsequently deepened, forming the benches. A further slot was cut along the centre of the excavation, to allow easy access to the sample locations. The soil profile was visible in the side of the excavation (Figure 6.17). The rock is clearly completely weathered to a depth of ~1.15m, this is made up of approximately 0.3m of top soil and 0.85m of yellow mudrock. Below this the mudrock grades from light to dark clay, probably representing the increase in moisture content of the material. Further evidence of weathering can be seen below the 1.15m completely weathered horizon, this is mainly focused along joints. No indication of bedding was visible within the pit, the only other main feature other than the joints were the nodules. The nodules could be divided into two types: the smaller 5-25mm buff coloured phosphatic nodules (Figure 6.5), and the sandy rust coloured, weathered pyritic nodules (Figure 6.6) discussed earlier. Both of the nodule types were dispersed throughout the excavation (Figure 6.18). Some references show nodules concentrated on specific horizons (Gallois and Morter, 1982). The nodules are dispersed throughout the Gault Clay, they also occur concentrated on specific horizons indicating an erosive event in the depositional history, which removed the fine grained material.

None of these horizons were observed within the excavation. The large pyritic nodules cause some problems with the excavation of the samples because they were associated with open vertical joints (Figure 6.18). These often caused block samples to fall apart. Eight block sample were cut out of the sides of the slot in the base of the excavation (Figure 6.19) using the standard method (Appendix B). During the process a gazebo was erected within the excavation, this was then covered by tarpaulin which was pinned into place so that rain water did not enter the excavation. No water ingress was observed during the excavation.



Figure 6.15 Top, a triangular pit containing 6 old pile like projections, facing south. Bottom, a box connected to one of these edifices. This whole area has been completely overgrown by trees.

Moisture content samples were taken from the sides of the excavation; water content measurements were taken using the oven at the Cambridge University Schofield Geotechnical Centre, on site.

At the end of sampling works, a joint survey was carried out on the southern end of the excavation (Figure 6.20). The joints observed were mostly high angle following two major directions clustering around $\sim 210^\circ$ and $\sim 150^\circ$. The roughness of the surface produced $\sim 15^\circ$ scatter. The joint spacing was 60-100mm, and they became less apparent with depth. This sort of pattern indicates these joints are desiccation polygons probably produced by the combination of the drawdown associated with the M11 cutting and the effects of the trees on the site. Although most of the joints were closed, some were open (especially those associated with nodules). Several times the edges of block samples opened up along vertical joints. Fortunately at all times there was enough material behind joints from which a block sample could be taken. Most of the joint surfaces were rough, on a few occasions roots were found taking advantage of the joints (Figure 6.21). On completion of sampling the excavation was filled in and the ground surface levelled.

Borehole sampling

Analysis of CPT data from the site and the laboratory behaviour of the mudrock showed the true extent of the desiccation experienced by the samples. A second sampling campaign was designed using boreholes with the aim of collecting samples from depth below the block sampling location. The date of this sampling campaign was late July 2009. By this time the options for the location of boreholes was extremely limited due to a badger sanctuary and roosting birds. Samples were recovered from two $\sim 14\text{m}$ deep boreholes located at (TL 42201 58864) & (TL 42203 58856), the method used is described later (Appendix B).



Figure 6.16 The initial stages of the excavation, 1m depth topsoil is 20-30cm thick going directly into the weathered top of the Gault Clay

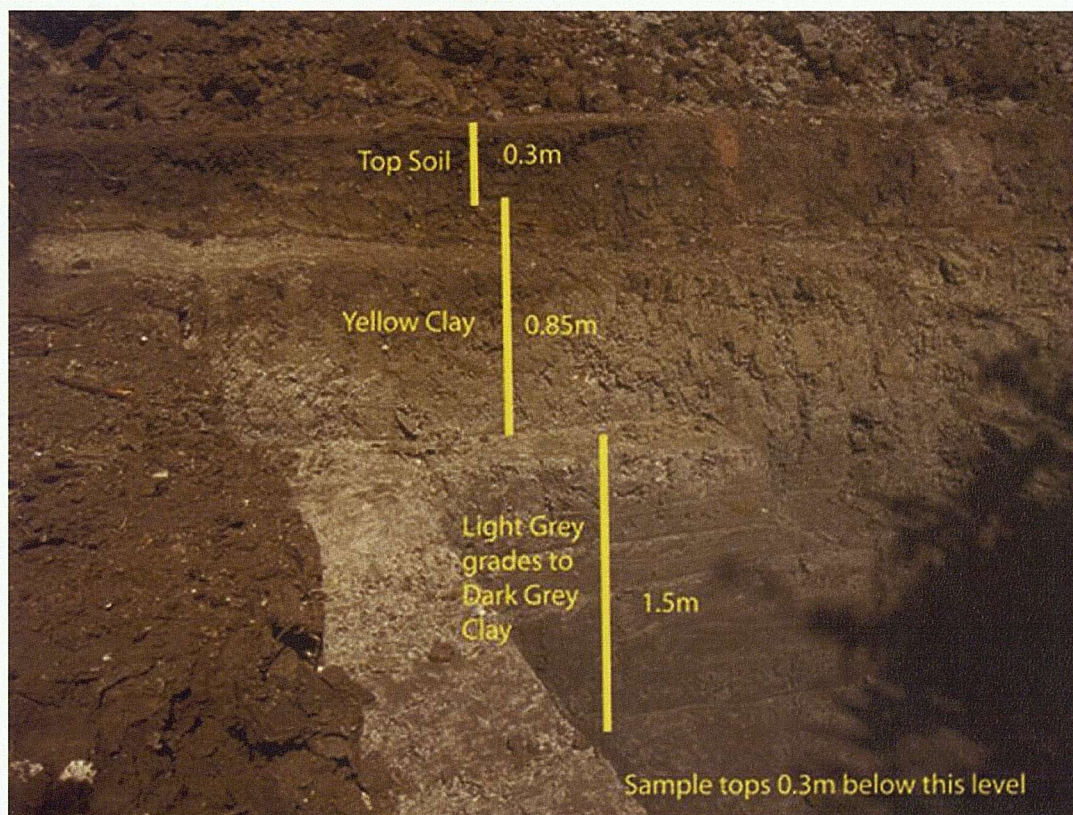


Figure 6.17 Profile of the Gault Clay within the excavation at High Cross. The weathered clay extends down to ~1.15m this can be seen from the change in colour of the clay from yellow to grey. Weathering along joints extends to a much greater depth (roots found on joints at depth). The extent of weathering was originally identified on the basis of the change in colour of the clay with depth.

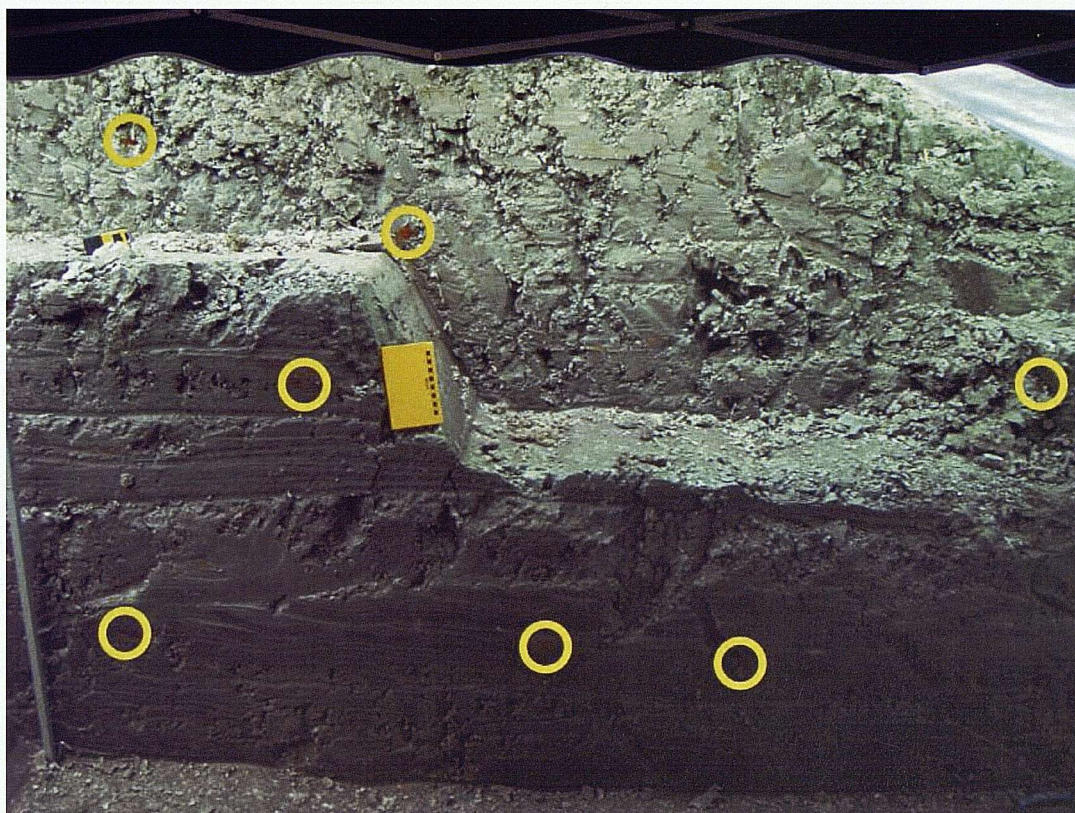


Figure 6.18 The smoothed surface of the clay on the edge of the excavation brown staining indicates scraping the iron rich nodules, inset: one of the brown nodules within a block sample. Notebook for scale (divisions = 10mm).

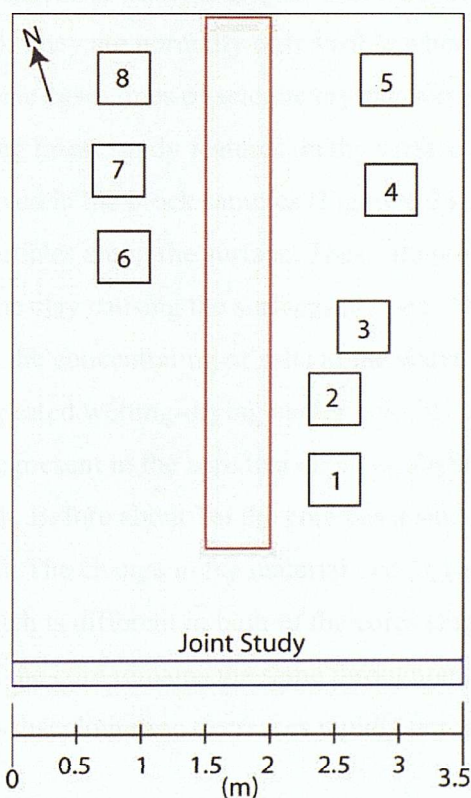


Figure 6.19 Layout of the block samples of Gault Clay in the base of the benched excavation at High Cross. Red area marks location of central slot cut into base of excavation. Blue area is location of joint study (Figure 6.20)

The stratigraphy of the boreholes as measured on site was as follows: reinforced concrete 0-0.29m, light grey mottled yellow completely weathered Gault Clay 0.29-2.01m, light grey slightly sandy Gault Clay weathered along joints, with weathered nodules 2.01-3.46m, light grey slightly sandy clay 3.46-7.31m, light grey clay with several holes on core surface 7.31-9.23m, grey clay with several holes on the core surface 9.23-13.62. This stratigraphy can be interpreted as: 0.29-2.01, completely weathered Gault Clay. 2.01-7.31 Gault Clay weathered along joints. This is indicated in the shallow part by the weathering of nodules and in the deeper part by the formation of selenite crystals (sand) along joints. A darkening of colour of the Gault Clay at 9.23m probably indicates a change in mineralogy.

Logging

The logging was performed according to the method described in the appendix (Appendix B). The features of the logs are described here. At a shallow level the core is weathered and mottled yellow (Figure 6.22). The weathering appears to follow horizontal bands; it is probably picking out the underlying bedded structure of the mudrock at the coarse scale. The amount of mottling decreases with depth, becoming concentrated around inclusions in the core (Figure 6.23). Several joints are present in the core, most are closed. They are normally only visible where the drilling has disturbed the joint. In some cases lines of selenite crystals have been found aligned along joint planes forming linear sandy features on the surface of the core. These surfaces were also observed in the block samples (Figure 6.24). There is a high concentration of sand particles along the surface. These are probably formed by the removal of water from the clay causing the surfaces to open. The removal of water from the mudrock also causes the concentrating of salts in the water. This allows the formation of crystals, repeated wetting-drying cycles possibly causing their growth. These sandy planes were present in the core to a depth of about ~7.4m, they become less numerous with depth. Before about 7m the core has a smooth surface (Figure 6.25) no holes can be observed. The change in the material occurs between 7.2 and 7.5m it is a gradational change which is different in both of the cores (Figure 6.26 and Figure 6.27). The grey shade of the core remains the same throughout, the top of the core is discoloured yellow. This discolouration decreases rapidly across the transition.

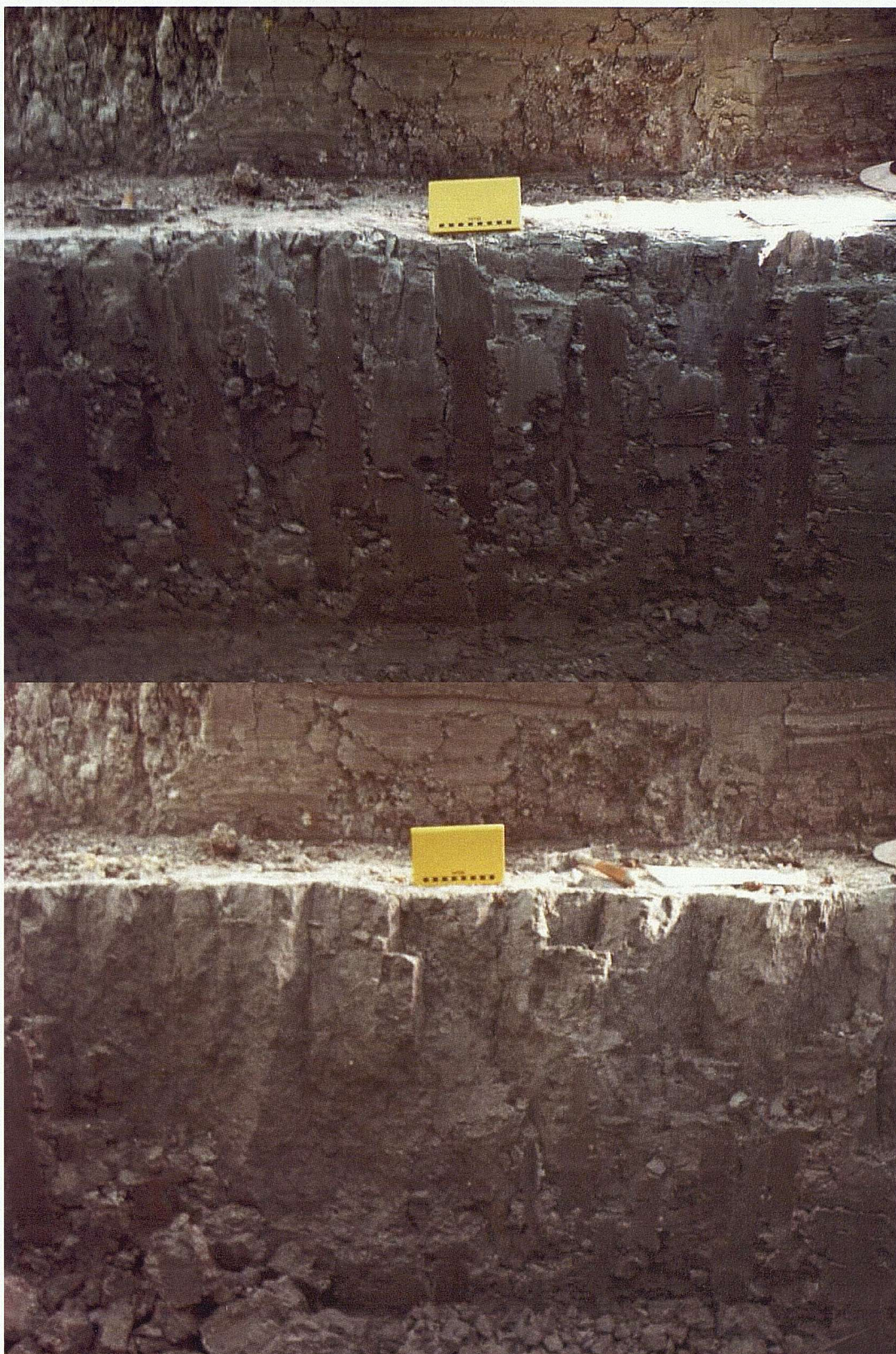


Figure 6.20 Both images show the side of the benched excavation in the Gault Clay. Top image main feature claw marks of the mechanical excavator used to dig the pit. Bottom image after the surface was cleared by hand revealing two main sets of closely spaced (50-100mm) joint planes. Notebook for scale (divisions = 10mm).

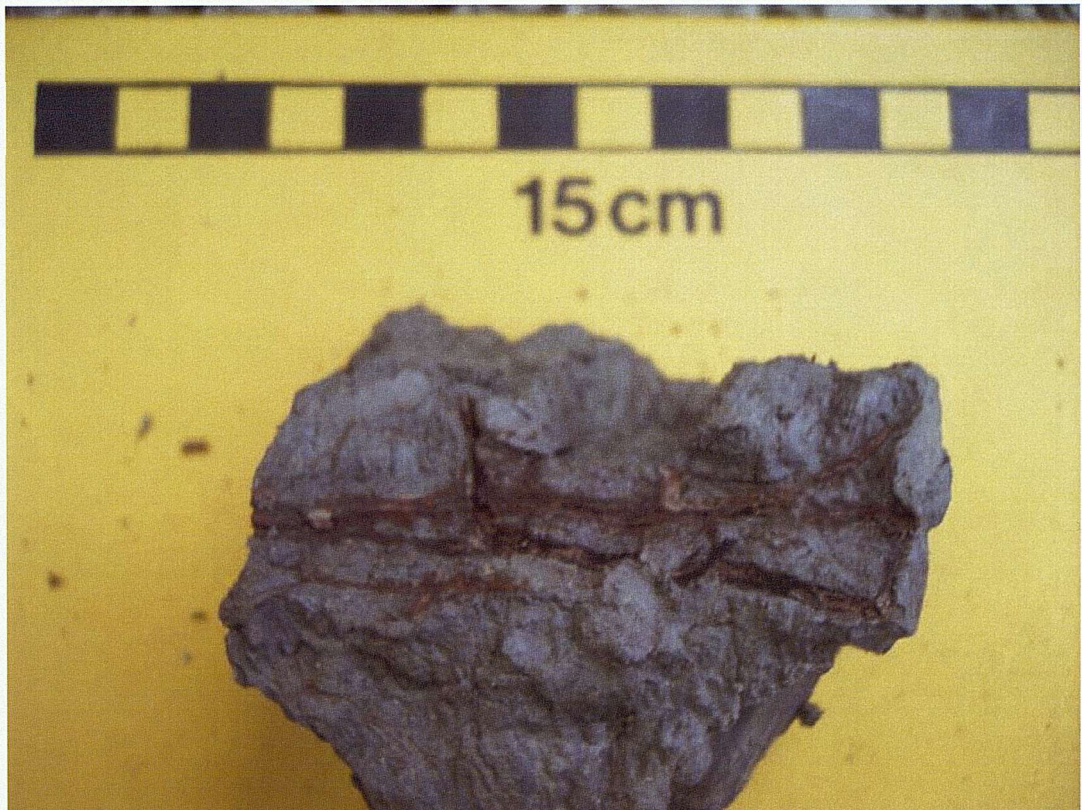


Figure 6.21 A broken joint surface of the Gault Clay from just above the sampling depth, showing roots within the joints of the Gault Clay to at least three metres depth.

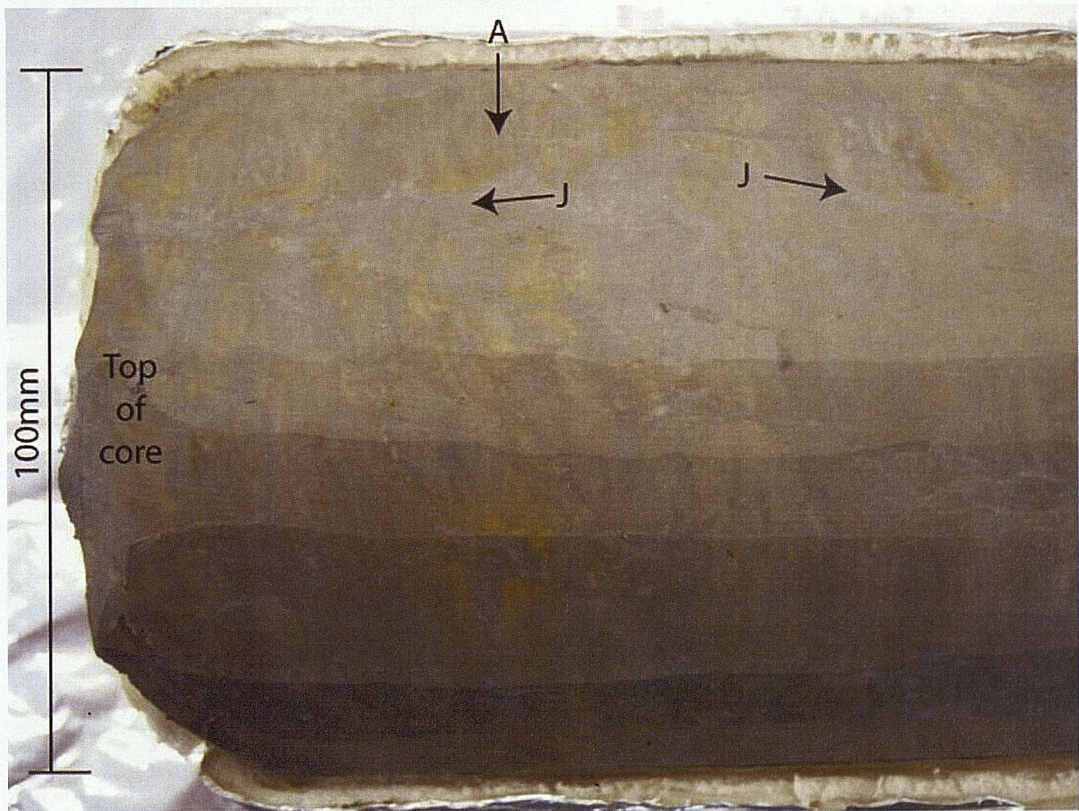


Figure 6.22 Gault Clay core HC2, 1.8m (sample top) at left edge. Yellow mottling showing some alignment to the horizontal (A) at one point breached by a rough grey vertical line (probable joint (J), possible desiccation crack). Lines parallel to sides of the core are scrape marks.

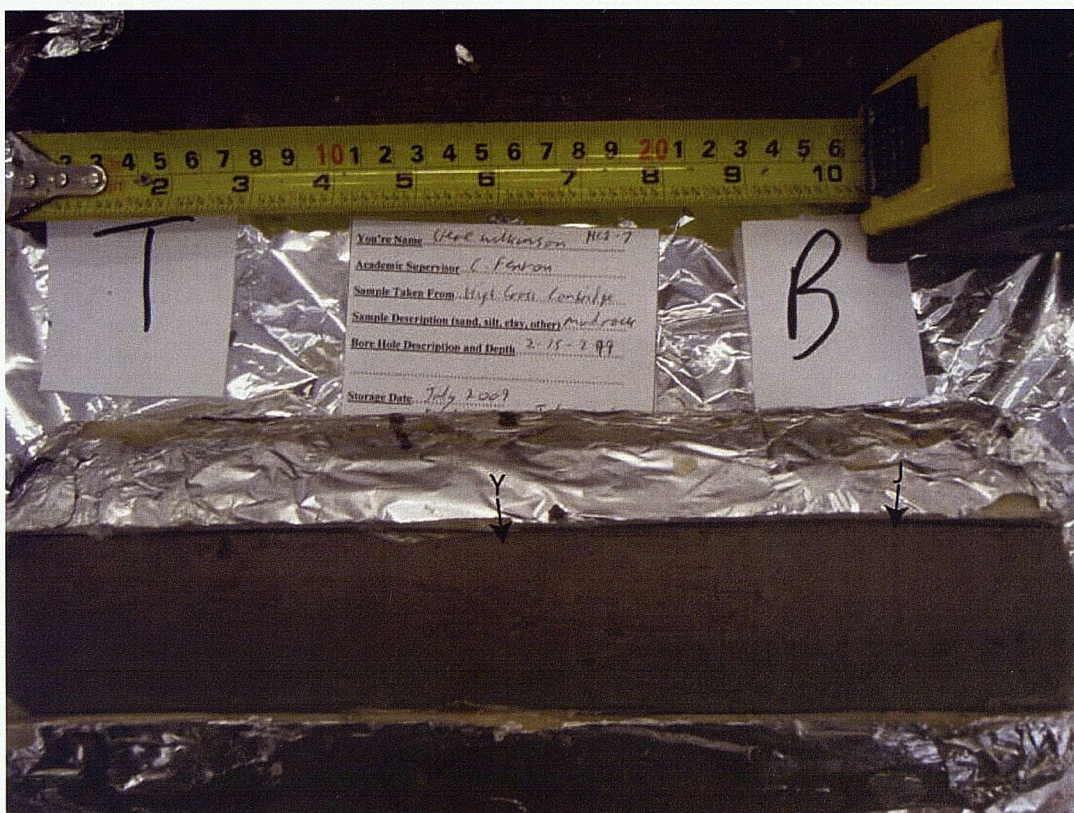


Figure 6.23 Gault Clay core HC1, 2.75-2.99m yellow mottling is present at this depth as spots on the core (Y), mainly associated with nodules or inclusions in the core. Near horizontal joint (J) 50mm from the base of core, sand particles are associated with joints. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

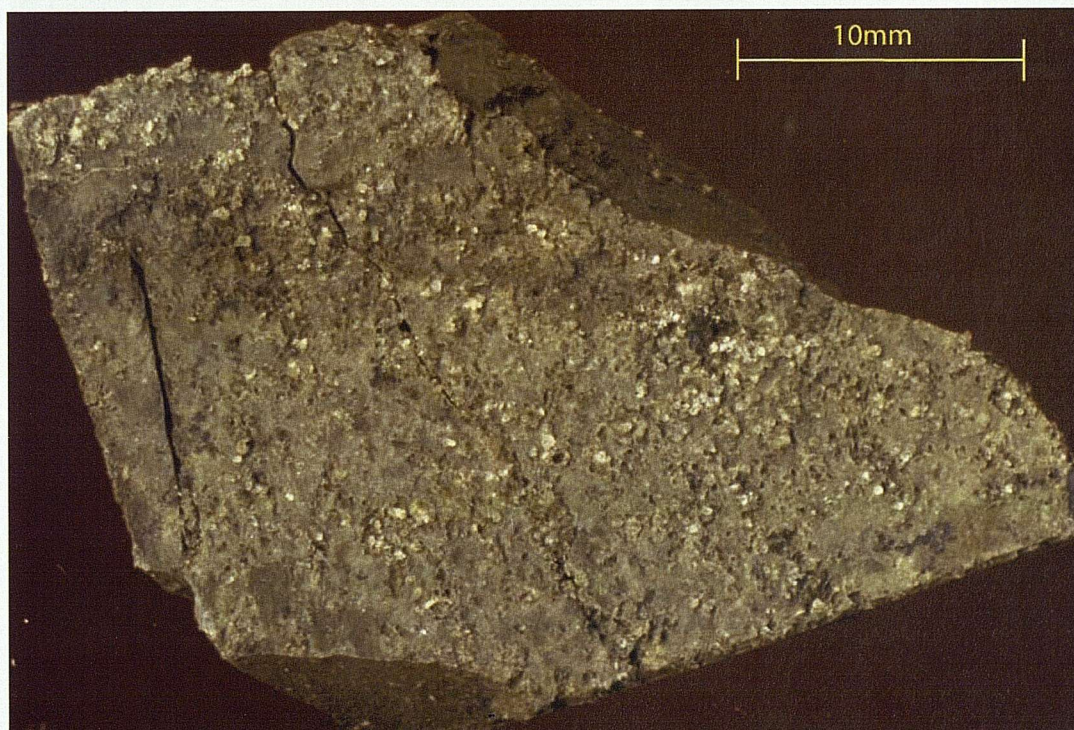


Figure 6.24 Joint surface from the block samples of the Gault Clay joint is covered in selenite crystals.

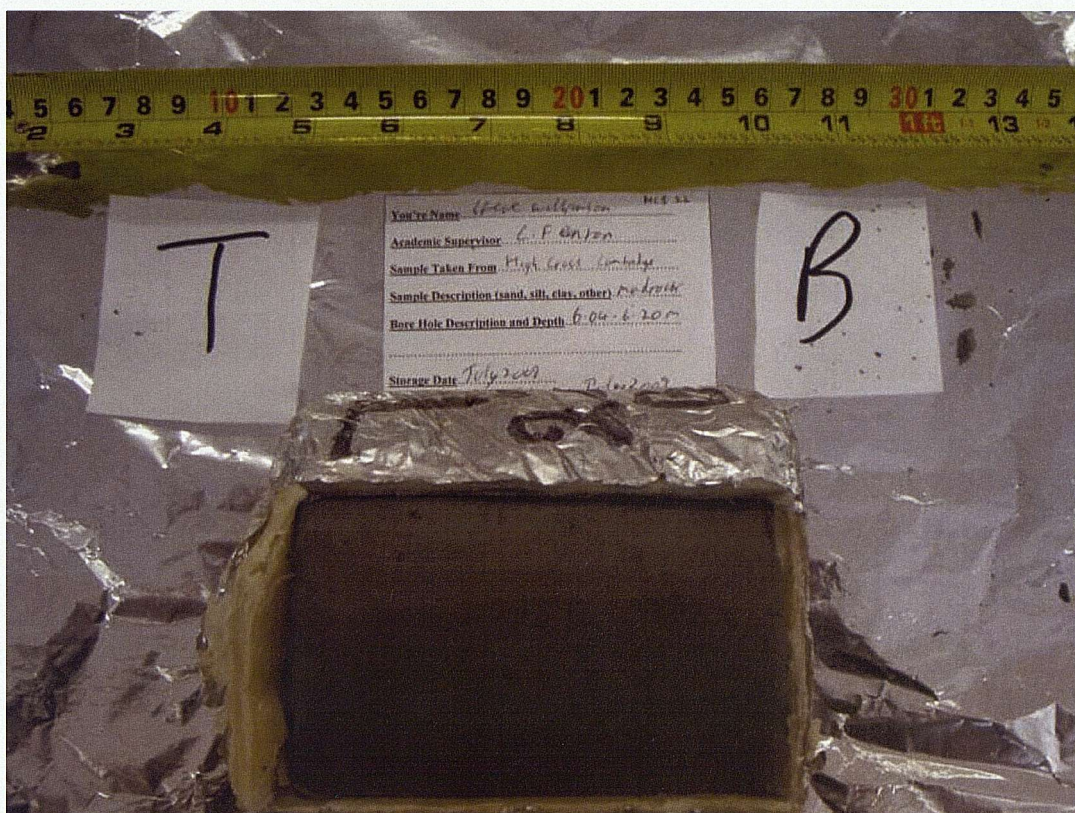


Figure 6.25 Gault Clay core HC1, 6.04-6.20m. Mottling becomes rare some sand particles in core. Core surface generally smooth. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

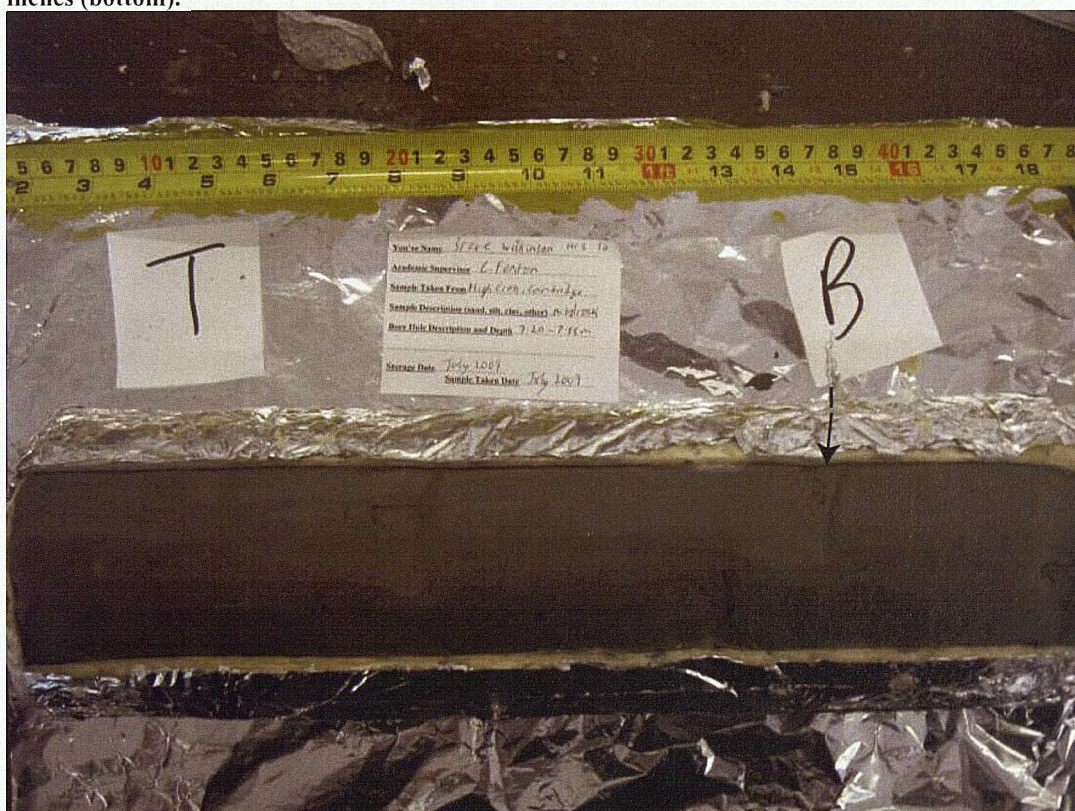


Figure 6.26 Gault Clay core HC1, 7.20-7.55m. The mudrock changes slowly from about 100mm along to core to 220mm along the core. The main change is one of colour, the sample also shows indents (I) in the lower portion. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

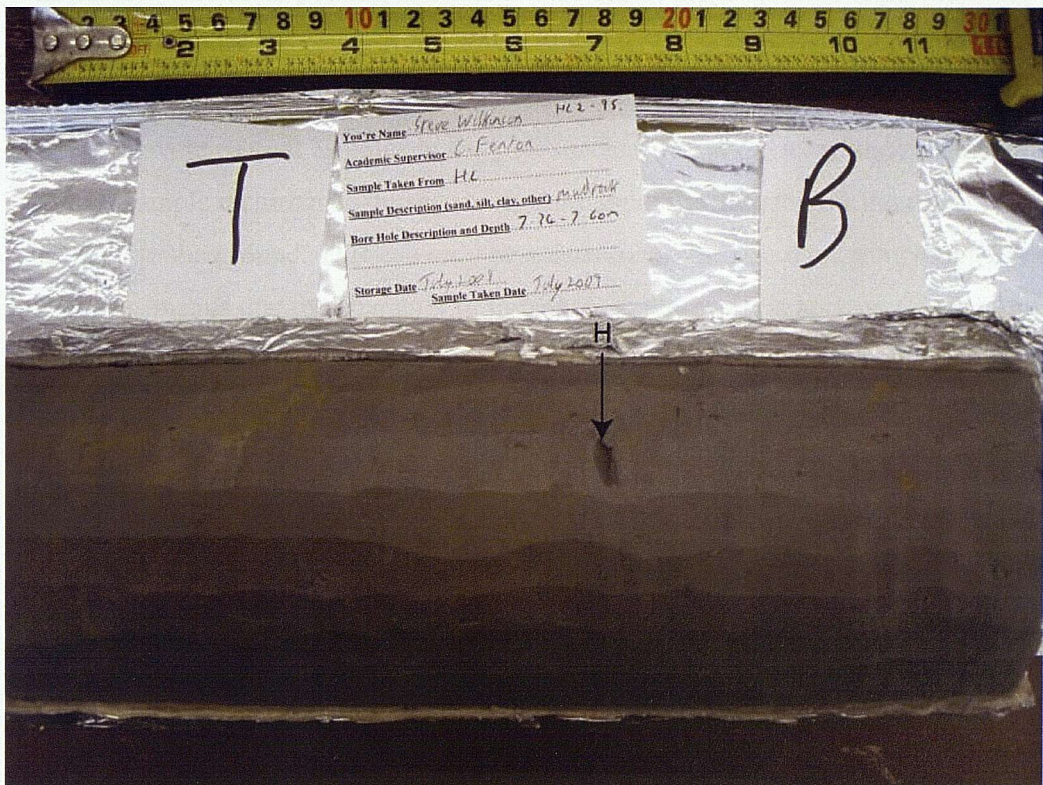


Figure 6.27 Gault Clay core HC2, 7.34-7.60m. Same transition as Figure 6.26 the gradational colour change is shown to be a decrease in yellow mottling of the surface. A hole (H) is present in the centre of the core length. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

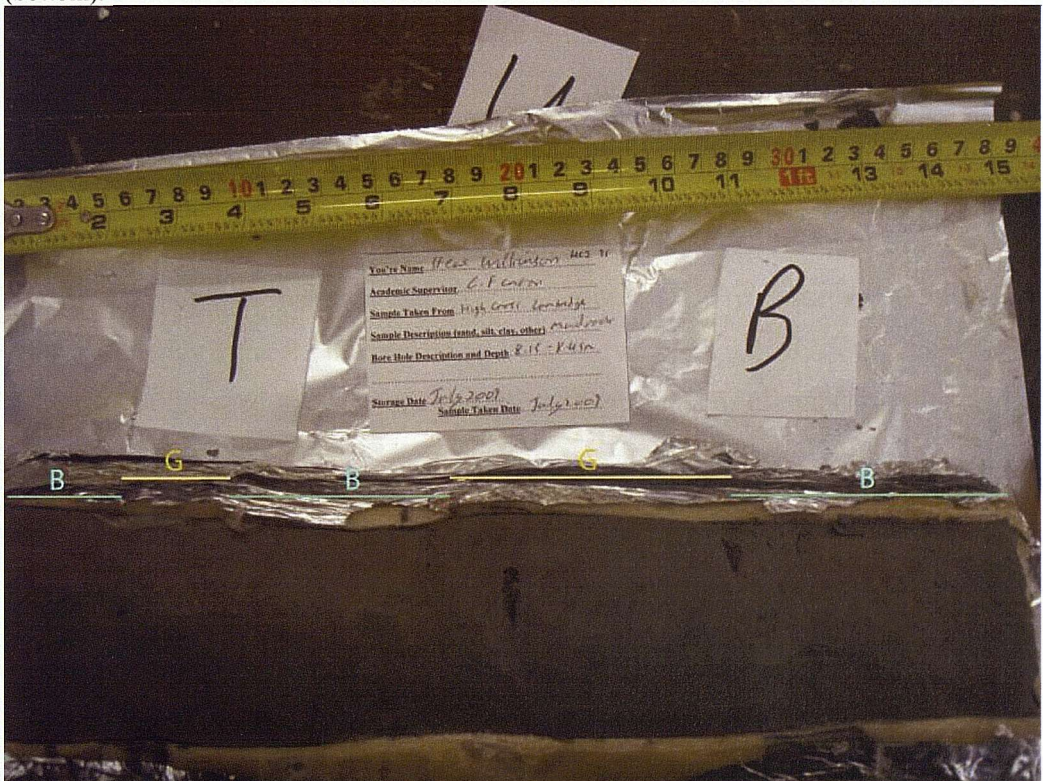


Figure 6.28 Gault Clay core HC1, 8.15-8.45m. Variations in shading, correlating with changes in the thickness of the core, three sections brown (B) shade, two sections grey (G) shade. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

This possibly indicates pooling of water at the base of the desiccated region forming a layer of enhanced weathering. In the lower non discoloured portion several indentations are visible in the surface (Figure 6.26). These are filled with soft material. In the field these areas were present as holes in the core surface. The presence of the soft material indicates sample disturbance during storage. At about this horizon during drilling, flush was lost into the ground. This can only be achieved through fluid entering the mudrocks joints. This means that the character of the joints changes at this horizon. The wetting-drying cycles causing the opening of joints in the upper layers possibly caused deposition of fine grained material within the joints (smectite?). As the water table was high at the time of drilling, the upper joints will have been sealed by this material. The lower joints were open and in many cases material from these joints was washed out producing holes in the core (Figure 6.26). The drilling fluid which had entered the joints during drilling has now produced areas of soft material which is now infilling the holes.

Between 8.15m and 8.45m metres depth another transition occurs, this one is characterised by the inter-bedding of units of varying shade and properties (Figure 6.28). The shade change coinciding with a change in the thickness of the core (which correlates with engineering properties). This may correlate with a change in the mineralogy of the Gault Clay. Holes in the core appear to be more numerous in the darker shade thicker (stronger?) core sections. These holes become larger at depth (Figure 6.29).

Nodules are present at various horizons within the Gault Clay, some of these are scattered through the material, and others are concentrated on specific horizons (Figure 6.30). These horizons are interpreted as being erosive. Where multiple nodules are preserved in the core, these boundaries are easily identified (Figure 6.30). The nodule bands identified appear to be associated higher quantities of sand.

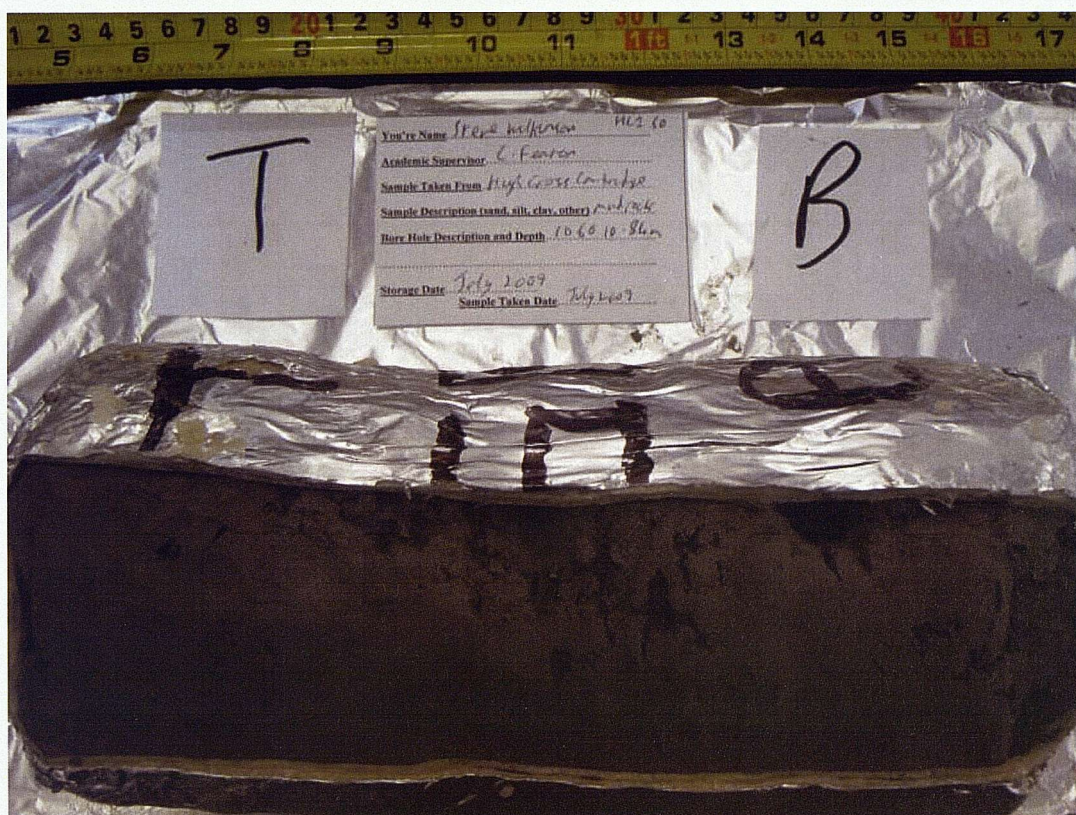


Figure 6.29 Gault Clay core HC1, 10.60-10.84m. Holes observed in the core below ~7m, many holes filled with soft material especially smaller holes. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

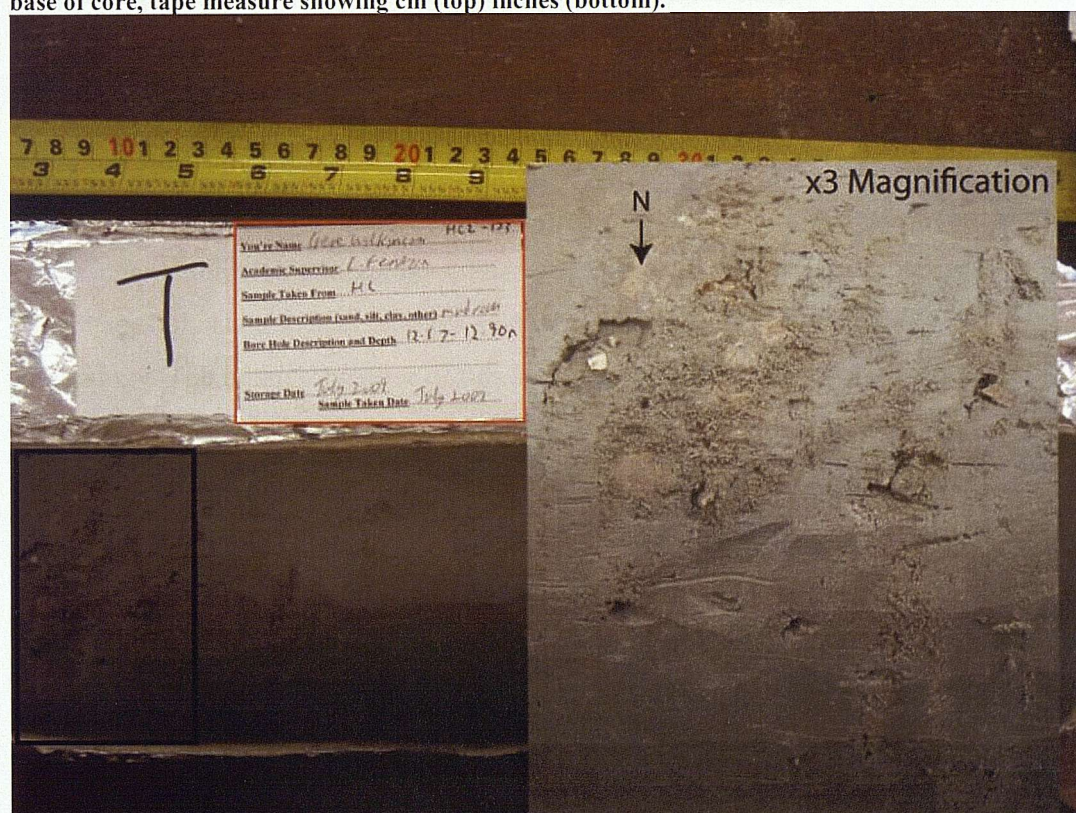


Figure 6.30 Gault Clay core HC2, 12.57-12.90m. Some sections of the core contain bands of phosphatic nodules shown in high magnification in inset. T = top of core, tape measure showing cm (top) inches (bottom).

Gault Clay (depth)	illite & mica	illite-rich illite-smectite	smectite-rich illite-smectite	kaolinite	quartz	K feldspar	chlorite	calcite	Fe dolomite	Pyrite
1.55- 1.72	7%	3%	38%	3%	32%	0%	0%	17%	0%	Tr
3.5	19%	1%	8%	17%	15%	4%	7%	27%	1%	1%
6.82- 6.87	5%	7%	26%	3%	26%	0%	0%	32%	0%	0%
9.99- 10.02	5%	6%	27%	3%	26%	0%	0%	32%	0%	0%
13.46	9%	3%	14%	7%	27%	2%	3%	36%	0%	0%

Gault Clay (depth)	illite	illite-smectite I-rich	illite-smectite S-rich	chlorite	kaolinite
1.55- 1.72	13%	0%	83%	1%	3%
3.5	21%	0%	69%	1%	10%
6.82- 6.87	18%	0%	75%	1%	7%
9.99- 10.02	28%	0%	61%	2%	10%
13.46	73%	0%	2%	10%	16%

Table 6-3 Top table bulk mineralogy of the Gault Clay, bottom table, percentage of minerals contained within the clay grade (<2µm grain size) part of the Gault Clay.

Mineralogy

As boreholes are profiles through geology, multiple samples are required to characterise the change in mudrock mineralogy with stratigraphy. Quantification of relative percentages of clay minerals requires a separate sample to that used for bulk mineralogy. The relative expense of this analysis limits the number of samples that could be analysed in this project.

The mineralogical analysis identifies the changes to the mudrock associated with weathering in the 1.55m sample (Table 6-3). These include a higher quantity of smectite and a lower quantity of calcite due to dissolution. Quartz increases by default as it is not dissolved or removed. The samples at mid depth (6.82 & 9.99m), reflect the mineralogy of the upper part of the Gault Clay. Samples are generally rich in calcite which originates from the large number of micro and nano fossils (especially coccoliths) present in the mudrock. The Gault Clay is also rich in quartz probably in the form of detrital silt particles (Figure 6.31).

XRD Bulk Mineralogy

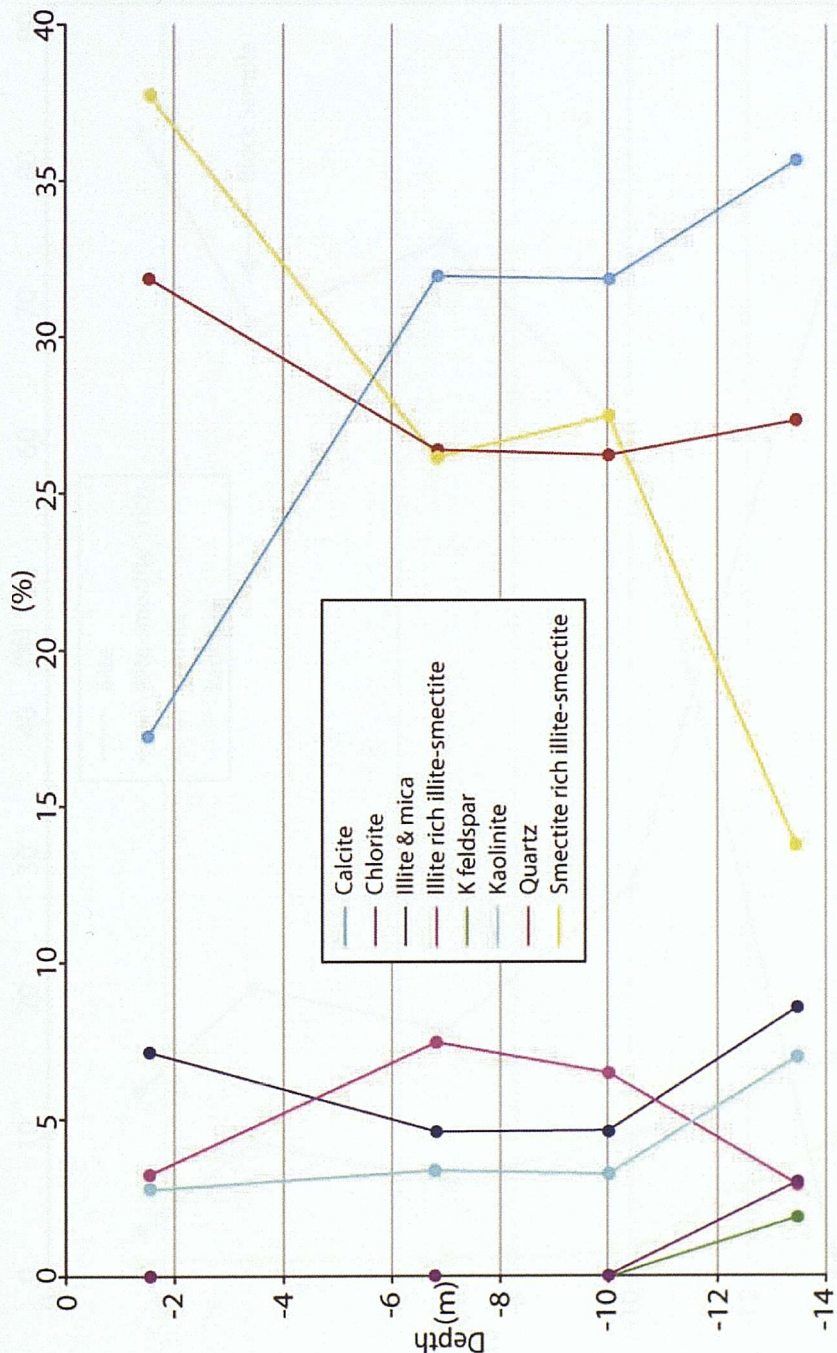


Figure 6.31 Profile of the bulk mineralogy of the Gault Clay with depth, omitting results from block samples. The shallow sample reflects weathering in the shallow layer (1.55m), the mid depth samples (6.82 & 9.99m) have very similar, near identical mineralogical assemblage. The deepest sample (13.46m) is very different probably coming from a different mineralogical system. Geological summary provided for comparison.

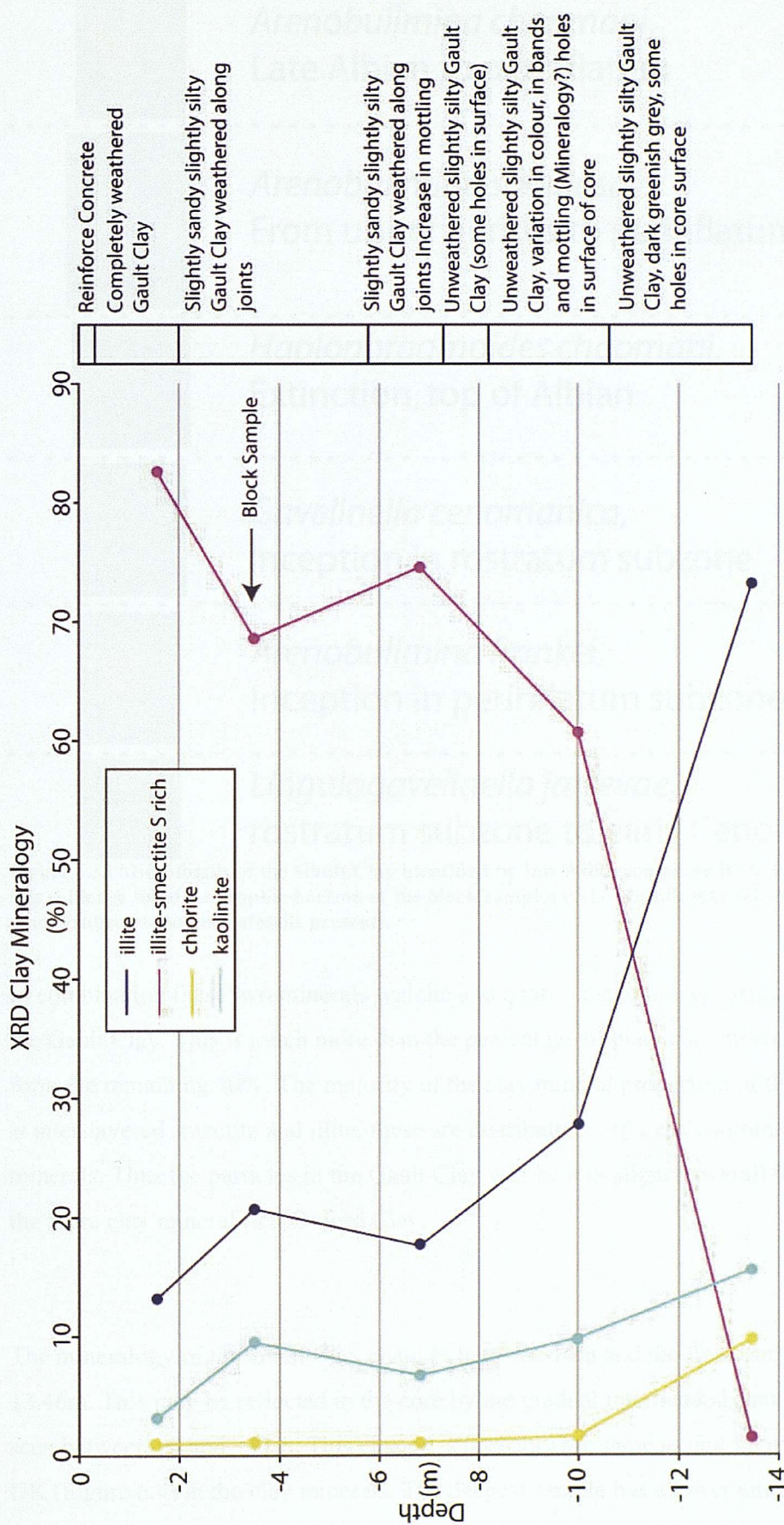


Figure 6.32 Profile of clay mineralogy of the Gault Clay with depth, the mineralogical changes enhance the general trends seen in the bulk mineralogy (Figure 6.32). A sudden change is seen in the clay mineralogy in the deepest sample.



Figure 6.33 Microfossils of the Gault Clay identified by Ian Wilkinson of the BGS. On the basis of this evidence the stratigraphic horizon of the block samples is the perinflatum subzone to the no macro subzone (no macrofossils present).

In combination these two minerals (calcite and quartz) make up approximately 60% of the Gault Clay. This is much more than the percentage of platy clay minerals which form the remaining 40%. The majority of the clay mineral proportion of the Gault Clay is inter-layered smectite and illite, these are distributed ~50% each within the interlayer minerals. Thus the particles in the Gault Clay will be less aligned overall than those of the more clay mineral rich Oxford Clay.

The mineralogy of the Gault Clay changes between 10m and the final sample at 13.46m. This may be reflected in the core by the gradual interbedded change in colour seen between ~8 and ~11m. This change is probably the same as that seen across the UK (Figure 6.4) in the clay minerals. The deepest sample has a lower amount of smectite and a higher amount of kaolin and non inter-layered illite (Figure 6.32). There

is also a small increase in the relative amounts of quartz and calcite, which will probably decrease the relative alignment of particles.

Microfossils

The stratigraphic horizon of the block samples could not be identified by macrofossils, as none were present in the excavation. The upper part on the Gault Clay is known for its relative scarcity of fossils. The stratigraphic horizon was determined based on microfossils using the standard procedure (Appendix B). The block sampling horizon is within the perinflatum subzone to the no macro subzone (Figure 6.33).

6.4 CPT investigation

Five CPT profiles were created on the site across two locations, three profiles in the same location as the block samples and two reference profiles away from the wooded area (Figure 6.13). As there is a lateral difference between the locations of the CPT sites of ~150m it is expected that there will be a vertical difference in stratigraphy. Profiles which are close together are similar in broad terms however the detail does not match especially at depth. The only major and distinctive change across the site is that at 6m in the reference profiles and 7m in the block sampling profiles. This is seen as a near doubling of sleeve friction (f_s) and a nominal increase in cone end resistance (q_c). An increase in the ratio of f_s to q_c is normally related to a fining of the materials grain size. This boundary is difficult to pick out in the boreholes. Two things occur around this horizon, first a grain size change and second a weathering change. The disappearance selenite grains from the joint planes occur at 7.35m in BHHC1 and 6.26m in BHHC2. Enhanced discolouration of the core ends at 7.35m in BHHC1 and at 7.50m in BHHC2.

One major difference is seen between the CPT profiles for both sites. This is the elevated level of both f_s and q_c in the near surface level at the block sampling site due to desiccation. This effect has produced an increase in q_c of ~2MPa and an increase in f_s of ~100kPa. This has a major influence on the engineering properties of the block samples taken from 3.5m in this location.

Some of the geological changes seen in the boreholes are not represented in the CPT profile. None of the nodules or nodule bands has any major visible effect on the profile as they are not coherent or continuous enough. The colour changes from ~7-10m seen in the core (interpreted as a clay mineral change) cannot be correlated with any major

changes in the CPT profile, although minor variations are visible. This may be because the material properties are governed by other factors, such as joints. Or it may be that a change in the fine grained material's mineralogy, which does not affect the overall grain size of the mudrock, does not have an impact on the material properties measured during a cone penetration test.

6.5 Comparison sites

Barrington Quarry was visited to describe the large scale exposures of the Upper Gault Clay, Cambridge Greensand and Lower Chalk. The quarry is located 8km to the south of High Cross (TL 3935, 5114) and was visited during September 2008.

6.5.1 Barrington Quarry

The main material excavated at Barrington is Chalk. The base of the pit goes through the Cambridge Greensand and into the Upper Gault Clay (Figure 6.35). Barrington is owned by Cemex UK. The Imperial College team were kindly escorted around the site by John Drayton. The main aim of the visit was to characterise the change in nodules between the Lower Chalk and the Gault Clay.

The nodules observed at Barrington have already been described (Section 6.1), a small fault was also observed in the base of the quarry suggesting some local tectonic loading in the past in this area (Figure 6.36). The aim was to return to the site to carry out a joint survey, unfortunately due to the closure and abandonment of the pit no further work was possible.

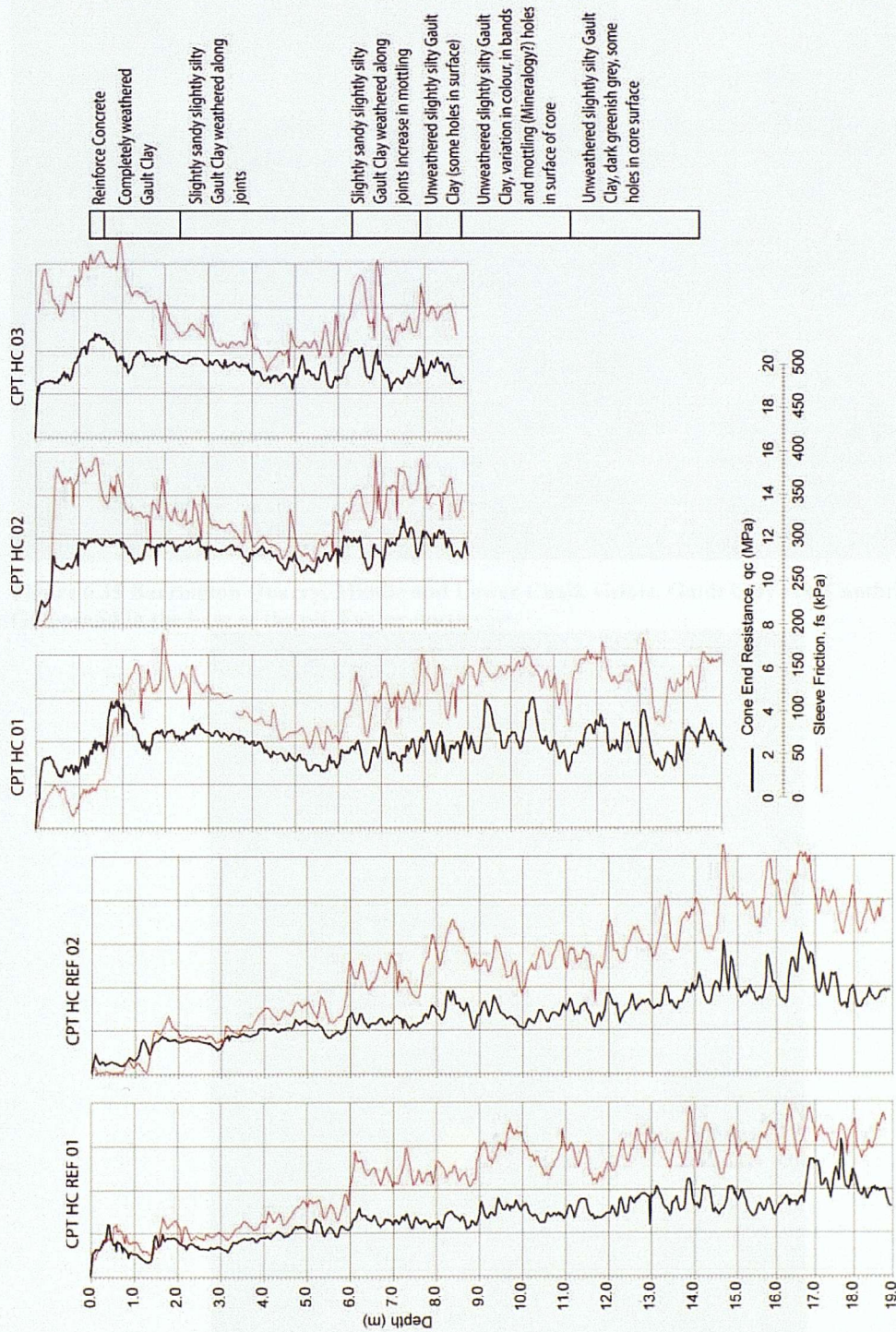


Figure 6.34 CPT results from High Cross locations shown on map (Figure 6.13) profiles from the reference site and the block sampling site have been aligned based on the sudden change in sleeve friction. The geological profile has been levelled with the CPT reference site, they may be up to 0.5 m out of place. Note the elevated signals at the block sampling site due to desiccation.



Figure 6.35 Barrington Quarry, Middle and Lower Chalk visible, Gault Clay and Cambridge Greensand in the base of the pit. Facing north east.



Figure 6.36 Fault crossing the Cambridge Greensand, crossing the boundary between the Lower Chalk and the Gault Clay. Notebook size: 0.15m

6.6 Overall material behaviour

The main factors influencing the Gault Clay are: pre-existing joints, nodules and high quantities of smectite. The pre-existing joints appear to be closed and filled with clay minerals and selenite crystals in the upper layers. In the lower layers some of the joints were either open, or were opened during drilling. The re-examination of the core suggests that water seeping into the joints during drilling has caused some swelling of the surrounding material. Discontinuities (including joints) are areas of weakness, their close spacing in the block samples is likely to play a major role in the sample behaviour. They are rarer at depth in the borehole samples, however based on the holes observed in the cores (intersection of joints) there will probably be at least one joint present in each sample. Nodules which are harder than the surrounding material cause stress concentrations to form, reducing the maximum strength the material can sustain. The nodules occur at regular intervals in the core, they are only present in a few of the samples. The amount of smectite throughout the core is greater than in all of the other mudrocks studied. Although it is more prevalent in the upper portions of the core, it is still present in the deeper levels (Figure 6.31). Smectite is able to take large quantities of water into its structure. Thus mudrocks containing large quantities of smectite tend to exhibit swelling behaviour.

The Gault Clay is the youngest and most shallowly buried mudrock (except for the London Clay). It has a smectite rich mineral assemblage, which is relatively weaker than the quartz-illite assemblage of the Oxford Clay. It is expected to exhibit one of the lowest strengths of the mudrocks studied. The relatively large amount of quartz and calcite compared to the amounts of clay minerals will probably result in a reduction in preferred particle orientation, which is likely to limit its anisotropy.

CHAPTER 7

THE KIMMERIDGE CLAY AT WILLOWBROOK FARM

The Kimmeridge Clay is present from Dorset all the way across the UK to Yorkshire. It is subsumed by the Cretaceous overstep both in Dorset and part of the midlands. Its thickness varies greatly from ~450m in Dorset to ~50m on the East Midlands Shelf to ~150m in Yorkshire (Brenchley and Rawson, 2006). The samples for this project were taken from a green field site near Steventon in Oxfordshire. The Kimmeridge Clay is known to be laminated in the south of the UK. It becomes increasingly silty towards the London Platform.

7.1 *Geological setting*

The literature for the Kimmeridge Clay can be divided by subject into the following categories: stratigraphy, depositional environment, mineralogy/chemistry, engineering behaviour and petroleum (Table 7-1)

Stratigraphy

In the Wessex Basin on the Dorset Coast the Kimmeridge Clay has a stratigraphic thickness of 450m. It is composed of several small scale mudstone-limestone rhythms. In the earliest work the Kimmeridge Clay was not subdivided (Fitton, 1836). The early division of the Kimmeridge Clay into two units (the Upper and Lower Kimmeridge Clay) was a result of the division of the Jurassic into stages. The Kimmeridgian Stage was created (d'Orbigny, 1842-1849), this stage was defined in continental Europe, and thus it did not coincide with the formation defined in the UK. The Lower Kimmeridge Clay was the part assigned to the Kimmeridgian Stage and the Upper Kimmeridge Clay assigned to the Bolonian Stage. The difference between these units being the ammonite used to define the zone. Physically the material making up the Kimmeridge Clay is the same in the upper and lower divisions. It is not surprising therefore that when calls came from the continent to further subdivide the Lower Kimmeridge Clay, forming a middle division, these calls were rebutted (Blake, 1875).

Subject	References
Stratigraphy	Early work (Blake, 1875; d'Orbigny, 1842-1849; Fitton, 1836) Ammonite Stratigraphy (Cope, 1967, 1978) Bed numbers across UK (Cox and Gallois, 1981; Gallois, 1994, 2000; Gallois and Cox, 1976) Correlation across UK (Cope et al., 1980a) Wireline correlations (Ahmadi, 1997) General comments (Chambers, no date; Wright and Cox, 2001) Thickness across UK basins (Sellwood and Sladen, 1981) Kimmeridge Clay on the London Platform (Cox et al., 1990) Discrepancies between systems (Cope, 1995) Sequence stratigraphy southern UK (Taylor et al., 2001) Silt content as a stratigraphic indicator (Williams et al., 2001) Combination of stratigraphical methods (Cope, 2009)
Depositional Environment	High energy environment (Wignall, 1989) Modelling sediment accumulation (Wignall, 1991) Facies-environment relationship (Macquaker and Gawthorpe, 1993) High resolution correlation to annual cycles (Chambers et al., 2000) Volcanic origin (Jeans et al., 2000) Environmental marker bands (Pearson et al., 2004) Mapping rapid environmental changes Swanworth Quarry (Gallois, 2000; Morgans-Bell et al., 2001; Weedon et al., 2004) Formation of mudrocks by flocculation (Macquaker and Bohacs, 2007; Schieber et al., 2007)
Mineralogy & chemical analysis	Geochemistry of organic matter (Tribovillard et al., 1994) Overview of clay mineralogy across the UK (Jeans, 2006b) Water column geochemistry (Piper and Calvert, 2009)
Engineering Behaviour	Undrained shear tests (Petley, 1999) Compaction and microstructure (Nygård et al., 2004) Compaction (Woodman, 2005)
Petroleum geosciences	Within the North Sea (Baird, 1986) (Cooper et al., 1995) Petroleum maturation, burial depth estimation (Scotchman, 1987, 1989, 1991a, b, 1994) Prospectivity of onshore basins (Swann and Gray, 2007)

Table 7-1 Summary of the literature on the Kimmeridge Clay, this table is not a complete list of all the available literature; it only included the sources consulted by the author.

Source	Liquid Limit (%)	Clay Fraction (%)	Plasticity Index (%)	Φ' peak (weathered)	Φ' peak (unweathered)	Φ' residual
(Cripps and Taylor, 1981)	70-81	57	24-59	14-23°	-	10-18°

Source	Moisture Content %	Plasticity Index (%)	Bulk Density Mg/m³	Dry Density Mg/m³	Shear Strength Su (kPa)
(Reeves et al., 2006)	18-29	16-59	1.95-2.14	1.46-1.70	70-500

Table 7-2 Example engineering properties for the Kimmeridge Clay.

More recently stratigraphers have assigned bed numbers to the rhythms where they can be correlated across the UK (Cox and Gallois, 1981; Gallois, 1994, 2000; Gallois and Cox, 1976), sixty three have been identified (KC1-KC63). Unfortunately these beds (rhythms) do not extend onto the Midlands Platform. The London Platform was a landmass during Kimmeridge Clay deposition, the sequence is condensed with proximity to the London Platform (Figure 7.1) and its silt content increases. The rhythms defined in Dorset and Eastern England are no longer identifiable (Cox et al., 1990). Most foraminifera species cannot be used to identify the mudrock's precise stratigraphic horizon for material on the London Platform (Section 7.3). It may be possible to get a more precise date by identifying fossil spores/pollen within the sample. However due to time and cost implications this method was not attempted. In the absence of stratigraphic data the samples are defined by location and depth.

Depositional environment

The environment of deposition of the Kimmeridge Clay is of great interest to the petroleum industry because the environment of deposition is the main cause of the rich oil and gas contents of the mudrock. The geological processes which formed the Kimmeridge Clay are thought to be high energy (Wignall, 1989). The high energy mechanisms for mudrock formation involves the combination of clay particles to form larger grain size colloids allowing deposition in floccule ripples (Macquaker and Bohacs, 2007; Schieber et al., 2007). Others have suggested that annual cycles can be identified through electron microscope studies of the Kimmeridge Clay (Chambers et al., 2000). Variations can be seen with depth, however in electron microscope images the variations observed are rarely laterally continuous, and are unlikely to relate to annual changes. Volcanogenic material is present in the upper silty part of the Kimmeridge Clay and is probably present at the sampling site (Jeans et al., 2000).

Mineralogy and chemical analysis

Very few studies of the bulk mineralogy of the Kimmeridge Clay have been conducted. The available bulk mineralogy measurements show a range of measurements (Nygård et al., 2004). This may be due to the variable nature of the Kimmeridge stratigraphy (Gallois, 2000). The layers included in the mudrock are limestone bands, which are high in calcium carbonate, calcareous mudstones, which are high in calcium carbonate and clay minerals, siltstones, which are normally high in quartz, and bituminous mudstones which are high in organics. In contrast the clay mineralogy of the Kimmeridge Clay is

relatively well known. The lower part of the formation is normally dominated by illite with ~20-30% (illite is called mica in the paper (Jeans, 2006b), petrologists often group illite with the micas. The other micas are expected to form a minor percentage of the overall material) kaolin and ~20-30% poorly defined collapsible minerals (Jeans, 2006b). The very top of the formation contains assemblages rich in illite (mica) and smectite with minor (or absent) quantities of kaolin. The geochemistry of the Kimmeridge Clay is studied with the aim of reconstructing the material's depositional environment (Piper and Calvert, 2009; Tribovillard et al., 1994). These analyses can be used to identify environments into which petroleum rich material are deposited (discussed below).

Engineering behaviour

The land area of the Kimmeridge Clay outcrop is smaller than that of the other mudrocks as it is overstepped (buried) by Cretaceous materials. There are few cities founded on the mudrock so it is unsurprising that there is little geotechnical information available (Table 7-2). One major geotechnical investigation has taken place; this is for the Upper Thames Reservoir (which may not go ahead). The ground investigation data for this project are not currently publically available. When they become available they will be a useful set of data for comparison. Until this time a few test points are available from others (Nygård et al., 2004; Petley, 1999; Woodman, 2005). The samples were taken from the south western end of the Midlands Platform, on the boundaries of the London Platform. This location is close to the proposed site for the Upper Thames Reservoir. The Kimmeridge Clay here is a shallow water facies up on the flanks of the London Platform part of which was above sea level. Shallow facies materials contain a coarser grain size of material; they may have more intense bioturbation and shallow water sedimentary structures depending on their exact depth.

Petroleum geosciences

A large number of papers have been published concerning the Kimmeridge Clay as it is the onshore equivalent to the North Sea oil source rock. Many of these papers are published in conference proceedings. The main factor of interest in these papers is the material history which is required for the extraction of oil to be possible. One of the main factors of interest is the depth to which the material has been buried (Scotchman, 1987, 1989, 1991a, b, 1994), which has been discussed elsewhere (Section 3.4).

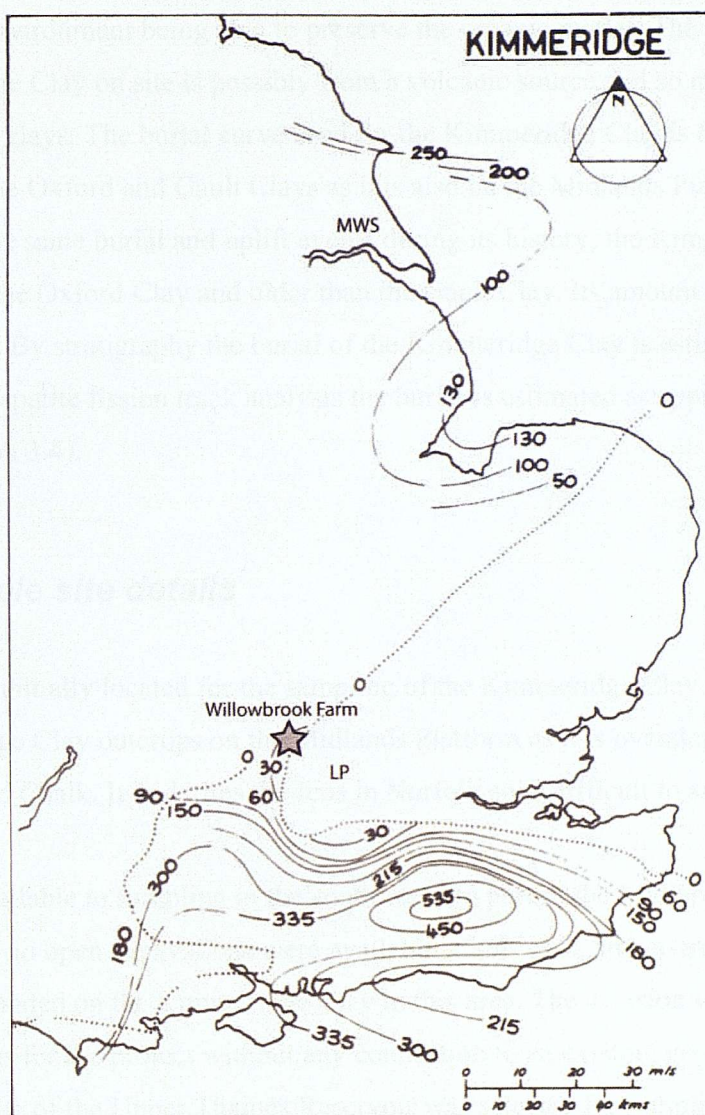


Figure 7.1 Isopachs for the Kimmeridge Clay (m) from (Sellwood and Sladen, 1981). Showing the London Platform (LP) the Market Weighton Structure (MWS) and the sampling site (Willowbrook Farm).

7.2 Reconstruction of material history

The available literature is of little use for the location from which the samples were taken, as it is a local facies. It was deposited on a relatively shallow slope on the boundary of the London Platform. It was surrounded by the deeper deposits which are classically described as the Kimmeridge Clay. It is a greatly condensed sequence compared to the surrounding material. This could either be produced by limited deposition, or more likely through the presence of erosive horizons within the sequence. High organic content is one of the major features of the Kimmeridge Clay. This is partially due to the prolific organic life in the oceans and partially due to the

sedimentary environment being able to preserve the organic matter. The upper part of the Kimmeridge Clay on site is possibly from a volcanic source and so may contain some swelling clays. The burial curve used for the Kimmeridge Clay is the same as the one used for the Oxford and Gault Clays as it is also on the Midlands Platform. It experienced the same burial and uplift events during its history; the Kimmeridge Clay is younger than the Oxford Clay and older than the Gault Clay. Its amount of burial is between them. By stratigraphy the burial of the Kimmeridge Clay is estimated as about 410m, and by apatite fission track analysis the burial is estimated as approximately 1080m (Section 3.4).

7.3 *Sample site details*

No sites were initially located for the sampling of the Kimmeridge Clay. Very little of the Kimmeridge Clay outcrops on the Midlands Platform as it is overstepped by the Gault Clay and Chalk. It underlies the fens in Norfolk so is difficult to sample here.

It becomes available to sampling in the south western part of the Midlands Platform. Unfortunately no open excavations were available in this area, and no major population centres are founded on the Kimmeridge Clay in this area. The decision was taken to collect samples for the project without any connection to an existing geotechnical project. The site of the Upper Thames Reservoir was selected for sampling due to the large number of boreholes (site data) present on the site.

The site is located in a field to the northern side of the road between Steventon and East Hanney (Figure 7.2). To gain access to this site, several applications were made to the Land Registry to identify owners of land in the area of Kimmeridge Clay. Letters were then sent to land owners asking if it would be acceptable for mudrock to be taken from their land. Neil Walker very kindly allowed samples to be taken from his fields (Figure 7.2).

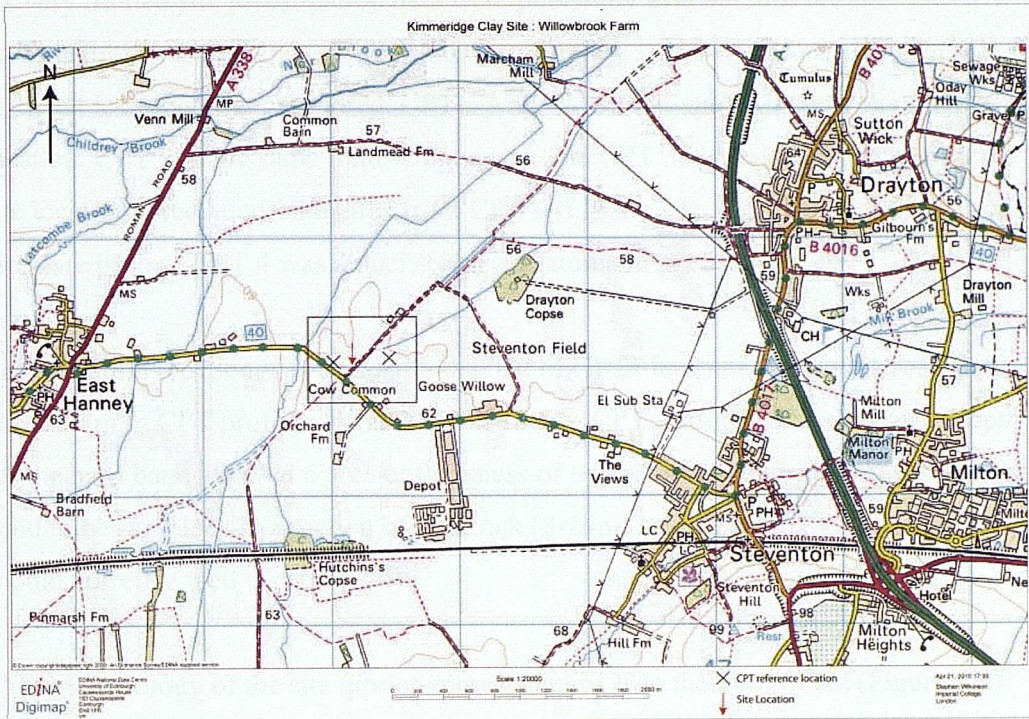


Figure 7.2 Site location for Kimmeridge Clay sampling: Willowbrook Farm (Ordnance Survey, 2009b). Square shows location of (Figure 7.3).

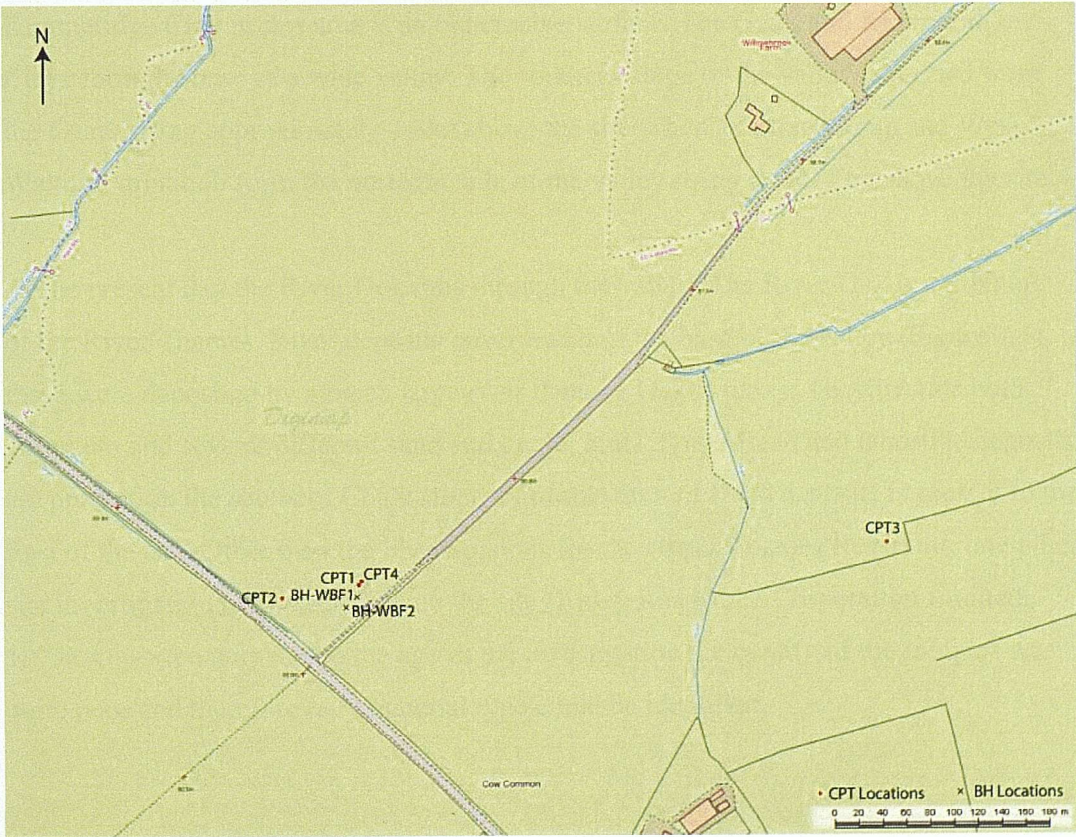


Figure 7.3 Locations of investigations undertaken during this project, CPT and boreholes (Ordnance Survey, 2010)

As very limited site investigation data were publically available for the site, the CPT investigation was undertaken to explore the sampling location options. Thus three different locations were explored. CPT's 1 and 4 were in approximately the same location, CPT 2 in the same field ~50m away, and CPT 3 was taken in the alternative site location three hundred metres from CPT 1 (Figure 7.3). CPT 1, 2 and 3 were soil moisture probes. CPT 4 was a dual seismic dilatometer profile.

The sampling location was decided to be that of CPT1 because there is a strong hard band at 8m in CPT profiles 1 and 2 and at 10m in CPT profile 3. The shallower depth for the hard band allowed a greater thickness of unweathered mudrock below the hard band to be sampled. The section of mudrock (8-14m) has similar CPT properties so should correlate well (Section 7.4).

The solid geology of the site dips at approximately 2° to the South-East (Figure 7.4). The boundary between the Lower Greensand/Gault Clay and the Kimmeridge Clay is an unconformity, which is part of the Cretaceous overstep, not completely covering the Kimmeridge Clay in this area. It can be inferred therefore that the top of the Kimmeridge Clay in this area is an old erosive surface. The Gault and Kimmeridge Clays form the base of a wide valley. The southern slope of the valley is formed from the Chalk rising approximately 140m above the site. The Corallian Group and West Walton Formation form the northern side of the valley rising to 30-40m above the site.

At the present day the River Ock runs through the valley. The River Ock is a tributary of the River Thames. River deposits cover much of the base of the valley (Figure 7.5), these were deposited by a much larger river than the Ock is today. They include both Alluvium and several different sand and gravel units. Some Head and landslide deposits are present on the southern Chalk slope. A further area of Head deposits is present to the East of the site. Other than the investigations for the Upper Thames Reservoir, one other site investigation has taken place on the site (Amey Roadstone Corporation Limited, 1973). Unfortunately due to the age of the investigation the quality of the samples was quite poor and therefore only material type could be identified.

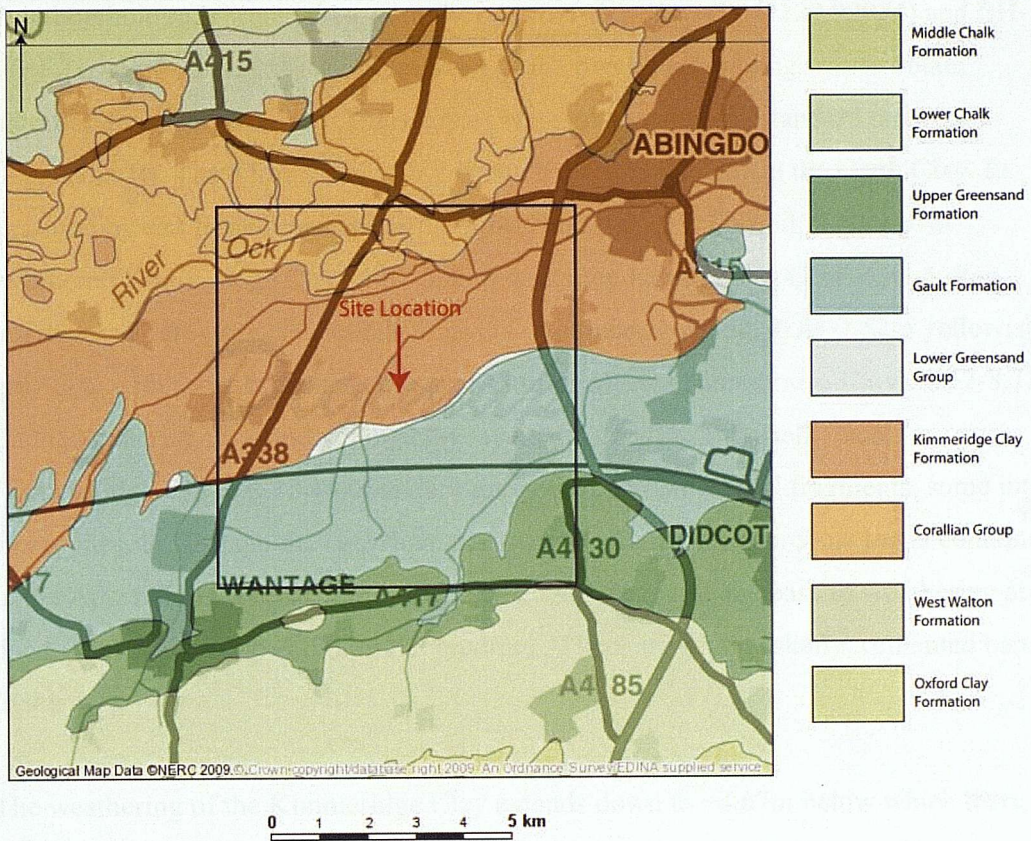


Figure 7.4 Solid geology around Willowbrook Farm (British Geological Survey, 2009c). Box indicates location of Figure 7.5.

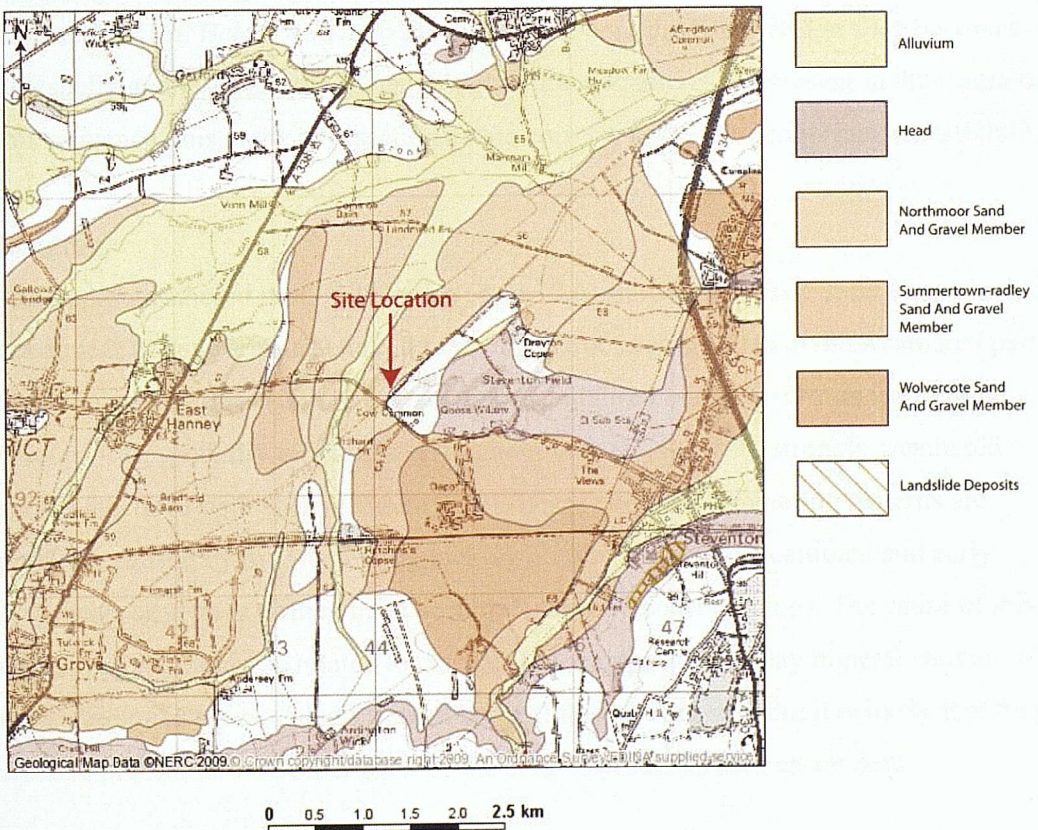


Figure 7.5 Drift cover around Willowbrook Farm (British Geological Survey, 2009f)

Two borehole runs were taken from the site: BH-WBF1 (SU 441129 92974) and BH-WBF2 (SU 44120 92958). The main aim of the borehole sampling was to obtain mudrock from 8-14m depth. The samples were taken using the standard method (Appendix B). The Kimmeridge Clay is much more variable than the Gault Clay. Its stratigraphy can be summarised as the following: 0-0.10m Topsoil, 0.10-4.67m weathered Kimmeridge Clay, 4.67-5.32m dark grey Kimmeridge Clay, 5.32-6.46m interlayered dark grey clay and shell layers (Kimmeridge Clay), 6.46-7.52m yellowish grey clay with some broken shells some forming layers (Kimmeridge Clay), 7.52-8.71m Banded material, grey and yellow clay layers mixed with hard shelly bands (Kimmeridge Clay), 8.71-14.06m dark grey clay containing shell fragments, some intact ammonites, larger than the core diameter from 11-12.7m. The borehole log is contained in the Appendix B. The features observed in the core below the base of weathering are due to the original deposition of the mudrock. There are several shelly, cemented bands in the core.

The weathering of the Kimmeridge Clay extends down to ~4.67m below which there is a thin layer of unweathered mudrock to 5.32m. Below this horizon the mudrock starts to contain shelly horizons, the main hard layers are between 5.32 and 6.46m and between 7.52 and 8.71m. Below 8.71m to the base of the core the Kimmeridge Clay becomes less shelly and less variable. Even though a few ammonites are present in the centre of this sequence, this is the best section of the core for testing due to its lack of variability.

Logging

The upper weathered part of the core (Figure 7.6) shows patches of yellowing which do not appear to have any structure. Several odd structures are seen in the weathered part of the core: wiggly lines (Figure 7.7), compartmentalised sections surrounded by sand (Figure 7.8), weak discolouration throughout core (Figure 7.9), strongly weathered spots associated with shells (Figure 7.10). These complex weathering patterns are probably caused by the weathering picking out pre-existing depositional and early diagenesis structures (which have been deformed by consolidation). The cause of this colour variation may be related to changes in the depositional clay mineral content. If these structures are revealed by weathering at this level in the core it is likely that they are also present in the deeper parts of the core where no structures are seen.

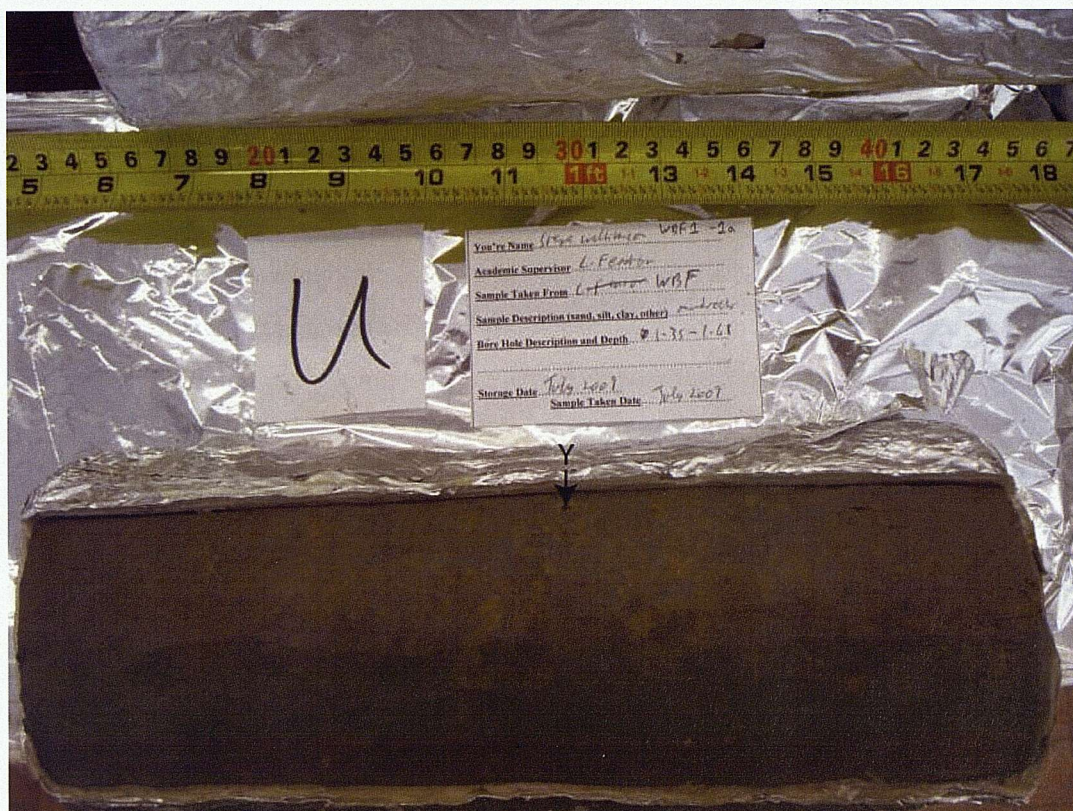


Figure 7.6 Kimmeridge Clay, core WB1 1.35-1.65m yellow mottling on the core (e.g., Y), no specific structure. U = unknown orientation, tape measure showing cm (top) inches (bottom).

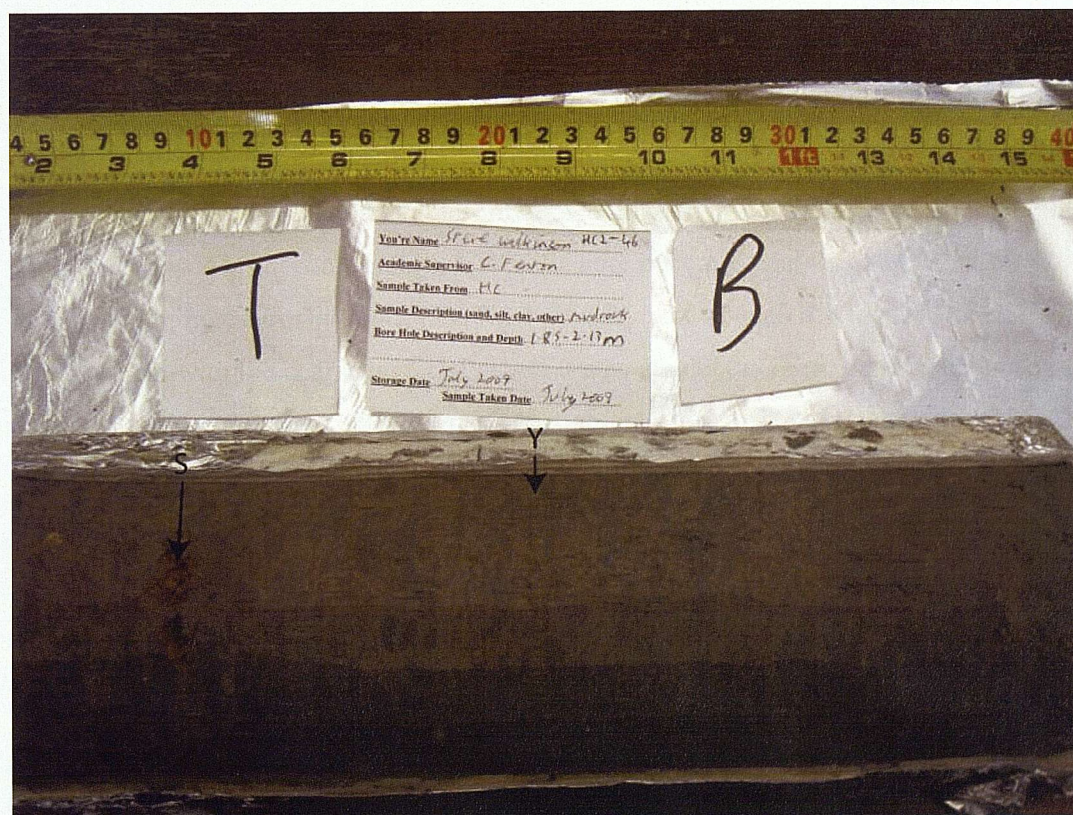


Figure 7.7 Kimmeridge Clay, core WB2 1.85-2.13m yellow mottling forming wiggly lines on core (e.g., Y), one sandy inclusion (S) near top of sample mottling decreases towards base of sample. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

The shallowest core is weathered such that all pre-existing structure has been removed (Figure 7.6). The wiggly lines (Figure 7.7) are likely to be weathering features. The horizontal lines of sand may be deposited during the normal sedimentary sequence. Vertical lines of sand are normally indicative of later alteration, crystal growth within joints for example; however these lines are probably depositional and do not cross the preserved burrow in the core (Figure 7.8). These complex structures indicate a high energy, depositional environment with varying depositional mechanisms, probably affected by reworking due to low sediment supply or limited accommodation space. Below this horizon the extent of weathering lessens. Core discolouration greatly reduces (Figure 7.9); shells are broken up forming shelly regions of the core. Other areas are devoid of shells (Figure 7.9). The lowest evidence of chemical weathering in the core profile is yellow discolouration of the core contained within shell fragments, or around inclusions (Figure 7.10). The lowest of these occurs at about 4.20m, which has been selected as the base of weathering. The variations in the weathering profile highlights that there are several weathering processes occurring, each process gains dominance and diminishes at different depths.

Below the weathered horizon the mudrock is fine grained, containing lenses of darker material (Figure 7.11). Very little shelly material is present in this part of the core. The lenses are possibly formed by floccules ripple migration (Schieber et al., 2007), or they may be the result of soft sediment deformation acting on minor density variations in the initially deposited mudrock. Below 5.18m the core becomes coarser and the broken shell content increases (Figure 7.12). The boundary is inter-leafed wedges of coarse and fine material with the coarse material dominating with depth. These coarse sections may indicate shallowing of the depositional environment or high energy events (debris flows, and turbidity currents).

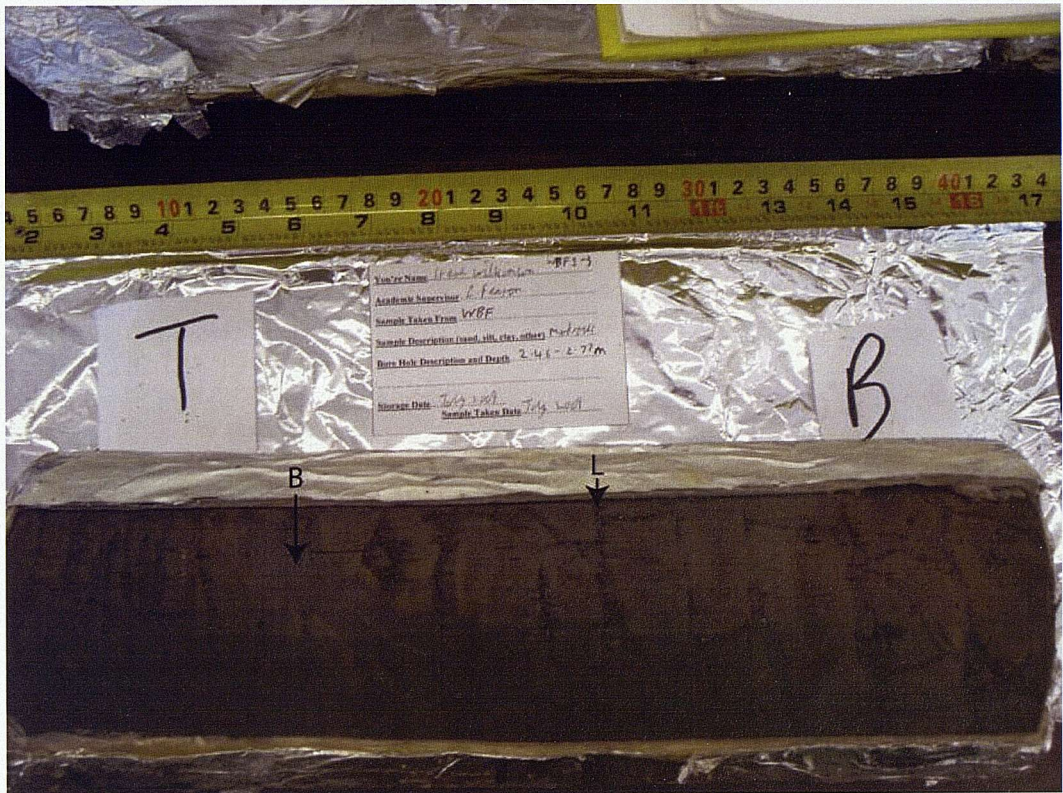


Figure 7.8 Kimmeridge Clay, core WBF1, 2.46-2.77m mostly yellow in colour containing lines of sand (e.g., L) many sub horizontal, some vertical, grey areas surround sand. Sand lines become more spaced out with depth. Probable preserved burrow (B) in core (grey area). T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

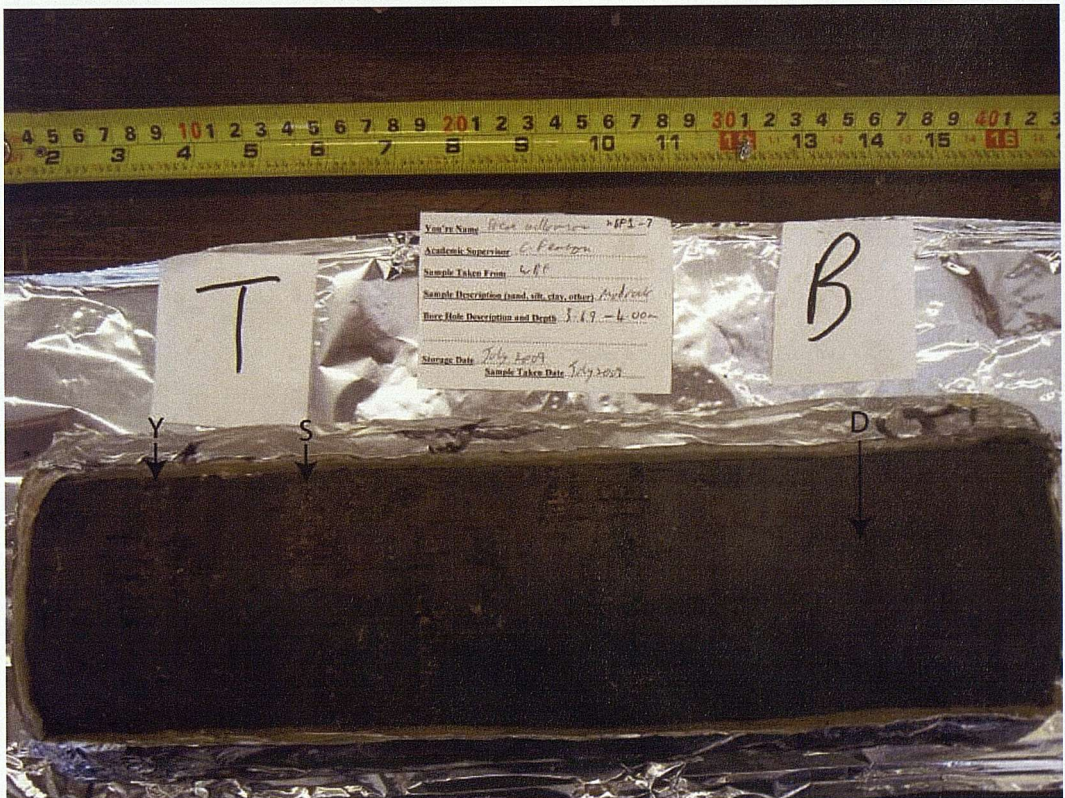


Figure 7.9 Kimmeridge Clay, core WBF1, 3.69-4.00m some weak yellow mottling (Y), broken shell fragments (e.g., S) visible in the top of the core becoming rarer with depth, some darker lenses (D). T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

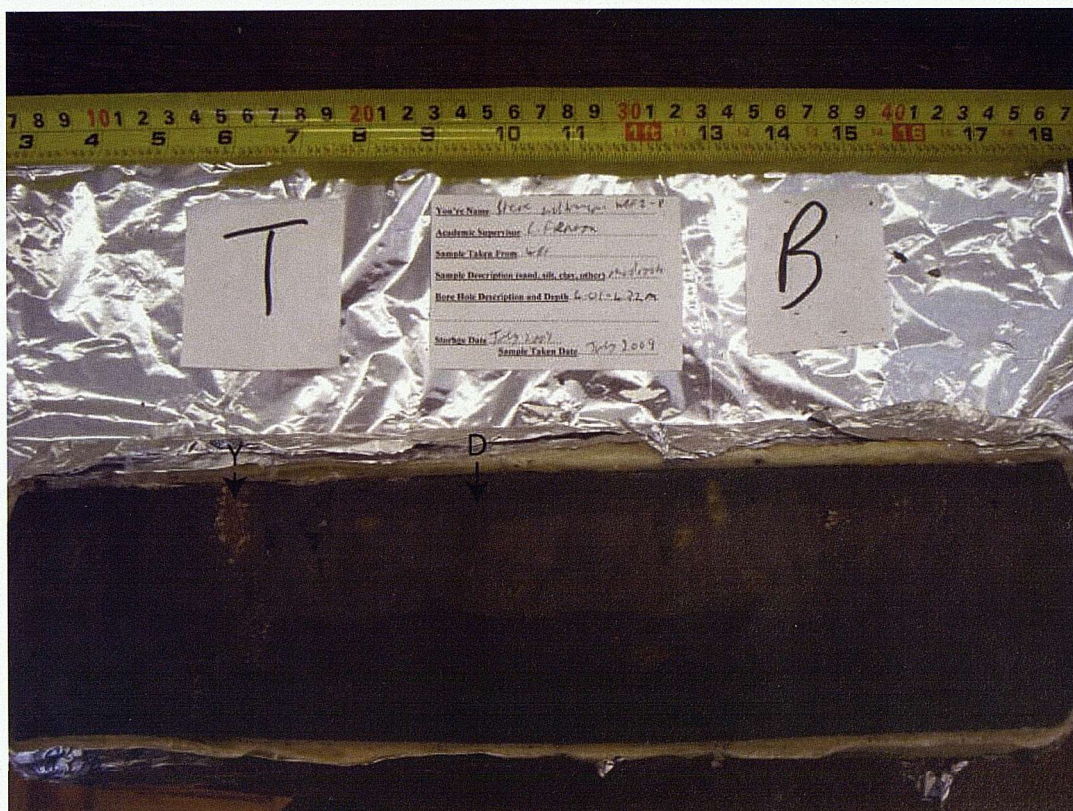


Figure 7.10 Kimmeridge Clay, core WBF1, 4.01-4.32m yellow discolouration associated with shell fragments (Y), some darker (D) lenses on core. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

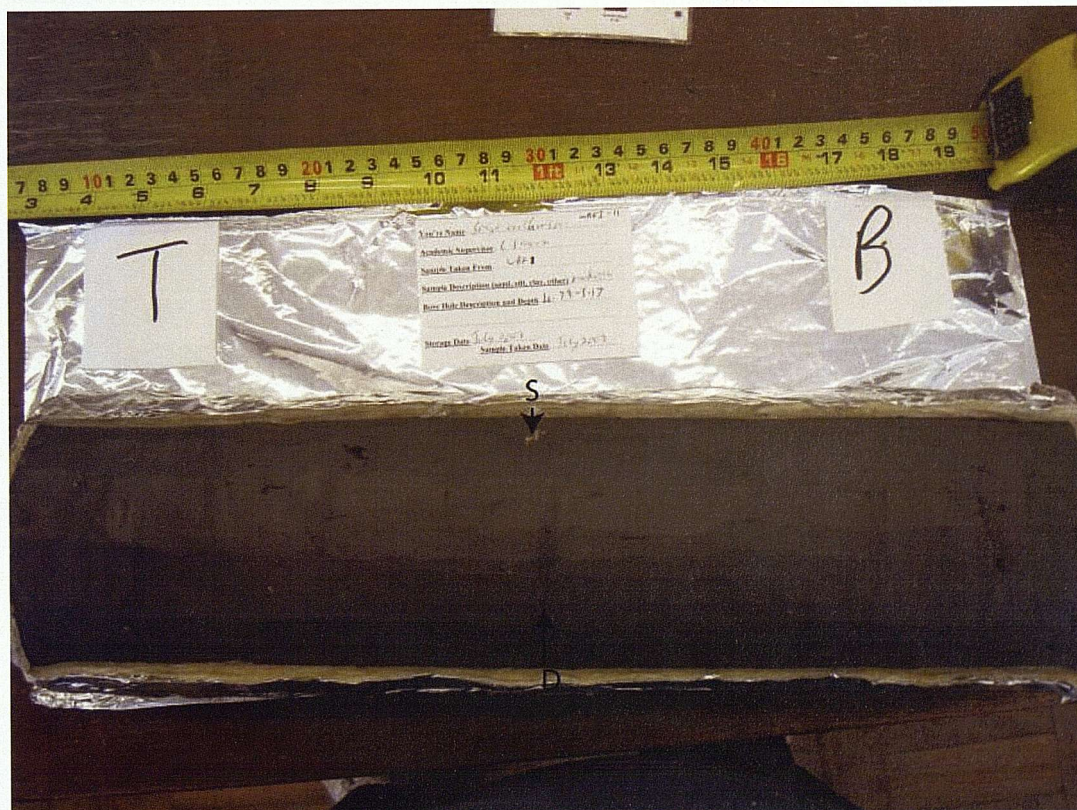


Figure 7.11 Kimmeridge Clay, core WBF1, 4.79-5.17m very few shell fragments (S), some dark shaded lenticular patches (D). T = top of core B = base of core, tape measure showing cm (top) inches (bottom).



Figure 7.12 Kimmeridge Clay, core WBF1, 5.18-5.51m mudrock becomes coarser grained, and contains some shell fragments. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

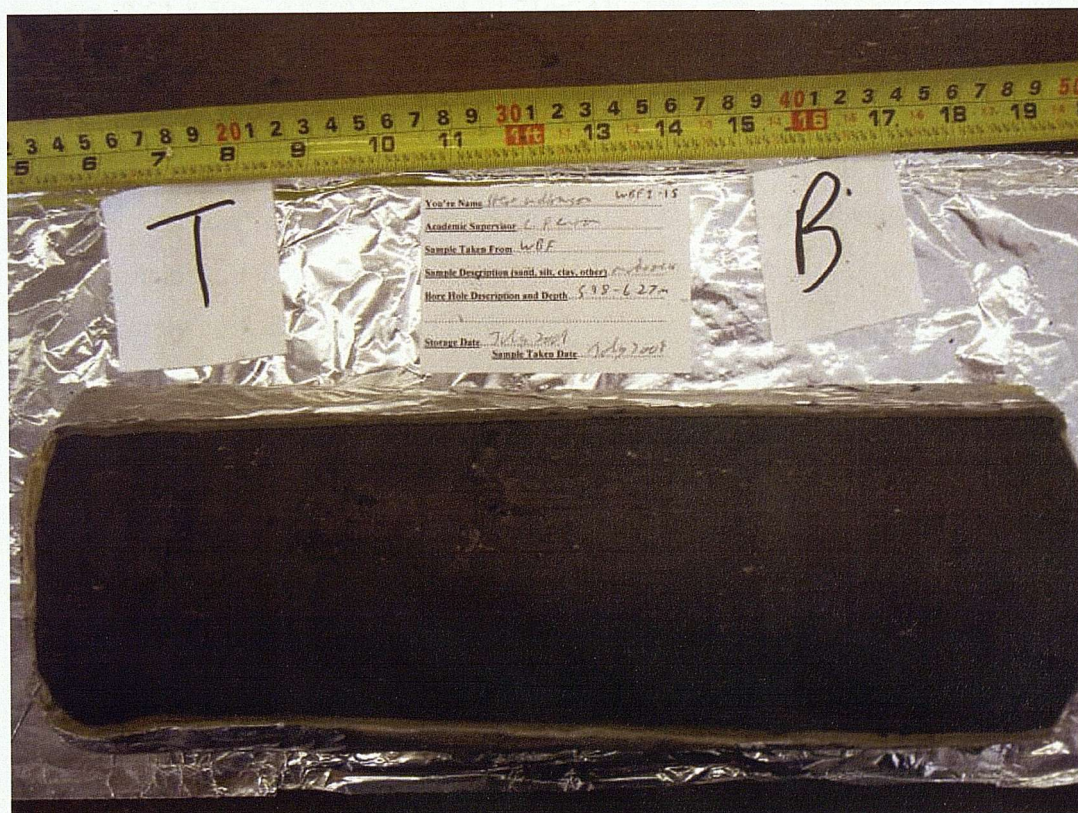


Figure 7.13 Kimmeridge Clay, core WBF1, 5.98-6.27m mudrock becomes fine grained again. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

At 6.12m the core becomes fine again indicating the return to the earlier environment (Figure 7.13), although several small shells are present. A few small coarser horizons are present between 6.12 and 8.00m (*e.g.*, Figure 7.14) each of these is approximately 100mm in length, probably representing the same sort of event as between 5.18 and 6.12m. Between 8.00 and 8.11m there is a sand horizon, this sand horizon is loosely cemented with calcite (Figure 7.15). The sand was possibly the original depositional material with the calcite being deposited due to its high hydraulic conductivity. Below the cemented layer the mudrock becomes fine again for the remainder of the core (Figure 7.16). The main details of this portion of the core are sections of ammonite shells (Figure 7.17). There are also lenses of silty material, in some of these lenses coarse-fine variations form dipping layers some of which show cross-bedding style terminations. Below about 12.53m there is a small elevation sand and silt content. The laboratory testing horizon is from the base of the lowest hard band (8.11m) to the bottom of the core.

Mineralogy

As the boreholes cross stratigraphy several samples are required to characterise the mineralogy of the core. These samples were all taken from the laboratory testing horizon, 8.54-8.59m, 9.85-9.87m and 12.71m. All of the samples are very rich in quartz (Table 7-3); this is consistent with the material being derived from volcanic ash. Most of this quartz is microcrystalline. In the 12.71m sample there is a greater sand content than there is in the other horizons. The majority of the coarse material (greater than 160µm) in the core is lithic fragments (Figure 7.18). These fragments are quartz rich which explains the 79% quartz content for this fraction. The upper two samples have approximately 25% clay minerals (Table 7-3), of which the majority is illite with lesser amounts of smectite and very little kaolinite. The deepest sample contains 14% clay minerals (Table 7-3). This sample is slightly richer in smectite and kaolinite, although it is still dominated by illite. The relatively low amount of clay minerals within the Kimmeridge Clay has a great affect on its microstructure, as a lack of platy particles does not allow the measurement of the appropriate particle long axis orientations.

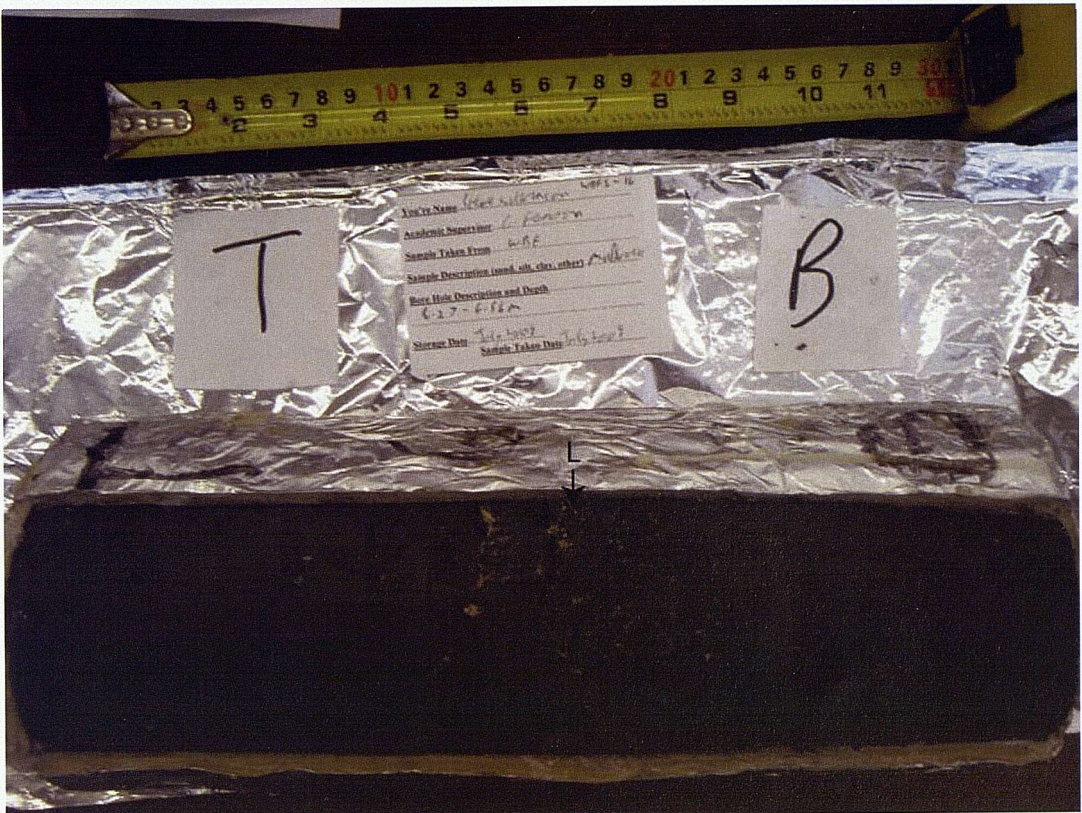


Figure 7.14 Kimmeridge Clay, core WBF1, 6.27-6.56m some thin coarser grained, broken shell rich layers (L). T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

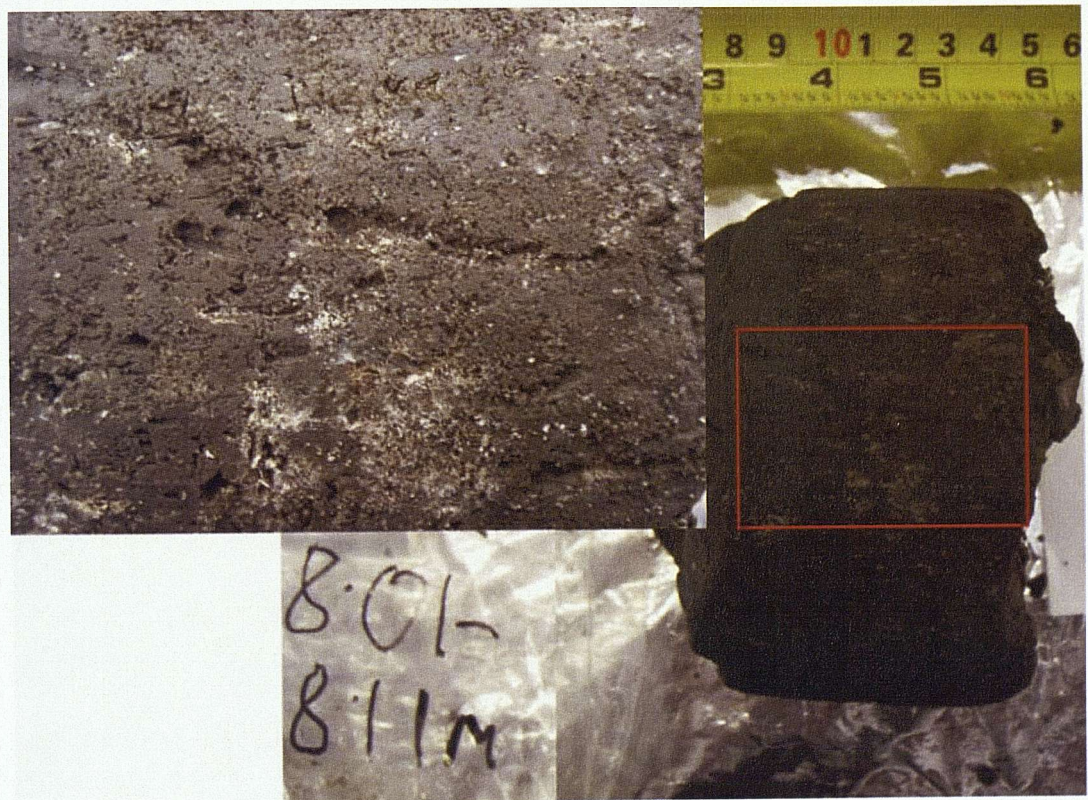


Figure 7.15 Kimmeridge Clay, core WBF1, 8.01-8.11m, hard band of carbonate rich partially cemented sand size grains.

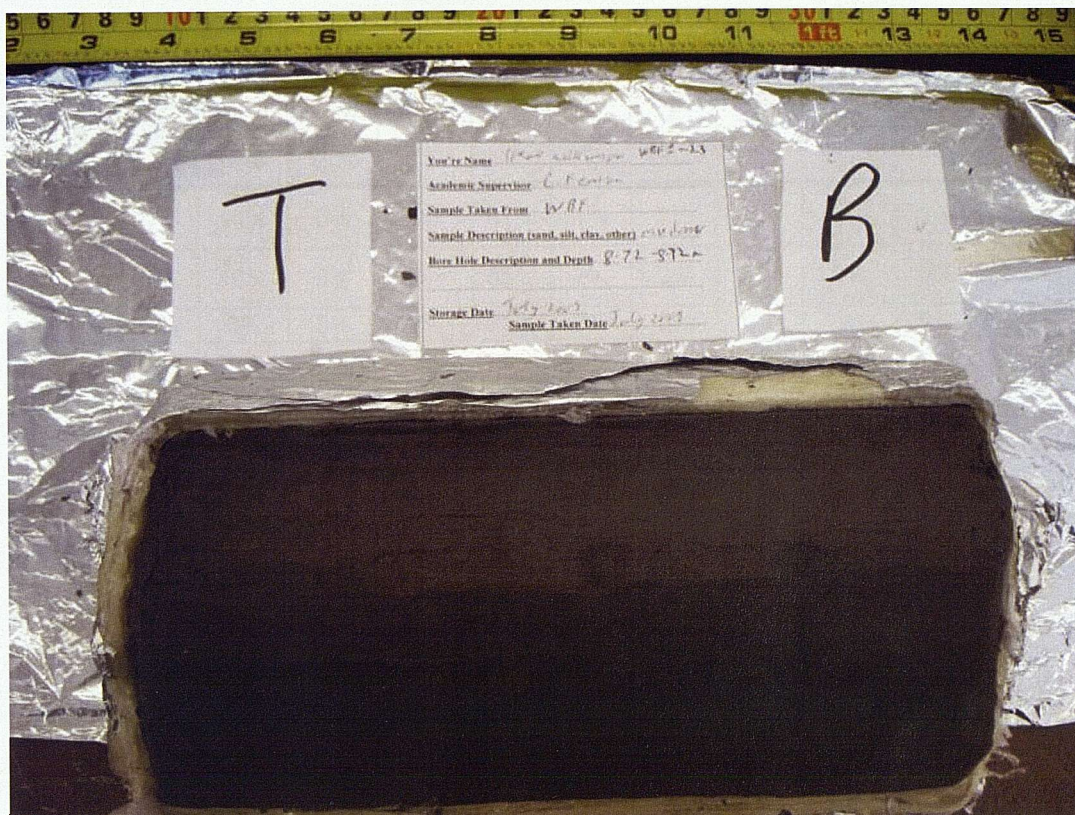


Figure 7.16 Kimmeridge Clay, core WBF1, 8.72-8.92m core becomes fine grained, no indications of any structure. T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

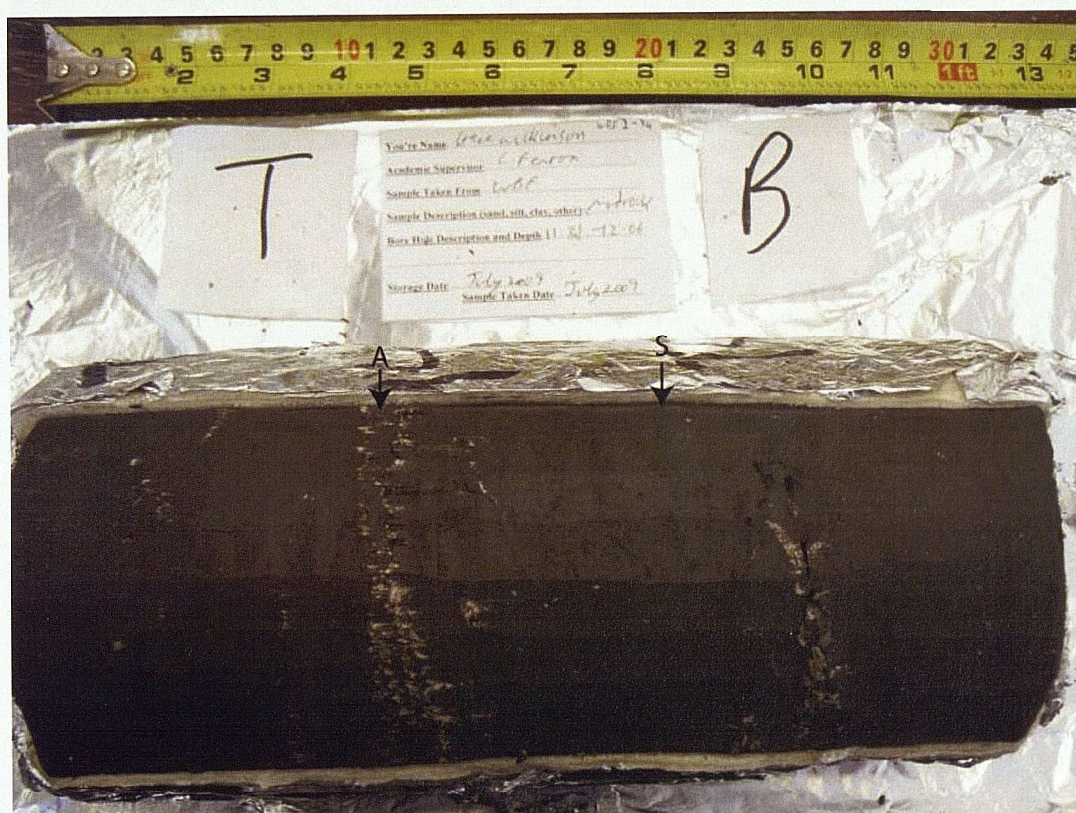


Figure 7.17 Kimmeridge Clay, core WBF2, 11.81-12.06m some ammonite shells (A) and some silty lenses (S). T = top of core B = base of core, tape measure showing cm (top) inches (bottom).

Kimmeridge Clay (depth)	illite & mica	illite-rich illite-smectite	smectite-rich illite-smectite	kaolinite	quartz	K feldspar	chlorite	plagioclase	Calcite	siderite	pyrite
8.54-8.59m	9%	14%	0%	1%	61%	1%	1%	tr	9%	1%	2%
9.85-9.87m	7%	16%	0%	2%	65%	2%	2%	1%	0%	0%	4%
12.71m	4%	0%	8%	2%	79%	3%	0%	0%	tr	0%	4%

Kimmeridge Clay (depth)	illite	illite-smectite I-rich	illite-smectite S-rich	chlorite	Kaolinite
8.54-8.59m	74%	11%	0	10%	5%
9.85-9.87m	47%	34%	0	5%	15%
12.71m	55%	0%	16%	11%	17%

Table 7-3 Top table bulk mineralogy of the Kimmeridge Clay, bottom table, percentage of minerals contained within the clay grade (grain size) part of the Kimmeridge Clay.



Figure 7.18 The greater than 160 micron fraction residue of the 12.71m sample, the residue is rich in polyminerale lithic fragments. There are a few broken shell fragments, and very few quartz grains.

Microfossils

The Kimmeridge Clay is one of the few mudrocks where the microfossils which are present are normally all long ranging. They are of little use in identifying the stratigraphic horizon. Very few fossils were found within the Kimmeridge Clay (Figure 7.19), none of them were of particular note. This lack of microfossil variation produces a problem for stratigraphic variation. From the very rare foraminifera in the sample (12.30-12.33m) Dr Ian Wilkinson identified *Epistomina parastelligera*, *Lenticulina subalata* and *Vaginulina prima*. Dr Wilkinson suggests that the best age which fits the distribution of these fossils is Mutabilis (or possibly Eudoxus) Zone (Wilkinson, 2011). This equates to the middle of the Lower Kimmeridge Clay. Due to the condensed and unusual nature of this section a grid reference and depth are provided for future correlations.

7.4 CPT investigation

The purpose of this CPT investigation was for sampling site selection from the three sites which were available (Section 7.3). For this reason the CPT data are spread across a 300m area. The events preserved in these sequences can be correlated very well across this local area (Figure 7.20). As the samples were taken from the site of CPT1 all of the depths are given in relation to this site. The top of the core has a low cone end resistance (qc), and low sleeve friction (fs). There is a small spike in both qc and fs at 2.5m, this is in the same location as the burrow in core WBF1, in core WBF2 there are some small pockets of coarse sand at this horizon. At approximately 3.7m there is an increase in fs with very little response in qc, caused by core fining. The highs in the CPT profile between 5 and 8m are hard bands. At 8 m there is a very high signal in both fs and qc caused by the cemented sand band. Below 8m the core is less variable maintaining similar fs and qc, there is a slight elevation in qc between 10.8 and 12m this is probably due to the silt lenses at this horizon. Ammonites in this section produce small elevation in both qc and fs. From 14.5m there is a large elevation in qc and fs probably produced by the next cemented band in the sequence



Figure 7.19 Microfossils from 12.31m sample of the Kimmeridge Clay. The image shows pyrite spheres (A), fish teeth (B), plant remains (C), possibly *Marginulina* (D), possibly *Citharina* (E), and *Citharina serratocostata* (F)

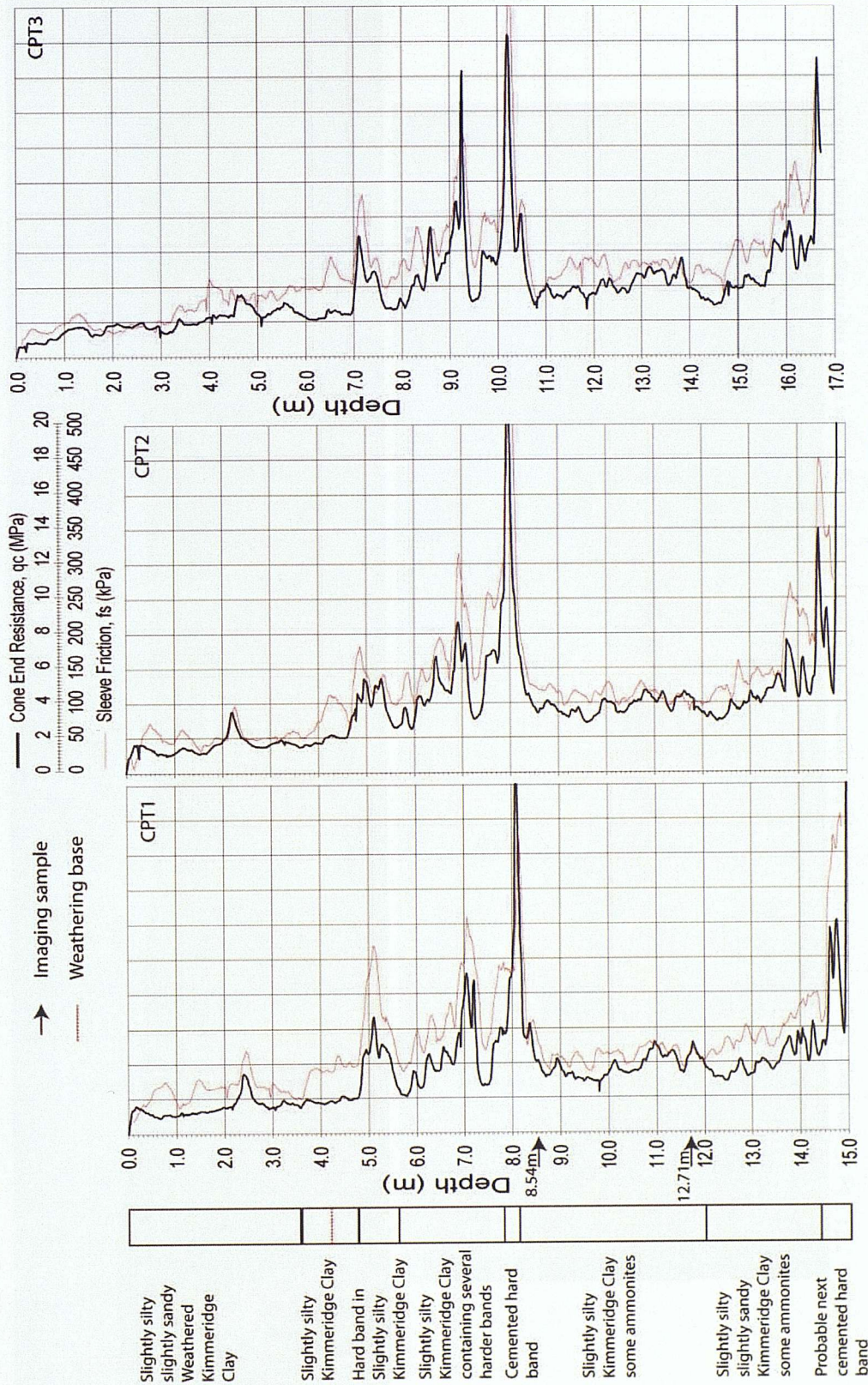


Figure 7.20 CPT results from Willowbrook Farm, locations shown on map (Figure 7.3) the distance between CPT1 and CPT2 is about ~50m, and CPT3 ~250m the events recorded in the core have been matched up, CPT3 is ~100m in the direction of dip, this stratigraphic change represents a dip of 1.15° assuming unstructured parallel beds.

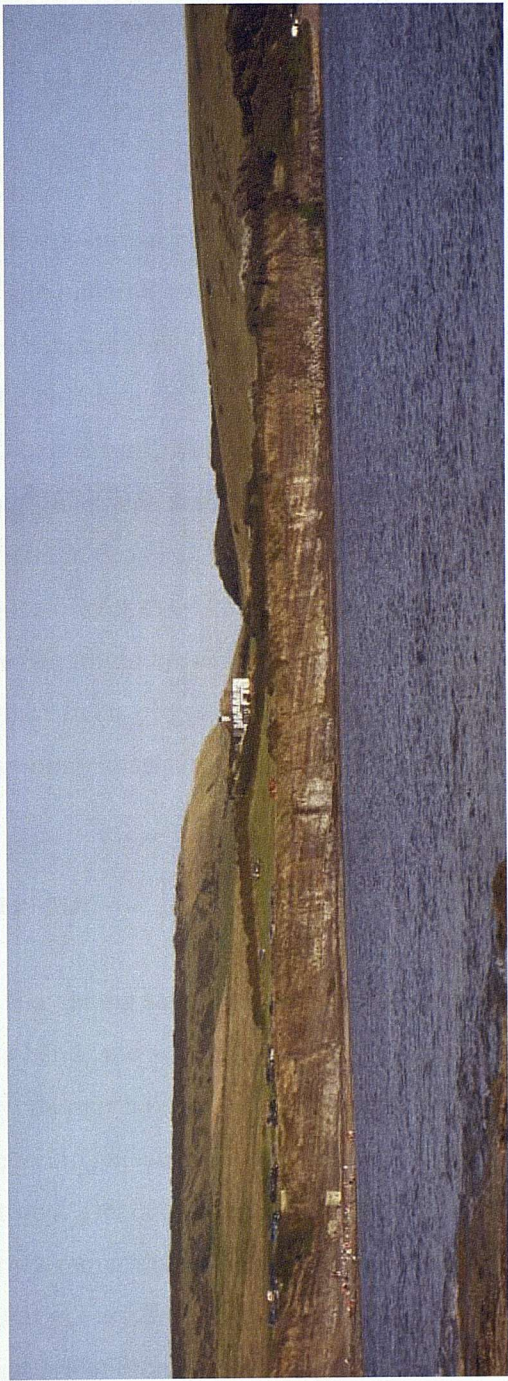
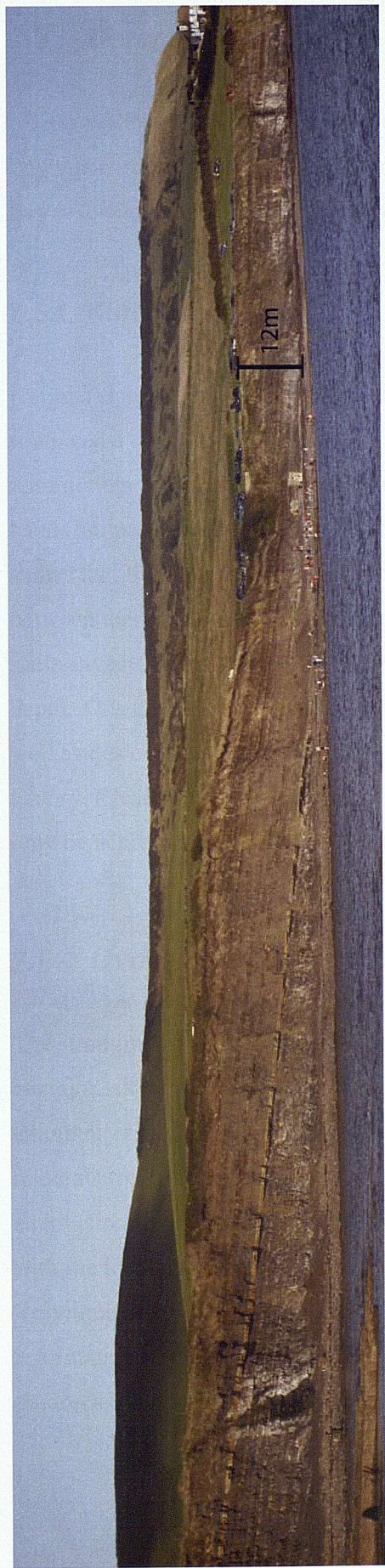


Figure 7.21 The Kimmeridge Clay at Kimmeridge Bay, bottom image continues from the right side of the top image. Facing North West.

7.5 Comparison sites

No excavations could be found in the Kimmeridge Clay near to the site. Kimmeridge Bay was visited to see how the Kimmeridge Clay changes between basins. Kimmeridge Bay is located on the south coast of the UK (SY9067, 7917)

7.5.1 Kimmeridge Bay

Kimmeridge Bay is a long term costal exposure of part of the Kimmeridge Clay on the south coast of the UK (Figure 7.21). The material exposed here is part of the Kimmeridge Clay, in this location the Kimmeridge Clay is 450m thick. The limestone bands are particularly prominent in the cliff face (Figure 7.21). A close-up of the cliff shows that the limestone bands are closely spaced with seams of mudrock running between them (Figure 7.22). The limestone bands are much harder than those at the sampling site because here the Kimmeridge Clay has been buried to a much greater depth. This deeper burial is also responsible for the fissile nature of the mudrock (as well as desiccation). These differences highlight the effects of a mudrock's geological history. Clearly an understanding of the effects of geological history on a mudrock can only be achieved through multiple sampling sites across a number of geological basins.

7.6 Overall material behaviour

The stratigraphy of the Kimmeridge Clay at the sampling site is very variable. It contains silty mudrock a cementstone band, and several sandy hard bands. Within the laboratory testing horizon (below 8m) there is a relatively low percentage of clay minerals (platy particles). This reduction in anisotropic minerals will probably produce a reduction in overall material anisotropy. The clay minerals are likely to be aligned with the lenses observed in the mudrock rather than being aligned to the horizontal. Very limited amounts of smectite result in very limited swelling behaviour. No information is available about the joint orientations on the site; their effects on the mudrock's behaviour are unknown.

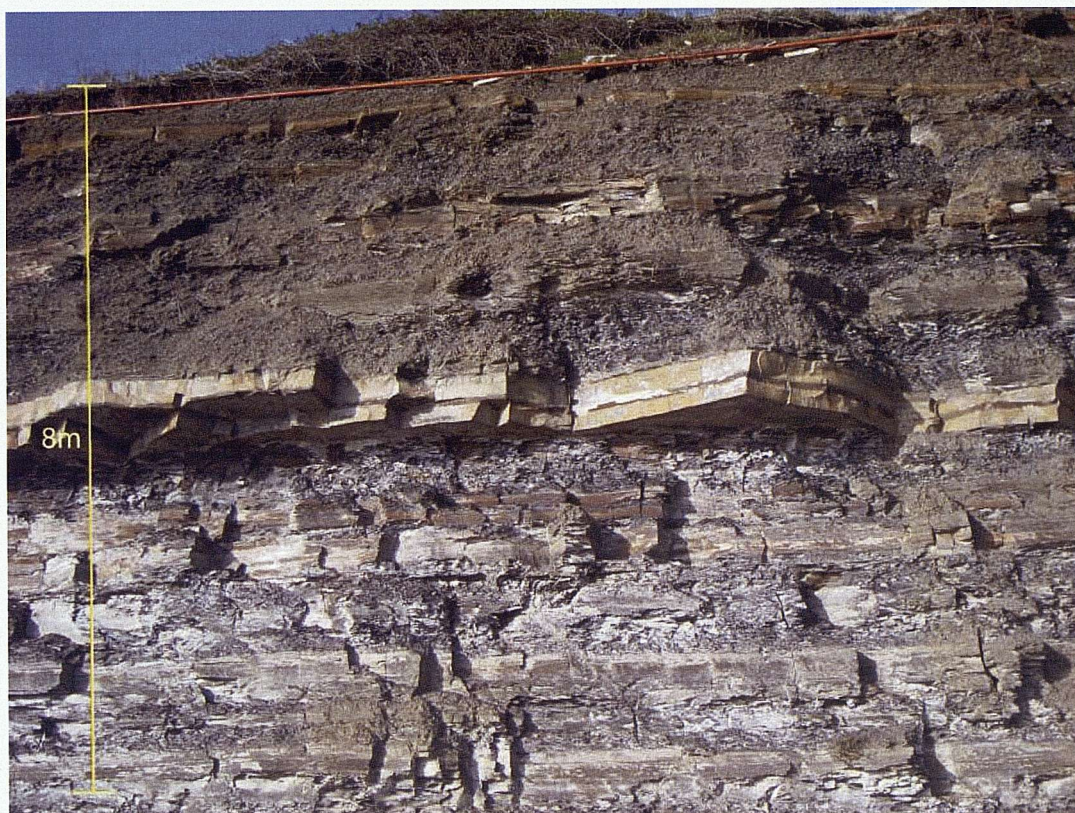


Figure 7.22 Close up image of the Kimmeridge Clay cliff face in Kimmeridge Bay, showing both hard limestone bands and fissile mudstone layers. Facing North West.

CHAPTER 8

THE LIAS CLAY AT M5 JUNCTION 13

The Lias Clay is a very different material to the other mudrocks in this thesis. All of the other mudrocks are defined as formations, the Lias is defined as a group. It is divided into twelve formations, of which five contain fine grained (mud grade) material (Figure 8.1). Each mudrock formation could be considered Lias Clay, and each one is different. The Blue Lias and Scunthorpe Mudstone Formations have about an equal amount of limestone and mudrock. The interbedding of limestone and mudrock makes the sampling of mudrock from this material impractical. The Redcar Mudstone Formation is confined to the Cleveland Basin, and is thus unsuitable for sampling due to the movements of the Market Weighton Structure (Section 3.3.2). The Whitby Mudstone Formation is thickest in the Cleveland Basin and thins southwards across the East Midlands Platform. The Charmouth Mudstone Formation is thickest in the Wessex Basin and thins northwards across the East Midlands Platform. Both of these formations are present in the preferred sampling area

The Middle Jurassic is formed mainly of limestone as this is a harder material than the surrounding mudrock. Thus it forms an area of elevated topography (the Cotswald Hills). The Whitby Mudstone was deposited directly before (and therefore under) the Middle Jurassic and so normally forms on slopes beneath this hard material. It has been greatly affected by glacial processes (Section 3.2.7), the structure of this mudrock is of interest in terms of glacial effects, however this complex history would make comparisons between mudrocks difficult. Thus the Charmouth Mudstone was selected as the ideal mudrock from which to take samples. As this material is mainly present in the south of the UK there is only a small area of outcrop of the Charmouth Mudstone on the Midlands Platform. Unfortunately no sampling options were available in this area. The only sampling opportunity which arose was in association with a site investigation at junction 13 of the M5. This is in the Worcester Basin (Section 3.3.2), which is not ideal as this basin experiences a slightly different burial history compared to the other mudrocks. However it gives the opportunity for studying some of the effects of deeper burial on mudrock structure.

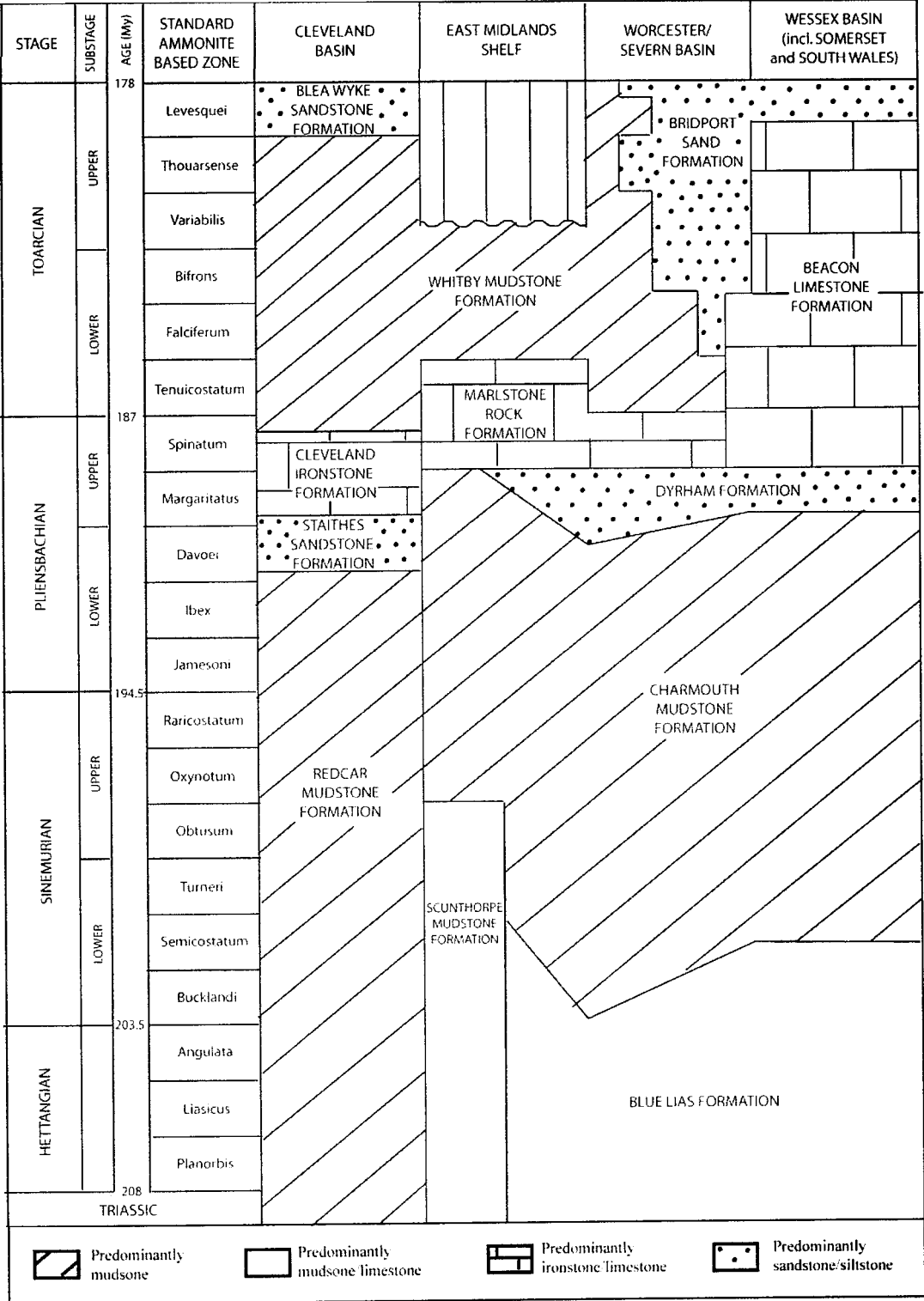


Figure 8.1 The twelve formations of the Lias Group, the Whitby Mudstone, Redcar Mudstone, the Charmouth Mudstone, the Scunthorpe Mudstone and the Blue Lias Formation, all contain mudrock, based on (Cox et al., 1999).

Subject	References
Stratigraphy	Early work (Arkell, 1933, 1947) Material classification (Morris, 1979) Correlation across the UK (Cope et al., 1980b) Thickness (Sellwood and Sladen, 1981) Microfossil stratigraphy (Copestake, 1989; Copestake and Johnson, 1989) Descriptions (Gaunt et al., 1992; Simms et al., 2004; Taylor, 1995) Subdivision based on gamma-ray character (Bessa and Hesselbo, 1997) Formal classification (Cox et al., 1999) Blue Lias (Ambrose, 2001) Tilton Railway Cutting (Wilkinson, 2001) Ammonite zones for Worcester Basin (Donovan et al., 2005) Warwickshire: Southam quarry (Radley, 2003, 2005, 2008)
Depositional Environment	Structure of depositional basin (Chadwick, 1985) Origin of rhythms: Milankovitch cycles (Smith, 1989)
Mineralogy & chemical analysis	Chemical origin of cementstone bands (Bottrell and Raiswell, 1989) Copperhill Quarry (Mitchell, 1992) M5 Gloucestershire (Kemp and Hards, 2000) SEM petrography (also qualitative micro-structure) (Bouch, 2003) Clay mineralogy across the UK (Jeans, 2006b) Spatial distribution of clay minerals (Dera et al., 2009)
Engineering Behaviour	Effect of weathering (Chandler, 1972) Whitby Mudstone long term stability (Chandler, 1974) Coastal landslide Charmouth (Denness et al., 1975) Pressuremeter testing (Stevenson et al., 1990) General properties (Bell, 1994) Dowdeswell Dam (Hutchinson and Coope, 2002) BGS shrink swell and formation review projects (Freeborough and Jones, 2003; Hobbs and Sumbler, 2001; Kemp and McKervery, 2001; Rowlands and Jones, 2002) Effects of sulphates and sulphides (Czerewko and Cripps, 2006) Empingham Dam (Kovacevic et al., 2007a) Use in landfill liners (Binns et al., 2008)
Burial history	Burial in Gloucester Basin (Kemp et al., 2005) Variation in organic matter: early diagenesis (Taylor, 1998)
Preferred particle orientation	Preferred particle orientation and imposed magnetic fabric (Hounslow, 1985)

Table 8-1 summary of the literature on the Lias Clay, this table is not a complete list of all the available literature; it only included the sources consulted by the author.

8.1 Geological setting

The literature on the Lias Clay can be divided into: stratigraphy, depositional environment, mineralogy/chemistry, engineering behaviour, burial history and preferred particle orientation (Table 8-1)

Stratigraphy

The division of the Lias Clay into twelve formations has already been discussed (Figure 8.1). Although some further informal divisions into members are available (Cox et al., 1999), these are only locally defined and not completely in the Worcester Basin. One of the key stratigraphic boundaries for the samples is that between the Charmouth

Mudstone and Blue Lias. This boundary is highly diachronous within the Worcester Basin (Donovan et al., 2005) making its exact location difficult to identify. The microfossil analysis of the lowest samples in the core shows them to be from the Lower Semicostatum Zone (Section 8.3). Both the Charmouth Mudstone and the Blue Lias contains material of this age within the Worcester Basin. The formal definition of the boundary is: the top of a greater than 0.1m limestone band above which the mudstone unit is at least twice as thick as the underlying limestone mudstone unit. Above this there is no return to closely spaced interbedded limestone mudstone sequences (Cox et al., 1999). In the borehole from which the samples were taken this boundary is located at 14.95m below ground level. This suggests that the samples come from both formally defined materials.

Depositional environment

The mechanism of formation of interbedded limestone mudstone units is currently an unresolved and much discussed problem (Potter et al., 2005). Some limestone bands are formed by direct deposition of calcite; others are formed by post-depositional diagenetic processes (Bottrell and Raiswell, 1989). Diagenetic processes act on the original deposited structure of the mudrock. Even if they are formed by diagenetic processes their position is defined by depositional processes. The rhythm of beds seen in the Blue Lias has been correlated with orbital processes (Smith, 1989).

The thickness of the Lower Lias varies greatly across the country (Figure 8.2). One of the thickest parts of the Lower Lias is within the Worcester Basin. The eastern edge of the basin (closest to the Vale of Moreton) has the greatest thickness of material. This thins towards the West (sampling location). The basin is a half graben bounded by the Vale of Moreton fault, its movement creates this asymmetric accommodation space within the basin

Mineralogy and chemical analysis

The Blue Lias varies between mudrock and limestone, the same as the Kimmeridge Clay. This produces a rapid stratigraphic bulk mineralogical change. The Charmouth Mudstone has fewer limestone bands and has a more consistent mineralogy. The general clay mineralogy of the Lower Jurassic (Lias) is illite rich (Mitchell, 1992), with minor amounts of kaolin and smectite. In a few locations (such as the M5) sub equal quantities of kaolinite and illite have been observed (Kemp and Hards, 2000)

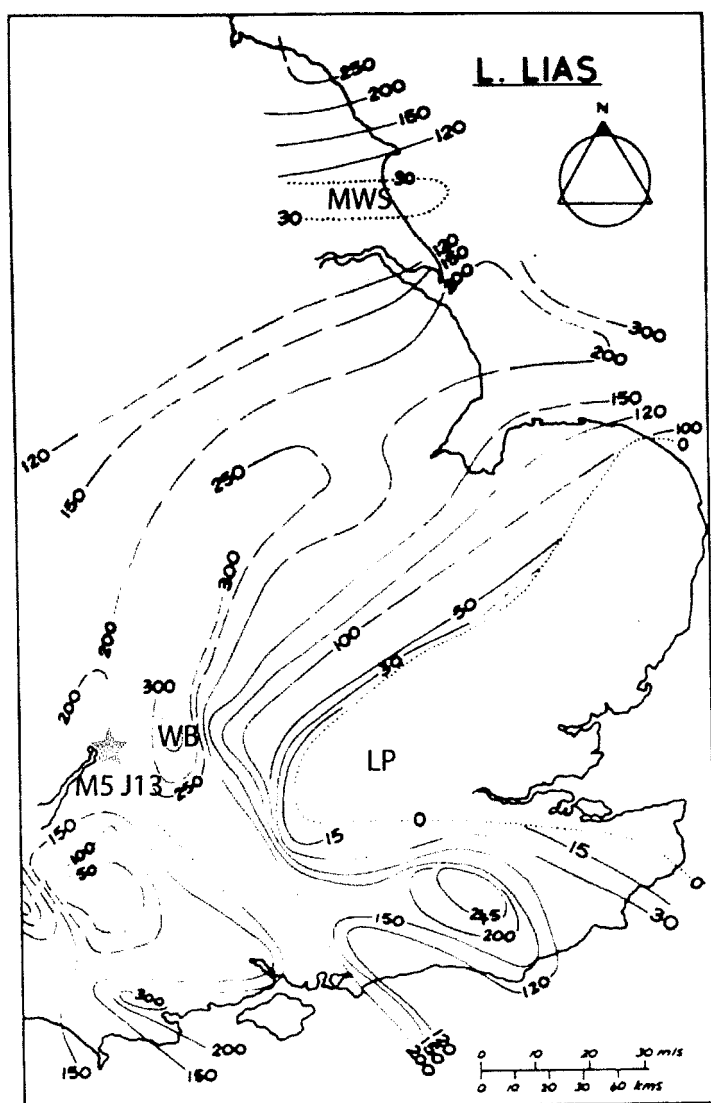


Figure 8.2 Isopachs for the Lower Lias Clay including the Blue Lias and the Charmouth Mudstone (m) from Sellwood and Sladen, (1981) Showing the London Platform (LP) the Market Weighton Structure (MWS), Worcester Basin (WB) and the sampling site (M5 J13).

(Chandler, 1974)	c' (kN/m ²)		Φ'	LL (%)	PL (%)	W (%)
Brecciated Upper Lias Clay	17		23°	68-61	31-27	35-20
(Cripps and Taylor, 1981)	Liquid Limit (%)	Clay Fraction (%)	Plasticity Index (%)	Φ' peak (weathered)	Φ' peak (unweathered)	Φ' residual
Upper Lias Clay	53-70	55-65	20-39	18-25	-	9-13.5
Lower Lias Clay	53-63	50-56	32-37	27	-	13-16
(Hobbs et al., 2005)	Moisture content (%)	Plasticity Index (%)	Clay Fraction (%)	Bulk Density Mg/m ³	Dry Density Mg/m ³	Shear Strength Su (kPa)
Blue Lias	19-26	24-34	37-50	1.94-2.08	1.59-1.70	88-250
Charmouth Mudstone	18-25	23-34	28-50	1.98-2.12	1.54-1.74	84-187

Table 8-2 Engineering properties of the Lias Clay (See Reeves et al., 2006).

Engineering behaviour

Several different Lias Clay horizons have been previously studied (Table 8-2). The Lias Clay is famous for cambering and valley bulge environments (Chandler, 1974; Hutchinson and Coope, 2002). The Whitby Mudstone at the top of the lower Jurassic is overlain by the hard limestones of the Middle Jurassic, thus due to the depositional sequence the Whitby Mudstone normally occurs on slopes. This made it particularly susceptible to glacial processes, the unfrozen material at depth flowing to produce a disturbed micro structure and macro features such as valley bulge and cambering. These features are especially well known at Empingham Dam (Kovacevic et al., 2007a). Valley bulge features are occasionally developed in the Charmouth Mudstone in deep valleys (Hutchinson and Coope, 2002). More usually the Charmouth Mudstone is distal from the slopes and so less disturbed. Recent work by the BGS has highlighted the minor amounts of smectite in the Lias mudrocks as potential causes of shrink-swell behaviour (Kemp and McKervey, 2001).

Burial history

The depth of burial of the Mercia Mudstone and the Lower Jurassic is studied well by multiple means to see the effect of early diagenesis on the maturation of organic matter. Measurement of burial of these rocks can be used to predict the burial of all younger materials (as they form the base of the Younger Cover). The change of mineralogy with burial (Kemp et al., 2005) and the reduction of iron (Taylor, 1998) are examples of previous depth of burial measurements used on this material.

Preferred particle orientation

Parts of the Blue Lias (the paper shales) have shown a similar level of preferred particle alignment to that of the Oxford Clay (Hounslow, 1985). The Blue Lias limestone bands further reinforce this trend in the macro environment. In units with high clay mineral content which have been buried as deeply as the Lias Clay has, strong preferred particle alignment is expected.

8.2 *Reconstruction of material history*

Sea level rises throughout the Jurassic mean many of the Mercia Mudstone deposits are shallow water and on land. The Lias Mudrocks are deposited in a seaway between many islands in the north west of the Tethys Ocean, in moderate depth water. The Lias transgresses onto the London Platform further than the Mercia Mudstone (Donovan et al., 1979). The movements of the Vale of Moreton Fault govern the thickness of material deposited in the Worcester Basin. Depositional variation between mudrock and limestone is probably formed due to changes in the earth's orbit (Smith, 1989). Limestone bands may be depositional or may be formed during early diagenesis.

By stratigraphy the depth of maximum burial of the Charmouth Mudstone can be calculated as ~800m. By apatite fission track analysis the depth of maximum burial is calculated as ~1700m. Because the sampling site is within the Worcester Basin a different burial curve is used for this site. The samples were buried steadily to a depth of ~560m from the time of initial deposition (~200Ma) to ~75 Ma, and then they were rapidly buried to a depth of ~1700m by 65Ma. The samples were uplifted again to ~560m by 50Ma, and then re-buried to ~1130m by 25Ma before being returned to the surface today (Section 3.4). Thus the Lias Clay was buried to its deepest point at the end of the deposition of the Chalk, earlier than all of the other mudrocks.

8.3 *Sample site details*

The site from which the Lias Mudrock came is junction 13 of the M5 (Figure 8.3). The site is located in the flat base of the valley on undifferentiated Blue Lias and Charmouth Mudstone (Figure 8.4). These two formations are differentiated in the south of the map. As this map is constructed from several separate maps, some variations in colour scheme are present, the Whitby Mudstone Formation and the Blue Lias/Charmouth Mudstone Formation Undifferentiated are represented by two colours (Figure 8.4). The rapid geological changes on the east of the map are due to the steep slope forming the edge of the valley. The river Severn, which formed the valley, is located to the west of the site. The much smaller river Frome, a tributary of the River Severn runs from east to west 500m to the south of the site. Alluvium associated with this river is present within 100m of the site (Figure 8.5). The base of the valley contains several drifts of sand and gravel deposited by the rivers.

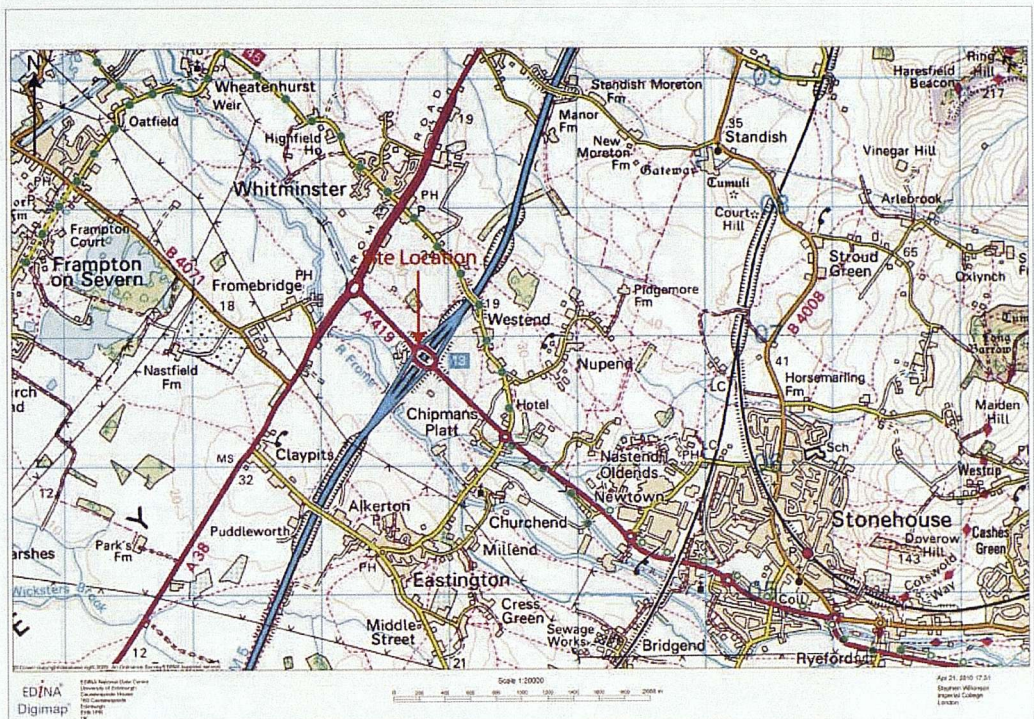


Figure 8.3 Sampling site location for Lias Clay, M5 Junction 13 (Ordnance Survey, 2009a).

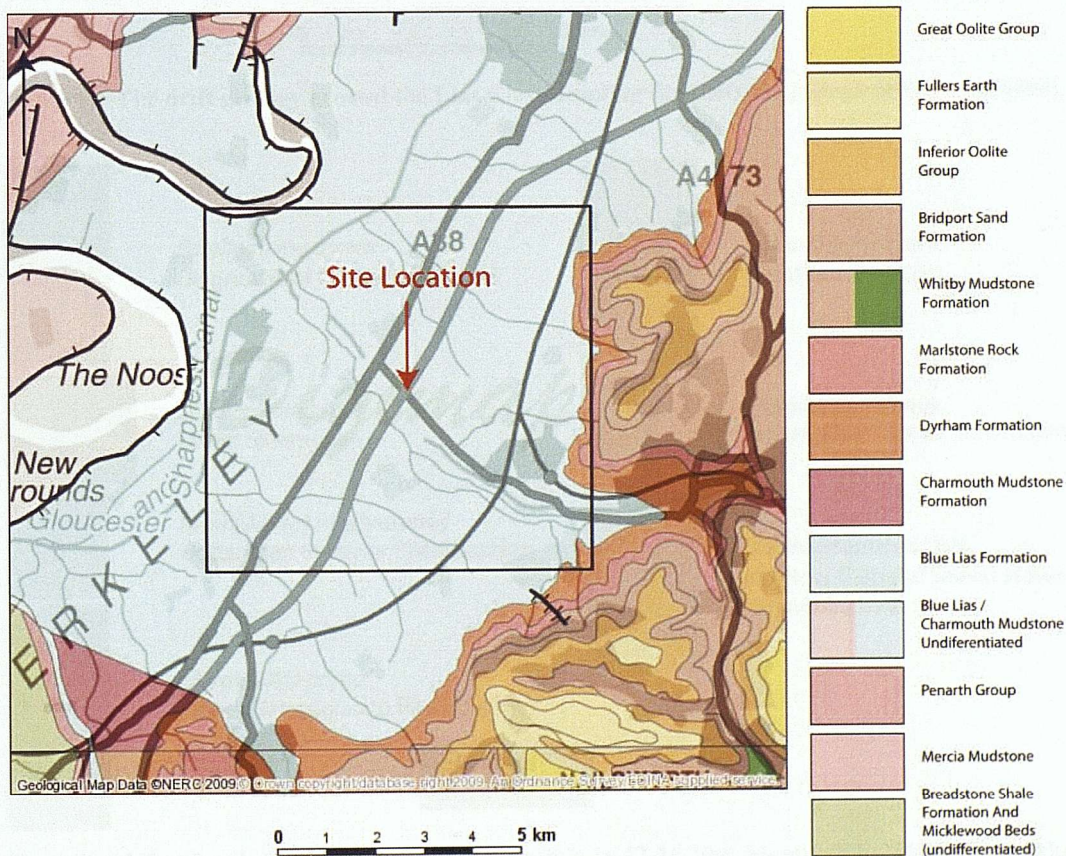


Figure 8.4 The solid geology around the Lias Clay sampling site (British Geological Survey, 2009b).

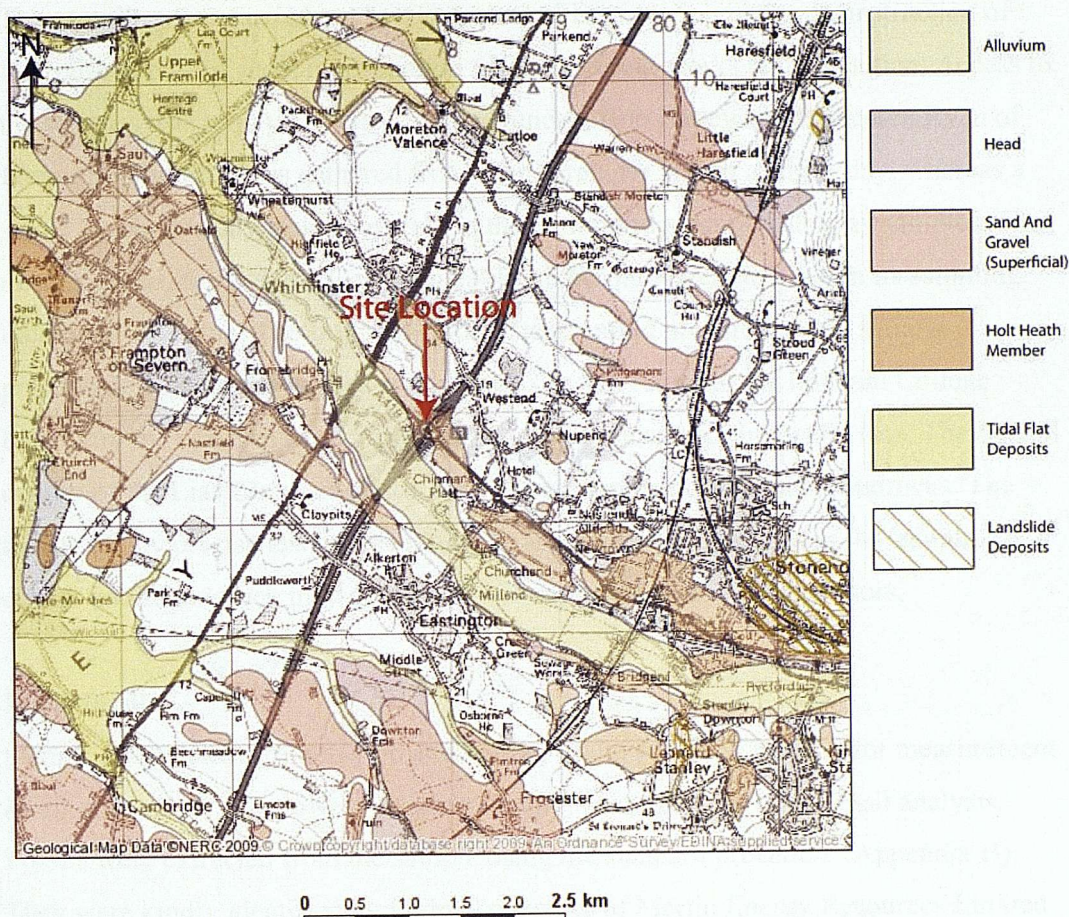


Figure 8.5 The drift geology around the Lias Clay sampling site (British Geological Survey, 2009e).

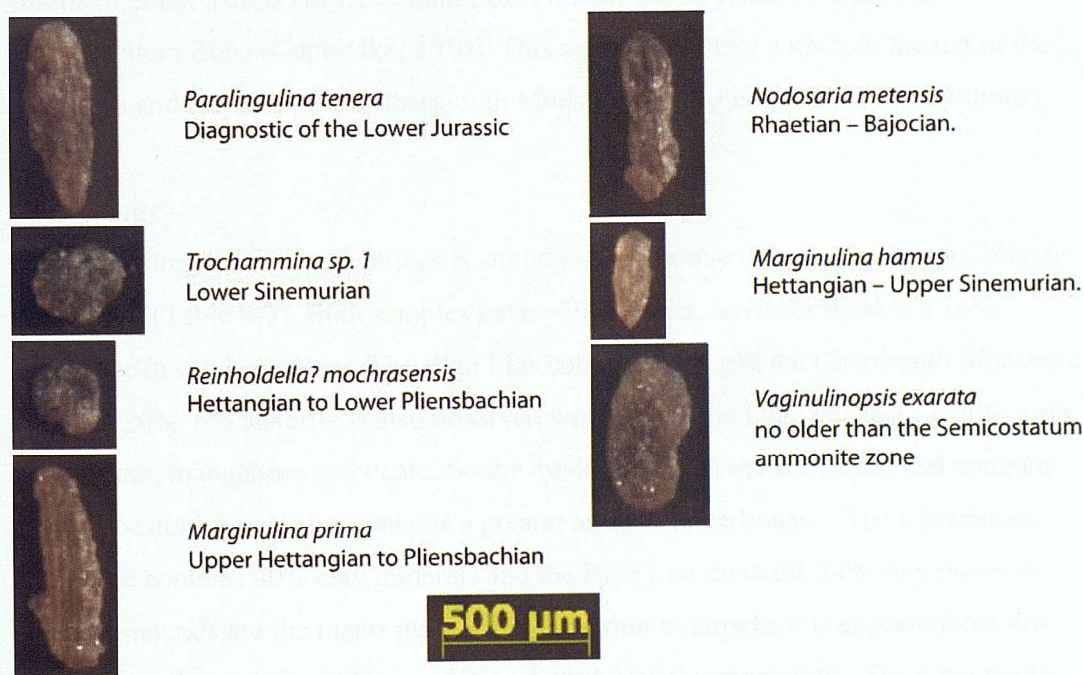


Figure 8.6 Microfossils from the Lias samples, from a 16.27-16.30m, identified by Philip Copestake of Merlin Energy Resources Limited. The sample is of Early Sinemurian age.

The sampling was carried out by Geotechnical Engineering under the instruction of Interoute as part of a site investigation for engineering works at the junction. Access to the samples was kindly provided by Ian Duncan, Rob Bailey and Francesca Ryan of Interoute. Mudrock was gathered from four boreholes within a single day, whereas a single borehole a day was recovered on the Kimmeridge and Gault Clay samples. The mudrock was stored in the Geotechnical Engineering warehouse within its sampling tube prior to testing. The Imperial College team could not attempt any sample preservation due to the limited space at the site. On return to the Imperial College laboratory the samples were preserved in wax to prevent further water loss. The overall quality of the Lias Clay samples is clearly less than that of the other mudrocks. The results of any precise test on this mudrock are difficult to interpret as the sampling effects on the mudrock may dominate over depositional and burial factors.

Microfossils

Samples were taken from the top and bottom of the sampling horizon for measurement of microstructure. The lowest imaging sample was reused for microfossil analysis. Fossils were extracted from the sample using the standard procedure (Appendix B). They were kindly identified by Philip Copestake of Merlin Energy Resources Limited (Figure 8.6). The sample is of Early Sinemurian age. Due to the absence of diagnostic fossils of either Turneri or Bucklandi Zone, the sample is probably from the Semicostatum Zone (Copestake, 2010). This age is consistent with both the top of the Blue Lias and the base of the Charmouth Mudstone (as the boundary is diachronous).

Mineralogy

The mineralogy of the two samples is slightly different due to being from two different formations (Table 8-3). Both samples have ~30% quartz, however there is a 16% difference in calcite content. The Blue Lias contains 36% and the Charmouth Mudstone contains 20%. 6% ankerite is also observed within the Blue Lias, this is a calcium, iron, magnesium, manganese carbonate. So the mudrock within the formation that contains many limestone bands also contains a greater amount of carbonate. The Charmouth Mudstone contains 40% clay minerals and the Blue Lias contains 24% clay minerals. As clay minerals are the major platy particles within mudrocks it is expected that this difference will result in a difference in preferred particle orientation. The Charmouth Mudstone should be better aligned at the micro scale. The Blue Lias less well aligned, however due to limestone bands it may be more anisotropic at the larger scale.

Lias Clay (depth)	illite & mica	illite-rich illite-smectite	smectite-rich illite-smectite	kaolinite	quartz	K feldspar	chlorite	Plagioclase	Calcite	Fe dolomite	ankerite	Pyrite
11.6m	11	14	9	6	32	1	3	1	20	0	0	2
16.4m	9	10	4	1	28	0	1	0	36	3	6	2

Lias Clay (depth)	illite	illite-smectite I-rich	illite-smectite S-rich	chlorite	Kaolinite
11.6m	60	10	6	7	17
16.4m	71	15	Tr	4	10

Table 8-3 Top table bulk mineralogy of the Lias Clay, bottom table, percentage of minerals contained within the clay grade (grain size) part of the Lias Clay.



Figure 8.7 The Blue Lias at Hock Cliff the cliff is heavily weathered, the main features visible are three limestone bands shallowly dipping away from the camera (East).



Figure 8.8 The Blue Lias at Hock Cliff, limestone bands (L) 100-150mm in size, some of the layering in the mudrock is visible, the surface is very hard and dry.

8.4 Comparison sites

The Lias Clay is exposed at Hock Cliff, 5km to the West of the Site. This material is Blue Lias (SO7285, 0904). In this location the boundary between the Blue Lias and the Charmouth Mudstone is very gradational the material present in the Blue Lias is essentially the same as the Charmouth Mudstone except the limestone bands are more closely spaced.

8.4.1 Hock Cliff

Hock Cliff, is a cliff exposure on the River Seven, it is formed by the Severn bore, which is a tidal phenomenon (Dreghorn, 1967). The cliff is an exposure of the top of the Blue Lias just prior to the transition to the Charmouth Mudstone (Figure 8.7). The most prominent feature visible on the cliff is the 100-150mm limestone bands. Between these the surface is covered with washed and hardened mud, some of the horizontal structure of the mudrock can be seen (Figure 8.8).

8.5 Overall material behaviour

As the Imperial College team was not able to be present on site during sampling due to its small size and location on a major road. Much less information is known about these samples compared to the others. That the horizon from which the samples were taken crosses the formal boundary between the Blue Lias and Charmouth Mudstone is unfortunate. It highlights the fact that this formation boundary is difficult to identify. These two mudrocks will probably behave differently because of the dominance of the limestone bands within the Blue Lias and the higher percentage of clay minerals within the Charmouth Mudstone. It is unfortunate that CPT data could not be gathered for this site, due to the high legal costs of carrying out investigations on a public road. Although it is unlikely that the cone would be able to penetrate the limestone bands, a good profile could have been created for the upper 15.4m of the Charmouth Mudstone prior to the first limestone band. Borehole logs cannot be included in this thesis as they were not organised by Imperial College and are proprietary data.

CHAPTER 9

METHODS

Twelve different imaging methods were evaluated prior to the selection of the environmental scanning electron microscope (E-SEM) as the primary imaging technique. The various methods and their application to mudrocks are briefly described in Appendix C (see Table 9-1 for a summary). The E-SEM was selected for the analysis because it provided both a high enough magnification, the possibility of measuring surfaces in three dimensions and allowed the sample moisture content to be maintained during imaging. The methods used for the analysis of microstructure using the E-SEM are described in this chapter.

9.1 *The scanning electron microscope (SEM)*

The electron microscope allows the imaging, at the micron or nanometre scale, of a very wide range of materials. By specialist adaptation of the basic SEM the range of materials that can be imaged is further widened. In essence the SEM is a highly focused beam of electrons which is scanned across the material surface (Figure 9.1). The number of electrons returned to the sensor is counted. By partitioning this incoming stream the variability of the electron return with location can be assessed. By displaying number of electrons on a grey level scale in a partitioned grid, an image can be created. The resolution of the image depends on the fineness of the grid. The number of electrons returned (and therefore the contrast of the image) is dependent on the topography of the sample and the atomic number of the elements in the sample (Goldstein et al., 2003). The images produced are very useful for the analysis of the structure of broken surfaces.

	Approximate Magnification	Advantages	Disadvantages
Optical Microscope	X1-X1,000	3D surface measurement possible	Maximum magnification too low
Confocal Microscope	X1-X1,414	3D volume imaging possible	Magnification too low, minerals cast shadows away from surface
Iterferometer	X1-X1,000 (vertical 0.1nm)	3D surface measurement possible (roughness)	Spatial resolution too low
Atomic Force Microscope	X1000- X5,000,000 (Vertical 5µm- 0.25nm)	Very high magnification surface measurements	Limited maximum variability in topography
Micro Computed Tomography (CT)	X1-X1,000 3D	Three dimensional volume measurements.	Maximum magnification too low
X-ray Ultra Microscope	X10-X3,000 3D	Three dimensional volume measurements.	Maximum magnification too low
X-ray Diffraction	-	Crystal orientations measured	Required a lot of time, does not provide spatial data
X-ray Goniometer	-	Crystal orientations measured	does not provide spatial data
Scanning Electron Microscope	X10-X500,000	Surface measurements possible	Cannot image 3D volumes
Transmission Electron Microscope	X10- X50,000,000	Very high magnifications possible	Very thin samples required
Focused Ion Beam	X10-X500,000	3D structure can be imaged	Prohibitively expensive
Porosymetry	-	Measures size of pores	does not provide spatial data

Table 9-1 Imaging methods tested (Appendix C). Scanning electron microscope produced good images at a useful magnification for mudrock.

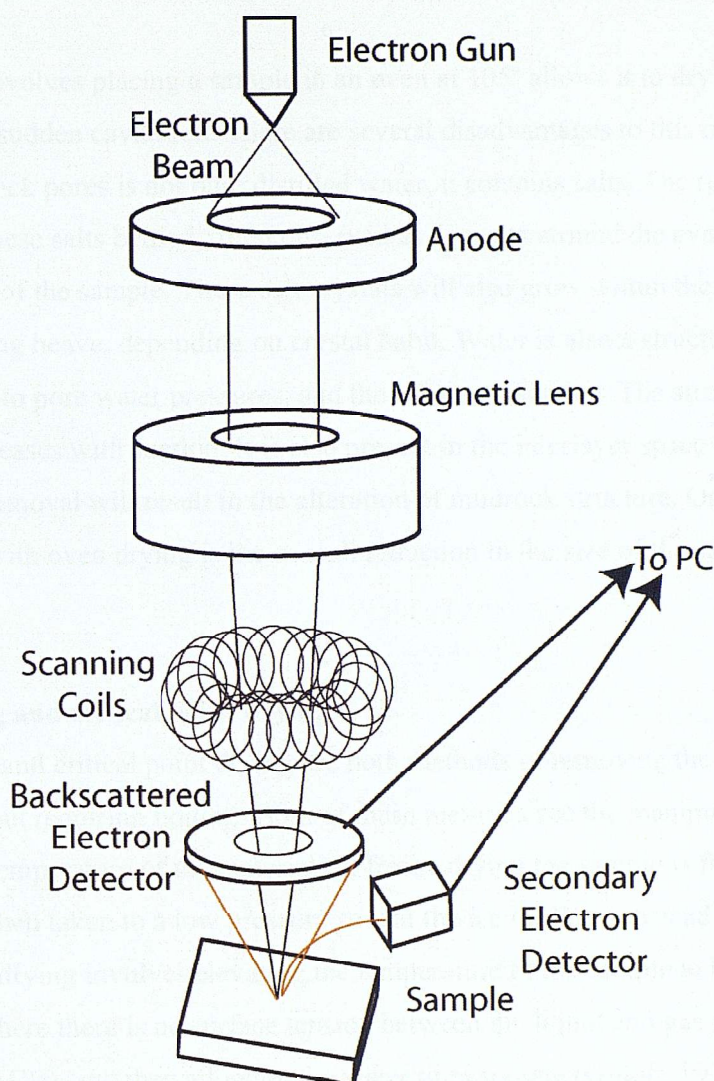


Figure 9.1 The components of a basic scanning electron microscope

9.1.1 Sample preparation techniques

Standard SEMs require samples to be imaged in a high vacuum environment. As water is an integral part of the structure of mudrocks, high vacuum is a particular problem. Placing a water rich sample under high vacuum causes immediate cavitation, effectively disrupting the micro-fabric.

This problem is dealt with either by the removal of water, or changes to the imaging process to allow the water to remain in the sample during imaging. Studies comparing multiple techniques show that measured material structure is dependent on the preparation method used (Gillott, 1970). Common methods include the following.

Oven drying

Oven drying involves placing a sample in an oven at 105° allows it to dry over a few hours without sudden cavitation. There are several disadvantages to this method. The water in mudrock pores is not pure distilled water, it contains salts. The removal of water leaves these salts behind, often observed as deposits around the evaporation sites on the surface of the sample. These salt crystals will also grow within the sample possibly causing heave, depending on crystal habit. Water is also a structural element of mudrocks due to pore water pressures, and the effects of suction. The strength of mudrocks increases with suction. It is also present in the interlayer space of clay minerals. Its removal will result in the alteration of mudrock structure. One of the main observations with oven drying is the overall reduction in the size of the samples (shrinkage).

Freeze drying and critical point drying

Freeze drying and critical point drying are both methods of removing the water in the material without requiring boiling. Both of these methods use the manipulation of the pressure and temperature of the material. In freeze drying the sample is frozen in liquid nitrogen and then taken to a low pressure so that the ice sublimates instead of boiling. Critical point drying involves elevating the temperature of the sample at high pressure to the point where there is no surface tension between the liquid and gas phases of water (374 °C, 22.5MPa), and then allowing the water to evaporate (Gillott, 1970).

Both of these pressure temperature conditions have never been experienced by the Jurassic mudrocks during their burial and uplift histories. Critical point drying is particularly excessive. Sometimes water is replaced by ethanol as this has a lower critical point (243°C, 6.5MPa) compared to water (Gillott, 1970). Even this pressure temperature condition is more than twice the highest estimates of that experienced by the Jurassic mudrocks. The use of ethanol also leads to problems of incomplete imbibition. Critical point drying has the possibility of causing mineralogical changes (low temperature metamorphism). This has the potential to greatly alter the structure of the mudrock.

The freezing of water results in the growth of ice whiskers; these have the potential to force particles apart. This is especially a problem where water is present in the interlayer space of clay minerals as its expansion can break the layers apart causing

some clay minerals to crumble (Perrott, 1977). Freeze drying has been commonly used as it does not cause macro scale shrinkage (Tovey et al., 1973). However the process can only achieve no shrinkage by disrupting the micro structural effect which also disrupts the micro structural fabric. This method can be improved by freezing the mudrock very rapidly (Gillott, 1969) so that unstructured vitreous (non crystalline) ice is formed (see Cryo-SEM below).

Air drying

Air drying involves placing a sample in a cool place allowing water to evaporate over a long period of time. The sample is not exposed to extreme conditions or boiling. The air dried samples experience bulk shrinkage. As drying has an effect on the samples irrespective of the method used it is better to avoid doing so where possible. If drying is necessary then air drying may have the least effect on the mudrocks microstructure.

Embedding

Embedding is the replacement of the water in the pores of a mudrock with a resin (or a liquid which will solidify). A large number of different resins are available (Smart and Tovey, 1982). Most of them are immiscible in water. Normally intermediate liquids (which are not immiscible) are used so that the minimum amount of water remains in the sample after imbibition. Due to the low permeability of mudrocks resin injection takes a long period of time. To speed up this process the resin is often forced into the sample under pressure. The resin is then allowed to set; some resins are thermosetting and so require heating to solidify. Selection of the right resin is important. Some resins are designed to alter the mudrock's structure, the most common causing the expansion of layered clay minerals (Kim et al., 1995).

Embedding is necessary for transmission electron microscope (TEM) studies of mudrocks as it allows the creation of a solid sample which a microtome can cut thin enough for it to be electron transparent. However for SEM studies, the extensive amount of sample preparation and the possible effects of fluid replacement make this method unsuitable.

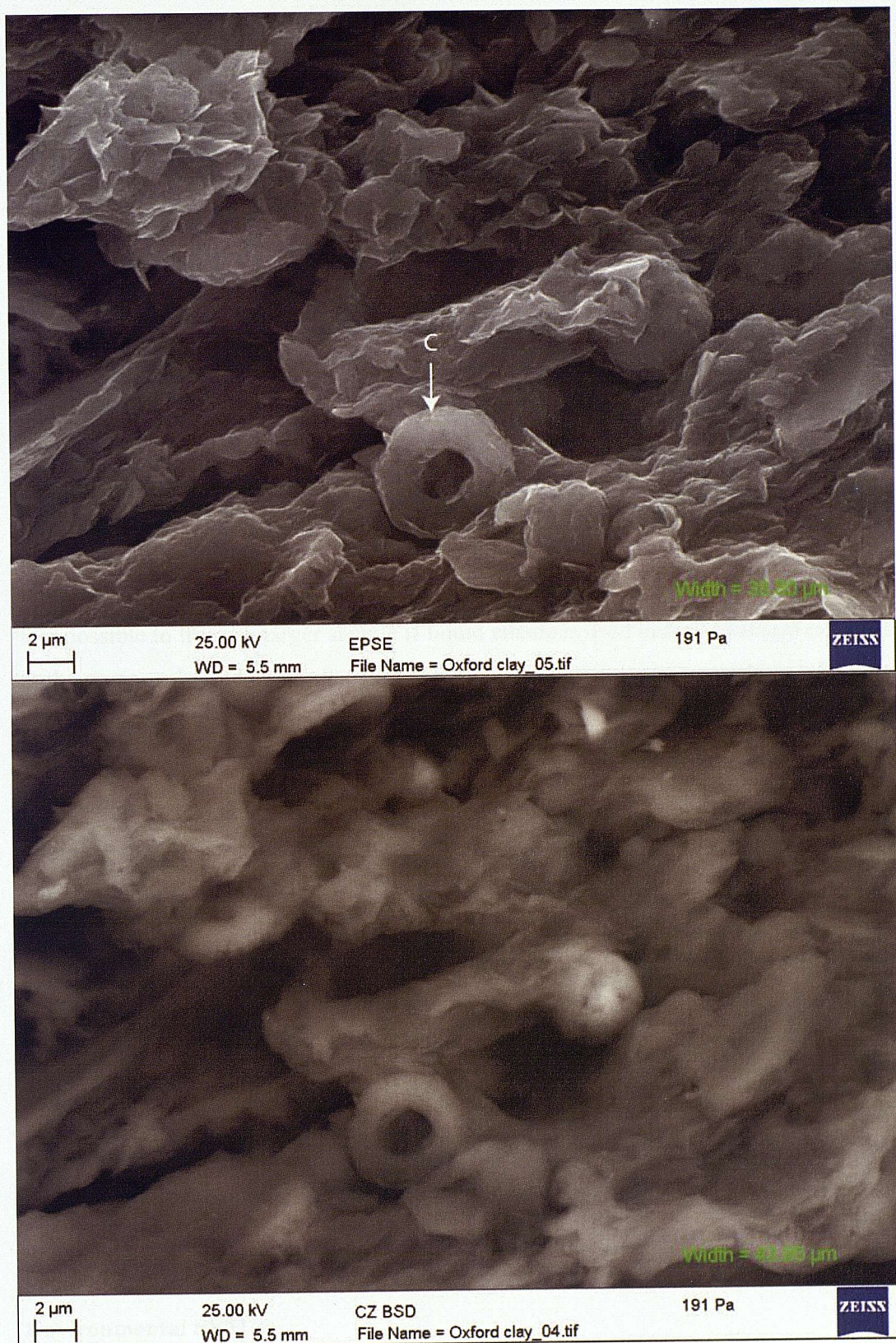


Figure 9.2 Both images are of the same location with a minor change in scale. Top image formed using secondary electrons, bottom image formed using back scattered electrons. Image is a vertical broken surface of Oxford Clay, taken in the Carl Zeiss Laboratory at Cambridge. The coccolith (C) in bottom centre of both images is visible using the surface penetrating back scatter electrons, however the secondary electrons cannot penetrate the thin clay mineral layer covering its surface.

Cryo-SEM

By rapid freezing of the sample in slushy liquid nitrogen at $\sim -210^{\circ}\text{C}$, water within the mudrock can be transformed into unstructured, vitreous ice. The sample can be fractured while frozen and then imaged in high vacuum, with the sample maintained at very low temperature. By this method the location of water within the sample can be imaged. Curved menisci and pendular links between soil grains have previously been observed (Gvirtzman et al., 1987).

As cryo-imaging involves the first stage of freeze drying, the images of mudrocks are often very different compared to those of air dried samples. Pores are much smaller in cryo samples, because they are filled with clay minerals (Velbel and Barker, 2008). Clay minerals normally collapse to the edge of pores as water is removed.

For a sample to be frozen without ice crystal formation the sample must be very small. It is possible to freeze a larger sample if liquid ethane is used instead of liquid nitrogen as the cryogen. Liquid ethane is a highly flammable material, so a specialised laboratory is required for its use. This method was not selected because the required small sample sizes are too small for the easy maintenance of sample orientation, especially during the freezing process.

Variable pressure SEM

Most samples of high vacuum electron microscopy need to be coated with an electrically conductive material to stop electrons building up on the material surface and either discharging unevenly or deflecting the scanning beam through electric repulsion. When a gas is present within the chamber the gas conducts this surface charge away. The gas however also deflects some of the electrons in the scanning beam causing it to interact with a wider area. This leads to an increase in the noise in the image, and therefore a minor decrease in the resolution of the images. The higher pressure can slow the rate of the removal of water from the samples and greatly improving the images.

Environmental SEM

The environmental SEM is a variable pressure SEM with a temperature controlled stage and where the gas within the chamber can be water vapour. By careful control of the pressure and temperature within the microscope chamber the moisture content of the sample can be maintained. As this is an equilibrium process, pressure and temperature

conditions for 99% relative humidity are preferred. Evaporation is only slightly faster than precipitation. Moisture content is maintained without the risk of condensation on to the material surface. This method was selected as it maintains the material's water content during imaging and requires very little sample preparation.

9.1.2 Measurements taken by SEM

Image creation

The grey level of any point on an SEM image is dependent on the number of electrons returned to the detector while the electron beam is scanning across that location. By only counting electrons within specific energy ranges it is possible to create different images. Two main energies of electron are commonly used. High energy electrons (back scattered electrons) originate from the initial electron beam after they have interacted with the material's surface. They are able to penetrate the surface to some depth and still return to the detector. They can therefore penetrate through thin clay minerals and provide detail about the material beneath (Figure 9.2). The number of electrons returned to the detector is dependent on the elevation of the surface and the atomic number of the elements in the material.

Low energy electrons are produced when the electron beam knocks electrons from the samples atoms (secondary electrons). These electrons do not have enough energy to penetrate the materials surface and return to the detector. They can only return to the detector from the surface of the material. Thus secondary electron images show much more detail of the surface of the material than back scattered electrons, they are also less affected by the materials atomic number (Figure 9.2). The secondary electron detector is normally place to one side of the material. Surfaces angled away from the detector return fewer electrons (have a lower grey level) than surfaces angled towards the detector. Pinnacles return many more electrons than troughs. All of the analysed images are secondary electron images.

X-ray analysis

A beam of electrons fired at a material causes the emission of x-rays. Different atoms produce different energy x-rays. By collecting these produced x-rays a chemical map of the material surface can be created. This data can be used for the identification of

specific crystals. When applied across a wide area, it can be used for mineralogical analysis (Clayton and Pearce, 2007).

9.1.3 Application to mudrocks

The electron microscope is the most commonly used method for the imaging of mudrocks. One of its earliest uses was for imaging the shapes of clay minerals (Bates, 1952; Bates and Comer, 1954; Taggart et al., 1954). It is perfectly suited to this due to the potential for very high magnification imaging of materials. It is also of great use for the measurement of properties of intact mudrocks: preferred particle orientations (Foster and De, 1971; Gillott, 1970; Gipson, 1965; Ho et al., 1995); structures caused by shearing (Agar and Prior, 1989; Kim et al., 2007; McKyes and Yong, 1971); permeability anisotropy (Clennell et al., 1999; Dewhurst et al., 1996b), including that of oil shales (Nix and Feist-Burkhardt, 2003); the effects of structure on engineering behaviour (Rajasekaran and Rao, 1997; Romero and Simms, 2008); and of course the study of depositional micro-structure which has already been described (Section 4.3) . In short the electron microscope has been used regularly to study the microstructure of geological materials for multiple applications. It is therefore an appropriate technique to be used for the study of the UK Jurassic mudrocks presented here.

9.2 Sample preparation for E-SEM

The preservation of the moisture content is important for the successful imaging of the intact structure of mudrock. Samples are preserved in a double layer of cling film and wax after they are cut from the field sampled blocks to retain as much moisture as possible.

The procedure followed in the E-SEM room is as follows. The mudrock's wax covering is removed in the microscope room (Figure 9.3b). An elongate cuboid of mudrock is cut out of the sample using a razor blade (Figure 9.3c). The long axis of the cuboid must be perpendicular to the desired orientation of broken surface. The cuboid sample is then snapped so that a surface in the correct orientation is formed (Figure 9.3d). If the formed surface is not in the correct orientation the process is repeated until an acceptable surface is produced.

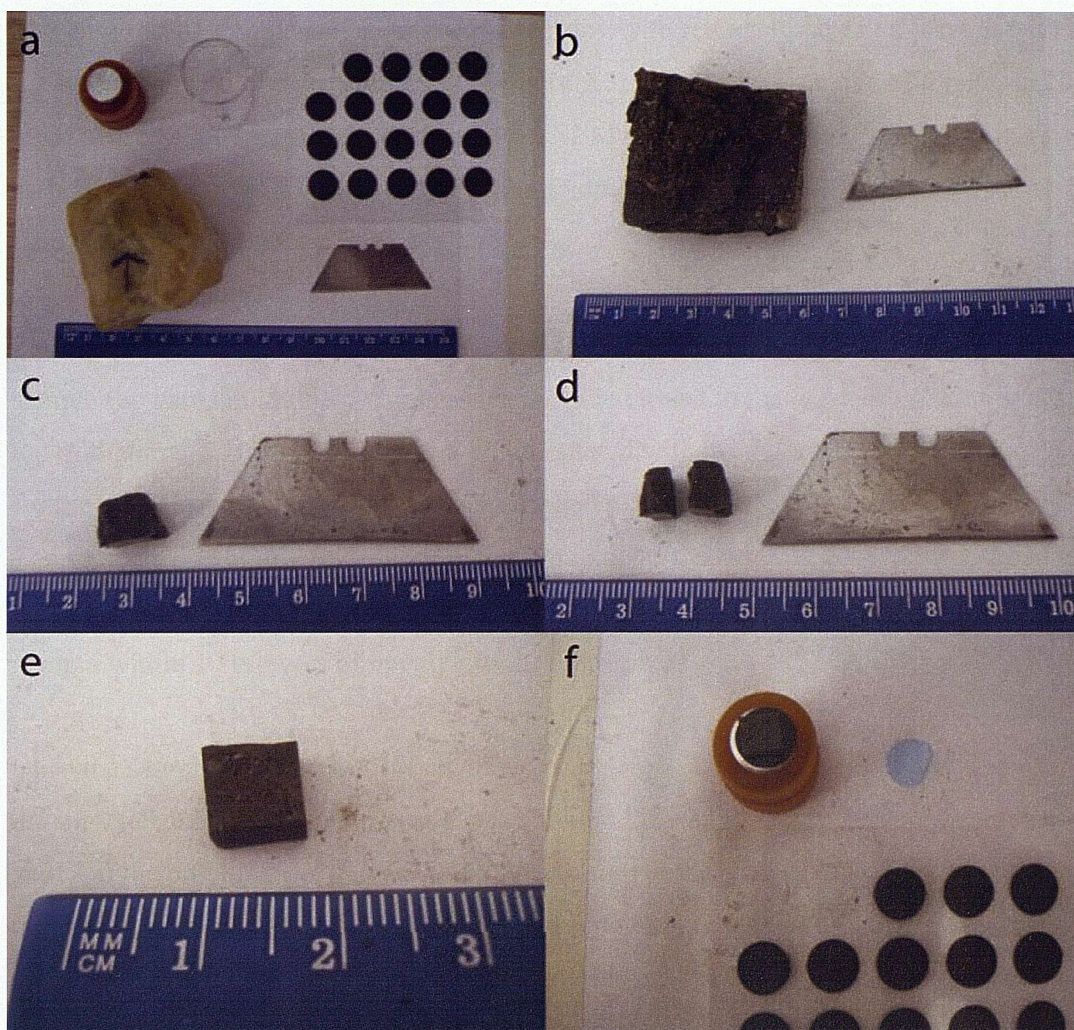


Figure 9.3 Sample preparation. a). materials: SEM stub, double sided sticky carbon tab, wax wrapped sample, blade. b). mudrock sample is removed from wax. c). mudrock cuboid cut from sample. d). broken surface created in horizontal orientation. e). four sides and base of sample shaved down to smaller than SEM stub. f). sample attached to stub by double sided sticky carbon tab.

The base of this sample is then shaved down slowly to reduce its size and leave it with a flat base parallel to the broken surface (Figure 9.3e); normally a sample of thickness 2-3mm is used. The sample is mounted onto a SEM stub using a double sided sticky carbon tab (Figure 9.3f). The surface of the sample is cleaned using a weak electrostatic field (Hulbert and Bennett, 1975).

The sample is then placed into the E-SEM. If it is a vertical break then the top of the sample (upwards in the field) is placed as the side closest to the detector, making it the top of the image. Horizontal samples are orientated with north to the top of the images for the block samples; no orientation is assigned to horizontal borehole sample images. The samples do not require coating before imaging as the water vapour in the E-SEM is very effective at removing charge from the materials surface. Samples are normally imaged within 10 minutes of opening to the atmosphere.

Once the sample is in the E-SEM chamber, four drops of water are placed into the dimples surrounding the microscope stage around the sample. Then the E-SEM door is shut and the pumping cycle starts. This cycle removes the air from the chamber and replaces it with water vapour. It involves pumping down to 0.8KPa (6 Torr) then up to 1.3KPa (10 Torr) during this cycle the drops of water evaporate keeping the atmosphere around the sample high in water vapour. The sample stage is temperature controlled to keep the sample at a constant temperature of 4°C. The pressure was set to 0.8KPa (6 Torr) which equates to 99% relative humidity, the pressure was varied on a few occasions according to sample response. For example the Lias Clay 16.46m sample was very dry going into the microscope, and started saturating during imaging so the pressure was reduced to lower the relative humidity. The sample takes time to reach the correct temperature; this time is related to the size of the samples, hence small samples are recommended. This adjustment of the samples to the right temperature can be seen as a drift of the sample surface during imaging. So no images are taken during the first few minutes, to allow the mudrock to equilibrate.

During sample preparation, the broken surface will form using the natural planes of weakness of the mudrock, for this reason it is expected that the natural weaknesses (joints) will be imaged where present instead of the stronger structured areas. As natural failures will also normally propagate through the weakest parts of the material, it is appropriate that these areas are more commonly imaged.

One of the major accessories of electron microscopes is an EDX detector; this detects x-rays produced by the interaction of electrons with atoms. By counting x-rays produced in this fashion it is possible to approximate the chemical structure of mudrocks, this is especially useful where electrons are concentrated on a small area of a material of interest instead of blanketing the surface. This method can be used to identify the mineralogy of unknown features or crystals. It is also possible to collect information about the variation of chemicals across an area of a surface, producing a chemical map of the surface; this can be used to identify changes across features. Unfortunately at the time of imaging it was not possible to collect chemical data while in E-SEM mode. On a few occasions high vacuum mode was used to collect chemical map data.

9.3 *Image analysis techniques*

Images can be processed by the human eye; relevant qualitative data can be extracted by description and explanation. Quantitative data can only be extracted by selecting specific factors for measurement. Quantification of images leads to specific data about one aspect of the material, whereas qualitative observation allows a wider range of factors to be taken into account. Images are large matrices of numbers, where each pixel in the image is represented by a number. This dataset must be summarised for information to be extracted.

9.3.1 Method for analysing single SEM images

The method selected for image processing is dependent on the information required from the image. Normally there are specific features in the image that need measuring (*e.g.*, particles or voids). The first step is to separate the features of interest from the remainder of the information present in the image. If the image has a large range of contrast then the features of interest can often be highlighted automatically by setting a grey level threshold. This can be easily achieved using a program such as ImageJ (Rasband, 2009). If images have a limited range of contrasts or if the feature of interest varies throughout the contrast range, either complex processing or manual interpretation is required. It is useful to illustrate this process with a simple example of sand before going on to the more complex mudrock images.

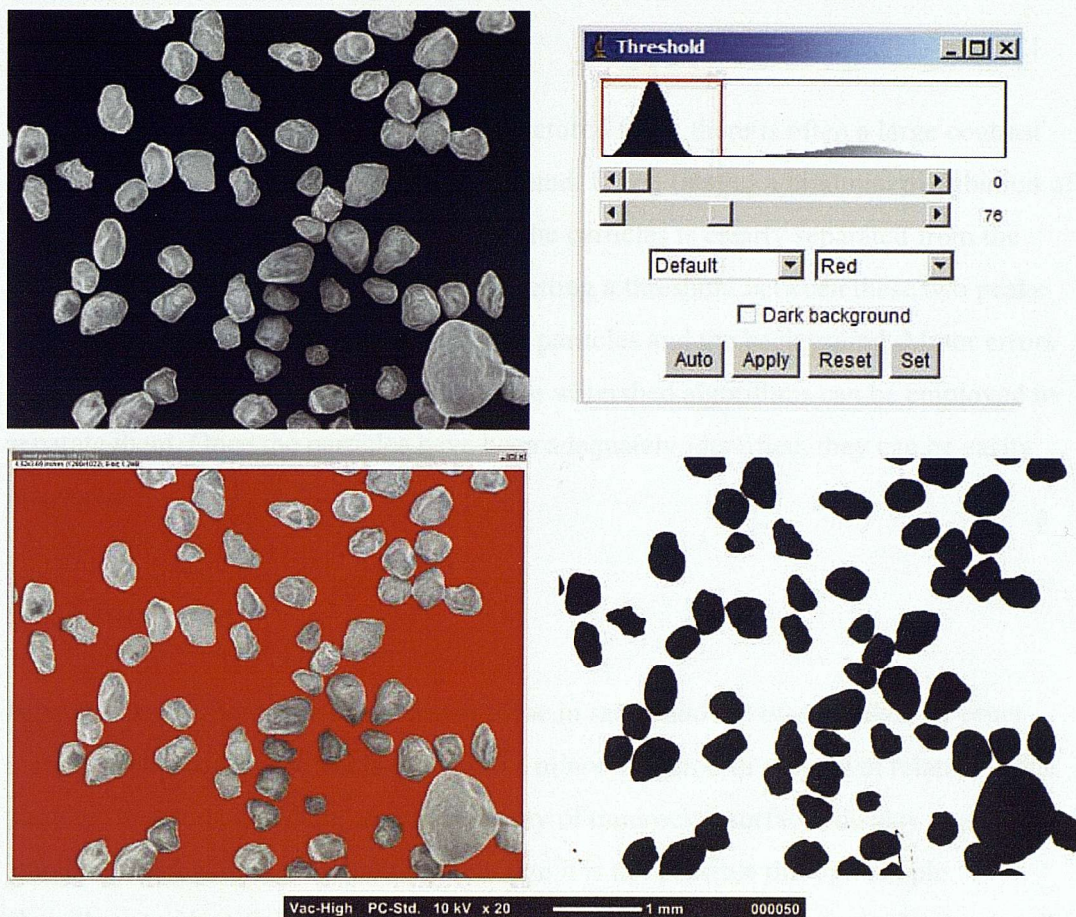


Figure 9.4 An electron microscope image is composed of a grid of pixels each of which can have a grey level between 0 and 256. By selecting a specific grey level as a threshold a binary image (black and white) showing only the features of interest can be created (Rasband, 2009).

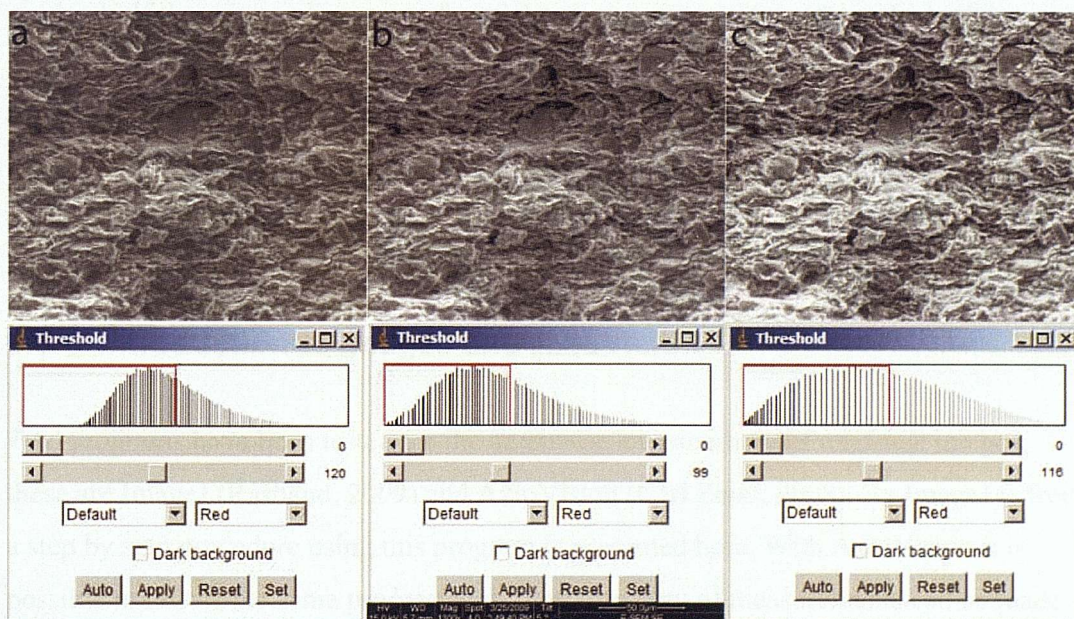


Figure 9.5 Grey level histograms for mudrocks. a). original image. b). after normalisation. c). after equalisation. This single peak histogram contrasts with the dual peak histogram from sands (Figure 9.4). Spreading the grey levels throughout the entire spectrum allows thresholds to be set at a constant grey level irrespective of the original contrast (Rasband, 2009).

9.3.2 Sands

When observing sand particles in an unstructured form, there is often a large contrast between the sand particles and the background. Using ImageJ a binomial distribution of grey levels can be observed. The peak for the particles is clearly separated from the peak for the background (Figure 9.4). By setting a threshold between these two peaks the image can be easily partitioned into the particles and the background. Minor errors occur where particles are touching, and here watershed algorithms can be employed to separate them. Once the particles have been adequately identified, they can be easily measured.

9.3.3 Mudrocks

In mudrocks the pores are smaller than those in sands and are often backed by other particles. This causes the voids to exhibit a minor variation in grey level relative to the particles that surround them. As the majority of mudrocks' surfaces display a greater amount of topography than their particle size it is not possible through simple algorithms to identify individual particles or voids. In other words, instead of two well separated peaks of grey levels mudrocks tend to display a single peaked grey level distribution (Figure 9.5). It is possible to identify trends in the broken surface of the mudrock. If there is an alignment of particles then peaks and troughs in the broken surface of the mudrock will often also be aligned. As grey level partially correlates with topography (Section 9.1.2), the alignment of peaks and troughs can be identified out using a grey level threshold. This analysis can be carried out using software such as AxioVision (Carl Zeiss, 2008) or ImageJ (Rasband, 2009).

9.3.4 Introduction to ImageJ and AxioVision

Two programs have been tested for the automatic measurement of mudrock images, these are ImageJ (Rasband, 2009) and AxioVision (Carl Zeiss, 2008). As ImageJ is free, a step by step procedure using this program is presented here. With AxioVision it is possible to follow the same process, and a larger variety of measurements can be made. However it takes much longer to process each image. This decreases its usefulness where a large number of images must be analysed. With ImageJ a single image can be processed in 30-45 seconds. Macros can be written to bulk process multiple images.

9.3.5 Step by step analysis of mudrocks with ImageJ

The first step is normally to select a threshold. This poses a difficulty for automated measurement of these materials as the contrast of an image is dependent on what the SEM operator thought would make the best image at the time it was taken. The distribution of grey levels is a function both of operator preference and of the features in the image. Electron microscope imaging is an expert skill, it cannot be standardised. The grey level spectrum of the resultant images can be standardised by stretching across the full spectrum through contrast enhancement. Two methods of contrast enhancement are available in ImageJ, these are to normalise, or equalise the grey level histogram. The equalise histogram method was used to analyse the mudrock images (explained below).

Normalise, this method stretches the grey level distribution such that the maximum and minimum values of the original distribution correspond with the maximum and minimum possible values (Figure 9.5). Outliers of the distribution cause problems for this method as they limit the amount of stretching that can be achieved. This is overcome by the parameter “% saturated pixels”, allowing this percentage of pixels to pass the minimum and maximum possible values when stretching the contrast. The value of these pixels is then set to the minimum or maximum grey levels respectively. Because of this process these pixels lose the relative image information that they once contained, and the process becomes irreversible. For this reason it is undesirable to allow a large percentage of pixels to saturate. The final distribution achieved by the method has the form of the original distribution stretched out through the entire grey level spectrum (Figure 9.5).

Equalise histogram, This method also stretches the grey level distribution across the entire spectrum. The spacing between new grey levels is dependent on the number of pixels in the original image sharing a grey level (Figure 9.5). If few pixels of the original image have a given grey level then there will be little spacing to the next grey level in the new histogram. If lots of pixels share a given grey level then there will be a large space either side of the grey level in the new histogram. This means that common grey levels in the image become more defined so differences which were originally small become more visible. This method is commonly used for processing images with little contrast variation between features of interest (Rasband, 2009).

Once the image's contrast has been enhanced a threshold can be set. From observation, particle orientations in images, which would be selected by visual inspection, often correspond with the higher grey levels or lighter colours. This is because the majority of particles which can be identified tend to be near the peaks in topography. For this reason the upper quartile (25%) of grey levels (191-255) is an appropriate measure which corresponds well with particle orientations.

The lower quartile of grey levels (0-64) was also selected for comparison, as in structured material both darker and lighter areas tend to align. Only where measurements taken from both of these quartiles correspond can the structure be real, *i.e.*, only where peaks and troughs align. Using these thresholds produces two binary images which can be processed for measurements.

An alternative threshold method considered for this work is to select based on the number of pixels. For example, the 25% of pixels with the lowest grey level values, and the same for the upper 25%. This was not attempted as no program could be found which applied such a filter and no program was written. It is believed that this would produce similar results to the process shown here, it would however be a more computationally complex process.

ImageJ has a suite of measurements available. The most important measurement for mudrocks is particle long axis orientation. Two such measures exist, the Feret's maximum length and ellipse long axis orientation, the ellipse long axis orientation is used for this work.

Feret maximum length: A Feret length is the distance between two parallel lines which just touch either side of the particle. To find the maximum length the lines are rotated at intervals through 360° and multiple measurements are taken; the Feret maximum length is the largest distance between the two parallel lines. The angle corresponding to this maximum distance is the long axis orientation. As there are an infinite number of orientations in which the parallel lines could be orientated it can be a very time consuming. For this reason it is the convention to consider 32 orientations through 360°. This is the process adopted by ImageJ. The data are divided into intervals of 11.5°, which is quite a broad descriptor of particle orientation. It also means that standard rose diagrams drawn of the distribution of orientations using a 10° interval, will contain four

partitions of no data. Thus, primarily because of the level of inaccuracy in particle orientation this method was not used.

Maximum ellipse angle: Particles of any shape can be modelled as ellipses. This is achieved by finding an ellipse which has the same moment of inertia as the particle. The orientation of the long axes of these ellipses is a measure of particle orientation. All areas touching the edge of the image are not measured as their true orientations cannot be confirmed. The ellipses used by the program can be output as a spatial diagram for comparison with the original binary image.

The measurements of long axis orientation are heavily biased by smaller particles. Particles of one pixel in reality have no orientation; this process treats them as having an orientation of zero. Particles made of two pixels can form only four orientations. Those of three can form eight orientations and those of four can form at least twenty four orientations including the circular group of four which is considered by the program to be zero although it has no orientation. As these particles are small there is potential for a large number of them to exist across an image. In practice, these small particles are removed and not considered in the analysis, to prevent them biasing the data

The long axis orientations can be plotted on a rose diagram; the program Rosetta (Jack1024, 2004) was used. Unfortunately Rosetta does not partition the data completely symmetrically. All of the partitions are 10 degree intervals except for 350-0.5° and 0.5-10°. As this unusual partitioning is not used for the opposite direction (180-180.5°), the resultant rose diagrams are asymmetrical. To overcome this problem a Java program was written to pre-partition the data into ten degree intervals. This output was then entered into Rosetta to plot the rose diagram.

There are two different standard notations used to describe circular data. The mathematical system: zero is to the right and degrees are counted anticlockwise such that ninety degrees is to the top of the page. This is the notation used by ImageJ. The compass notation starts with zero towards the top of the page and counts clockwise such that ninety degrees is to the right of the page. This is the notation used by Rosetta. To realign the rose diagrams to the images they had to be rotated left and flipped through a vertical plane. This can be achieved using any image manipulation software. IrfanView (Skiljan, 2009) was used in this project. This program can also quickly crop off the

labels which are now in the wrong positions. New labels were added to the images using Adobe Illustrator (Adobe, 2008).

Measurements of the preferred orientation of a material cannot be made from a single image, as single images are often affected by local bias. Therefore data obtained from all images of a sample at a single scale are gathered together and plotted as a single rose diagram. This shows the structure of the materials as a whole and can be used to investigate the effect of scale. These rose diagrams, formed from multiple images, are discussed in Section 11.1. The rose diagrams were processed further to create a single number quantification of preferred particle orientation. The method of quantifying rose diagrams will be discussed alongside the results in Section 11.1.4.

9.3.6 2D image analysis procedure

Figure 9.6 shows typical results for a vertical break (*i.e.*, bedding perpendicular to surface) of the Oxford Clay. Such summary pages are available in Appendix D for all of the images taken during this work. This figure is presented to show the application of the fabric quantification/image analysis method (Section 9.3.5) to a single image.

Oxford Clay has a strong preferred particle orientation as indicated by the areas of light and dark in Figure 9.6. These areas are patches of high and low topography, respectively, on the clay surface. There are areas of irregularity on the surface, however they have a small effect compared to the major features. Figure 9.6 (a) is the original electron microscope image. The first step in the process was the equalisation of the grey levels to stretch out the contrast to the full grey level range (Figure 9.6b). Binary images of the upper (Figure 9.6c) and lower (Figure 9.6d) quartiles of grey levels pick out peaks and troughs respectively in the image. These are not individual particles, they are colour contrasts caused mainly by surface topography, but they are representative of the orientations of particles within the image. The top right corner of the lower quartile image (Figure 9.6d) shows a large area orientated at a different angle to the rest of the image, because of a sudden change in topography. The process used places the same weight on particles irrespective of size, so the greater number of medium and smaller particles in the correct surface orientation stop this large irregularity from having an affect on the measured preferred particle orientation. During early experimentation with this method particles were weighted by size. This gave large irregularities a more

prominent effect on the resulting material preferred orientation. Therefore this method was therefore not adopted. The selected particles are modelled as ellipses with the same moment of inertia for upper (Figure 9.6e) and lower (Figure 9.6f) binary images. These images show how different original particles are replaced by ellipses and the distribution of different sizes of particle, which is difficult to identify in the binary images because connections between selected areas may be only one pixel in size. These ellipses highlight the large number of smaller size particles which a human interpreter would then discount.

The rose diagrams plot the summation of long axis orientations divided into 10° partitions. They can be thought of as angular histograms. They represent the overall structure of the material for the upper (Figure 9.6g) and lower (Figure 9.6h) quartiles.

9.3.7 Possible errors

The main advantage of this method (Section 9.3.6) is that direct comparisons can be made between different samples from different depths, both qualitatively and quantitatively. This is achieved because all images are treated the same way. However, any standardised method will not work perfectly in all situations. Some images do not produce good results using this method. These “errors” commonly occur both due to variations within the mudrocks and where they deviate from the aligned peaks and troughs which commonly indicate a preferred particle orientation (Figure 9.6).

The most important consideration is the scale of the features being imaged. Any feature which fills more than half of an image will greatly affect the structures measured. This can include grains which are larger than the minimum magnification (Figure 9.7a) or images at high magnification where a single clay particle fills the image (Figure 9.7b). In these cases the large-scale feature dominates the image and the orientation data gathered are not representative of the mudrock as a whole. Fortunately, although these errors affect single images, the number of measurements which can be taken from these images is relatively low. Thus, these images have little impact on the overall measurement of the material when added to the other images of the surface.

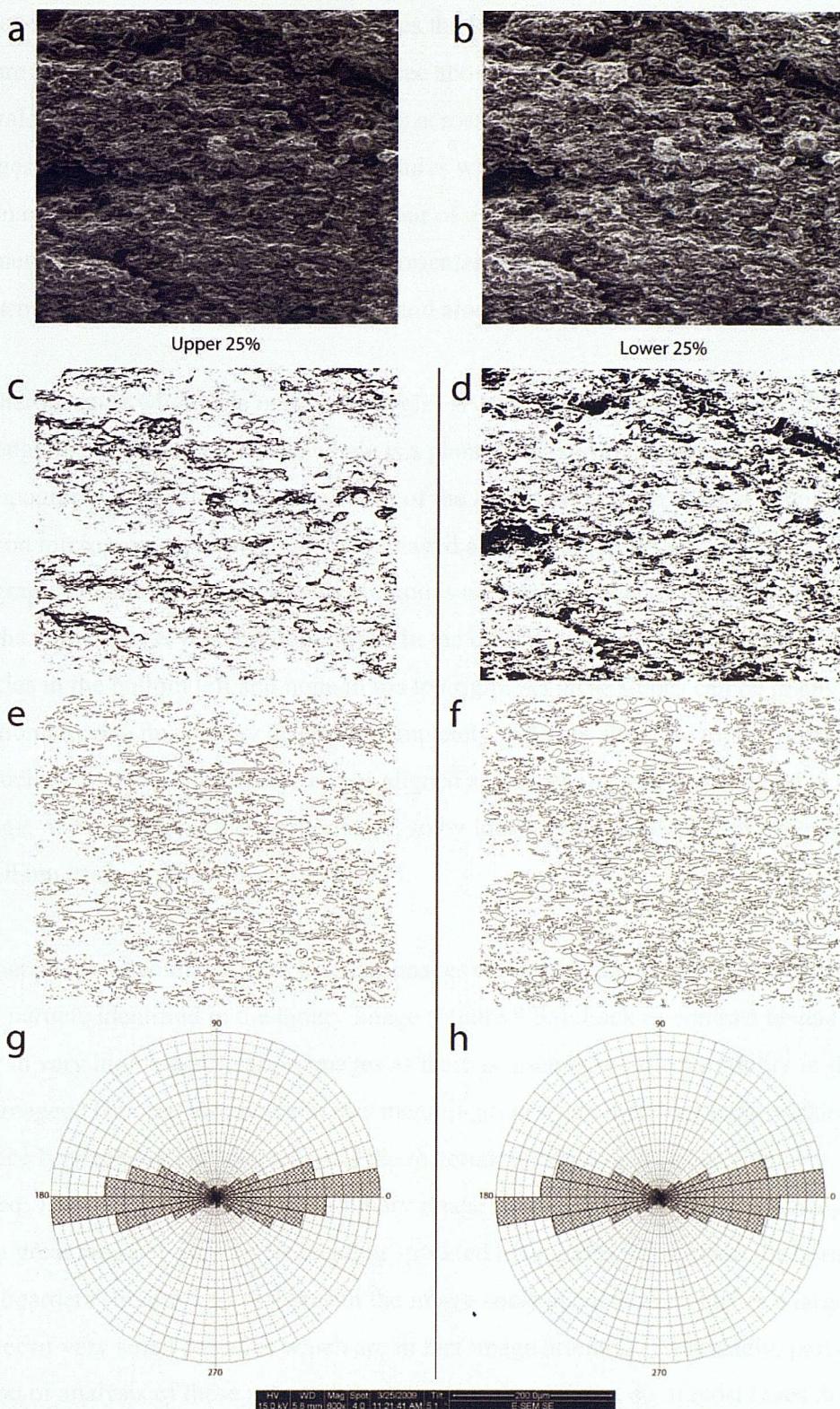


Figure 9.6 Typical preferred orientation measurements from vertical broken surfaces of the Oxford Clay. Original (a) and equalised (b) image. Upper quartile binary process in the left column and lower quartile in the right. From top to bottom, binary image (c & d), ellipses used to model particles (e & f), and rose diagram of orientations of long axes of ellipses (g & h). Original SEM image data bar at base. Produced using ImageJ (Rasband, 2009), Rosetta (Jack1024, 2004) and Adobe Illustrator (Adobe, 2008).

However, the method of processing ignores the localised complexity of materials (Figure 9.7c). This can be an advantage (see above) or a disadvantage; to understand the general preferred orientation of a mudrock across the entire sample surface, local changes must be ignored. However, structures which are imaged sometimes play a large role in controlling the mechanical behaviour of a mudrock. It should be highlighted that this method for quantifying the preferred orientation of a material is a tool to be used to augment human interpretation, not as a stand alone method.

Another instance where this method struggles is on sloping surfaces (Figure 9.7d). Although the broken surface as a whole is a plane in the desired orientation, the surface has topography. Focusing on a small area of the surface as is done using a scanning electron microscope may mean that the imaged area is sloping. In this case the topographic element selected by this method is not so much that of the peaks and troughs in the image, it is the slope itself. In the binary image there are many more particles in the bottom left and none in the top right. As these slopes can be in any direction any one image may display a completely different structure to that of the mudrock as a whole. Fortunately ridges aligned at the slightly larger scale tend to, on average, match those on the smaller scale, so by looking at a large number of images the overall mudrock structure is still extracted.

A general lack of contrast in the original images can greatly increase the number of very small particle identified in the binary image (Figure 9.8a). Lack of contrast tends to occur in very high magnification images as there is often very little variability in the area imaged. This can also occur at low magnification when there is a high working distance between the electron lens and the material, or when the surface is poorly imaged. This effect is highlighted in binary image (Figure 9.8). The contrast changes over a great range of grey levels creating speckled boundaries, rather than the normal crisp borders (Figure 9.6). As a result the image analysis method identifies a large number of very small particles which are in fact image artefacts. Fortunately, part of the method of analysis of these images is to ignore small particles, so in most cases this effect is ignored. Regardless, this adds noise to the real data, so it is important that images are taken with a good contrast variation.

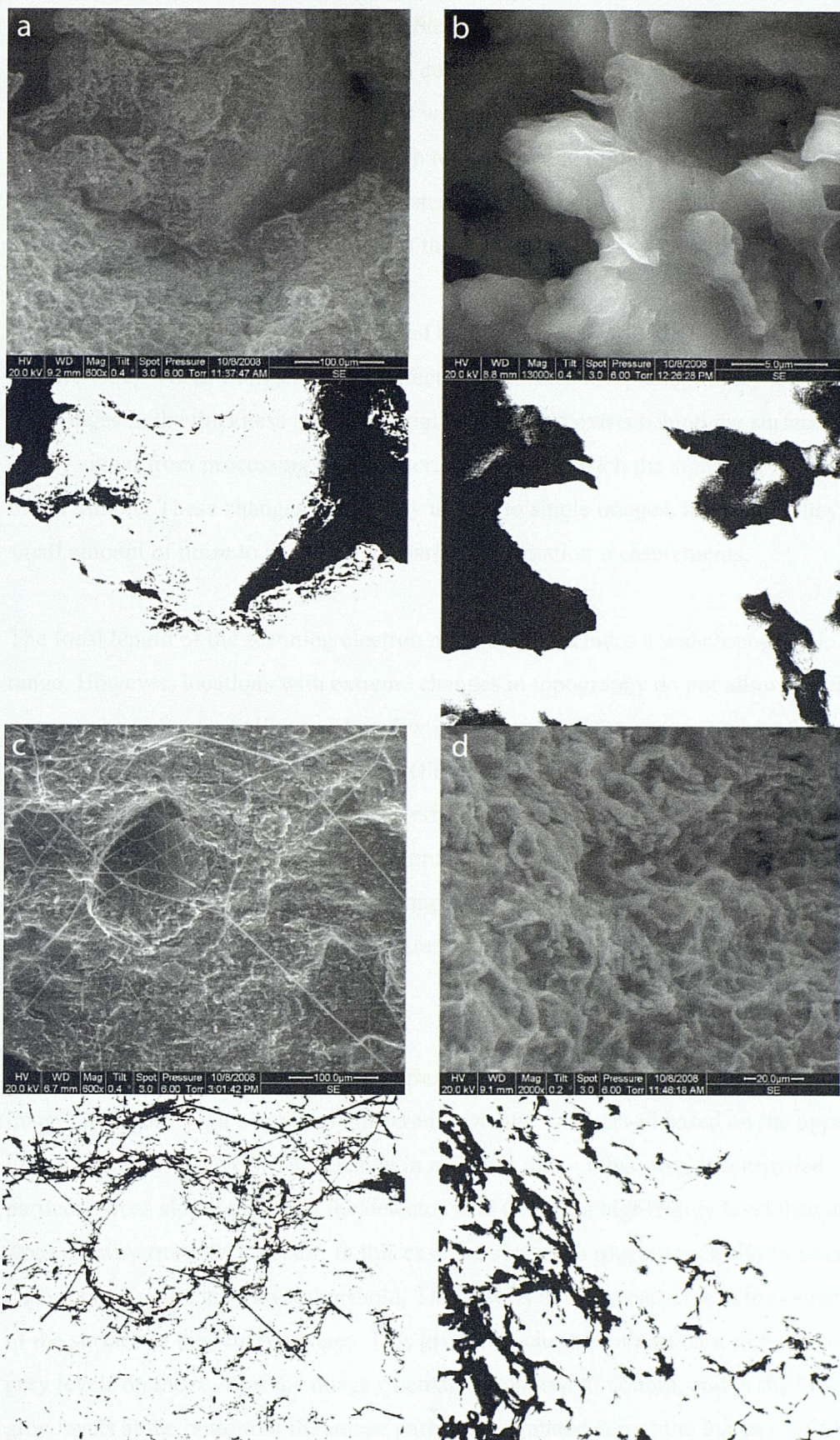


Figure 9.7 Possible errors in binary processing of SEM images, each error shown as two images, original image top, binary image bottom. Errors: large grains (a), high magnification (b) complex structures (c) sloping surfaces (d).

Whenever possible the edges of the cut block should be avoided (Figure 9.8b).

Disruption of the mudrock fabric due to cutting the sample has the potential to affect the analysis of the mudrocks. Figure 9.8 (b) was taken on the edge of a montage of 16 images. Even in this case particles which touch the edge of the image are ignored in the measurement process. As long as the material is not disturbed, then the measurements taken will not be affected by imaging of the edge.

Biological differences within the material cause changes in the grey level of the image (Figure 9.8c). These changes are not affected by surface morphology, they are caused by changes in the thickness in the material when a void exists behind the surface. The binary signal from processing these materials does not match the signal for the majority of the sample. These changes are usually unique to single images. In general, they add a small amount of noise to the preferred particle orientation measurements.

The focal length of the scanning electron microscope includes a wide topographic range. However, locations with extreme changes in topography do not allow the entire image to be in focus at all times. In a few images parts of the image are out of focus because of extreme surface topography (Figure 9.8d). In Figure 9.8d the top left and right of the image are out of focus. The effect of out of focus areas is to spread out and lower the grey level of areas. This is normally not a problem as it only affects a small proportion of images. Poorly focused images, where out of focus areas include a higher proportion of the image, would have a great effect on the structure measured on individual images.

The threshold selected can also have a great effect on the interpretation of a single image. In Figure 9.9 a completely different structure is observed based on the upper and lower 25% of grey levels. This change in results is due, in this case, to a rounded particle. Areas sloping towards the detector tend to have a higher grey level than areas sloping away from the detector. In this case the surface is rough which allows many changes across the grey level threshold. This results in many particles in the orientation of the surface in the binary images. This gives two clusters of particles, in the upper grey levels on the right of the image orientated from top to bottom, and in the lower grey levels at the bottom of the image particles orientated across the image resulting in the dual orientation signals.

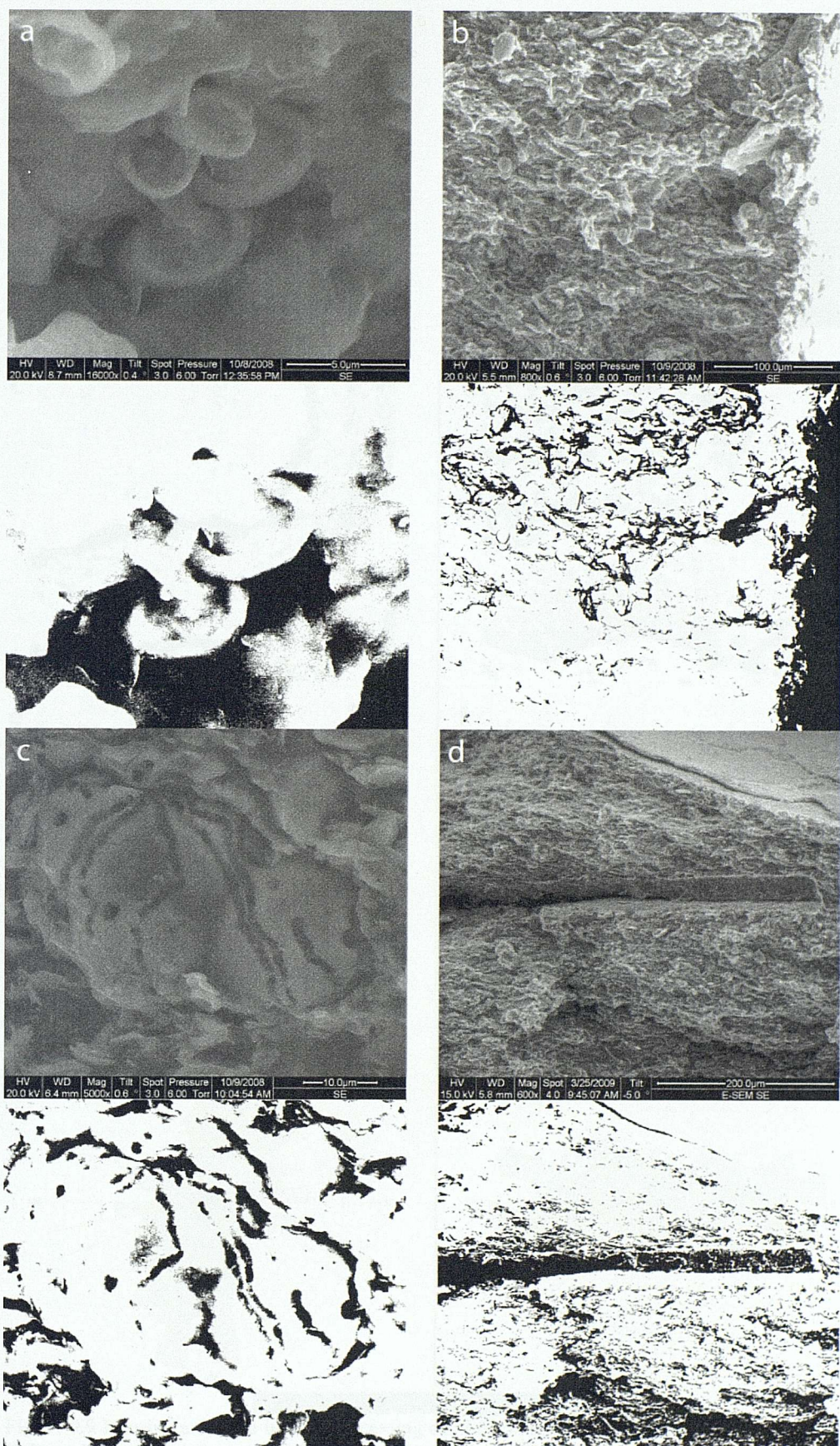


Figure 9.8 Possible errors in binary processing of SEM images, each error shown as two images, original image top, binary image bottom. Errors: low image contrast (a), edge of sample (b), compositional changes (c), loss of focus due to large topographic variations (d).

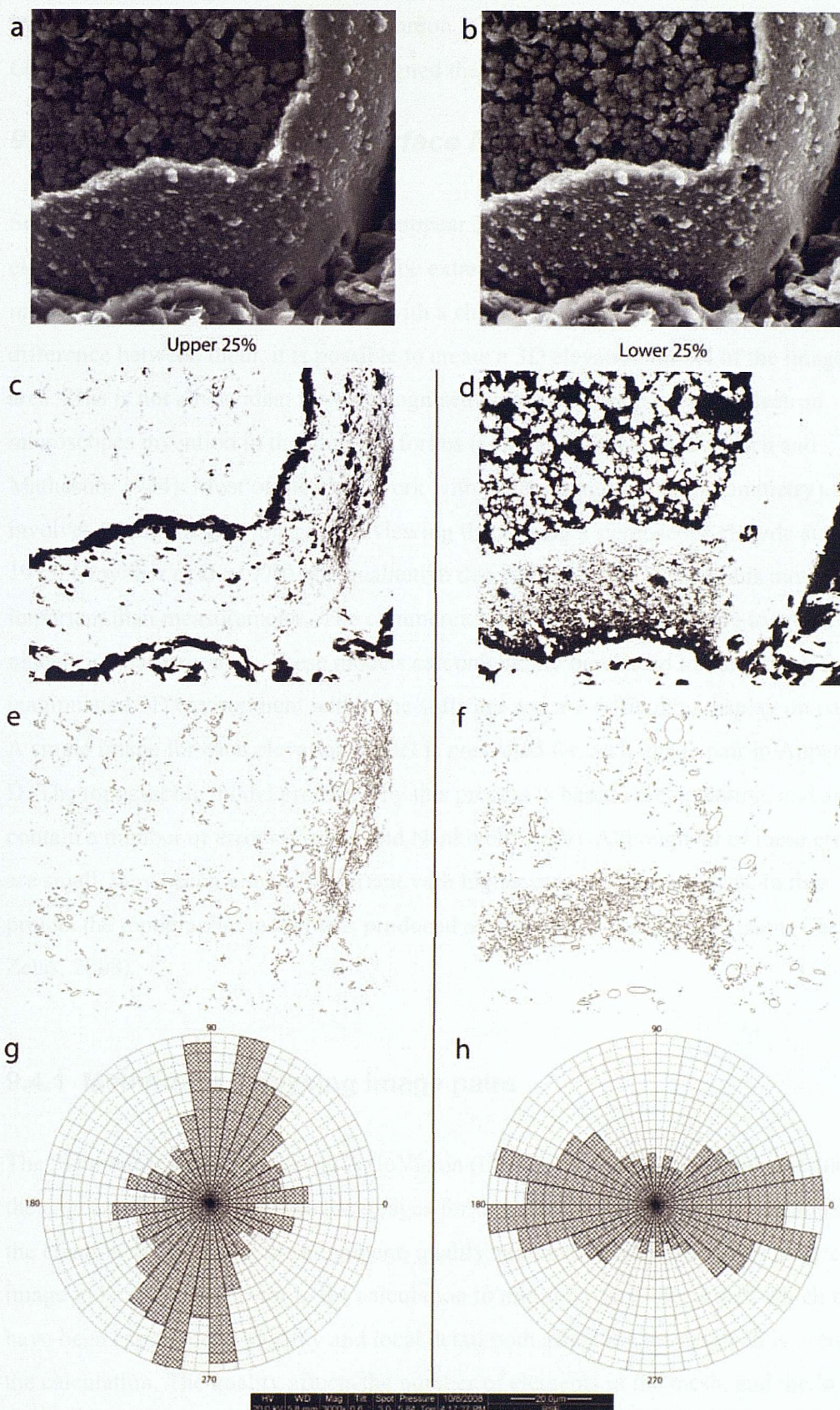


Figure 9.9 Possible errors in binary processing of SEM images. Both the rose diagrams of the upper (left) and lower (right) quartile of grey levels must show the same structure for it to be true preferred particle orientation. Original (a) and equalised (b) image. Upper quartile binary process in the left column and lower quartile in the right. From top to bottom, binary image (c & d), ellipses used to model particles (e & f), and rose diagram of orientations of long axes of ellipses (g & h).

Images of features such as this are common in the Gault Clay. These are part of the Gault Clays structure which is less aligned than many other mudrocks.

9.4 *Three dimensional surface imaging and analysis*

Scanning electron microscope images appear 3D because of the large focal length of the electron beam. However, 3D data can be extracted from a single image. When two images are taken of a single location with a change in the tilt of the stage as the difference between them, it is possible to create a 3D elevation model of the imaged area. This is not a new idea; it was recognised only a few years after the electron microscopes invention in the nineteen forties (Gotthardt, 1942; Heidenreich and Matheson, 1944). Most of the older work with stereo imaging (photogrammetry), involves taking the two images and viewing them using a stereoscope (Boyde and Ross, 1975; Crowther et al., 1970), the qualitative description of the 3D image is more important than measurements. The commercial development of software to create (3D) models is relatively new. These models can only really be viewed in the virtual, freely manipulated 3D environment within the software and are difficult to display on paper. A single image for each elevation model is presented for each image pair in Appendix D. The topographic model produced by this process is based on calculation, and as such, contain a number of errors (Garrod and Nankivell, 1958). Although all of these errors are small, they become more important with higher magnification images. In this project the topographic model was produced automatically using AxioVision (Carl Zeiss, 2008).

9.4.1 Method for analysing image pairs

The 3D models are created using AxioVision (Carl Zeiss, 2008). AxioVision requires the angular difference between the images for this calculation. The options available for the creation of the model are alignment, quality and local detail. Alignment allowed the image to be re-aligned prior to the calculation to minimize any lateral drift which may have been experienced. Quality and local detail both affect the mesh which is used for the calculation. The quality affects the number of elements in the mesh, and the local detail parameter affects the refinement of the calculated model.

Once AxioVision has calculated the model it can output this data as a height map, an anaglyph or a 2D projection of the 3D model. The 3D model can be a mesh, a surface or a surface with the original image texture superimposed on top. AxioVision can also output elevation cross sections of the model and several standard measurements of surface roughness (Appendix D).

Macros for AxioVision (Sheppard, 2008) produced a numerical output of the topographic model (X, Y, Z coordinates for each pixel). This output allowed further analysis of the 3D data. Three programs were written in Java to analyse these data.

1. Gradient map

Every non-edge pixel is surrounded by eight other pixels (Figure 9.10). Gradients are calculated from the central pixel to each of the surrounding pixels. The distance between adjacent pixels is the pixel size. The distance between the central pixel and a diagonal pixel is the pixel size multiplied by the square root of two.

Eight positive gradients can be calculated for each pixel (either towards or away from the central pixel). The maximum gradient is selected and saved. The gradient can be calculated as difference in height multiplied by pixel size which produces a scale of gradients from zero to plus infinity. For ease of partitioning the gradient is calculated as an angle (zero to ninety degrees). The inverse tangent of the difference in height divided by the pixel size (the angle of dip), is used.

Ignoring the edge pixels of the original image a matrix of gradients is created two cells smaller than the original image on all sides. The gradients can then be plotted as an image by assigning a colour scale (Hinsley, 2008) which is spread across the zero to ninety degree values (Figure 9.11). Linear coloured areas indicate material sloping perpendicular to the orientation of the line. If these linear patterns are repeated across the image then a preferred orientation is present.

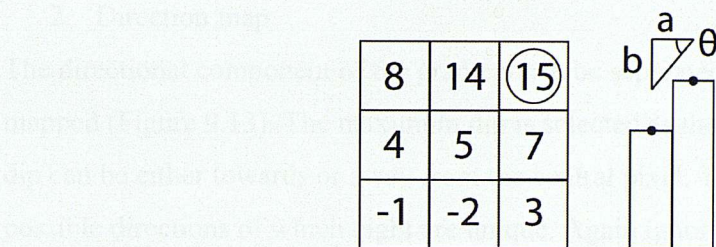


Figure 9.10 The calculation of gradient from a pixel matrix. Numbers indicate the height of each pixel, circle equals maximum gradient. Using a pixel size of 1 a gradient of $\sim 82^\circ$ can be calculated

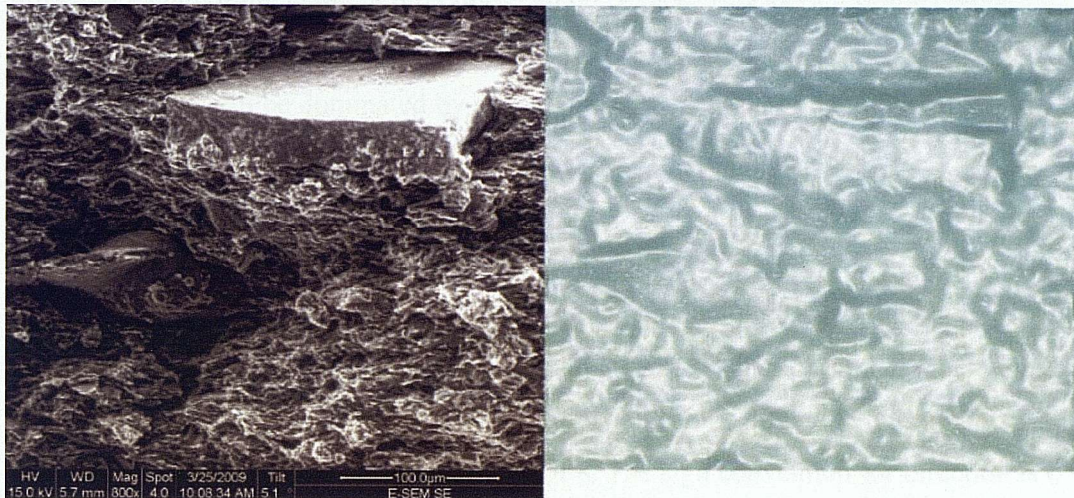


Figure 9.11 Original image: vertical surface from the Oxford Clay (left) and gradient map produced from the image (right). Darker blue equals higher gradient.

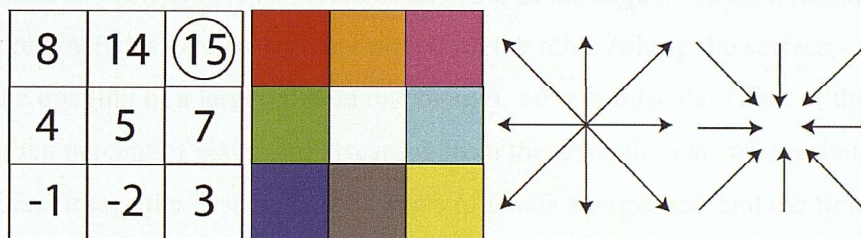


Figure 9.12 Calculation of direction of greatest dip of the 16 possible directions in this example 15 towards 5 is the greatest gradient, which is indicated by dark blue

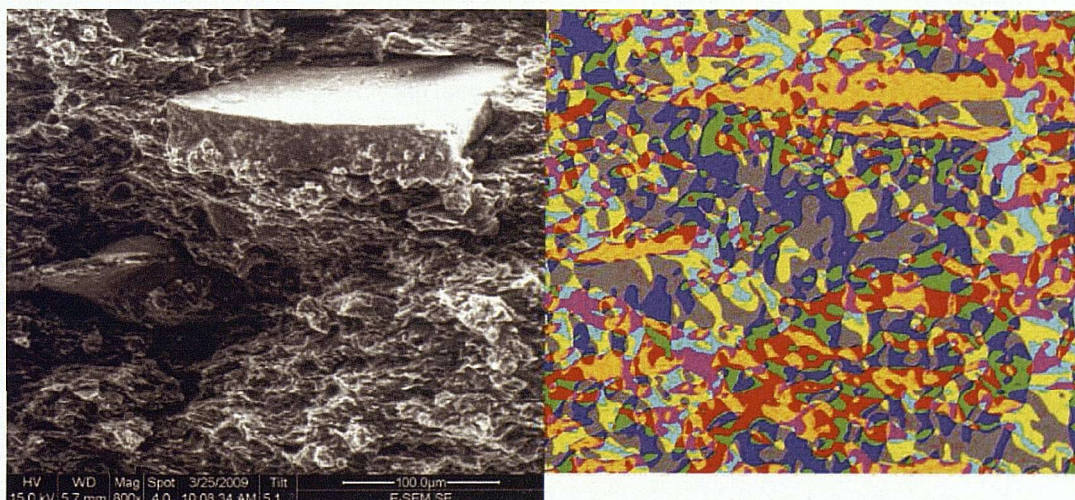


Figure 9.13 Original image: vertical surface from the Oxford Clay (left) and direction map produced from the image (right). Colours based on directions (Figure 9.12)

2. Direction map

The directional component of the gradient can be separated out (Figure 9.12) and mapped (Figure 9.13). The maximum dip is selected as the direction for the map. This dip can be either towards or away from the central pixel. There are therefore sixteen possible directions of which eight are unique. Again ignoring the edge pixels of the original image a matrix of directions can be created. By assigning a colour to each direction a map of the gradient directions can be created. Blocks of a single colour indicate a dominant dip direction in an area. Where an alignment is present the direction image will be dominated by six colours and the other two will be rare or absent.

3. Surface analysis

Combining the gradient map and the direction map produces a list of gradients with associated directions. By categorising the gradients into 5° divisions the frequency of pixels being attributed a given gradient and direction can be plotted. By using a 9×9 pixel grid 32 directions can be used instead of 8. The sign convention for direction is 0° = positive x-axis, 90° = positive y-axis. This provides a much clearer indication of the surface structure (Figure 9.14). Errors often occur at the edge of images; because one image often includes data that are not present in the other (tilting the surface sometimes allows the imaging of a larger area in one image). To minimise the effect of these errors, the outer ten percent of pixels are discarded from the analysis. This means that in a 1024 by 800 pixel image the first and last 80 rows of pixels are ignored, and the first and last 102 pixels of each row are also ignored. As a 9×9 grid of pixels is used, pixel 107 of row 85 is the first to be analysed.

On the resultant graph a planar surface in any orientation will result in a single peak, while surfaces which are ridged due to preferred particle orientations will have two peaks, and surface with no structure will have no specific peaks (a similar number of pixels dipping in all directions) (Figure 9.14).

Gradient map, direction map and surface analysis are all methods of qualitatively analysing the structure of three dimensional surfaces. Here they are used to confirm the results of the 2D analysis (Section 11.2). They also represent a step towards the full quantitative description of surface roughness.

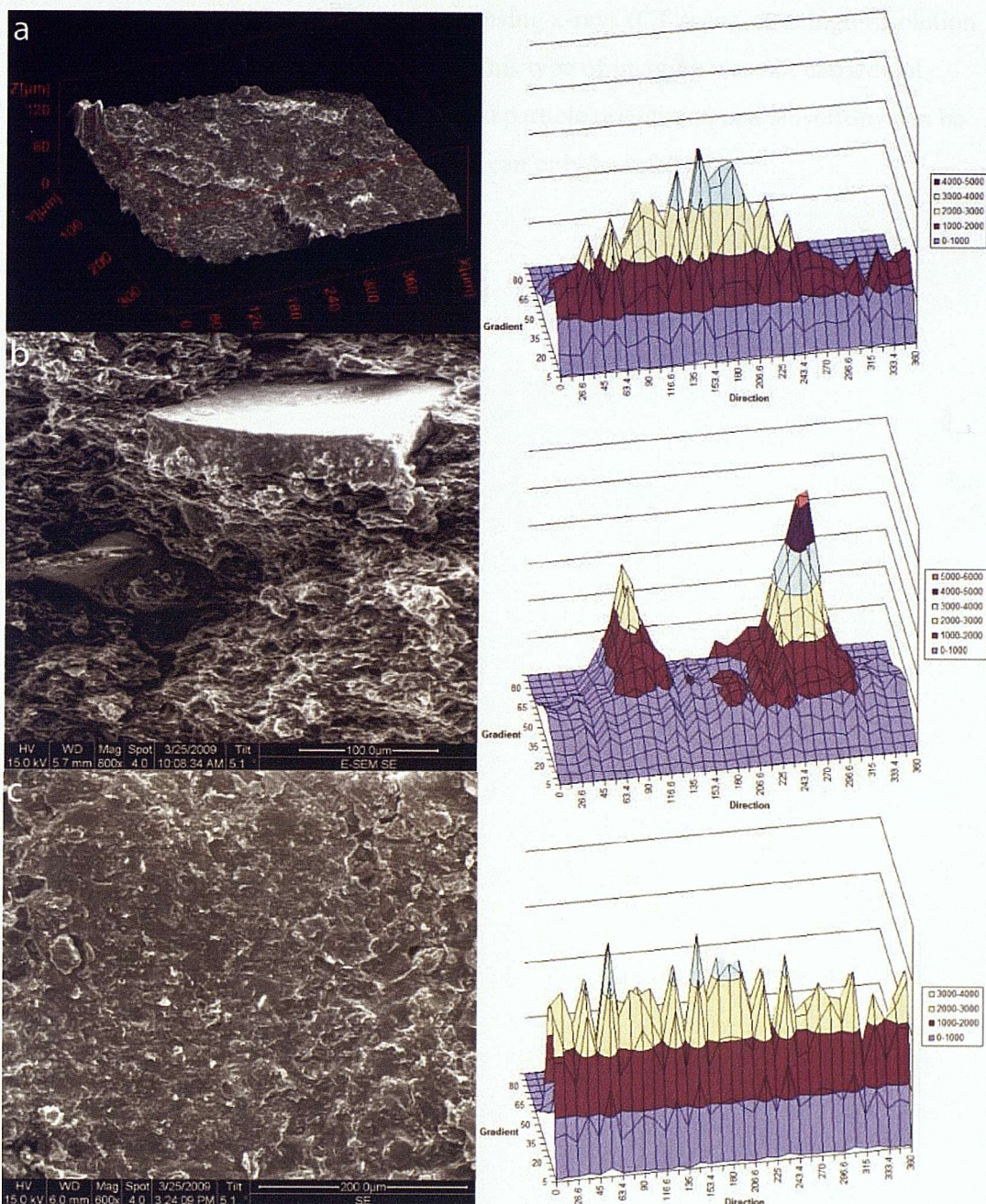


Figure 9.14 Surface structure plots, maximum gradient against direction of maximum gradient. a) Horizontal broken surface Gault Clay showing sloping surface. b) Vertical broken surface of Oxford Clay, showing a strong preferred particle orientation. c) Vertical broken surface within the Gault Clay showing no preferred orientation.

For a full particle scale analysis of mudrock behaviour a 3D block of material has to be imaged. This is possible at low resolution using x-rays (CT scan), or at high resolution using a focused ion beam (Appendix C). This type of imaging was not carried out during this project, therefore only preferred particle orientation and anisotropy can be quantified. Structure caused by cementing can only be inferred.

CHAPTER 10

MICROSTRUCTURE DESCRIPTION AND COMPARISONS

Thirty one different samples of UK mudrocks were imaged in order to examine their microstructure (Table 10-1). Samples were acquired from both trial pit (blocks) and borehole (core) investigations (Chapters 5-8). Multiple depths are imaged from borehole samples to investigate the variability of the mudrocks with stratigraphy. For some samples multiples were imaged at the same depth to test repeatability. In total nearly one thousand eight hundred images have been taken and analysed. From these a further six hundred and fifty one 3D models have been created and analysed (Table 10-1).

10.1 Material description

Using these techniques in the time available it is not possible to image and analyse the entire core. Each image is a fraction of a millimetre in size; each sample is a surface approximately 10mm in length. If three samples are imaged along a core length of 14m then 13.97m of the core (99.8%) remains unobserved by the electron microscope. The structures observed in these images are neither comprehensive nor conclusive; however they are indicators of the mudrock's behaviour. They can be understood in the context of the stratigraphy of each mudrock (Chapters 5-8).

The changes within borehole samples can be quite extreme (Figure 10.1). As they cut across stratigraphy, changes in stratigraphic horizon, including mineralogy, fossil content, grain size/shape and microstructure can be observed. In the extreme, changes from clay to limestone are present within the Lias clays over distances of 0.5m. The main imaging horizons are top and bottom of each laboratory testing horizon (Brosse, 2011; Hosseini-Kamal, 2011). A further sample was imaged at a shallow depth in the Gault Clay borehole to allow correlation with the block samples. The imaging locations in the London Clay were based on the horizons with relatively large amounts of pre-existing laboratory testing data (De Freitas and Mannion, 2007; Gasparre et al., 2007a; Gasparre et al., 2007b; Hight et al., 2007; Kovacevic et al., 2007b; Nishimura et al., 2007).

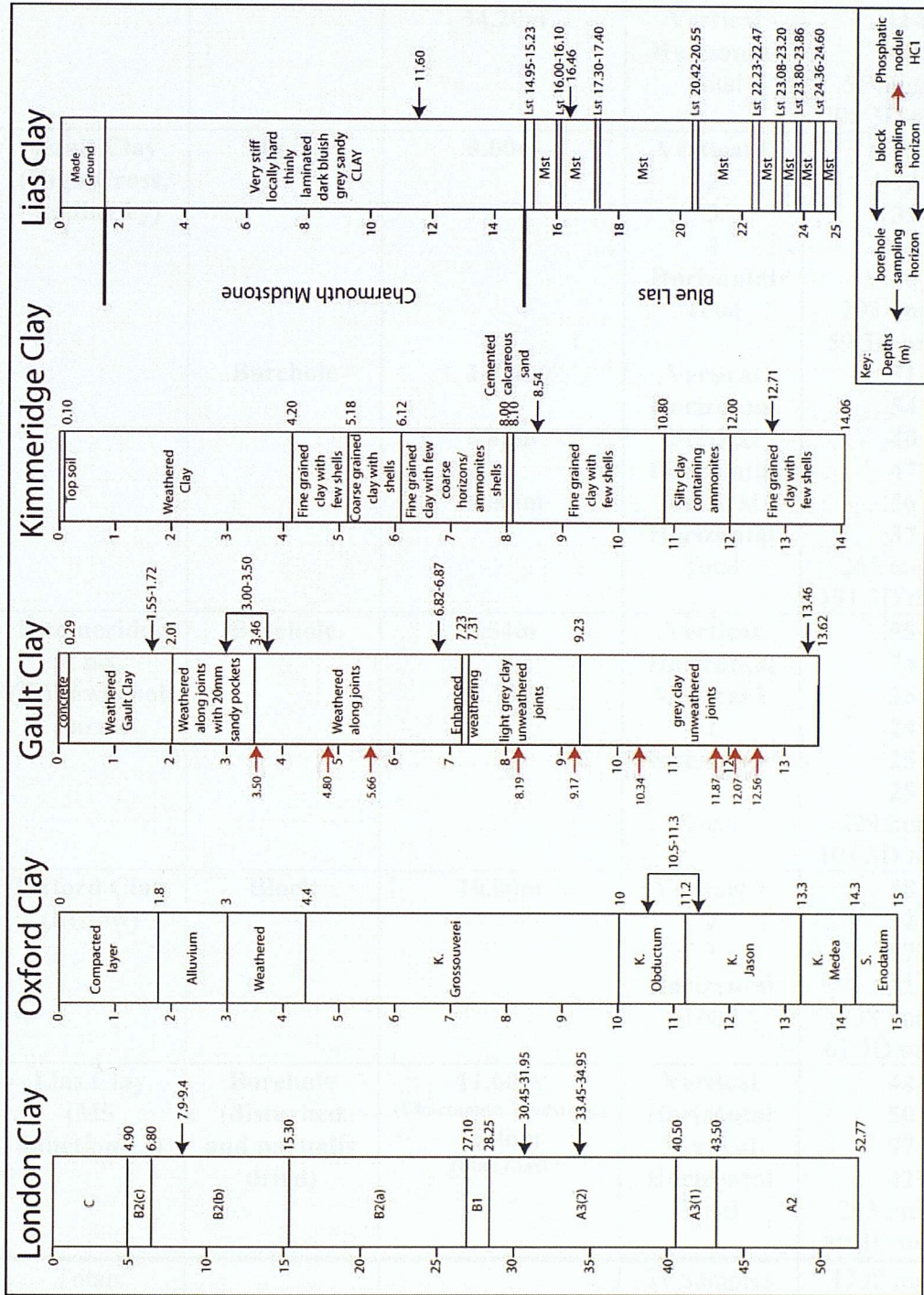


Figure 10.1 Depths of samples: Stratigraphy of London Clay from (Hight et al., 2007), Oxford Clay correlated from (Jackson and Fookes, 1974) using CPT data, Lias Clay rough on-site log (Geotechnical Engineering Limited, 2009), Gault and Kimmeridge Clay (Appendix B).

Material	Sample Type	Average Imaging depth (bgl)	Sample	Images
London Clay (Heathrow Terminal 5)	Borehole	8.65m	Vertical	74
			Horizontal	112
		31.20m	Vertical	57
			Horizontal	51
		34.20m	Vertical	114
			Horizontal	146
			Total	554 images 208 3D models
Gault Clay (High Cross, Madingley)	Block	3.00m	Vertical 1	95
			2	79
			3	39
			4	34
			Horizontal	46
			Total	293 images 59 3D models
	Borehole	1.72m	Vertical	51
			Horizontal	54
		6.82m	Vertical	40
			Horizontal	47
		13.46m	Vertical	36
			Horizontal	37
			Total	265 images 131 3D models
Kimmeridge Clay (Willowbrook Farm)	Borehole	8.54m	Vertical	55
			Horizontal	74
		12.71m	Vertical 1	26
			2	24
			Horizontal 1	25
			2	25
			Total	229 images 103 3D models
Oxford Clay (Elstow)	Block	10.00m	Vertical 1	48
			2	52
			3	97
			Horizontal	41
			Total	238 images 61 3D models
Lias Clay (M5 Junction 13)	Borehole (disturbed and partially dried)	11.60m (Charmouth Mudstone)	Vertical	44
		16.46m (Blue Lias)	Horizontal	50
			Vertical	77
			Horizontal	42
			Total	213 images 89 3D models
Totals			31 Samples	1792 images 651 3D models

Table 10-1 Mudrock samples imaged (Figure 10.1), at each depth images were taken of horizontal and vertical sections of the mudrock, the fabric of the block samples of Oxford and Gault Clay were imaged multiple times as these were the first samples taken. Bgl = below ground level. These images are presented in Appendix D.

Due to the large number of images not all are displayed and analysed individually in this chapter. However the main features of each mudrock at each sampling depth are presented. The remaining images are included for comparison within Appendix D. The mineralogy of particles within images is difficult to identify without EDex analysis which is very time consuming. Some minerals can be identified by morphology, but overall the chemical composition of the majority of particles remains unknown.

10.1.1 The Oxford Clay

The Oxford Clay has a strong preferred alignment of clay mineral particles parallel to bedding in vertical cut sections (Figure 10.2a). Some elevated nodular areas on the surface are probably weakly cemented aggregates; even these display an overall preferred particle alignment on their surfaces. On horizontal broken surfaces (Figure 10.2b), where images are taken perpendicular to the particle alignment, the structure is seen as steps in the material's surface between the various planes (top right of Figure 10.2b). High topographic areas are often surrounded by lines representing the edges of layers (centre right of Figure 10.2b). Sometimes shell fragments are aligned with the surface (centre left of Figure 10.2b).

The Oxford Clay contains a great number of shells mainly aligned with the predominant structure. Most of the shells are of a scale many times that of the mudrock's other particles. They act as horizontally aligned reinforcing elements. In a few cases shells are found out of alignment (Figure 10.2c). Often shells are separated from the surrounding material by a small gap. This probably formed during uplift of the mudrock. A gap can exist on either side of the shell or in the case of fossils composed of two shells can have an inter shell gap. The surface onto which the shell is pressed during burial becomes a mould which is easily seen on horizontal broken surfaces (Figure 10.2d). These moulds have the same shape as the shell, this creates a zone of weakness. Broken surfaces created for imaging often part along these planes. Their mode of formation may be due to clay surrounding the shell swelling more than the shell during uplift forming a local tensile stress on the shell surface. To create a vertical break in the mudrock shells must be broken, whereas a horizontal break may separate particles without breaking them. Thus horizontal breaks form more readily than vertical (Burland et al., 1977).

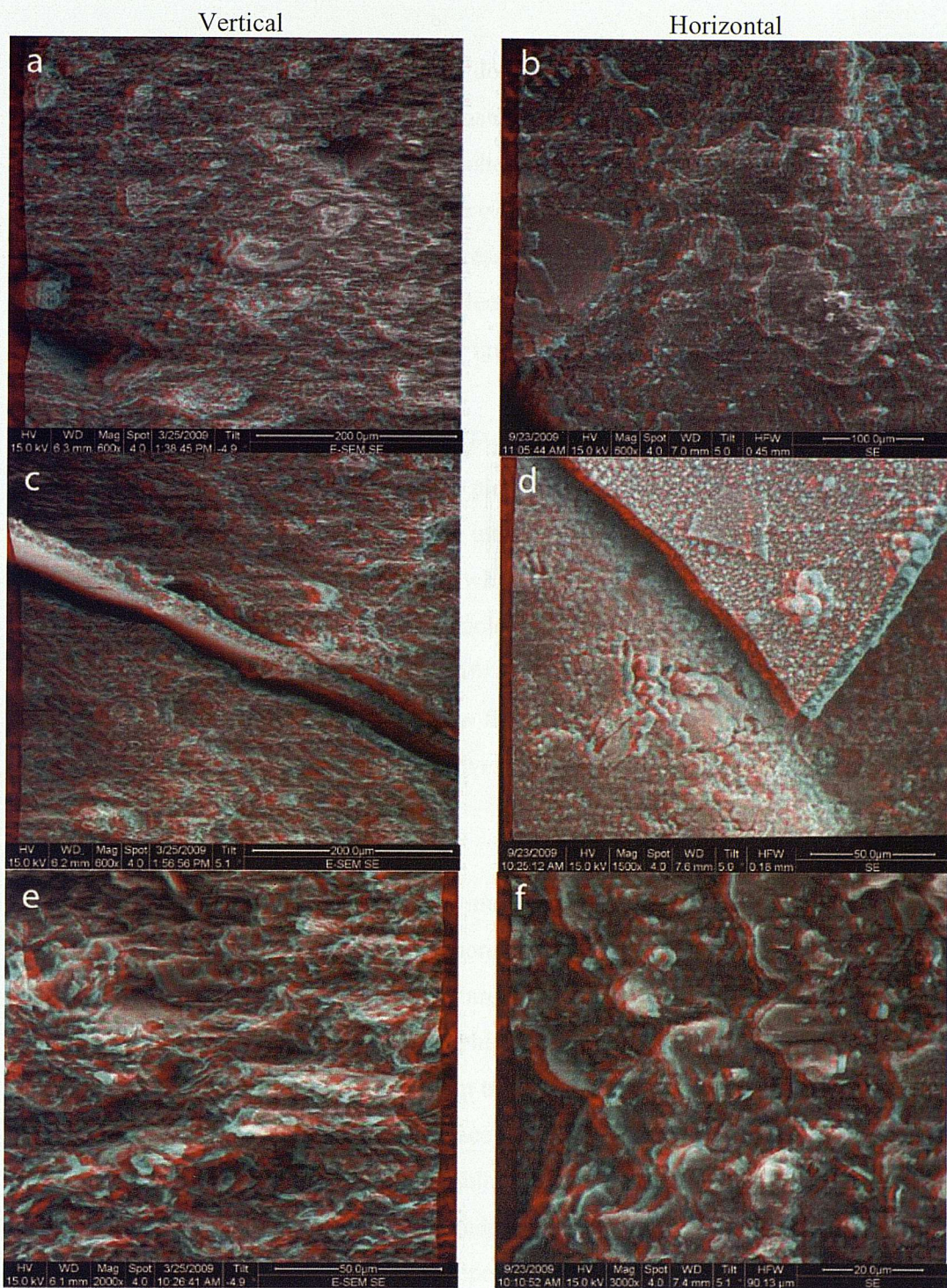


Figure 10.2 Oxford Clay block sample vertical break (a, c, e) horizontal break (b, d, f). Strong preferred orientation of platy particles (a) flaking, and flat shelved topography (b) shells acting as reinforcing elements (c, d) high magnification particle interactions (e, f). A triclinic crystal can be seen above and to the right of image f, possibly feldspar.

High magnification images of the material do not allow the delineation of individual particles, however distinct regions of the material are observed (Figure 10.2e).

Aggregated particles often show internal coherence and they tend to have a higher elevation than the average of the surface (*e.g.*, centre of Figure 10.2 (e)). Surrounding material may drape over the top of these areas. The inter-particle space appears to be empty voids (Figure 10.2 e), it could be filled with a mineral cement, or it may be filled with a finer grained/amorphous material (Figure 10.2f).

In all of the images of horizontal sections (Figure 10.2), the particles observed appear to have a more granular habit, rather than the platy shapes seen in the vertical sections. This is because aligned platy particles will intersect the vertical plane more often than with the horizontal. The granular material which dominates images of horizontal broken surfaces must be present between clay particles. Some stepping between planes in the material is observed. It is possible to identify some individual particles, on this broken surface, although most are subsumed below finer grained material. Several particles can be identified as small microfossils. A relatively large crystal with triclinic habit is possibly feldspar (Figure 10.2f).

By using a 4 by 4 montage image grid (16 images) the preferred orientation of the mudrock and the effects of shells can be more clearly seen (Figure 10.3). This image is approximately 1.2mm across. Three shells are present on the surface: One at the very top of the image is flat and has no topographic expression (Figure 10.3 S1). The next one down is slightly curved and has a slight topographic expression (Figure 10.3 S2). The one at the base has an undulating surface and marks a large change in the relief of the surface (Figure 10.3 S3). The more regular the occurrence of these shells, the more important they become in governing the mudrocks structure. Their disarticulated nature may indicate a reworking of the original deposited material. Overall shells increase the anisotropy of the Oxford Clay. This anisotropy has a greater effect at the macro scale, as shells are often found in high concentrations on specific layers of the mudrock. These layers occur either due to short breaks in sedimentation, during which animals continue to die, or to climatic/environmental changes to which a certain species cannot adapt. Both these processes can cause many shells to be represented on a specific layer. This alignment of multiple shells on a layer promotes horizontal slip on this horizon (Burland et al., 1977).



Figure 10.3 Montage of a grid of sixteen SEM images taken of the surface of the Oxford Clay. S1, S2 and S3 = shells. Scale bar bottom right image, approximately 1.2mm across image.

Strong ribs on a shell (Figure 10.4a) can act to lock a shell into its mould only allowing limited movement. The shell in Figure 10.4 (a) is also associated with a very large change in surface elevation. Several other thick shell fragments and microfossils (Figure 10.4b) are present as larger particles within the matrix. These particles may act to concentrate stress and as such they are likely to behave differently to an applied stress than the surrounding clay minerals matrix.

Pyritic framboids (Figure 10.4, c) are present in great numbers in the Oxford Clay. In some cases these framboids are in close association with the shells. This may be a result of enhanced fluid flow along the open shell boundaries, and the space around shells encouraging diagenetic mineralisation.

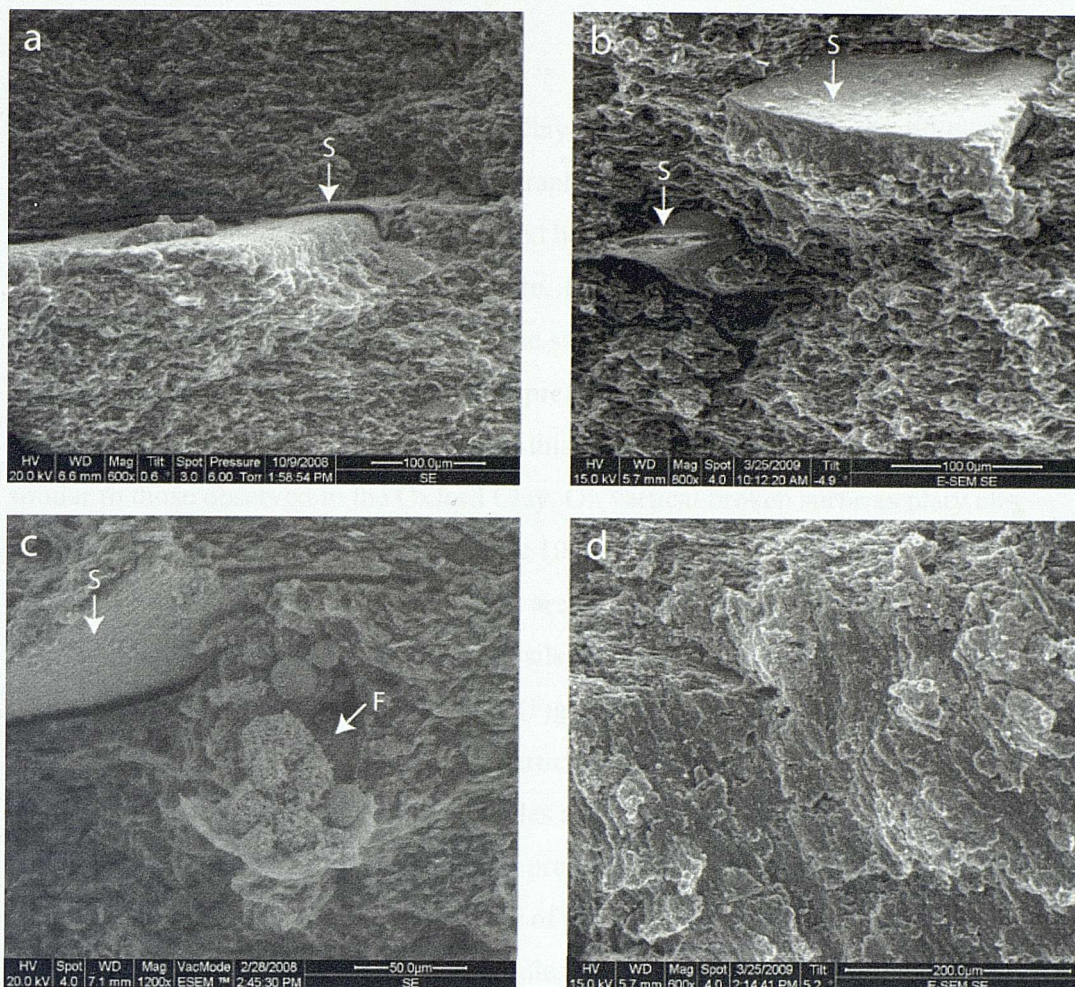


Figure 10.4 Oxford Clay, block sample: vertical break (a, b, c, d). Probably the most important feature of the Oxford Clay is its many shells (S). Some of the shells have ribs (a) which reduce the potential for there surface being a nucleating point for slip. Microfossils and shell fragments (b) are particles in the mudrock which have the potential to break up areas of preferred orientation. Pyritic particles in the form of framboids (F) are common through the fabric (c). Possible indications of movement (d) have been observed in one of the samples.

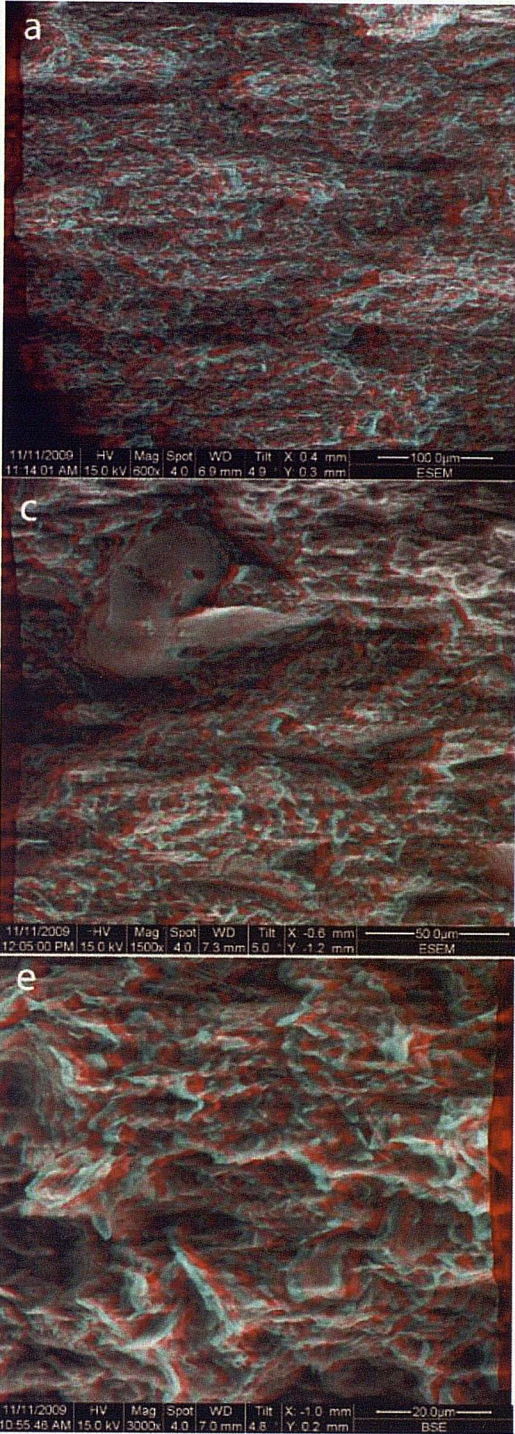
Field investigations show that joints at the macro scale are rare in the Oxford Clay.

Therefore the imaging of such a feature should be a rare occurrence. However, during sample preparation since broken surfaces preferentially split along pre-existing lines of weakness, it is possible that a joint could be selected. One possible joint has been identified. The top of Figure 10.4 (d) shows that away from this feature the structure of the material is horizontally aligned as it is elsewhere in the mudrock. In the area around the joint there is a slightly polished surface to the material. Striations on the surface suggest previous displacement.

10.1.2 The Gault Clay

In contrast to the Oxford Clay, the Gault Clay has a large amount of variability. This is in part due to the greater number of stratigraphic horizons studied (Table 10-1), and the degree of weathering of some of the imaged horizons. The upper part of the intact Gault Clay has a reasonably strong microstructure. This structure is disrupted in the near surface by recent processes at several of the sampling horizons. The Gault Clay at 6.82m bgl at High Cross has the strongest preferred particle orientation. The material appears to have been the least disturbed at this horizon. The structures observed are similar to those observed in the Oxford Clay. On vertical broken surfaces platy clay particles appear aligned to bedding (Figure 10.5 a). Some elongate ridges and troughs are present; the long axes of these features are aligned with the long axes of the particles. There are several small microfossils (characteristic of the Gault Clay) on the surface. In the horizontal broken surfaces (Figure 10.5 b) steps between levels are observed. There are indications of platy particle edges on sloping surfaces, and indications of lifting of the edges of particles away from the surface. All of these features are indicators of the presence of a preferred particle orientation. The Gault Clay at the macro scale contains a large number of phosphatic and pyritic nodules; at the micro scale these are present as micro nodules (granular area upper centre of Figure 10.5 b). The horizontal surfaces of the mudrock are covered with microfossils, granular crystals and shell fragments. These indicate a similar mode of deposition to that of the Oxford Clay. Bedding parallel shells, like those in the Oxford Clay do not form a major part of the Gault Clay at any horizon studied in this work. The biological life observed is dominated by fauna with rounded shells, mostly microfossils or coccoliths and other planktonic organisms (Figure 10.5 c). In the Oxford Clay the shells reinforced the mudrock's natural structure because of their flat planar shape, however in the Gault Clay the shells are mainly rounded, so they disrupt the preferred particle orientation. Platy particles drape over the top of microfossils instead of remaining bedding parallel. The tests (rounded shell surrounding the animal inside) of microfossils tend to be intact and in many cases remain unfilled. They form the major proportion of the void space of the Gault Clay. Lack of connectivity leads to a high porosity with a low permeability (Bachman, 1984). Rounded microfossils intersect the surface of the material (Figure 10.5 d), elongate fossils are often aligned. In Figure 10.5 (d) a trough in the surface appears to be caused by a microfossil. These shells are locally strong elements.

Vertical



Horizontal

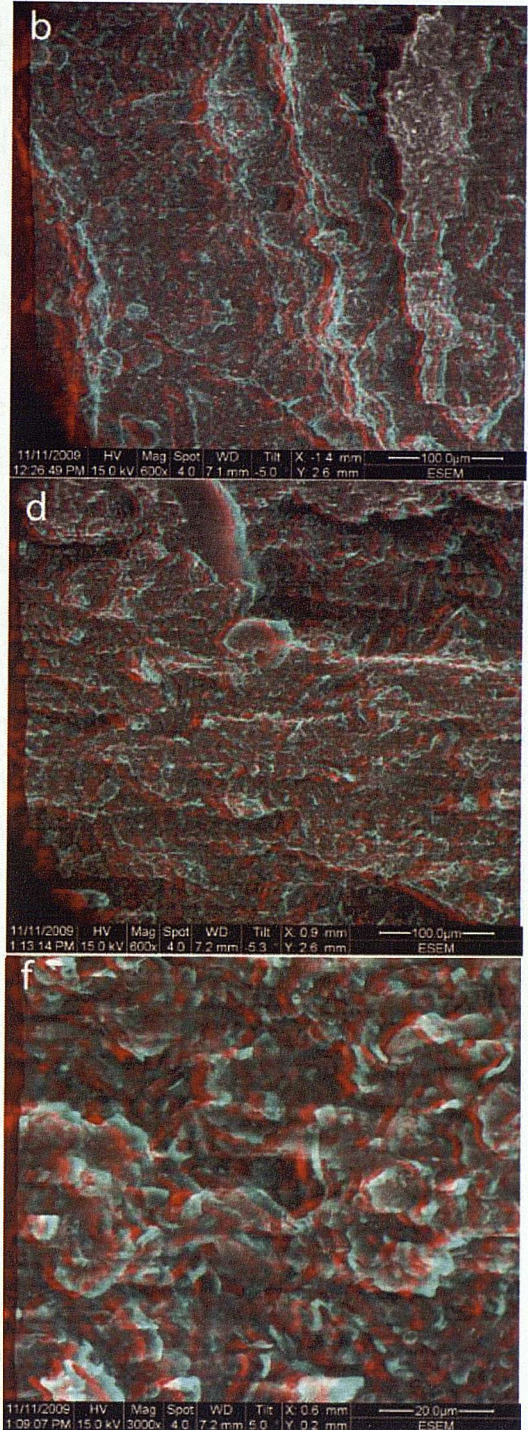


Figure 10.5 Gault Clay borehole sample 6.82m vertical break (a, c, e) horizontal break (b, d, f). Preferred particle orientation (a), flaking and flat areas of surface (b), shell interaction with fabric (c, d), high magnification images (e, f).

High magnification images are filled with a great number of planktonic microfossils (Figure 10.5 e, f). These particles (most are annular, some bisected), account for a large quantity of the small-scale particles in the mudrock. As individual fossils are small they do not disrupt the fabric of the platy particles to the extent of the larger microfossils (Figure 10.5 e). In larger numbers they can have a greater effect (Figure 10.12). Although many fossils are visible in the vertical plane they are not normally aligned with the fabric. A greater number of these fossils are visible on horizontal broken surfaces (Figure 10.5 f). Much of the surface is covered with an unresolved material, reducing the visible details of the fossils. The unresolved material may be the same as the fine grained/amorphous material observed in the Oxford Clay.

The Gault Clay sample, from a depth of 1.72m bgl, is chemically weathered. This could be identified in the field as a yellow discolouration to the sample. Although studying the effects of weathering was not originally an objective of this project, some conclusions about micro structural changes due to weathering may be drawn from images of the Gault Clay. The main aim in taking this shallow sample was to compare material from the same stratigraphic horizon as the block samples. Due to changes in ground level between the two sampling sites, the stratigraphic horizon of the borehole sample was shallower than the block samples. More extensive chemical weathering is observed in the borehole samples. At the micro scale, weathering causes a great reduction in the degree of the mudrock's preferred particle orientation. Some of the observed vertical surfaces at this level are almost completely flat (Figure 10.6 a). This may be due to the preferential selection of joint surfaces in this orientation, as there are a large number of vertical joints (desiccation features) observed in the near surface. Although Figure 10.6 (a) has little topography, scattered crystals, shell fragments and microfossils are visible within the unresolved material covering the surface. Some ridges of fine grained material are also observed. Images of horizontal breaks in the surface show possible indications of a preferred particle orientation parallel to the surface orientation (Figure 10.6 b). Linear troughs orientated from top to the bottom of Figure 10.6 (b) show particle edge terminations, and some shell fragments appear aligned with this orientation. These indications are much weaker than the structure seen at greater depth.

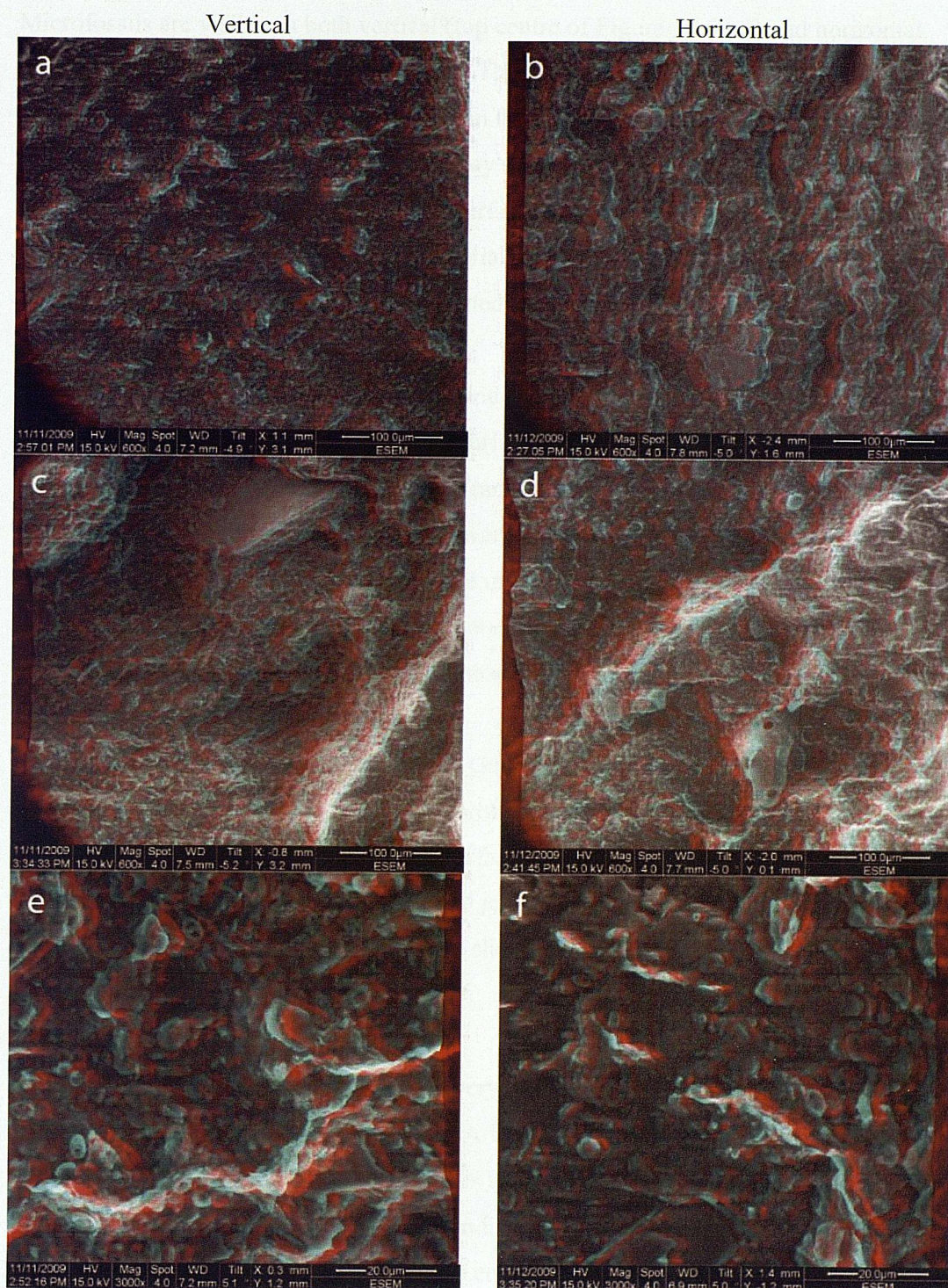


Figure 10.6 Gault Clay borehole sample 1.72m vertical break (a, c, e) horizontal break (b, d, f) weathered sample, no preferred particle orientation observed (a & b), most shells are rounded instead of flat (c & d), high magnification images with many microfossils (e & f).

Microfossils are visible in both vertical (top centre of Figure 10.6 (c)) and horizontal (bottom centre of Figure 10.6 (d)) images. The vertical image contains a sub-linear high area; possibly a vein of material harder than the surrounding area. This is probably a calcite vein. There does not appear to be any strongly preferred orientation to this sample. However, the horizontal broken surface, as before, shows weak indications of structure. A step in the surface of the material does show platy particles intersecting the sloping surface indicating particles orientated perpendicular to the direction of viewing.

At high magnification images on vertical and horizontal sections do not show significant variation. The majority of the surfaces are covered by a very fine grained/amorphous material (Figure 10.6 e & f). Granular material/tiny microfossils and shell fragments are visible on the surface, however no preferred orientations can be measured from these particles. Ridges of fine grained material are present across the surface and in some cases small particles/microfossils are entrained within the ridges (Figure 10.6 e). These ridges are more prevalent on vertical surfaces.

The images from the block samples of the Gault Clay, taken from 3m bgl, show a large range of variability. Most of the vertical broken surfaces show little or no preferred orientation. However horizontal broken surfaces show a strong preferred orientation. As vertical breaks preferentially select vertical joint surfaces, they sample the structured materials less often. The horizontal breaks show the true preferred orientation of the intact material.

Most of the images of vertical cuts show very little topography (Figure 10.7 a). Few features are clearly visible on the surface: A few fossils can be seen where they poke above the broken surface. In other instances where some topography is present on the surface (Figure 10.7 c), there is still little indication of a preferred particle orientation. Even this rugged topography appears to be composed of very fine grained material, with some small ridges on the surface.

Images of horizontal breaks in the material show a very different structure. There is clear evidence of preferred particle orientation perpendicular to the direction of imaging (Figure 10.7 b). In Figure 10.7 (b) separation of material from the surface is observed (delamination).

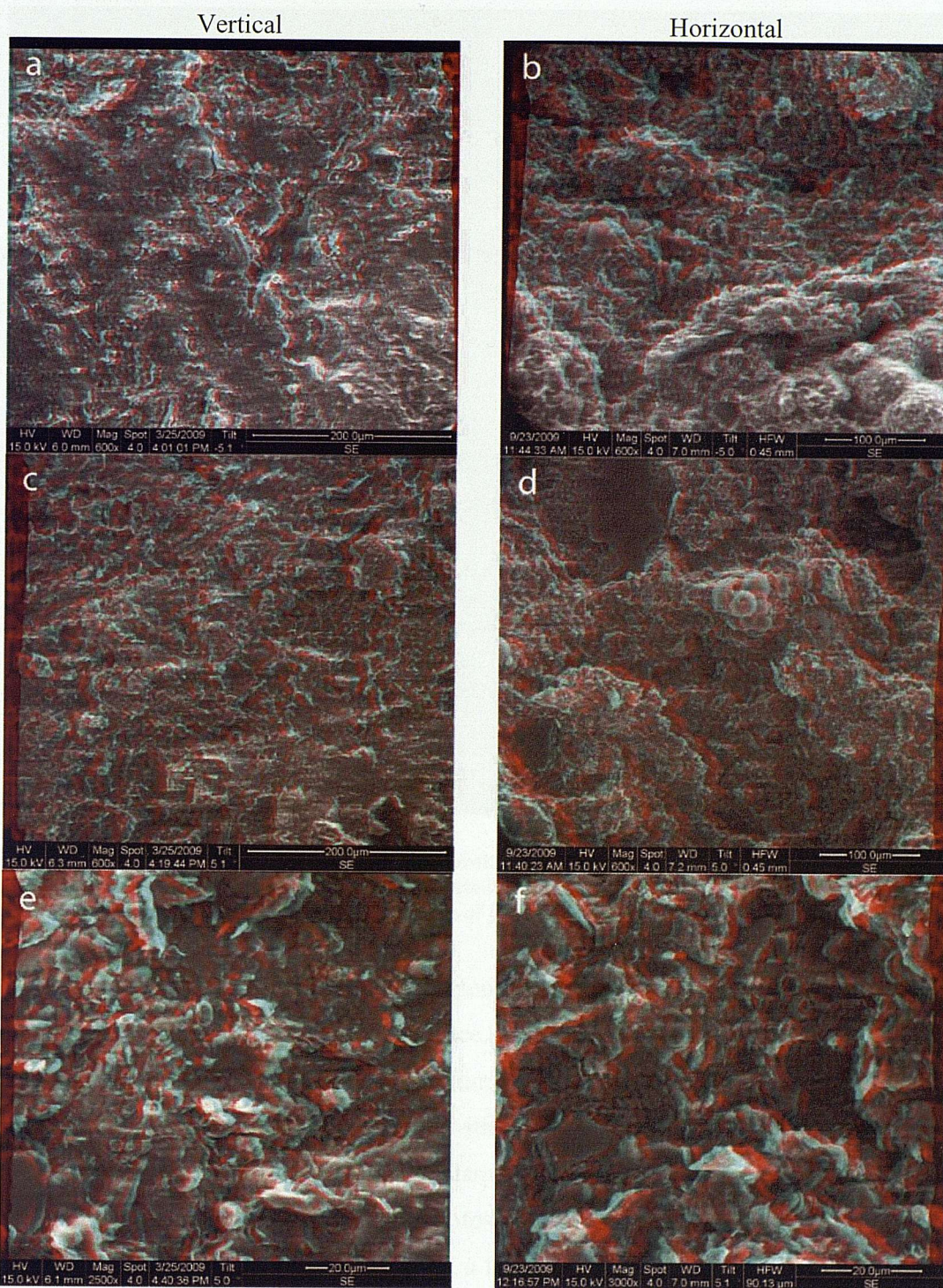


Figure 10.7 Gault Clay block sample vertical break (a, c, e). Horizontal break (b, d, f). Sample weathered near joints, very little preferred orientation is observed in the images of the Gault Clay. Many of the surfaces do not show significant topography (a), although some show ridges on the surface (c). Some of the horizontal surfaces show large amounts of topography, some indications of structure perpendicular to the image (b), lifting of part of the surface further indicating horizontal structure fossil bottom right. Microfossils, bedding parallel shells (d). High magnification images with a large number of microfossils in the fine grained matrix (e, f).



Figure 10.8 Montage of 16 images of a vertical broken surface taken from a block sample of Gault Clay. This samples shows a preferred particle orientation. It shows the particle orientations in an area which has not been altered by the presence of joints.

The mudrock has broken along a layer perpendicular to the direction of imaging and the action of the other side of the broken surface being lifted away has caused part of the surface to rise away from its previous position. Another indication of preferred orientation is the intersection of particles with the sloping surface. The top of Figure 10.7 (b) is not where it should be due to a large processing error in the creation of the 3D model by AxioVision. There is a microfossil in the bottom right of Figure 10.7 (b). Large flat shells are rare in the Gault Clay, a few are present on horizontal surfaces aligned with the parallel to the surface (Figure 10.7 d). There are also two more examples of delamination of the surface in Figure 10.7 (d).

At high magnification the mudrock appears very similar in both vertical and horizontal images (Figure 10.7 e, f). Numerous microfossils and shell fragments are present on the surfaces. Neither surface shows any obvious signs of a fabric within the mudrock; the vertical because there is no visible fabric on the surface and the horizontal because of

the high magnification. The vertical surface does seem to contain a larger amount of the fine grained amorphous material (possibly smectite) and the horizontal surface seems to have a greater amount of topography, although these factors are variable across the surface.

Some broken surfaces in the Gault Clay block samples do display a preferred particle orientation (Figure 10.8). The fabric in Figure 10.8, showing the Gault Clay at the large scale in a montage of images, shows a high level of alignment of platy particles. This broken surface is clearly not along a joint plane. The alignment of particles is not as strong as that seen in the Oxford Clay. Most of the fossils have rounded shells compared to the flat fossils of the Oxford Clay. They make up a great proportion of the particles of the Gault Clay. They act to locally disrupt the mudrock's fabric. On the left side of Figure 10.8 is an open hole supported by the shell of one of these fossils.

One of the major features observed in the vertical breaks of the shallow samples in the Gault Clay are small ridges on the surface (Figure 10.9). The majority of these ridges are formed from a very fine grained material, whose particles are too small to be resolved by SEM (Figure 10.9, d). The entrainment of some microfossils within these structures has been observed in a few cases (Figure 10.9, f). These ridges are often associated with recent fungal strands linking ridge tops (Figure 10.9, c). The ridges can stand out above the granular material (Figure 10.9, a & e) or the fine grained material forming the ridges can subsume the surrounding material such that a featureless surface remains on which the ridges are the only feature (Figure 10.9, b). In areas where large amounts of fine grained material are present next to a granular area (Figure 10.9, c) the ridges become intermittent in the granular area. These features also tend to occur in areas which are predominantly flat.

EDX analysis indicates that chemically there is very little difference between the ridges and the material on their margins (Figure 10.10). However, a chemical map of an area full of ridges showed a possible increase in oxygen on the ridges as compared to the material groundmass.

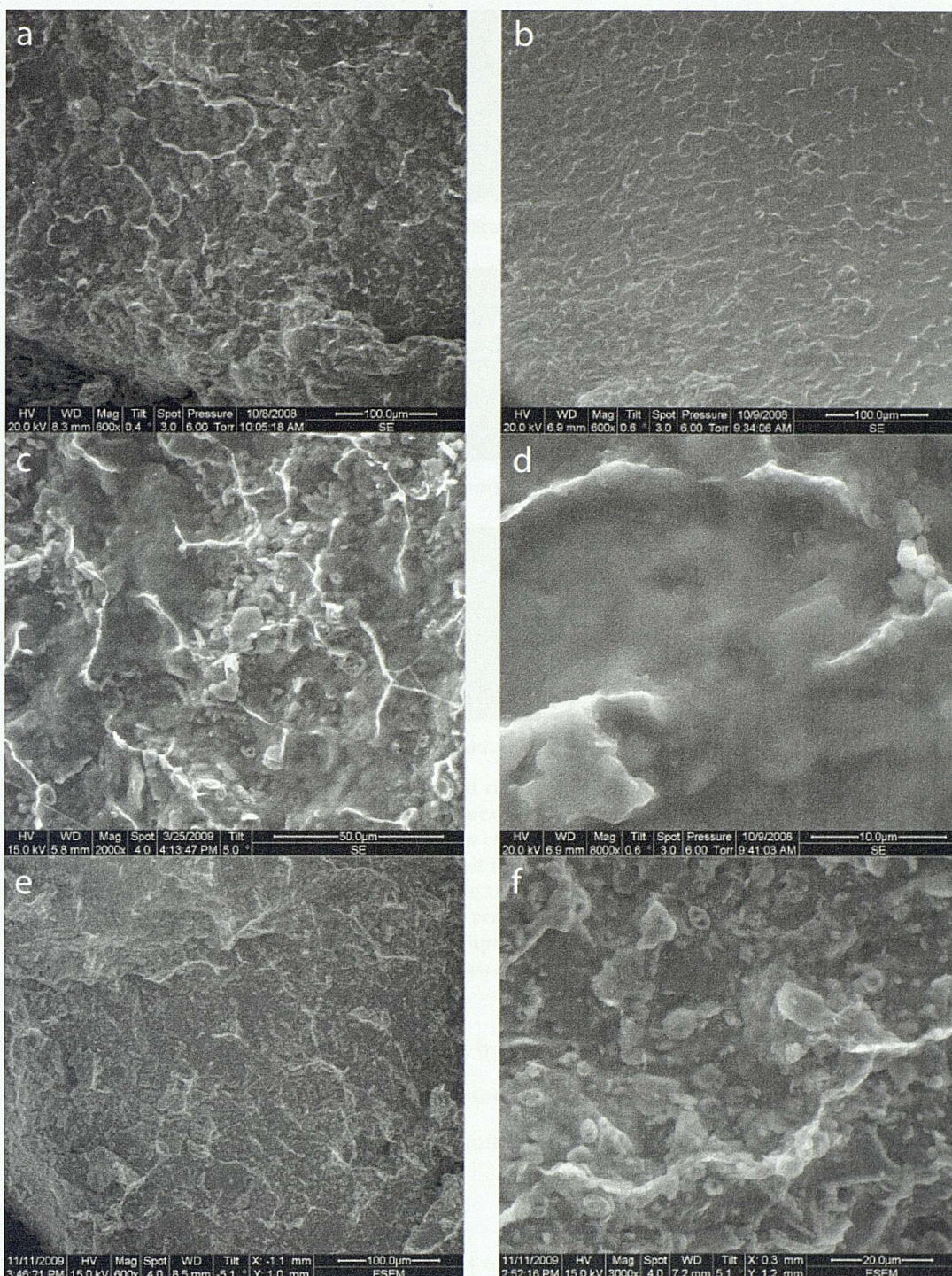


Figure 10.9 Gault Clay block samples vertical break (a, b, c, d), shallow borehole 1.72m vertical section (e, f). The ridges on the surface of the Gault Clay (light coloured areas) are a feature common to vertical samples from shallow depth. They are often related to a very fine grained material which is beyond the resolution of the SEM to pick out individual particles. Where the fine grained material is prevalent it subsumes the grains of the material (b, d) it can ride over the grains of the material (a, e, f) or it can occur in very local regions where it is completely absent a few microns away (c). Often these ridges are associated with biogenic fungal growths (surface of c) this suggests that these ridges are forming on slightly open joints, this is supported by the limited topography on many of these surfaces.

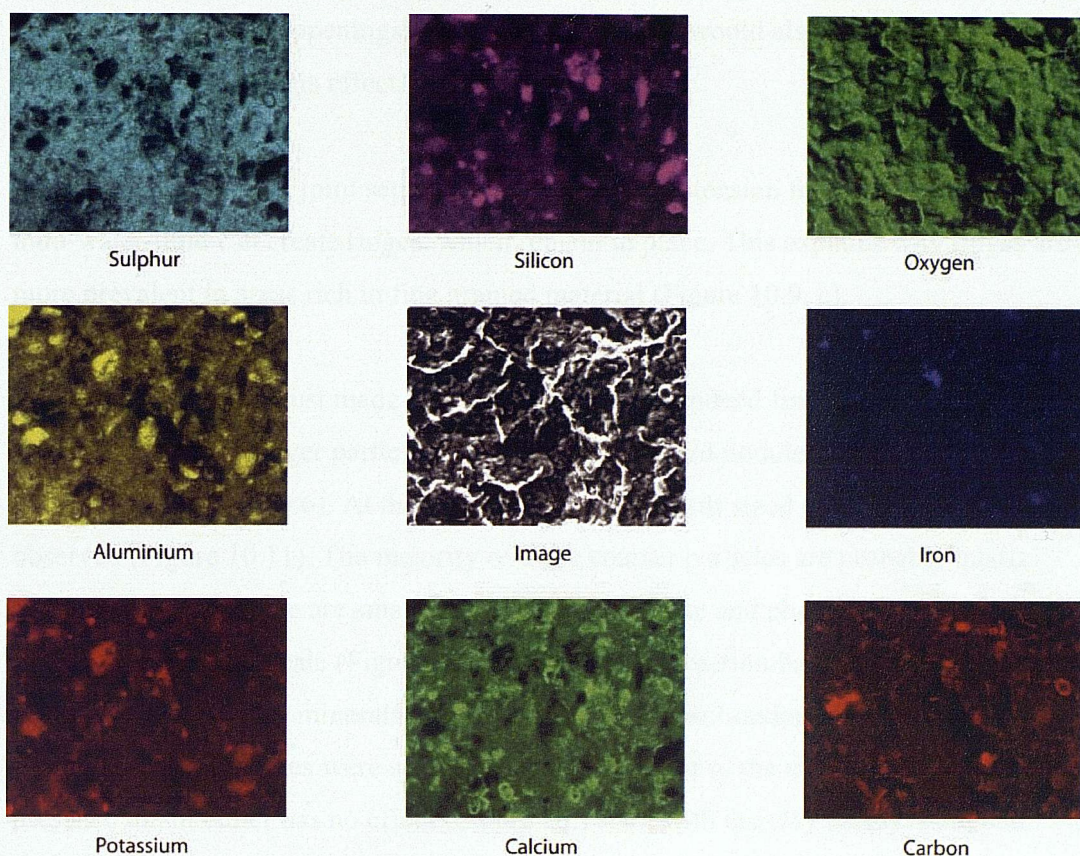


Figure 10.10 This is a chemical map of an area of ridges in the Gault Clay. Each image indicates the concentrations of a specific element in locations across the image. At first glance it appears that oxygen and carbon are highlighting the ridges on closer examination small filaments can be seen extended between the higher areas. It is suggested that these are fungal (biogenic weathering), or organic, which explains the high carbon signal on the pinnacles of the ridges but not encompassing them. Oxygen also seems to highlight the ridge areas; however this is more a function of the detection shadows behind the ridges. In general apart from a possible slight elevation in oxygen these ridges seem to be composed of a similar material to that of the groundmass (matrix) of the mudrock.

The ridges are commonly found in vertical broken samples in near-surface material. They may be related to the closely spaced dessication features observed at the field scale. The presence of biological material linking ridges supports the idea that these surfaces are open joint faces.

The pattern observed is probably a result of weathering of the mudrock. The mode of formation is possibility related to annual cycles in the groundwater regime. During summer water is removed from the ground by the trees, the roots of which are preferentially located along joints, this produces a low pore pressure in the joints compared to that of the soil, water would then be flowing from the soil to the joints. This process suggests one possible hypothesis for the formation of this structure (Figure 10.9): It is possible that this flow may drag some of the finer grained particles of the

mudrock into the joint openings. The drying of the soil would also cause the opening of the cracks increasing this effect.

As both surfaces of the joint separate at the microscale, tension forces in the viscous mud-water fluid can create ridges, which remain in place. This explains why ridges are more prevalent in areas rich in fine grained material (Figure 10.9, c).

The Gault Clay is not just made up of clay minerals or indeed fine silt sized particles, it also contains many larger particles, up to the cobble sized nodules observed at the outcrop scale (Figure 6.6). At the micro scale sand and silt sized particles are commonly observed (Figure 10.11). The majority of these coarser particles are probably quartz (Figure 10.11, a). Some are smaller versions of the pyrite and phosphatic nodules observed at the field scale (Figure 10.11, b, c). The interaction between silt-sized quartz particles and finer clay minerals has been observed in the London Clay (Dewhurst et al., 1998). The clay particles were splayed out perpendicular to the silt grains. Here the presence of silt either has no effect (Figure 10.11, c), with the clay particles aligned parallel to particle surfaces (Figure 10.11, a), or the edge of the particle can act as a detachment along which micro cracks can form (Figure 10.11, b). The nucleation of cracks on the boundary of pyritic nodules has been observed at the macro scale (Fouché et al., 2004). The alignment of clay particles parallel to silt particle boundaries (Figure 10.11, a) is likely to be a result of the stresses between the silt particles (alignment to local stress regime). Cracking at the particle boundaries is probably a result of the difference in compression properties of the soft clay and the harder nodule. The presence of these coarser particles suggests that high energy processes were occurring during the deposition of the Gault Clay. Possibly storms transported much of this coarser material further from land than usual, or debris flows transported coarser material further into the basin and mixed the different grain sizes together. A detailed study of features in the mudrock may be able to identify a sequence of events based on the relationship between the features of the Gault (nodules, joints, etc)

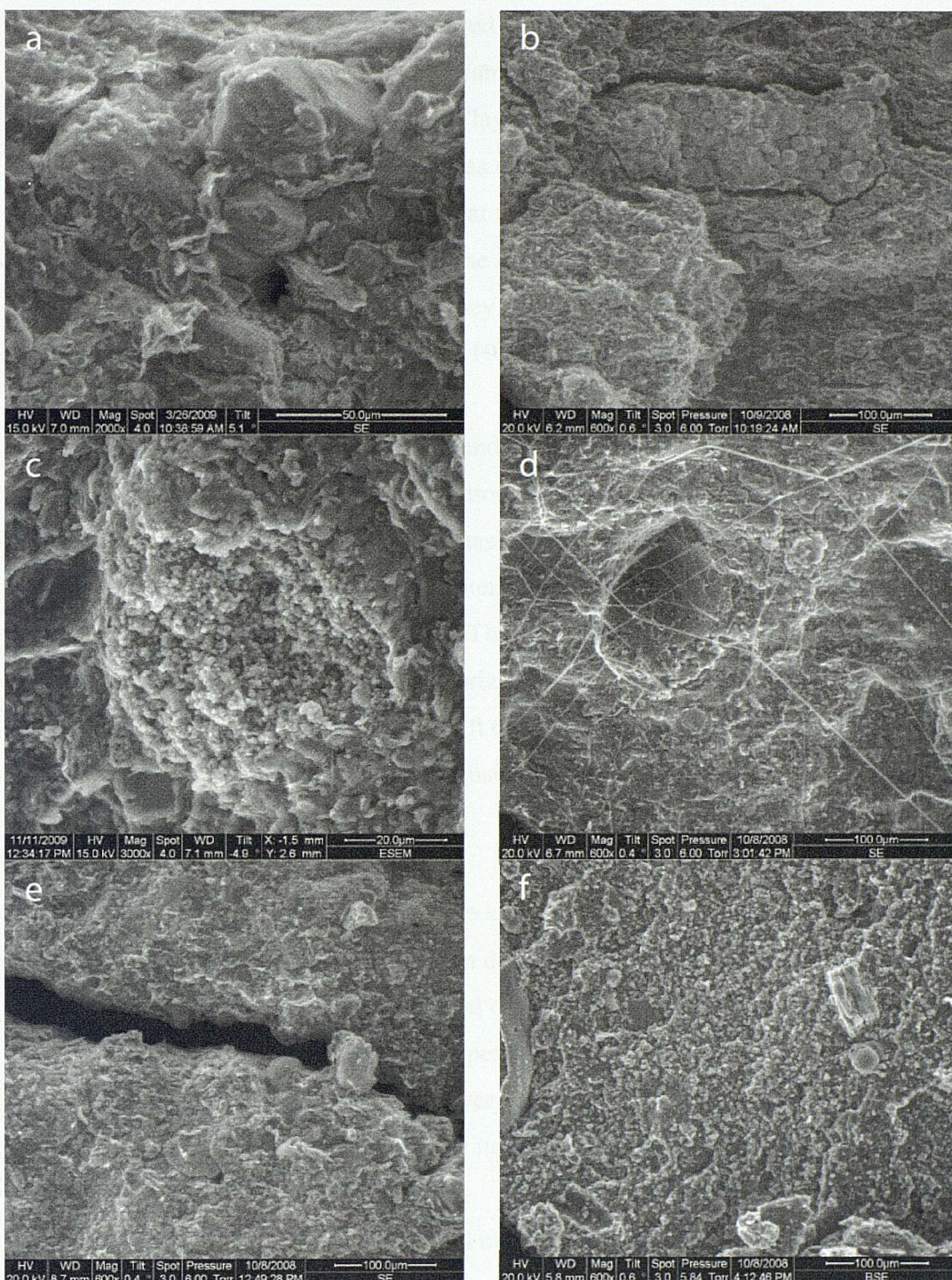


Figure 10.11 Gault Clay block sample vertical break (a, b, d, e, f) borehole 6.82m horizontal (c). Coarser than clay particles are regularly observed in the Gault Clay. Silt and sand sized particles (a, b, c) are present throughout the Gault Clay, although more regularly seen in the near surface. The phosphatic and pyritic nodules found at the field scale are also present at the micro scale (pyritic; b, phosphatic; c). Organic elements such as modern fungal filaments grown into the in-situ open joint space (d) have been observed in most of the shallow Gault Clay samples. This type of biological material can only occur in cavities in the material such as voids or open joints. Cracks (e) which are often held apart by pillars of material (not shown here, see Appendix D) are seen in the Gault Clay, these are often found in the absence of any fabric or feature. Some areas of the Gault Clay vary so much as to not contain any platy particles (f).

Recent biological material has been observed in the shallow samples, especially the block samples, because the joints in these samples are slightly open. The main type of biological material observed in the Gault Clay samples is filaments (Figure 10.11, d). This indicates that biological weathering has occurred at least to the depth of the block samples, probably confined to locations near the joint surfaces at this depth. Morphologically it is also possible that these filaments are mineral growths, such as halotrichite (Frost et al., 2007), however they are interpreted as biological due to their carbon-oxygen chemical signal (Figure 10.10).

In the Gault Clay, cracks are observed around silt and sand grains (Figure 10.11, b), and also some opening up the fabric of the mudrock, with no identifiable cause (Figure 10.11, e). Closer analysis of these cracks has revealed pillars of material present within the crack. These probably originated as material which fell into the crack, however they now appear to be holding the cracks open. These cracks are probably related to uplift/weathering, due to their generally horizontal orientation, and their general close association with hard particles (during uplift the expansion of clay particles is greater than that of the harder particles, creating tensile forces on the upper and lower hard particle-clay mineral interface).

The observed surfaces in the Gault Clay are exceptionally variable, this is further emphasised by the number of samples from different depths and locations that were imaged. This appears to be due to the lateral changes in the mudrock itself. Some of this variation is due to the weathering in the upper levels of the Gault Clay. One such unusual surface (Figure 10.11, f) shows a very fine grained granular material, with no indication of platy clay minerals and hence no preferred orientation.

At 13.46m bgl at High Cross the structured material observed at the shallower depth is not observed in the vertical broken surfaces of the material (Figure 10.12 a, c, e). The clay mineral content of the Gault Clay reduces with depth and the mineralogy changes from an illite-smectite dominated regime to illite-kaolinite dominated regime (Section 6.3). However the reduction in clay minerals between the highly structured 6.82m bgl sample (Figure 10.5) and the 13.46m bgl sample amounts to 8%. The reduction in illite and illite-smectite interlayers is 12%. The 13.46m bgl Gault Clay sample still contains 33% clay minerals. This drastic structural change is probably caused by something other than the mineralogical variation.

Vertical

Horizontal

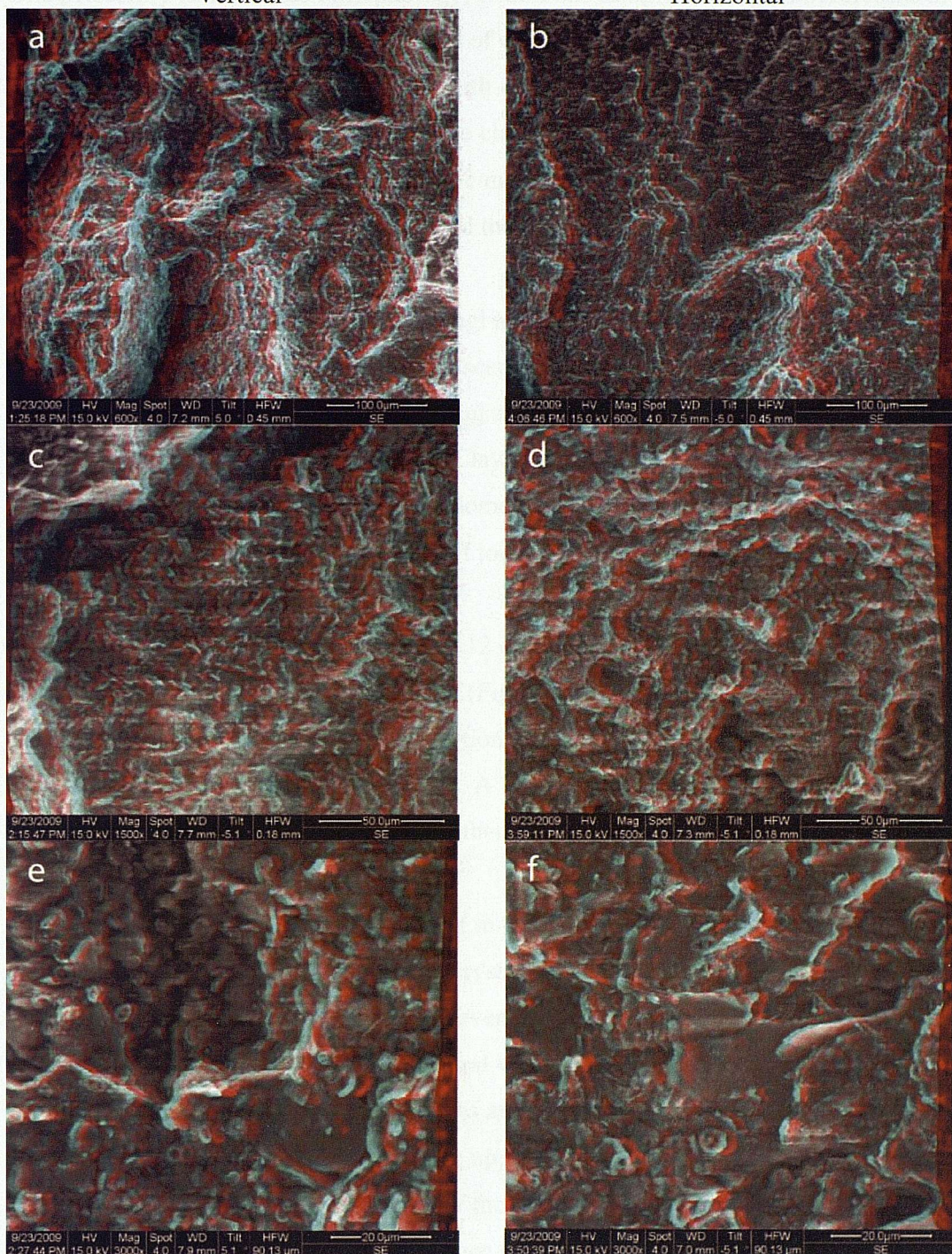


Figure 10.12 Gault Clay borehole sample 13.46m vertical break (a, c, e) horizontal break (b, d, f). No indication of preferred particle orientation on vertical surfaces (a), possible planar joint surfaces (a). Parts of the surface show ridges of fine grained material (c), the topography of the surface is different to that on shallower surfaces. The horizontal broken surfaces tend to be flat with steps between layers (b). Other locations on the surface have more rounded particles, and so are less flat (d). The high magnification images (e & f) have large numbers of coccolith sized microfossils within the finer matrix.

One possibility is microfossil morphology; within this sample the larger microfossils are rare if not absent, even though the amount of calcite in the material increases. The fine grained matrix of the material is packed with very small coccoliths (Figure 10.12 e & f). As these fossils are of a similar scale to the clay mineral crystals which formed the preferred alignment, their dominance in the material is probably enough to disrupt the orientation of clay mineral particles around them.

Most of the surfaces observed at 13.46m bgl are very irregular (Figure 10.12 a). There are rare examples of preferred orientation seen in the horizontal samples (Figure 10.12 b), such as the intersections with the sloping surface, and the possible flaking of the surface. In the field samples of the Gault Clay at this horizon holes were found in their surface which were interpreted as joints. Some features were observed at the micro-scale which are possibly the intersection of joints (Figure 10.12 a).

Some structures in the surface (Figure 10.12 c) appear similar to the ridges discussed earlier (Figure 10.9). On closer inspection (Figure 10.12 e & f) these are not ridges of fine grained material, they are the intersection of platy crystals with the surface, and clearly they show no preferred orientation. A fine grained material is present between the crystals, however it does not subsume them as in shallower samples.

The variability of structures is also present in the Gault Clay at 13.46m bgl. One area is dominated by angular particles, possibly crystals (Figure 10.12 d). These are covered with the thin veneer of the finer material, even so the morphology of this area is very different to the other images. Such structural variability is the key observation of the Gault Clay. It is seen on individual surfaces and between samples. A fairly strong preferred particle orientation is seen in the upper part of the core, its strength of expression increasing as the weathering of the material decreases. This structure then becomes absent at depth. On any one surface the structure observed appears compartmentalised or non pervasive. This makes prediction of material behaviour due to micro-features very difficult. However, the close spacing of joints at the macro scale will probably have the greatest effect on macro behaviour.

Vertical



Horizontal

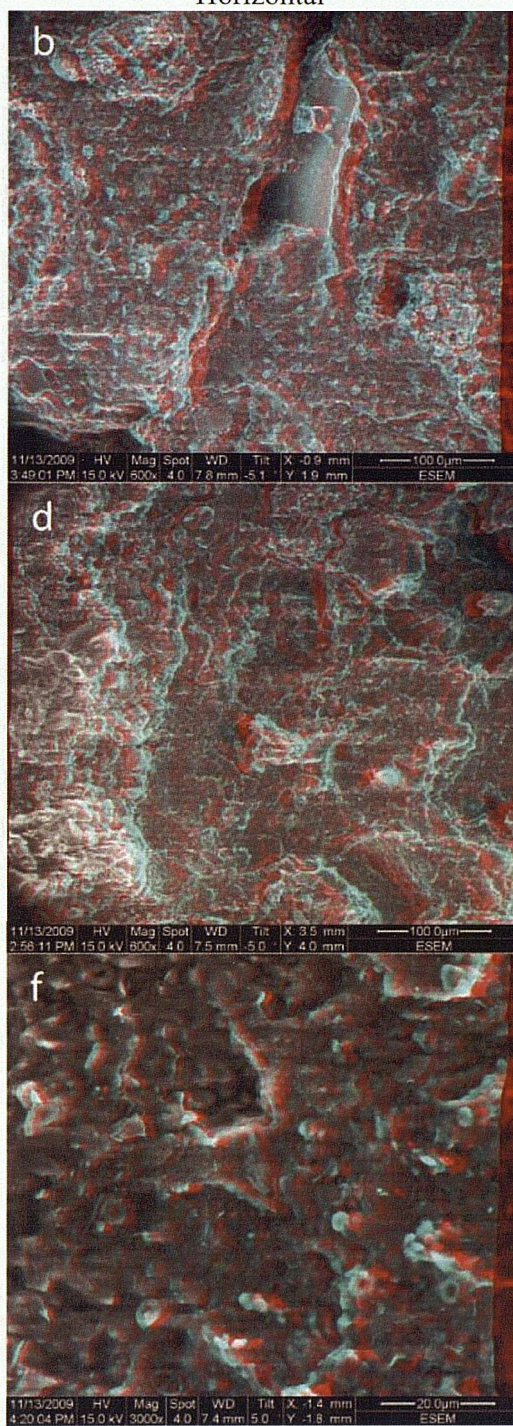


Figure 10.13 Kimmeridge Clay borehole sample 8.54m vertical break (a, c, e) horizontal break (b, d, f). The particles are orientated at an inclination to the horizontal (a), several small structures are present within the samples such as discontinuity planes (a & b). Shell fragments are present in several of the images (a, b & c). Some surfaces are encrusted with a coarse grained interlocking material (left of d). Inclined platy particles are clear at high magnifications (e). The horizontal surface shows flakes of particles, and very small microfossils covered in a fine grained material (f).

10.1.3 The Kimmeridge Clay

Many of the images taken of the Kimmeridge Clay do not show any specific preferred particle orientation. Some regions of the shallow depth Kimmeridge Clay probably have a preferred particle alignment of approximately 25° to the horizontal. However this does not correlate across all the images at that horizon. At the deeper level there is a large amount of silt present in the mudrock disrupting any fabric that might be formed by the clay minerals.

At a depth of 8.54m bgl the fabric of the mudrock is dominated by small structures (Figure 10.13 a) with two sub parallel discontinuities crossing the material's fabric. These discontinuities appear close to linear with limited surface roughness. There is a shell fragment in the bottom right corner. There is little evidence of a preferred orientation to the mudrock. Discontinuities also intersect the horizontal broken surfaces (Figure 10.13 b). Here the discontinuity follows the edge of a shell, with a small topographic step across it and some indication of particles intersecting with the sloping joint surface (possible preferred alignment). Smaller shell fragments are aligned to the broken surface. There is an area of interlocking granular material which appears to be encrusting the surface (centre right). In some places this encrusting material covers substantial areas of the surface (Figure 10.13 d). The entire left side of Figure 10.13 (d) is covered with the interlocking granular material, whereas the right side is the standard clay surface with some evidence of structure parallel to the surface. The encrusting material forms the high topography in Figure 10.13 (d).

Some images of vertical breaks from the Kimmeridge Clay show a preferred orientation of particles at $20\text{--}30^\circ$ to the horizontal (Figure 10.13 c). During core logging of the sample, silty lenses were observed in the material's surface. It is possible that the clay minerals on the edge of one of these lenses may have an inclined preferred orientation. Cross-bedding has also previously been observed within the Kimmeridge Clay (Macquaker and Bohacs, 2007; Schieber et al., 2007). Indications of terminations were observed on the cores surface. These structures are expected given the higher energy local facies from which the samples were taken.

Vertical

Horizontal

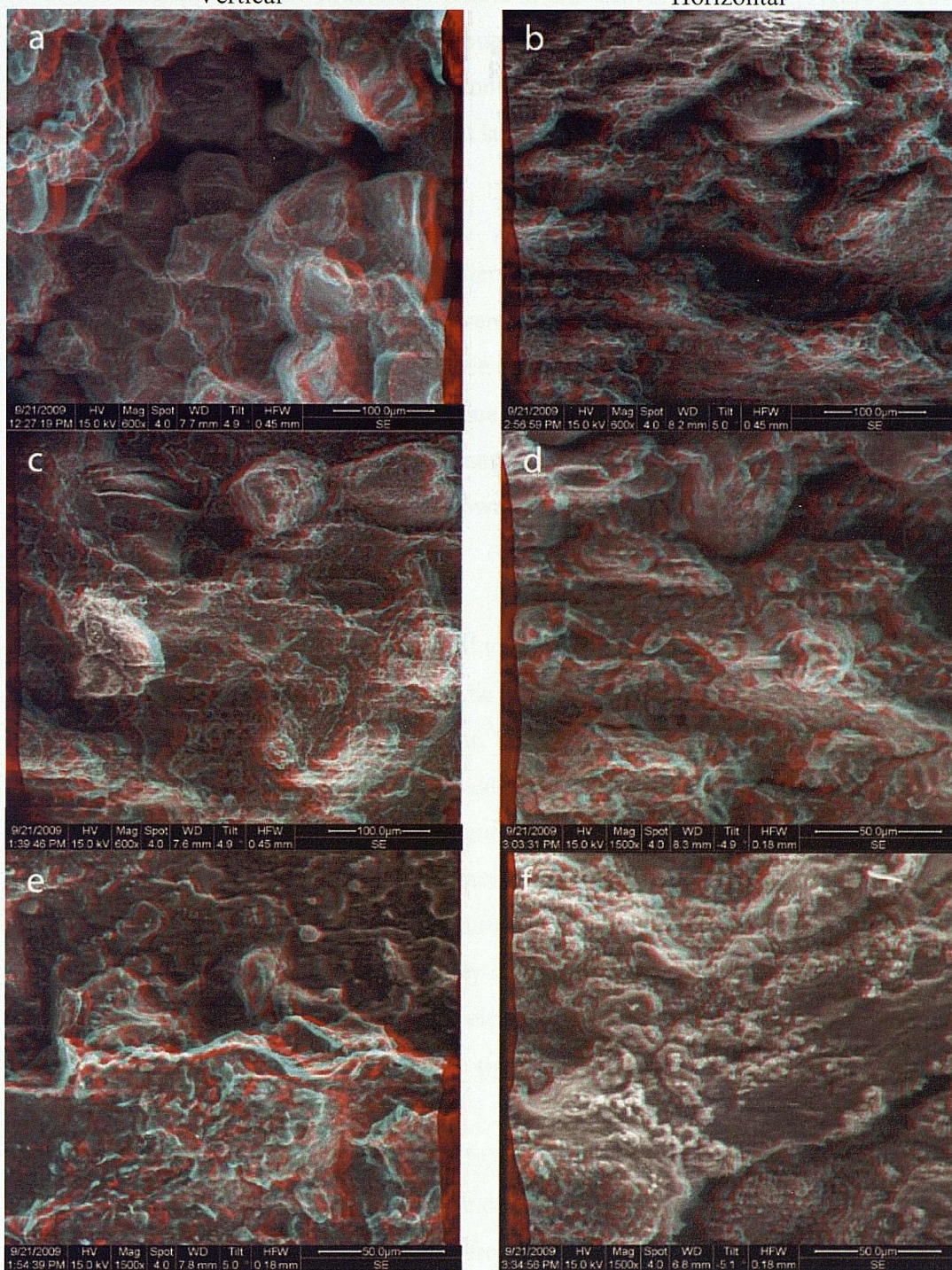


Figure 10.14 Kimmeridge Clay borehole sample 12.71m vertical break (a, c, e) horizontal break (b, d, f). The sample contains a lot of silt (a) clay minerals form a coating over the surface of the silt grains, there is also some overgrowth cements (c). Images of the horizontal surfaces do have a preferred orientation, due to cracks in the surface and ridges between silt particles (b & d). At high magnification no individual particles can be identified (e) possible particle edges intersect on the change in surface. Some pyrite crystals are present growing on the surfaces of some silt grains (f).

At higher magnification the structure is visible in the vertical cut sample (Figure 10.13 e). It does not remain parallel, it varies through about 25° a large number of platy particles are seen on the surface. The horizontal cut (Figure 10.13 f) shows microfossils and loose granular particles on a nearly flat surface which is covered with a very fine grained material.

At 12.71m bgl the Kimmeridge Clay is siltier than at the shallower horizon. The silt grains are angular (Figure 10.14 a) and covered by a thin layer of fine grainer material. This coating connects grains which are close together, however it does not fill large voids between particles. Some of these connections have a distinctive curved shape around the edge of the voids. The angular nature of the silt particles may be due to an overgrowth cement. This may then be followed by a fine clay infill transported into place by water flowing through the pores.

Silty horizons in the Kimmeridge Clay lead to permeability anisotropy. However, very little evidence of preferred orientation can be seen in these images. The mineralogy of this sample shows very limited clay content. With few platy particles no preferred particle orientation can be produced. Unusually a preferred orientation is observed on some of the horizontal broken surfaces (Figure 10.14 b). This preferred alignment is not as strong as that seen on other vertical surfaces, and is disrupted in places by silt particles. Aligned areas are not pervasive through the sample, they occur as separated ridges of material. These ridges are possibly areas of compressed platy particles present between silt particles. Most of the silt particles have been removed on the upper surface. One clear silt particle remains in the centre right of Figure 10.14 (b).

The quantity of silt varies quite substantially across Figure 10.14 (c) in some areas only very few silt particles can be seen. Here the cement coating around the silt grains is clearly visible where it has been separated from the grain (top left). In the lower part of Figure 10.14 (c) no silt is present. Even here no structure can be seen in the particles. An amorphous material containing several holes encrusts part of the surface (bottom middle).

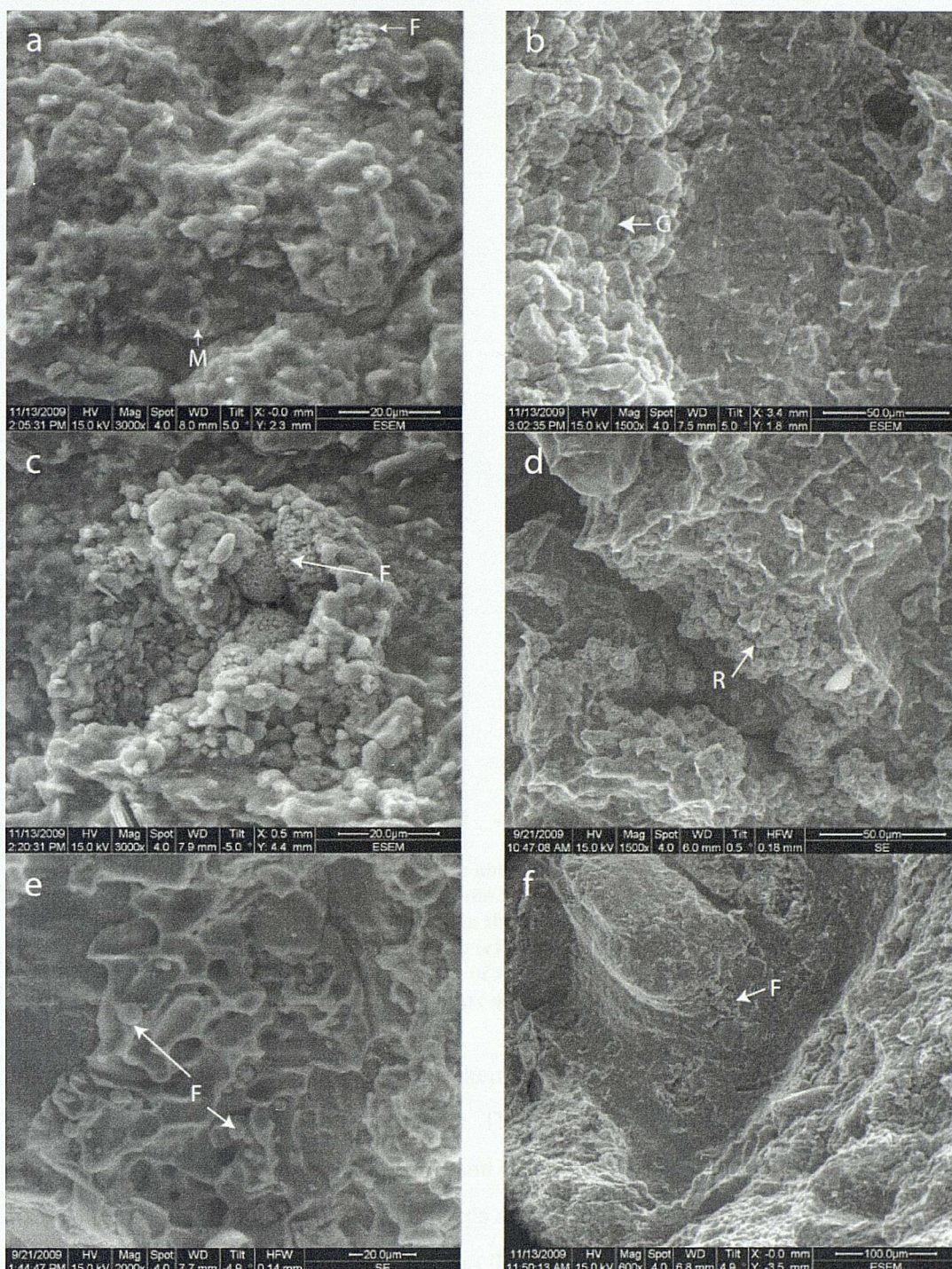


Figure 10.15 Kimmeridge Clay Borehole: 8.54m horizontal break (a, b, c) vertical break (d) 12.71m vertical break (e, f). Some of the small particles of the Kimmeridge Clay are very tiny microfossils (coccoliths) similar to but not as numerous as those observed in the Gault clay, 16 in this image, one example indicated (M) (a). In some areas there is a fine grained matrix beyond the resolution of the SEM (parts of all images). Several unusual material have been observed encrusting the surface, Coarse grained granular (G) (b) regular crystalline (R) (d) amorphous and full of holes (e). Examples of pyrite framboids (F) were picked out. Casts of shells have a great effect on the morphology of the surface (f).

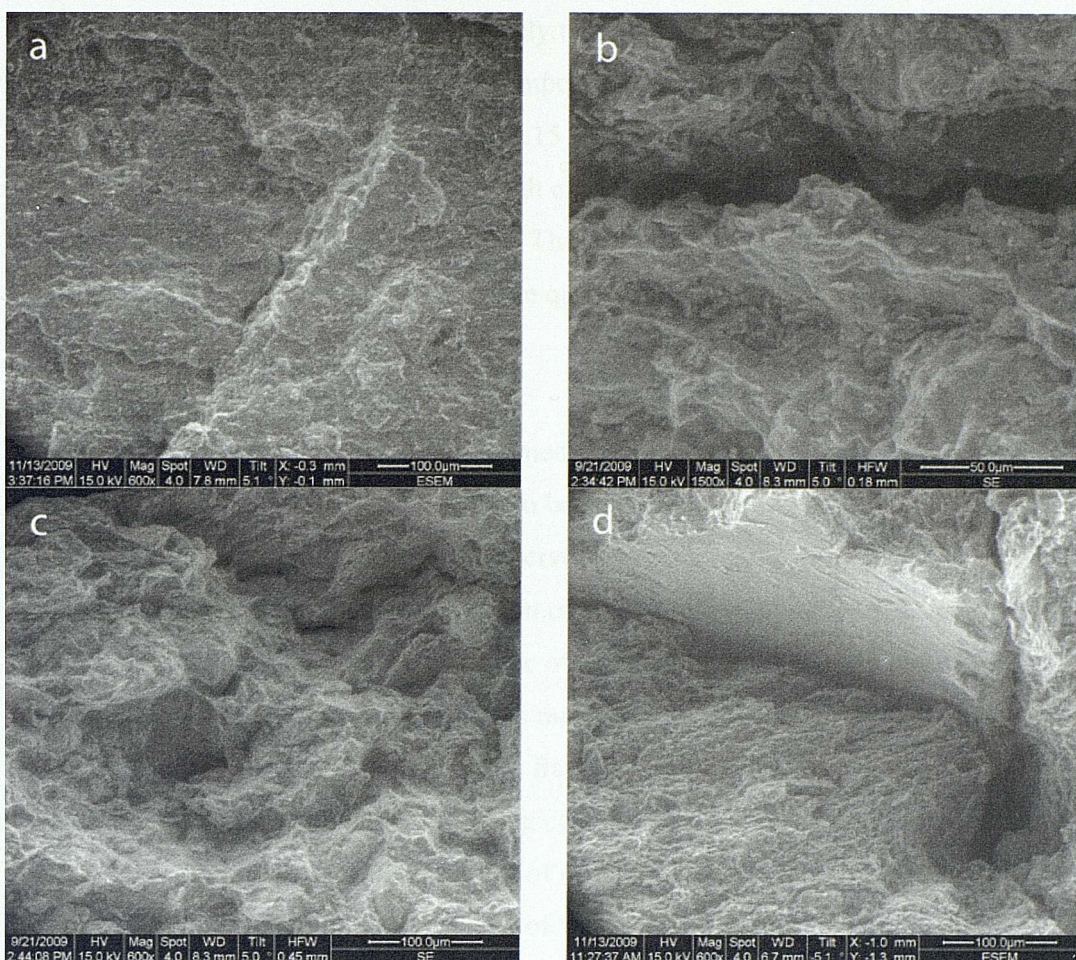


Figure 10.16 Kimmeridge Clay Borehole: 8.54m horizontal break (a) vertical break (d) 12.71m horizontal break (b, c). Joints running through the Kimmeridge Clay sometimes fine topographical expression on the mudrock surface (a). Cracks in the Kimmeridge Clay can be near planar (b) and open or they can have a more complex structure (c) jumping between small open segments. (d) Appears to be the intersection of two joints, one of which may run along the surface of a shell. The origin of these features is unclear as few joints are observed at the macro scale.

The aligned material on the horizontal broken surfaces (Figure 10.14 d) appears to be made of grains embedded in a finer matrix. This is a similar material to that seen on the horizontal broken surfaces of the Oxford and Gault Clay. Platy particles do not seem to be a major contributor to the structure. Steps are observed in the vertical broken surfaces (Figure 10.14 e). Particles intersect this surface suggesting a vertical particle alignment in this area. The boundary of the step is crenulated, not linear. Crystalline encrusting material is present on the surface of some of the silt grains (Figure 10.14 f). Possibly pyrite is growing into the space around the grain.

Tiny microfossils are common within the structure of the Kimmeridge Clay. They are present in large proportions on most of the horizontal broken surfaces (Figure 10.15 a). These microfossils and other material fragments are encased in a fine grained covering. Several different types of material have been observed encrusting the surface (Figure

10.15 b-e). These sorts of material are likely to have formed in the post deposition environment. One of these materials is composed of interlocking grains which appear to be cemented (bonded) together (Figure 10.15 b). This material can cover large areas of the surface, and occur in direct contact with clay rich areas, yet no evidence of the clay can be found within the granular material. This sort of material has also been observed in proximity to pyritic framboids which are quite common in the Kimmeridge Clay (Figure 10.15 c).

Another type of encrusting material is formed of bipyramidal crystals of octahedral habit, probably also pyrite. These appear to be covering an area of a joint's surface (Figure 10.15 d). They have also been observed covering a sand particle (Figure 10.14 f). These crystals probably grew in position on the joint from the fluids flowing through the material during diagenesis. A final type of encrusting material is non granular, possibly amorphous (Figure 10.15 e). This material is filled with holes and channels; some of the channels contain small pyritic framboids. This material has not been specifically identified; it is probably biological in origin. Other unusual structures on the surface of the mudrock are probably casts of microfossil shells (Figure 10.15 f). They form smoothed areas of the surface, some of which have unusual shapes. The shape depends on that of the microfossil which has been removed from the surface.

Several joints are present in the Kimmeridge Clay images. One joint causes a topographic step in the surface of the mudrock (Figure 10.16 a). It appears to be linear, with particles intersecting the surface causing a minor roughness. The joint is open but not along its entire length. Another joint, which is linear at the large scale, is rough at the smaller scale (Figure 10.16 b). This joint appears to have pulled open a surface, several particles poke out into the break causing some surface roughness. In other areas where it is not possible for a linear joint to form, the opening of the material is accommodated by jumps between several positions in the material (Figure 10.16 c). Several short linear sections have opened; these appear to be connected by short areas of unbroken material. Joints often occur along shell boundaries (Figure 10.16 d), as these are weak areas within the material fabric. Here two joints intersect; at the point of intersection there is an open hole in the fabric, probably formed due to erosion by water flowing along the intersection. The shell forms a smooth surface against its cast which allows joints to open easily. This is especially important where the shell cuts across the

material's fabric, as this orientation of break will allow the mudrock to fail under a lower applied stress.

10.1.4 Lias Clay – Charmouth Mudstone and Blue Lias

The Lias Clay generally has a high level of preferred orientation of particles. It also contains several unusual structures. The preferred particle orientation visible at 11.60m bgl is present in many images of the vertical broken surface (Figure 10.17 a). Platy particles are well aligned; they are only disrupted by shell fragments and microfossils. The topographic expression of this structure is not as strong as with the other structured mudrocks, due to the desiccation of the sample, as it was stored without wax prior to collection. The identification of aggregates/areas of stronger and weaker cement is difficult; they do not seem to be a feature of the Lias Clay. The structure is also present in the horizontal broken surfaces (Figure 10.17 b), particles intersecting with the sloping material surface. Broken shell fragments also aligned with the mudrocks overall fabric (centre of Figure 10.17 (b)). Locally the fabric may be orientated away from the horizontal (Figure 10.17 c). In this case near a linear feature running across Figure 10.17 (c). Possibly movement has occurred on the discontinuity allowing a local reorientation of the fabric. This discontinuity is associated with several holes in the mudrock, possibly this area has been affected by fluid flow and erosion. Many of the horizontal breaks have quite rugged topography, in some areas horizontal breaks occur on planes with very little topography (Figure 10.17 d) the top left corner shows a possible joint crossing the surface causing a jump in the surface topography.

Flat shell fragments align with the mudrock's fabric (Figure 10.17 e). Most of the platy particles are aligned; small microfossils and mineral grains disrupt the fabric. High magnification images of a horizontal break (Figure 10.17 f), show microfossils shell fragments and mineral grains with a large amount of fine grained material covering most of the surface. A change between two main horizontal planes is observed, highlighting the mudrocks structure perpendicular to the viewing direction.

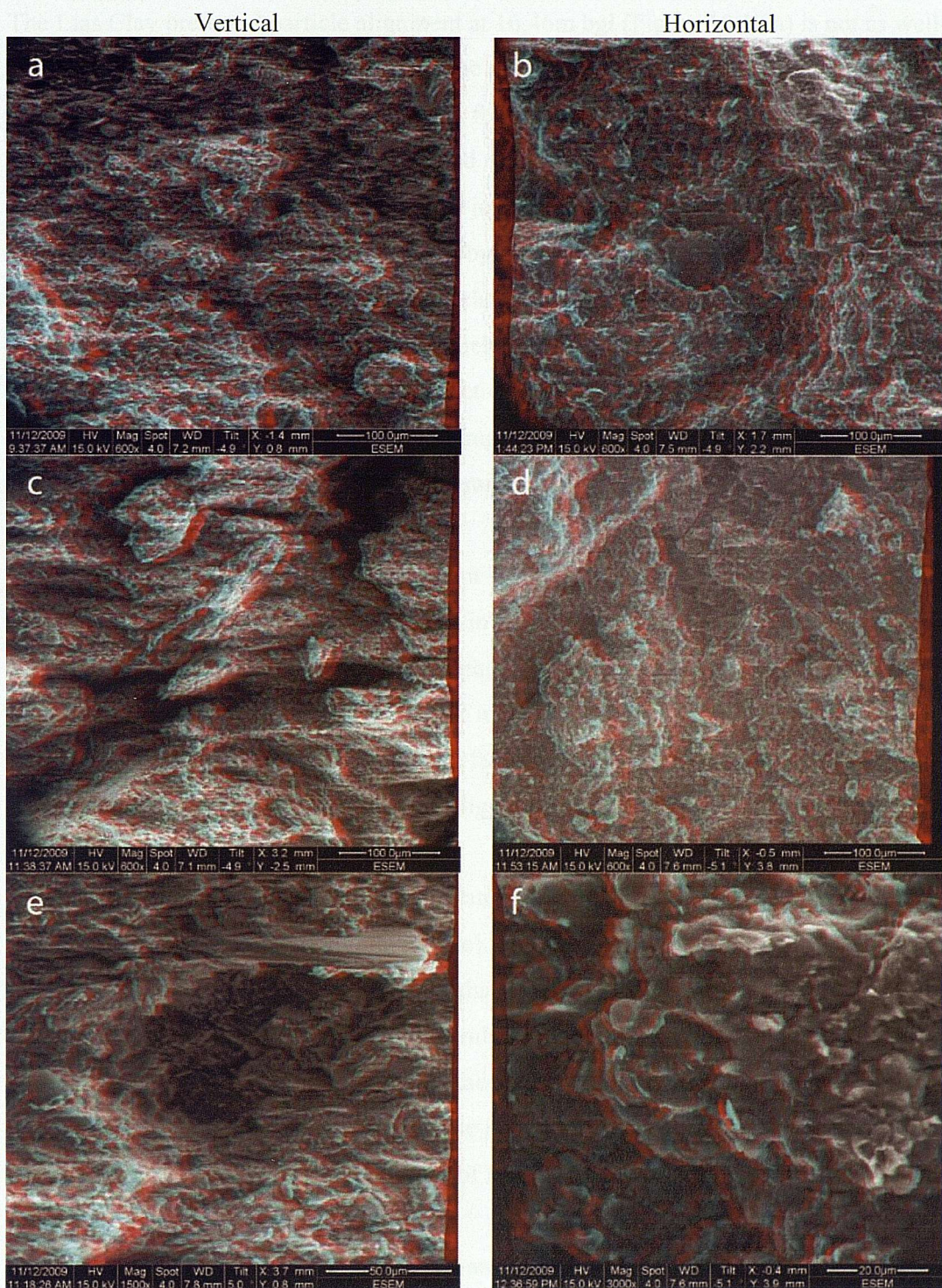


Figure 10.17 Lias Clay borehole sample 11.60m vertical cut (a, c, e) horizontal cut (b, d, f). Strong preferred particle orientation on vertical surfaces (a) some aggregated areas may be cemented. Platy particles orientated perpendicular to imaging directions, intersecting with sloping surface (b). Possible joints (c & d), a loose particle is orientated at 45°, probably having fallen from the joint as the broken surface formed. High magnifications images (e & f), showing preferred particle orientation, and large numbers of rounded particles on the horizontal surface.

The Lias Clay preferred particle alignment at 16.46m bgl (Figure 10.18 a) is not as well-developed as that at shallower horizons. The alignment of platy particles to the horizontal is disrupted by the presence of a few non-platy particles, most of which are shell fragments, with some microfossils and mineral grains. The fabric is also cross cut by a flat planar feature which is probably a joint surface. Alignment of particles is observed in the horizontal broken surfaces by the intersection of the platy particles with the sloping surface (Figure 10.18 b). The relatively large amount of topography on these surfaces increases the number of platy particles in these images. This increase in topography is probably a result of the reduction in preferred particle alignment in a partially cemented material. Several shell fragments are aligned to these planes. The very bright spots on the surface are loose grains which were not removed.

Some vertical breaks in the Lias Clay do not show any preferred orientation of platy particles (Figure 10.18 c). Local variations in the mineralogy of the mudrock, or close association with an inclined joint (lower right of Figure 10.18 (c)), may cause this effect. In this case the sample appears to be in the interior of a rounded shell. Some rounded shells align with the horizontal surface (Figure 10.18 d). Particles intersecting with the sloping surface also indicate the alignment.

The preferred orientation is caused by a relatively small number of platy particles dispersed across the surface (Figure 10.18 e). The shell fragments and mineral grains take up a much greater area of the surface than the clay minerals. This is possibly reflected in the surface's topography. The ridges seen in the aligned Oxford Clay are absent here. At high magnification the surface is covered with a fine grained material (Figure 10.18 f), through which a few of the large grains emerge (broken shell fragment centre of Figure 10.18 (f)). This is typical of most of the mudrocks studied.

Shells are quite common in the Lias, some are planar and thin (Figure 10.19 a) while others are more rounded (Figure 10.18d). There is a possible partial cast of a rounded shell on the bottom left of Figure 10.19 (a). Some of these shells are much thicker than those in the Oxford, Kimmeridge, Gault and London Clays (Figure 10.19 b). The example in Figure 10.19 (b) is nearly 20µm thick. As is common in several of the other mudrocks, displacements seem to localise near shell boundaries. These shells are also often aligned to the mudrock's fabric (Figure 10.19 c).

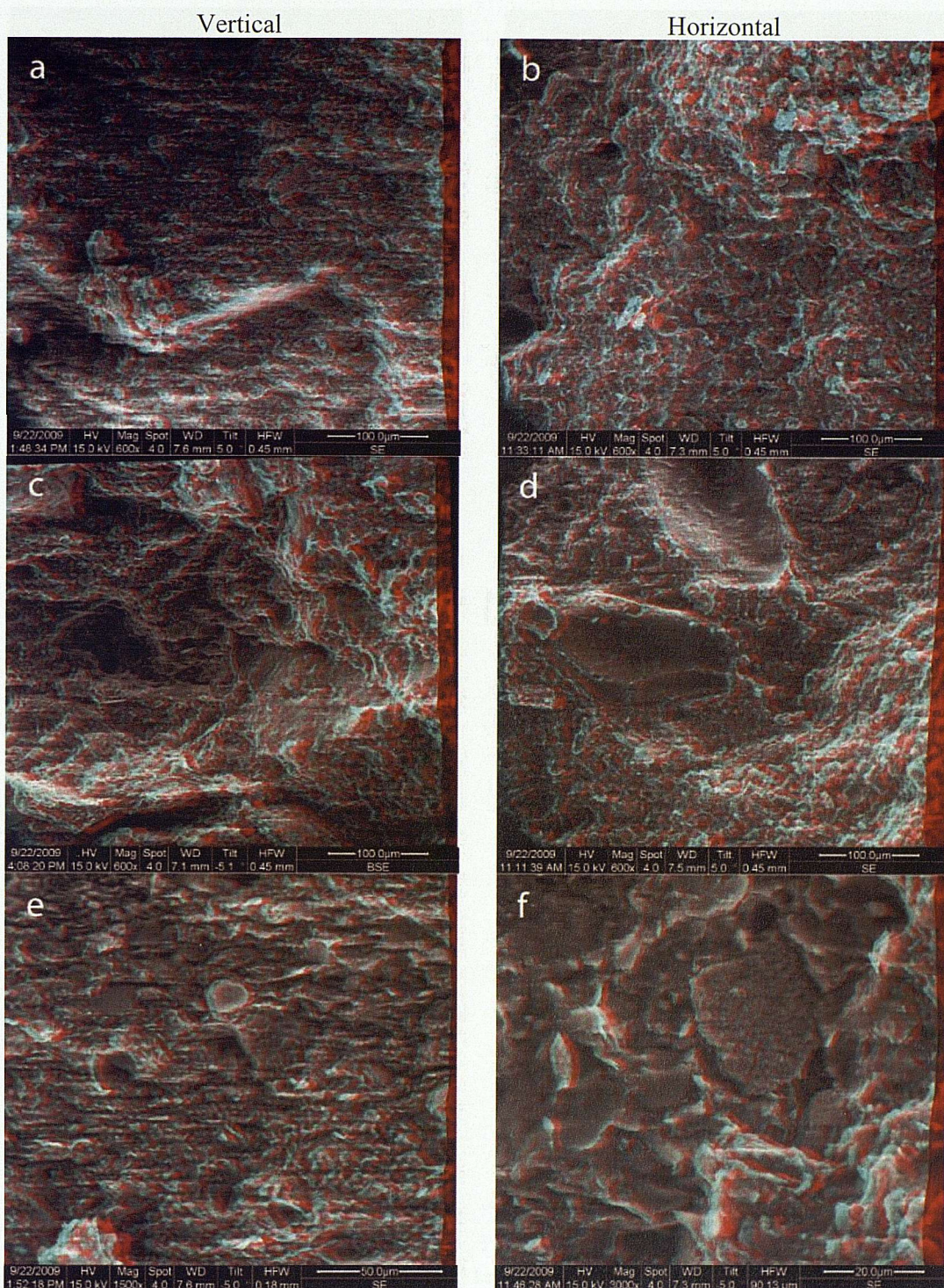


Figure 10.18 Lias Clay borehole sample 16.46m vertical cut (a, c, e) horizontal cut (b, d, f). Preferred particle orientation and joints (a & b). Some areas of no preferred orientation (c), shells relatively common (d), a large number of non platy particles are present in the material, the few platy particles carry the preferred orientation (e & f). High magnification images, one shell fragment and other granular material within a fine grained matrix.

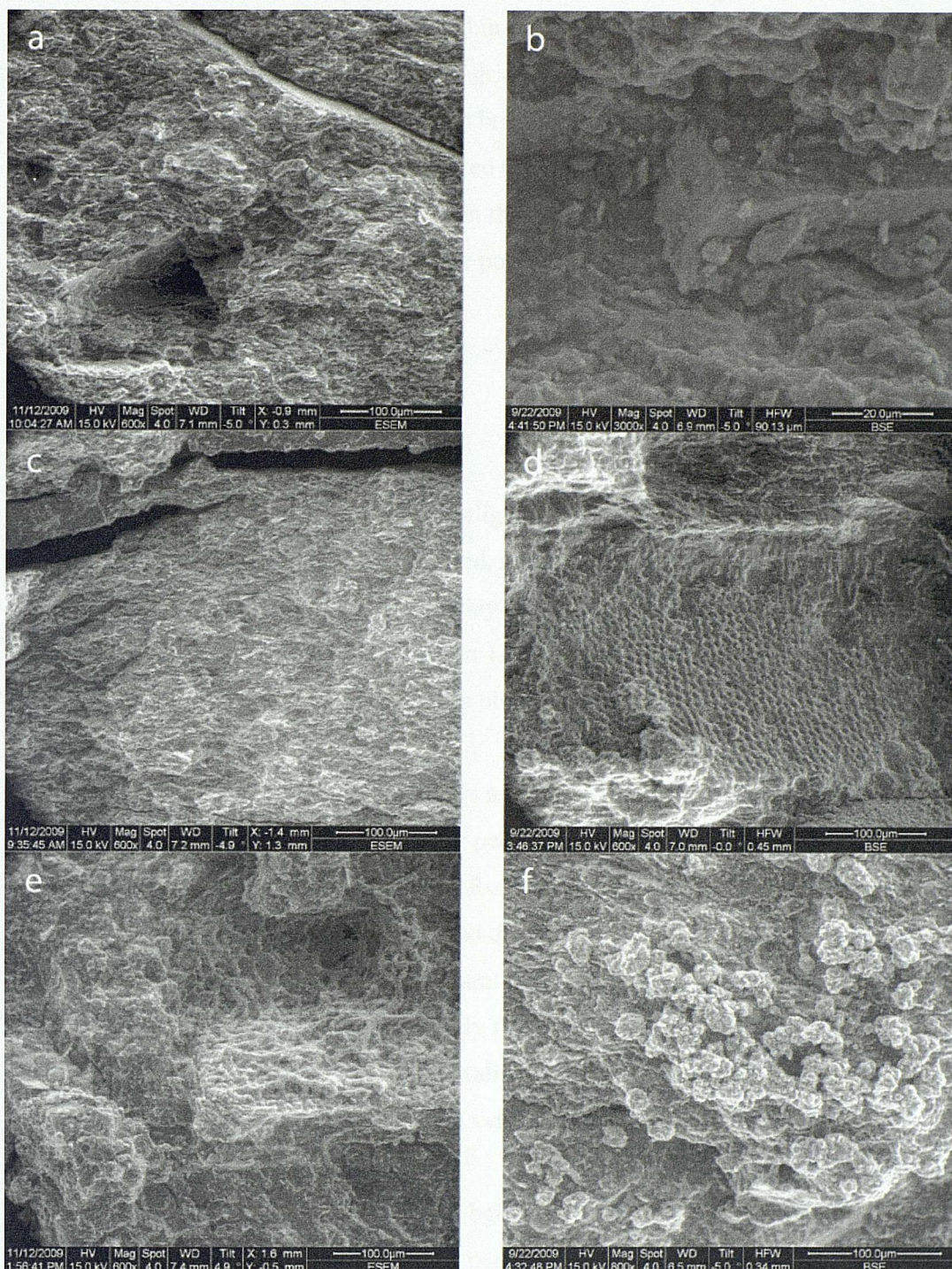


Figure 10.19 Lias Clay borehole: 11.60m vertical break (a) 16.46m vertical break (b, c, d) Horizontal break (e, f). Planar and rounded shell fragments are present in the Charmouth Mudstone (a). Some of the shells are thicker (b) than those observed in other mudrocks. Although these shells are often associated with cracks in the mudrock (c) as seen in other mudrocks also observed are diatoms (d) and several oddly shaped microfossils which form prominent features in places on the samples surface (e).

There are several unusual fabrics observed in the Lias Clay which are due to fossils. For example, sets of regular hollows in a tight grid (Figure 10.19 d), is a fossil diatom. Another fossil is formed from interlocking branching tubes (Figure 10.19 e). These fossils form areas of high porosity compared to the surrounding material. There are some areas of granular clusters in places on the mudrock surface. These granular areas are crystal growths probably formed in the post depositional environment (Figure 10.19 f).

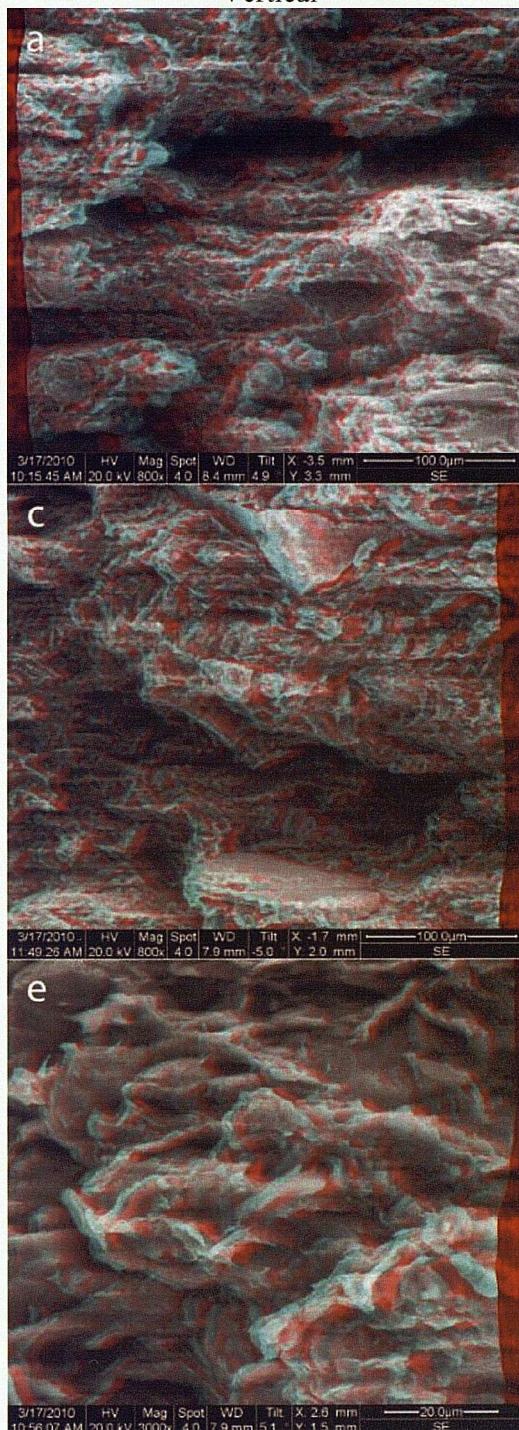
10.1.5 The London Clay

The London Clay has a preferred particle alignment in most of the studied material. The particle alignment is never very pronounced. In some cases the fabric is disrupted by non platy particles, however even where clay minerals dominate the structure is not especially strong. The cores were logged in 1.5m core runs, depths are identified as that of the core run from which the sample was obtained.

The London Clay from 7.9-9.4m bgl shows a moderate preferred alignment (Figure 10.20 a). This alignment is emphasized by peaks and troughs in the surface. Shells are aligned to the horizontal (Figure 10.20 c). Rounded silt sized particles make up a large proportion of the grains of the mudrock and disrupt the alignment at the particle scale (Figure 10.20 e). Preferred particle alignment is also present in the horizontally orientated samples: Mainly by the relatively flat shelved areas, as flat hollows in the surface (Figure 10.20 b) or as a surface parallel plateau (Figure 10.20 d). Particles appear to be interleaved, and covered by a very fine grained material (Figure 10.20 f).

In contrast the material from 30.45-31.9m bgl shows a much stronger preferred particle alignment (Figure 10.21). Both individual particles and the ridges on the surface form lines parallel to the horizontal (Figure 10.21 a) and the horizontal surface multiple flat horizons, and particles intersecting with sloping surfaces (Figure 10.21 b). On some surfaces the platy particles have a much greater variation in orientation. The lines they form a wave like shape, fluctuating around the preferred orientation (Figure 10.21 c).

Vertical



Horizontal

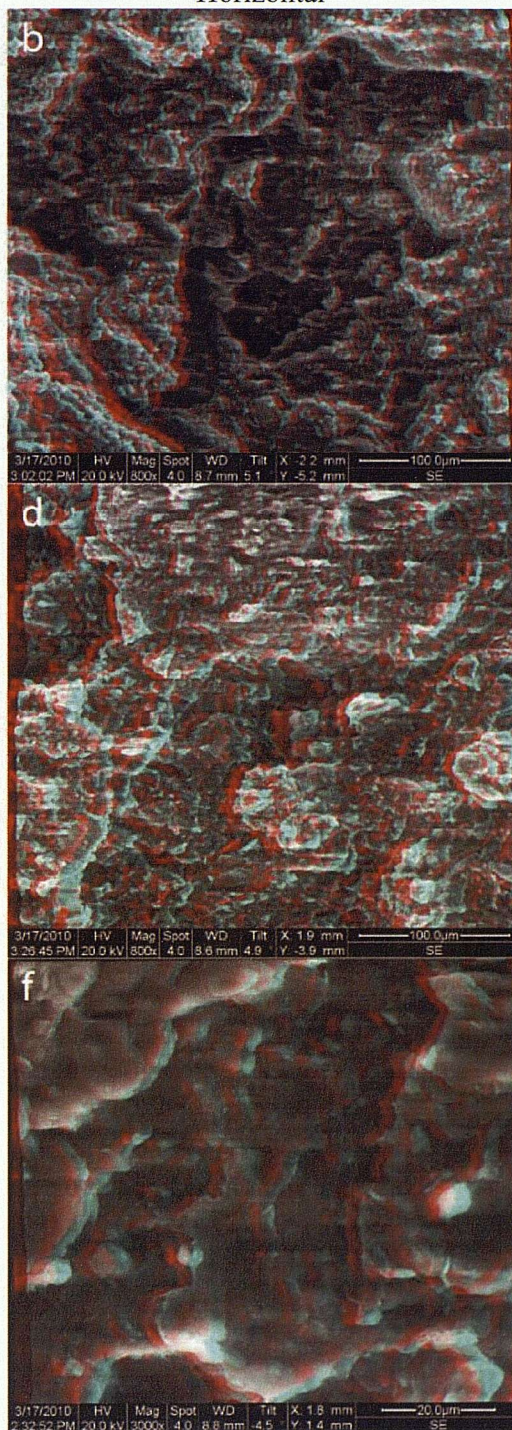


Figure 10.20 London Clay borehole: 7.9-9.4m, vertical break (a, c, e), horizontal break (b, d, f). Preferred orientation in ridges and cracks in material surface (a). Many rounded particles are present in the material matrix (c) shells and shell fragments are aligned to the horizontal. Horizontal surfaces contain flat steps, both hollows (b) and plateaus (d). High magnification images show rounded particles coated in platy clay minerals (e). Platy particles are covered with small crystals and fine grained materials (f).

Vertical

Horizontal

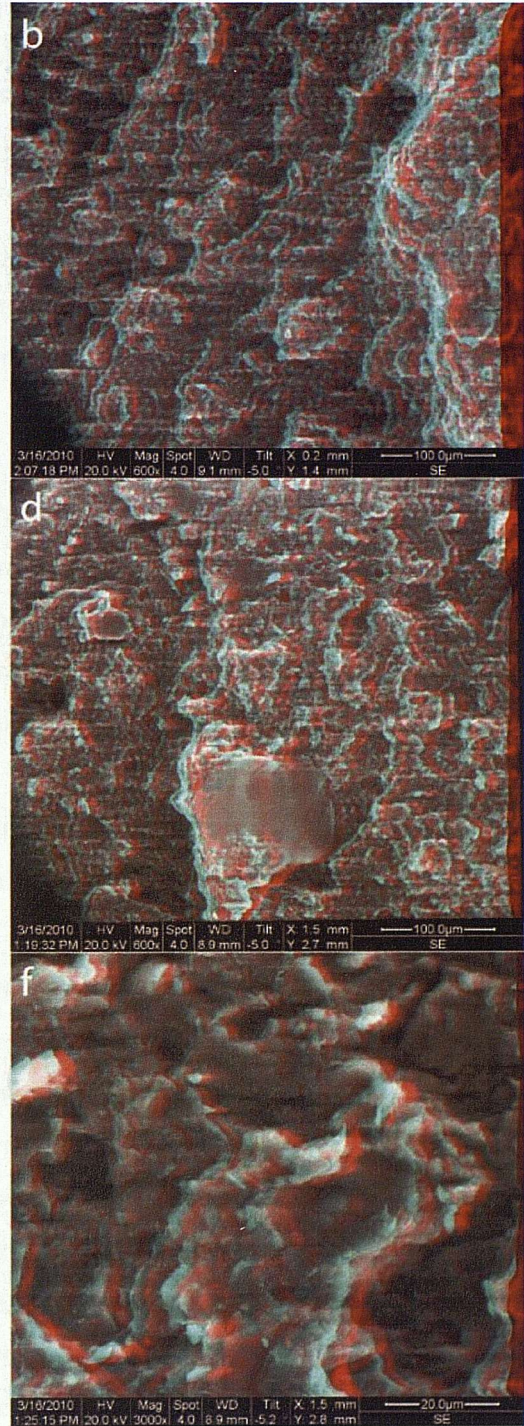
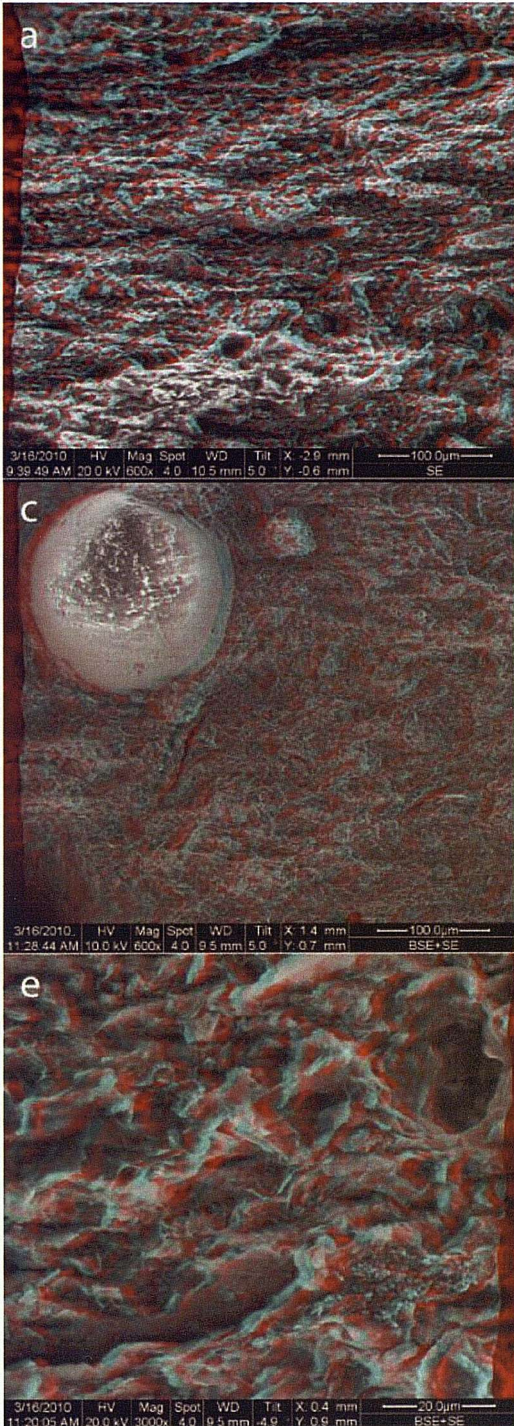


Figure 10.21 London Clay borehole: 30.45-31.95m, vertical break (a, c, e), horizontal break (b, d, f). Preferred orientation of ridges and troughs in the surface (a) and flat horizons on the horizontal surface (b) thin flakes intersect with the surface. Some very large rounded microfossils disrupt the local fabric of the materials (c), many small shell fragments align to the horizontal (d). At high magnification particle surfaces appear wrinkled, variations around the dominant preferred particle orientation are present (e), several very small rounded particles are present on the platy particle surfaces all within a fine grained matrix (f). No coccoliths or similar small microfossils are present in the high magnification images.

The other major feature of Figure 10.21 (c) is a large rounded shell. Although there are many broken shell fragments spread through the London Clay, intact shells are quite rare. Very few flat shells are observed in the mudrock. The majority of the aligned shell fragments are on horizontal surfaces (Figure 10.21 d). The variation in orientation of the platy particles is caused by a large number of small grains dispersed through the mudrock (Figure 10.21 e). In Figure 10.21 (e) there is also an inclusion formed of a large number of rounded grains (probably pyrite). The enhanced number of small granular particles presents itself in the horizontal break as an irregular surface (Figure 10.21 f).

The platy particles are still visible with the granular particles on their surfaces, all of which is subsumed by the finer grained material. The material from 30.45-31.95m clearly has a higher concentration of clay minerals than the other layers. It does not result in a stronger preferred orientation. Possibly this structure would develop more if the London Clay were buried to a greater depth.

At 33.45-34.95m the horizontal structure although present, is not strongly developed (Figure 10.22 a). The surface appears nodular, although it contains some clear ridges and troughs indicating preferred alignment. The nodular appearance continues on the horizontal surfaces (Figure 10.22 b); these nodules, which appear to be regions of loosely aggregated particles, form irregularities on the horizontal broken surfaces, increasing their surface roughness. The normal flat horizons of a mudrock with a preferred particle orientation are not formed. In Figure 10.22 (b) the bright cube is probably pyrite. Several fragments are present disrupting the mudrock's fabric (Figure 10.22 c); several shell fragment and angular grains are present. This sample has one of the few large shell fragments aligned to bedding (Figure 10.22 d) embedded within the granular fabric. Several unusual materials were observed within the London Clay. One such material is a cluster of smooth sub-rounded grains (Figure 10.22 e). Their smooth shape indicates that they probably grew in situ, into the pre-existing material fabric. Without mineralogical data they cannot be clearly identified.

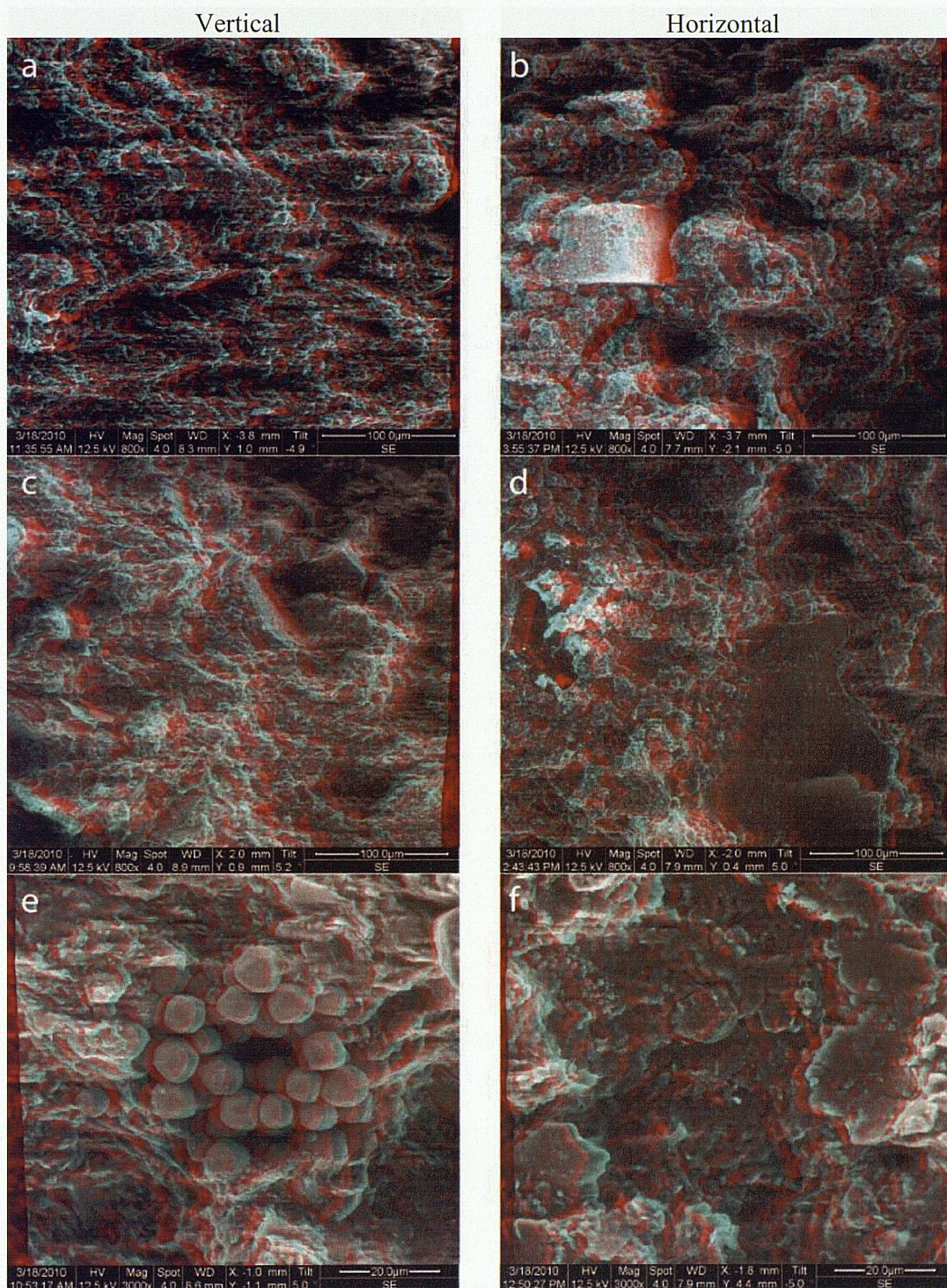


Figure 10.22 London Clay borehole: 33.45-34.95m, vertical break (a, c, e), horizontal break (b, d, f). Some surfaces show a relatively strong preferred particle orientation (a). Other areas are very granular in appearance (b & c), cube shaped mineral is probably pyrite (b). Of the few sell fragments in the London Clay, those that are found are aligned to the horizontal (d). Several odd inclusions (mostly pyritic) are present (e). Horizontal surfaces have flaky clay minerals peeling from the surface (f).

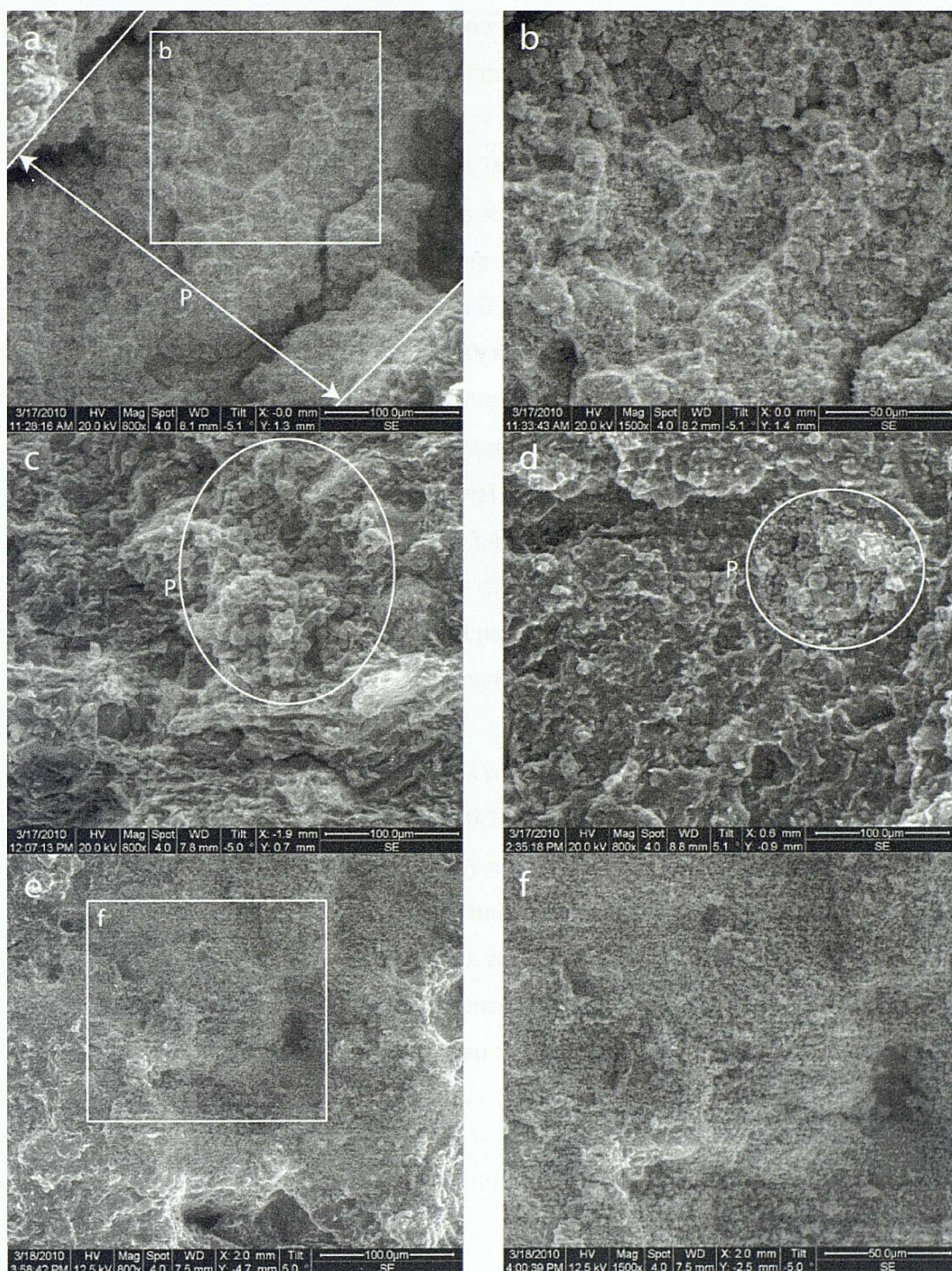


Figure 10.23 London Clay borehole samples: 7.9-9.4m, vertical break (a, b, c) horizontal break (d) 33.45-34.95m horizontal break (e, f). Pyrite nodules (P) are very common within the London Clay. Pyrite occurs in multiple habits within the material. Framboidal mass low magnification (a) high magnification (b). Bipyramidal crystals within the soil mass (c), framboidal enclosure within the soil mass (d). Mass of bipyramidal crystals low magnification (e) high magnification (f).

The alignment of the material appears to bow around this inclusion. The platy particles are interlocked with finer material between them (Figure 10.22 f)

Pyrite is very common within the London Clay (Figure 10.23). It forms large inclusions within the material's fabric (Figure 10.23 a & e). In the 7.9-9.4m sample the nodule is formed of rounded pyritic framboids (Figure 10.23 b), and in the deeper 33.45-34.95m sample it is formed of bipyramidal crystals (Figure 10.23 f). Smaller pyrite inclusions are found throughout the London Clay, both in bipyramidal form (Figure 10.23 c) and in the framboidal form (Figure 10.23 d) more commonly seen in the other materials. Pyrite nodules have previously been discussed as possible nucleation points for the propagation of micro-cracks into the material structure prior to failure (Fouché et al., 2004). Both the material at 7.9-9.4m and 33.45-34.95m are described as nodular or granular. However their structures are very different (Figure 10.24). The grains within the shallow sample are formed of silt sized particles (probably quartz). The grains in the deeper samples are agglomerates of platy particles.

At 7.9-9.4m a large hollow in the surface is connected to a couple of varying horizontal cracks in the material's structure (Figure 10.24 a). Several other horizontal cracks may be present across Figure 10.24 (a). The clay mineral particles arch around the silt grains. The deeper sample has a non linear crack running diagonally across Figure 10.24 (b). There are several shells in Figure 10.24 (b), at varying angles to the overall structure. There are also a number of silt grains present which have a minor contribution to the overall structure. The platy particles have an aligned structure, which is weakened by the nodular clay mineral aggregates.

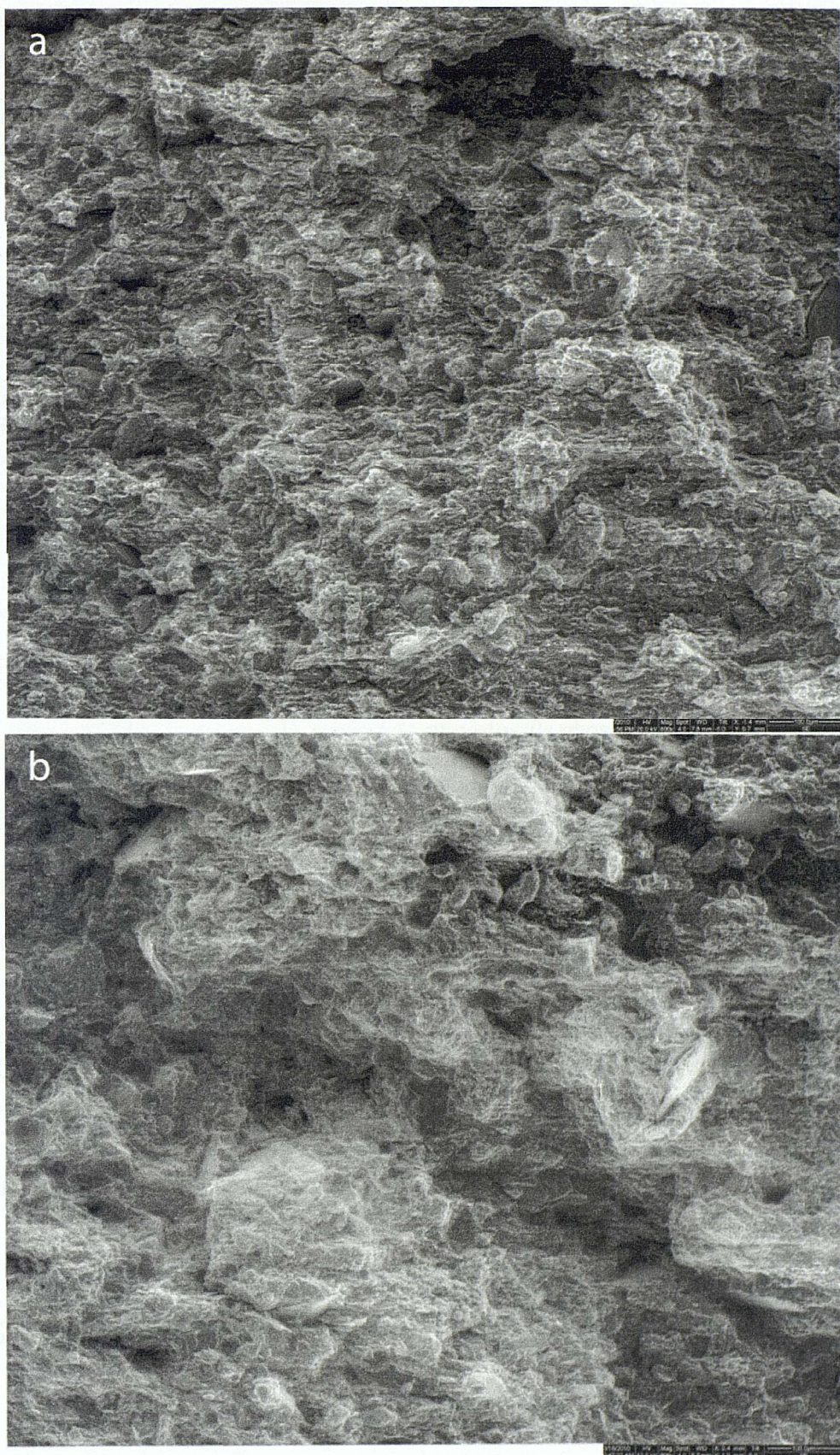


Figure 10.24 Montages of sixteen images of vertical surfaces of London Clay 7.9-9.4m (a) 33.45-34.95m (b). The shallower material contains many rounded quartz silt grains, which are covered in a layer of clay minerals (a). The deeper material also contains silt grains, however the clay minerals appear cemented, there is very little gap between particles.

Material	Average Imaging depth (bgl)	Images
London Clay (Heathrow Terminal 5)	8.65m	Vertical: Moderate preferred particle alignment, aligned shells, silt particles, pyrite, Horizontal: steps between layers, pyrite
	31.20m	Vertical: Moderate preferred particle orientation Microfossils, some small scale orientation variations. Horizontal: Steps between layers, no coccoliths
	34.20m	Vertical: Moderate, locally strong preferred particle alignment, granular areas. Horizontal: Steps between layers, shells, pyrite
Gault Clay (High Cross, Madingley)	3.00m Block	Vertical: No indication of alignment (Joint surfaces), some away from joints. Joint surfaces show regions of fine grained material and indications of biological growth into a void. Some open horizontal cracks, some pyritic nodules. Horizontal: Steps between layers and some lifting of the surface indicate alignment, many microfossils,
	1.72m	Vertical: No indication of alignment, some rounded fossils. Horizontal: No indication of alignment
	6.82m	Vertical: Strong preferred particle alignment, many microfossils. Horizontal: Steps between layers, microfossils phosphatic nodules
	13.46m	Vertical: No indication of preferred particle alignment, several joints and fine grained material, large number of coccoliths. Horizontal: Some indication of steps between layers, large number of coccoliths
Kimmeridge Clay (Willowbrook Farm)	8.54m	Vertical: inclined preferred orientation, shell fragments Horizontal: encrusting material, some pyrite, joints
	12.71m	Vertical: silty horizon, indications of cementation Horizontal: aligned cracks, pyrite, possible shells (cast), joints
Oxford Clay (Elstow)	10.00m	Vertical: Strong particle alignment, large flat shells also aligned, some pyrite. Horizontal: some steps between layers
Lias Clay (M5 Junction 13)	11.60m (Charmouth Mudstone)	Vertical: Strong preferred particle orientations, some aligned shells, joints Horizontal: particles intersect with sloping surfaces, joints
	16.46m (Blue Lias)	Vertical: preferred particle orientation observed in some locations, common shells Horizontal: steps between layers, shells on surface

Table 10-2 Summary of some of the structural features observed in the images of each material.

10.2 Summary

The main structural features are summarised in Table 10-2. The Oxford Clay has the strongest preferred particle alignment. The middle depth of the Gault Clay, the Lias Clay and the London Clay all show a tendency for preferred particle orientations. The Gault Clay at 1.72m has no preferred orientation because it is highly weathered. The Gault Clay block sample is weathered along joints, however still having strong preferred particle orientations away from the joints and on horizontal broken surfaces. The deepest Gault Clay samples also do not have a preferred particle orientation probably due to a large number of small microfossils. The platy particle orientations are disrupted in the Kimmeridge Clay due to the high silt content. The shallower sample has a preferred alignment 25° to the horizontal probably due to sedimentary structures, such as lenses or cross-bedding. Further away from the London Platform it is likely that the Kimmeridge Clay will have a very strong preferred alignment.

The microfossils within geological materials change with time due to evolutionary processes. The aligned planar shells of the Oxford Clay form horizontal reinforcing elements, the rounded shells within the Gault Clay form enclosed porosity, the high volumes of coccoliths greatly affect the mudrock's particle arrangement.

Vertical surfaces are commonly dominated by the platy particles; horizontal surfaces are often dominated by fine grained material which covers the platy particle surfaces. The fine grained material contains shell fragments, mineral grains and microfossils. This material is probably formed of small clay mineral grains, probably rich in smectite.

CHAPTER 11

MICROSTRUCTURE QUANTIFICATION AND COMPARISONS

The quantification of preferred particle alignment by the stated method (Section 9.3.5) for all of the mudrocks is presented in this chapter. The quantitative results correlate well with the qualitative results (Chapter 10). Abnormalities such as those of the Kimmeridge Clay can only be explained with reference to the images. The results presented here must be interpreted in the context of the SEM images (Appendix D). Materials which have a preferred particle alignment will show an alignment in rose diagrams from vertical breaks (ridged and troughs) and also a lesser alignment on horizontal breaks (the sides of steps between layers). Materials with no preferred particle alignment will have similar rose diagrams for both vertical and horizontal broken surfaces. Their rose diagrams will not be circular due to the statistical variability of the samples and the position of the detector within the microscope. Materials with no particle organisation will still have a measurable (if small) preferred particle orientation in both horizontal and vertical images, however the difference between horizontal and vertical images will be less pronounced.

11.1 *Two dimensional analysis*

The two dimensional analysis has been applied to all 1792 images; the results for individual images are presented in Appendix D. The results discussed here are for groups of images taken from the same horizon in each mudrock and taken at the same scale.

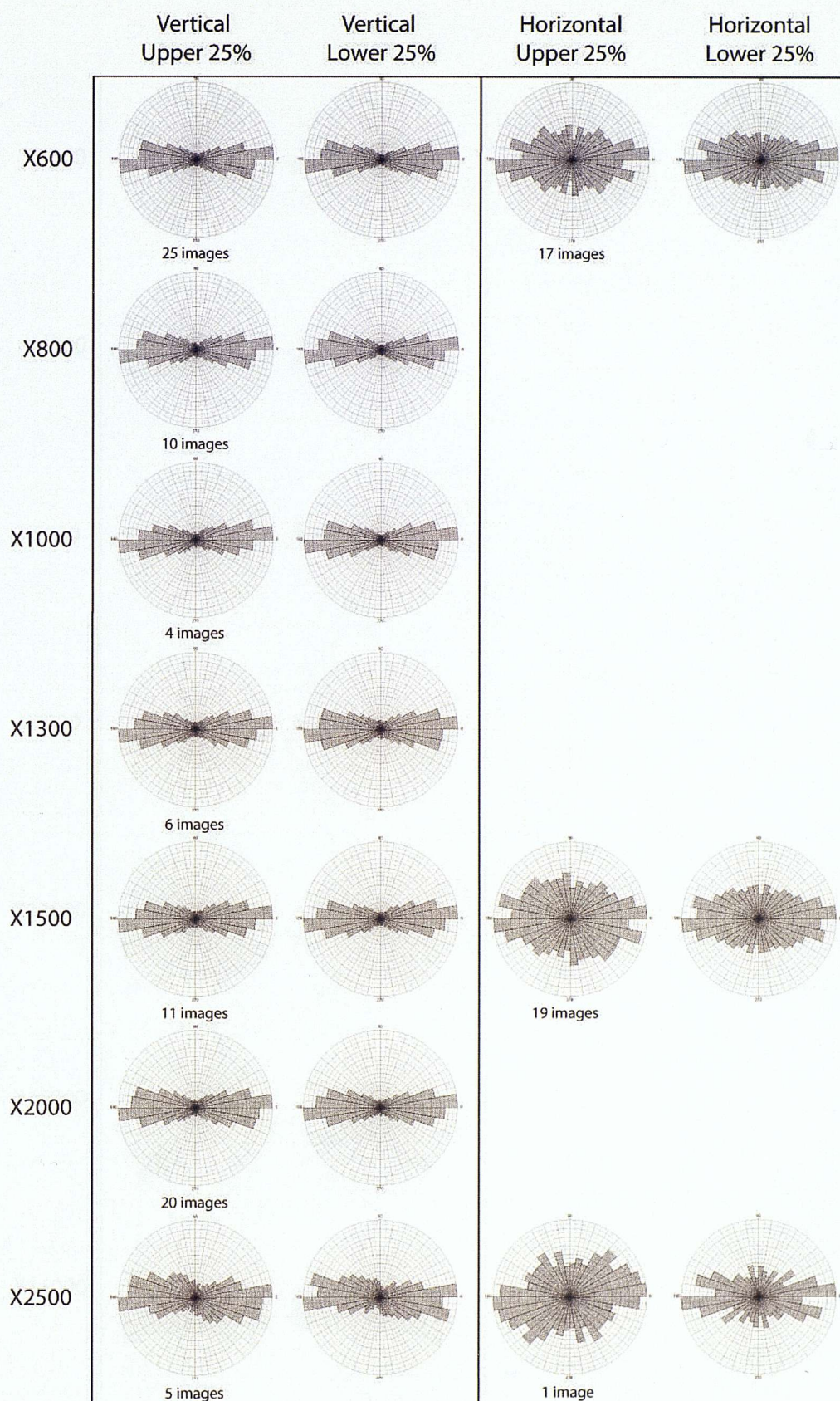


Figure 11.1 The effects of scale, above are rose diagrams of the structure of the Oxford Clay for both the upper and lower quartiles of grey levels for vertical surfaces (left) and horizontal surfaces (right). The number of images used in the creation of the rose diagrams is given under the lower quartile diagram, and applies to that for the upper quartile, this table for x600-x2500 magnifications.

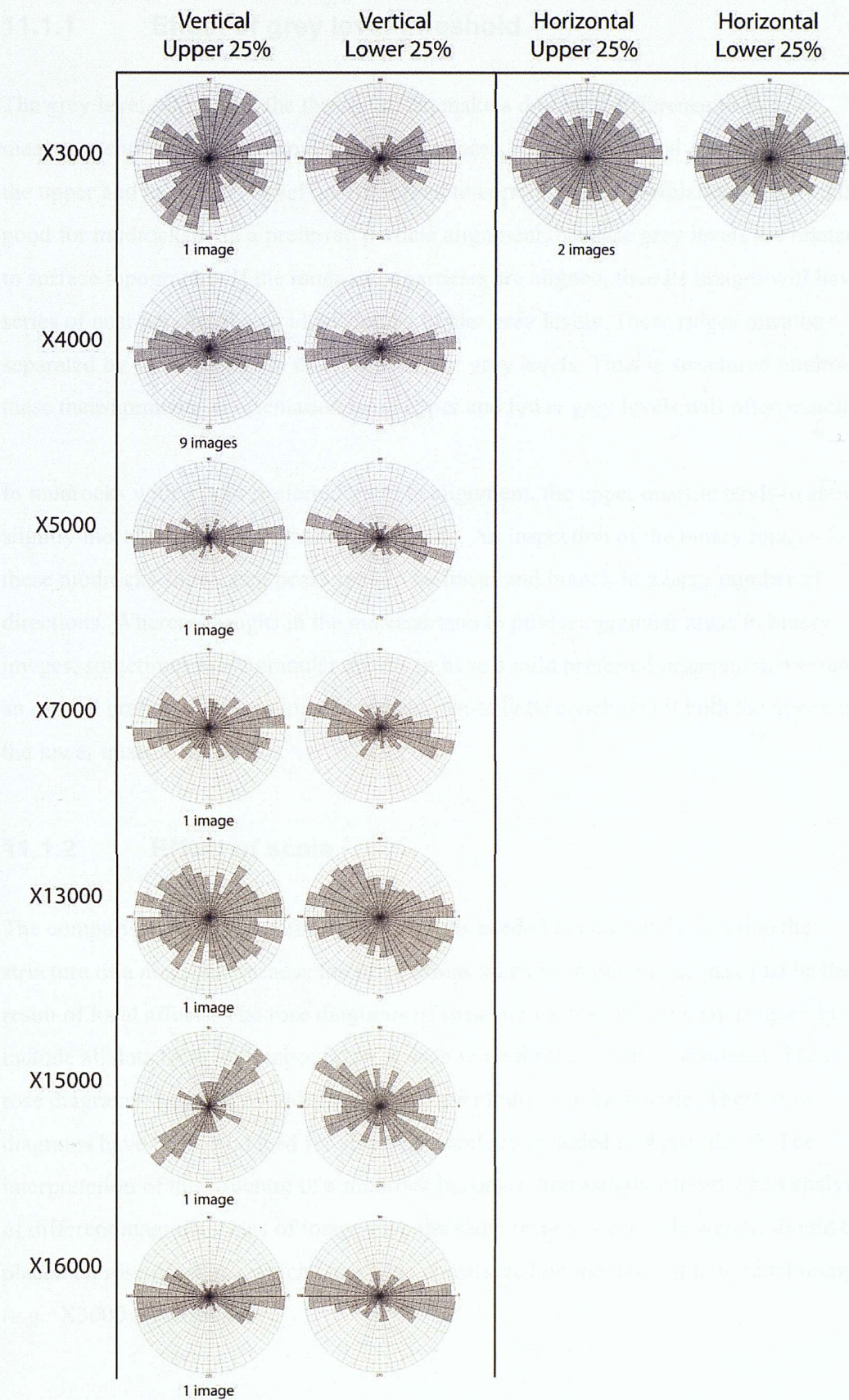


Figure 11.2 The effects of scale, above are rose diagrams of the structure of the Oxford Clay for both the upper and lower quartiles of grey levels for vertical surfaces (left) and horizontal surfaces (right). The number of images used in the creation of the rose diagrams is given under the lower quartile diagram, and applies to that for the upper quartile, this table for magnifications x3000-x16000

11.1.1 Effect of grey level threshold

The grey level selected as the threshold can make a dramatic difference to the measurements made from some mudrock surfaces. Except in special cases (Figure 9.9), the upper and lower grey level quartiles tend to correlate. This correlation is especially good for mudrocks with a preferred particle alignment, because grey levels are related to surface topography. If the mudrock's particles are aligned, then its images will have a series of near parallel ridges identified by lighter grey levels. These ridges must be separated by equally parallel troughs in darker grey levels. Thus in structured mudrocks these measurements of orientation from upper and lower grey levels will often match.

In mudrocks with a poor preferred particle alignment, the upper quartile tends to show slightly more variation than the lower quartile. An inspection of the binary images for these mudrocks shows that peaks tend to be linear and branch in a large number of directions. Whereas troughs in the material tend to produce granular areas in binary images, sometimes these granular areas can have a mild preferred orientation. Therefore an overall preferred orientation of particles can only be concluded if both the upper and the lower quartile agree.

11.1.2 Effect of scale

The comparison of a large number of images is needed to adequately describe the structure of a mudrock because the orientations taken from one image may just be the result of local affects. The rose diagrams of structure for the Oxford Clay (Figure 11.1) include all data from all images taken at each scale for the sample considered. These rose diagrams show the overall structure of the mudrock at each scale. These rose diagrams have been produced for all images and are included in Appendix D. The interpretation of the structure of a mudrock becomes increasingly certain when analysis of different magnifications of image give the same results. Very little weight should be placed on rose diagrams which have been constructed on the basis of individual images (*e.g.*, X3000 magnification).

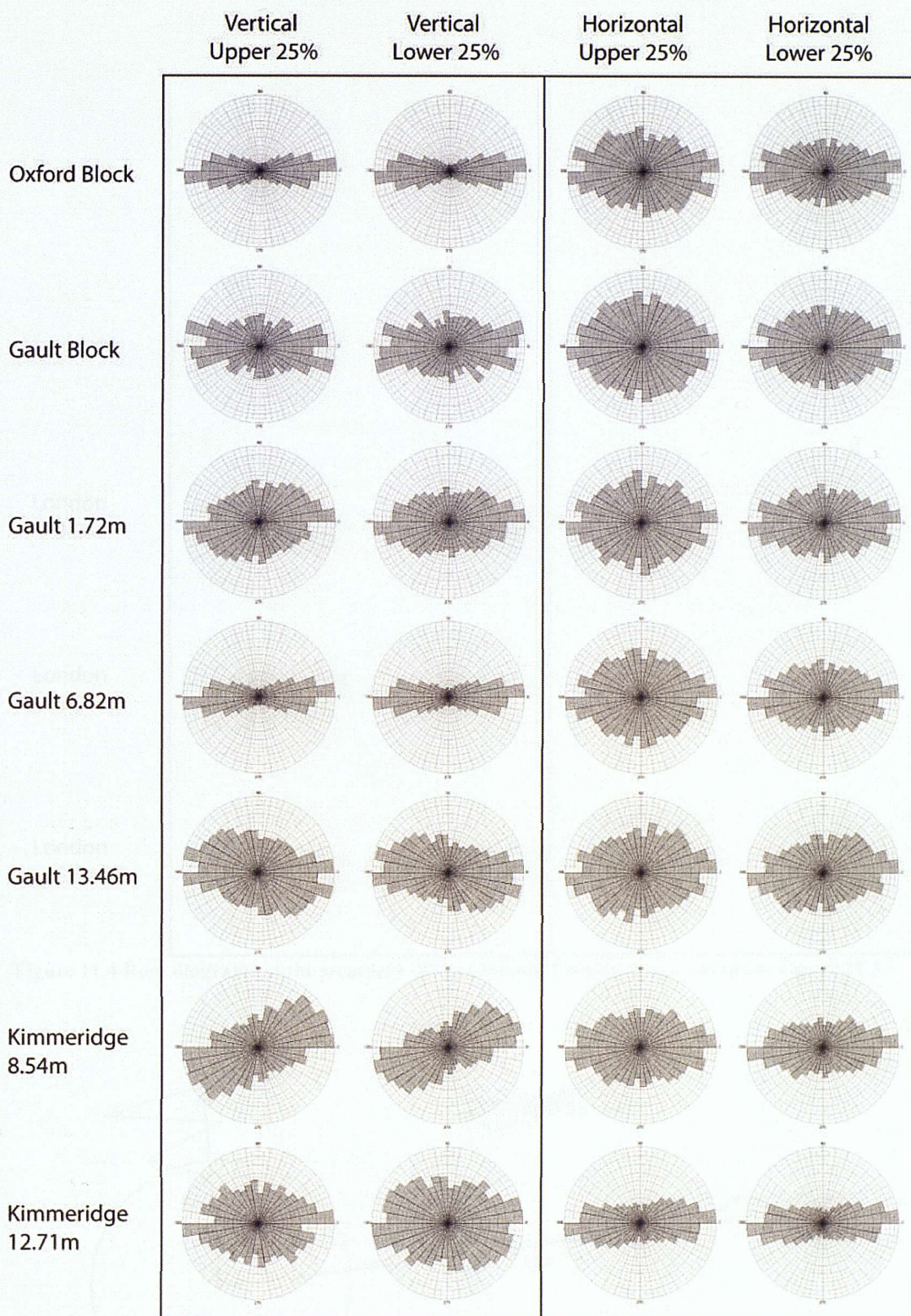


Figure 11.3 Rose diagrams of the summation of particle long axis orientations for all sampling horizons of the Oxford, Gault and Kimmeridge Clay. These rose diagrams are based on images at a magnification of X1500. Vertical broken surfaces on the left, horizontal broken surfaces on the right.

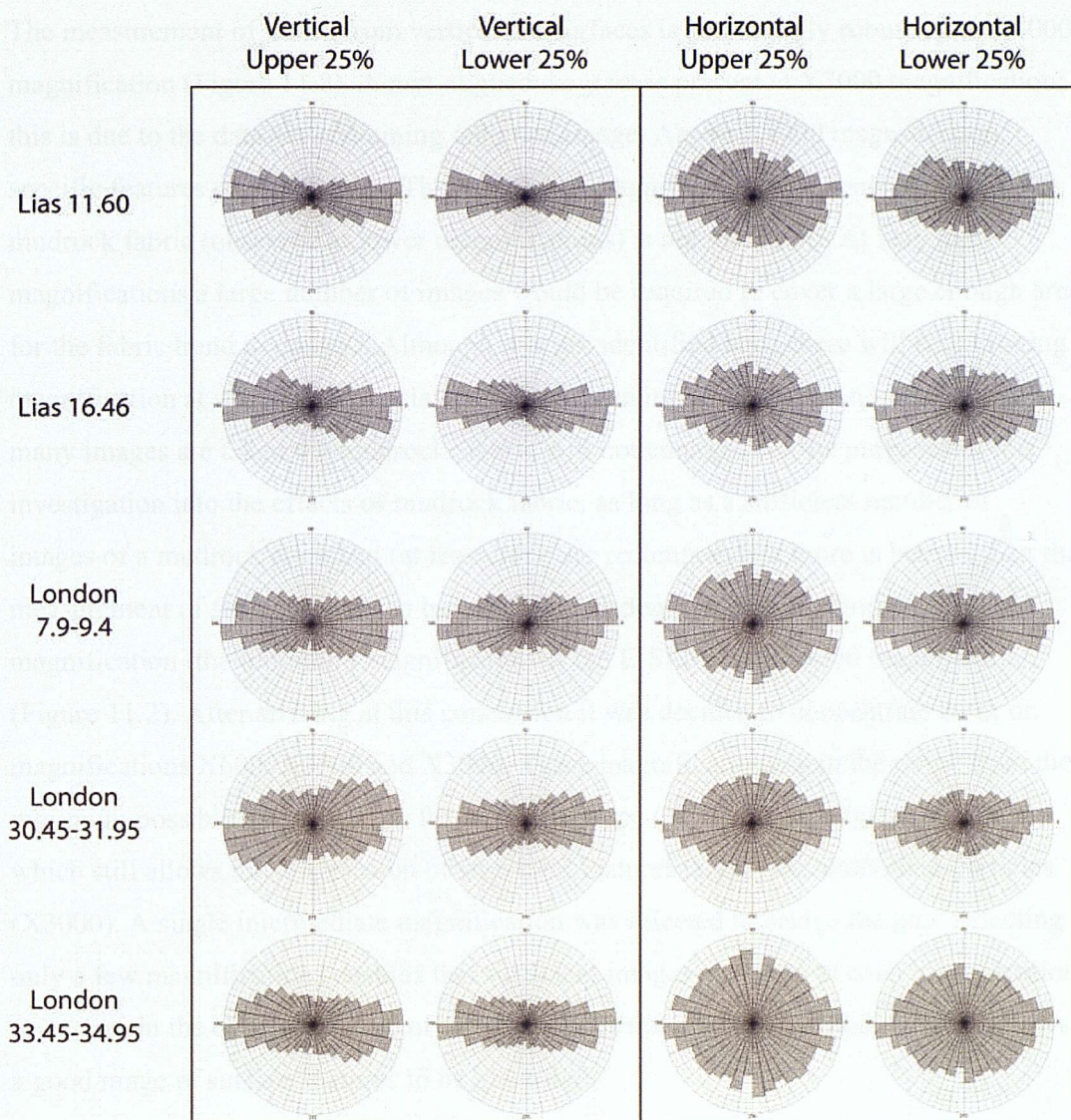


Figure 11.4 Rose diagrams of the structure of the Lias and London Clays, details as Figure 11.3

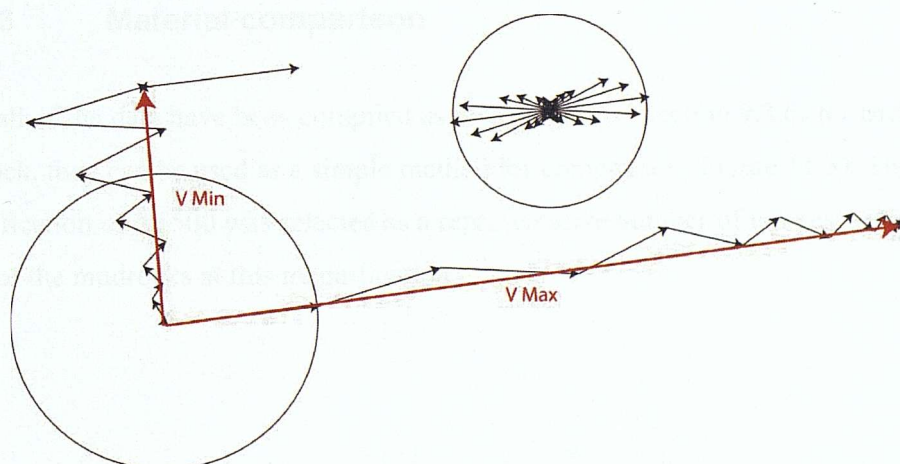


Figure 11.5 Vectors V max and V min formed by half circle addition, V max is the magnitude of the addition of all vectors within 90 degrees of the maximum vector. V min is the half circle 90 degrees from the maximum vector

The measurement of fabric from vertical cut surfaces is surprisingly robust up to X4000 magnification (Figure 11.2). A non-aligned diagram is present at X3000 magnification; this is due to the data-set containing only one image. Above X4000 magnification specific features may dominate. Thus, at higher magnifications the general trend of the mudrock fabric (observed at lower magnifications) is not identified. At very high magnifications a large number of images would be required to cover a large enough area for the fabric trend to emerge. Although it is not identified here, there will be a limiting magnification at which a single clay particle fills an image, such that no matter how many images are taken the mudrocks fabric will not emerge. For the purposes of this investigation into the effects of mudrock fabric, as long as a sufficient number of images of a mudrock are taken (at least three are recommended, more is better), then the measurement of fabric appears to be scale independent between the limits of X600 magnification (the minimum magnification of the E-SEM) and X4000 magnification (Figure 11.2). After arriving at this conclusion it was decided to concentrate work on magnifications X600, X1500 and X3000. These magnifications span the range from the minimum possible magnification for the microscope (X600) to a higher magnification which still allows the observation of structural features larger than individual particles (X3000). A single intermediate magnification was selected to bridge the gap. Selecting only a few magnifications ensures that sufficient images are taken at each magnification to be certain the observed fabric of the mudrock is not altered by outliers. It also allows a good range of surface features to be identified.

11.1.3 Material comparison

Once all of the data have been compiled as rose diagrams (Section 9.3.6) for each mudrock, they can be used as a simple method for comparison (Figure 11.3). The magnification of X1500 was selected as a representative number of images were taken of all of the mudrocks at this magnification.

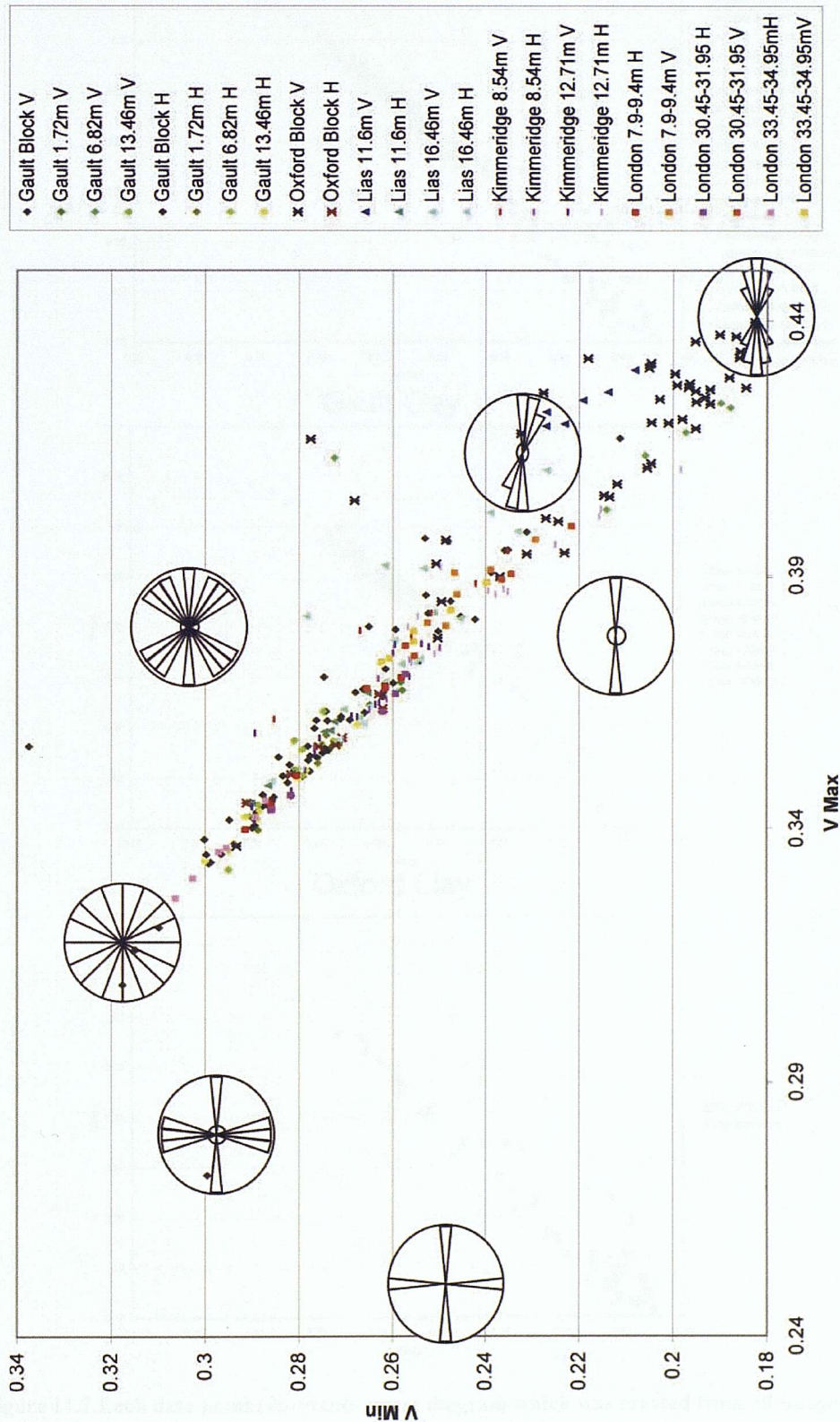


Figure 11.6 Examples of where rose diagrams of different shapes plot on the Vmax-Vmin graph strongest structure bottom right of graph.

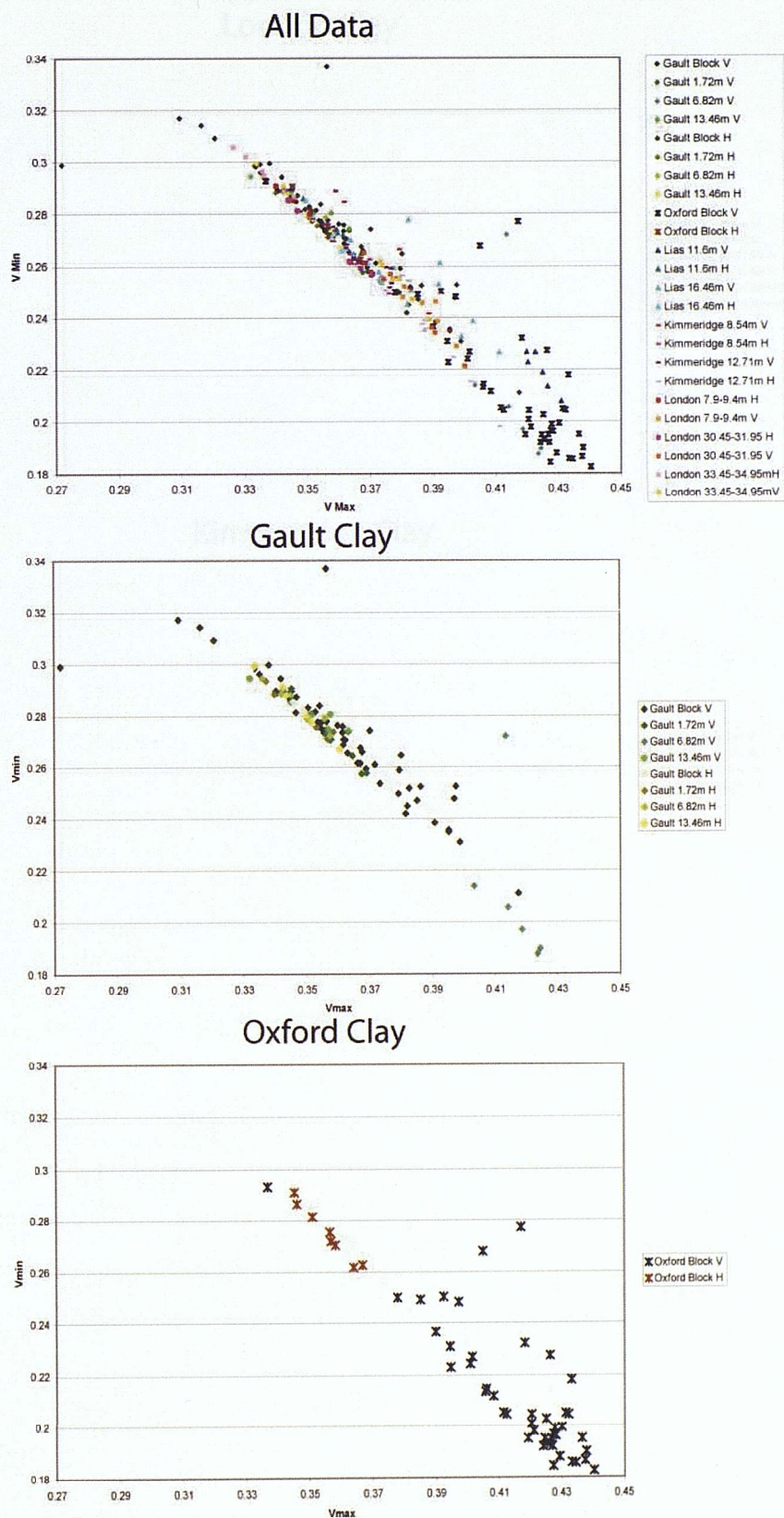


Figure 11.7 Each data point represents a rose diagram which was created from all images at a given scale for each mudrock. Rose diagrams from images with a magnification greater than 3000X are omitted. See Figure 11.6 for higher magnification.

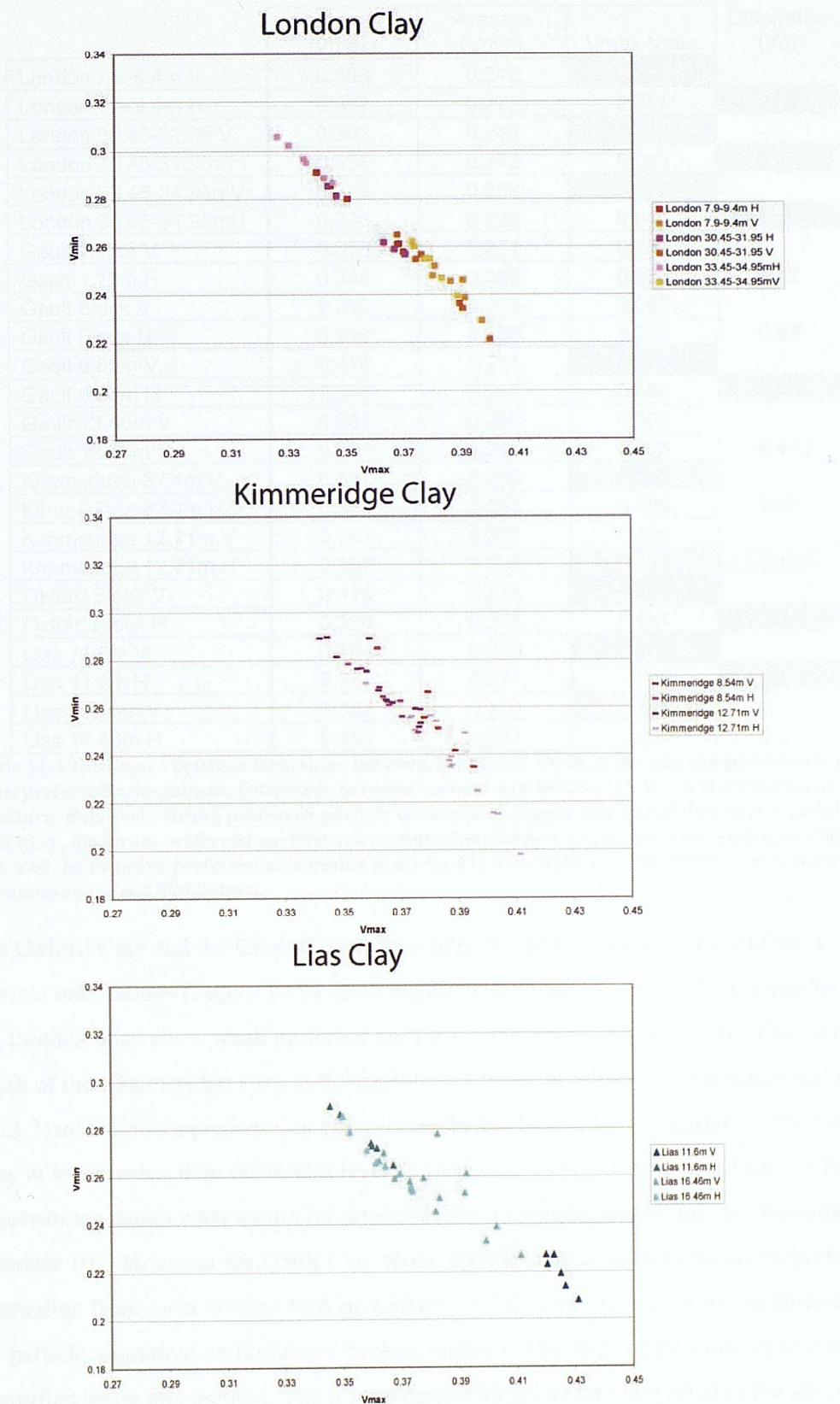


Figure 11.8 Continued from Figure 11.7 Each data point represents a rose diagram which was created from all images at a given scale for each mudrock. Rose diagrams from images with a magnification greater than 3000X are omitted. See Figure 11.6 for higher magnification.

	Average (Vmax)	Average (Vmin)	Vmax-Vmin	Difference (Vδ)
London 7.9-9.4m V	0.388	0.243	0.145	0.06
London 7.9-9.4m H	0.357	0.273	0.084	
London 30.45-31.95 V	0.383	0.245	0.139	0.05
London 30.45-31.95 H	0.356	0.272	0.085	
London 33.45-34.95mV	0.379	0.253	0.126	0.09
London 33.45-34.95mH	0.336	0.296	0.041	
Gault 1.72m V	0.358	0.271	0.087	0.02
Gault 1.72m H	0.348	0.282	0.066	
Gault Block V	0.360	0.273	0.087	0.03
Gault Block H	0.344	0.288	0.056	
Gault 6.82m V	0.416	0.211	0.205	0.14
Gault 6.82m H	0.348	0.283	0.065	
Gault 13.46m V	0.351	0.282	0.069	0.01
Gault 13.46m H	0.347	0.284	0.063	
Kimmeridge 8.54m V	0.381	0.256	0.125	0.03
Kimmeridge 8.54m H	0.364	0.265	0.099	
Kimmeridge 12.71m V	0.363	0.269	0.094	-0.05
Kimmeridge 12.71m H	0.387	0.239	0.148	
Oxford Block V	0.416	0.213	0.203	0.12
Oxford Block H	0.356	0.275	0.081	
Lias 11.6m V	0.424	0.219	0.205	0.12
Lias 11.6m H	0.357	0.277	0.080	
Lias 16.46m V	0.382	0.254	0.127	0.03
Lias 16.46m H	0.363	0.267	0.096	

Table 11-1 Difference between structures between Vmax and Vmin indicating samples strong and weak preferred orientations. Difference between vertical and horizontal (Vδ) is the strength of the structure. Relatively strong preferred particle orientations (upper quartile of data) are highlighted dark grey, moderate preferred particle orientations (second quartile of data) are highlighted light grey and the negative preferred orientation is marked in very light grey. Relatively weak preferred orientations are not highlighted.

The Oxford Clay and the Gault Clay from 6.82m bgl both display strong preferred particle orientations (Figure 11.3). Both depths in the Lias Clay and all three depths in the London Clay show weak preferred particles orientations (Figure 11.4). The vertical break of the Kimmeridge Clay at 8.54m shows a fabric at ~20-30° to the horizontal and at 12.71m it shows a preferred orientation on the horizontal broken surfaces. The Gault Clay at levels other than the 6.82m level shows no discernable structure. Many of these observations match what would be expected from the qualitative image descriptions (Chapter 10). However the Gault Clay block sample showed little evidence of preferred orientation from most vertical broken surfaces, it did however show a strong preference for particle alignment on horizontal broken surfaces. This type of structure cannot be quantified using this method. The results cannot therefore be interpreted in the absence of qualitative image analysis. From these images the preferred orientation of the Lias at 16.46m bgl and that of the London Clay at 30.45-31.95m bgl are relatively weak, their horizontal and vertical rose diagrams are almost identical (Figure 11.4) at X1500

magnification. Preferred alignment is the alignment of particles to a particular plane. A preferred orientation is seen only in the vertical or horizontal plane but not in both. In other words images perpendicular to a structure should not show the same structure. This difference between vertical and horizontal images can be used to quantify the strength of preferred orientations.

11.1.4 Rose diagram quantification

Several methods are available for the quantification of rose diagrams. The method used in this thesis is half circle addition (Fisher, 1993). The radial data from all of the rose diagrams are normalised by the total number of particle long axis measurements (2 directions per particle) in each rose diagram, so all the rose diagrams are comparable. Thus the sum of all the scalar measurements is equal to one. As all particles have a bidirectional long axis orientation the sum of all the vector measurements is zero. The sum of a half circle of scalars in any one direction will be 0.5; the sum of a half circle of vectors (Figure 11.5) will be a finite number with a maximum value of 0.5. By taking the half circle as all vectors within ninety degrees of the maximum direction (V_{max}), nineteen rather than eighteen directions are included, this allows the half circle ninety degrees from the maximum direction (V_{min}) to equal a minimum value of 0 due to the cancelling of the two end members. Thus the addition of nineteen vectors is equal to the addition of the closest seventeen. By plotting the maximum and minimum vectors against each of the relative levels of preferred orientation shown in the rose diagrams, they can be graphically compared and quantified (Figure 11.6).

By plotting V_{max} against V_{min} for all image sets with magnifications less than X3000 magnification for all of the mudrocks, comparisons between mudrocks can be drawn (Figure 11.7 & Figure 11.8). In these graphs the greatest preferred orientation possible would occur at $V_{max}=0.5$, $V_{min}=0$ towards the bottom right of the image. Most of the data points are spread on the line between strong preferred orientation (elliptical rose diagram) and no preferred orientation (circular rose diagram). A strong alternative to the method presented here which is more amenable to batch computer processing is the calculation of a fabric tensor (Ketcham and Ryan, 2004).

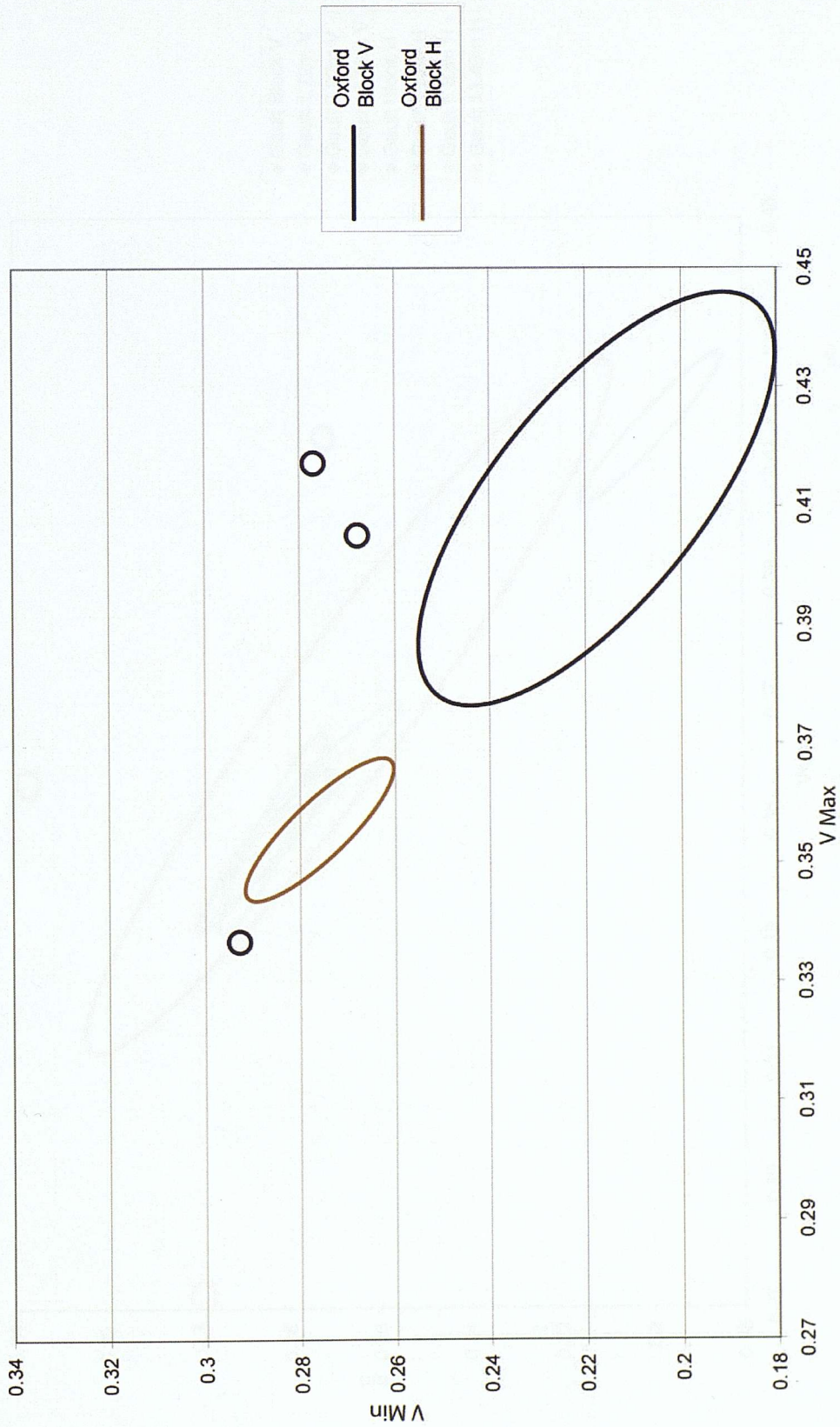


Figure 11.9 Distribution of orientation data for the Oxford Clay

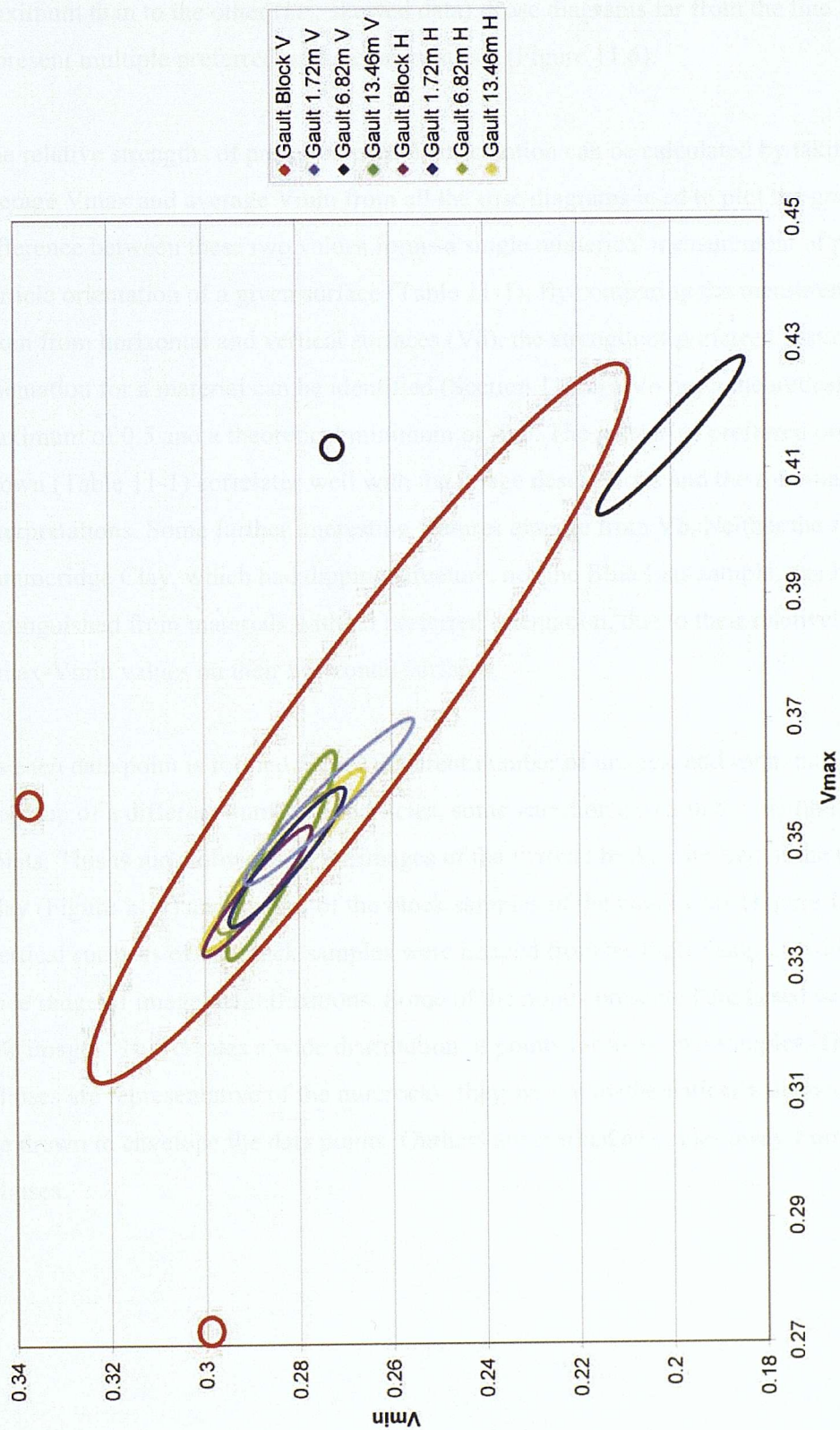


Figure 11.10 Distribution of orientation data for the Gault Clay

Results from this orientation analysis agree with the results presented here. Points above and to the right of this line represent rose diagrams with more data to one side of the maximum than to the other (*i.e.*, skewed data). Rose diagrams far from the line represent multiple preferred particle orientations (Figure 11.6).

The relative strengths of preferred particle orientation can be calculated by taking the average V_{max} and average V_{min} from all the rose diagrams used to plot the graph. The difference between these two values forms a single numerical measurement of preferred particle orientation of a given surface (Table 11-1). By comparing the measurement taken from horizontal and vertical surfaces ($V\delta$), the strength of preferred particle orientation for a material can be identified (Section 11.1.3). $V\delta$ has a theoretical maximum of 0.5 and a theoretical minimum of -0.5. The pattern of preferred orientation shown (Table 11-1) correlates well with the image descriptions and the rose diagram interpretations. Some further interesting features emerge from $V\delta$. Neither the shallow Kimmeridge Clay, which had dipping structure, nor the Blue Lias sample, can be distinguished from materials with no preferred orientation, due to their relatively high $V_{max}-V_{min}$ values on their horizontal surfaces.

As each data point is formed from a different number of images, and each image being made up of a different number of particles, some variation exists in the distribution of points. This is most obvious in the images of the vertical broken surface of the Oxford Clay (Figure 11.9) and images of the block samples of the Gault Clay (Figure 11.10). Vertical surfaces of the block samples were imaged from multiple samples, with a very wide range of image magnifications. Some of the points presented are based on very few images. This creates a wide distribution of points for these two samples. The ellipses are representative of the mudrocks, they are not mathematically defined; they are drawn to envelope the data points. Outliers are marked as circles away from the ellipses.



Figure 11.11 Distribution of orientation data for the Kimmeridge Clay

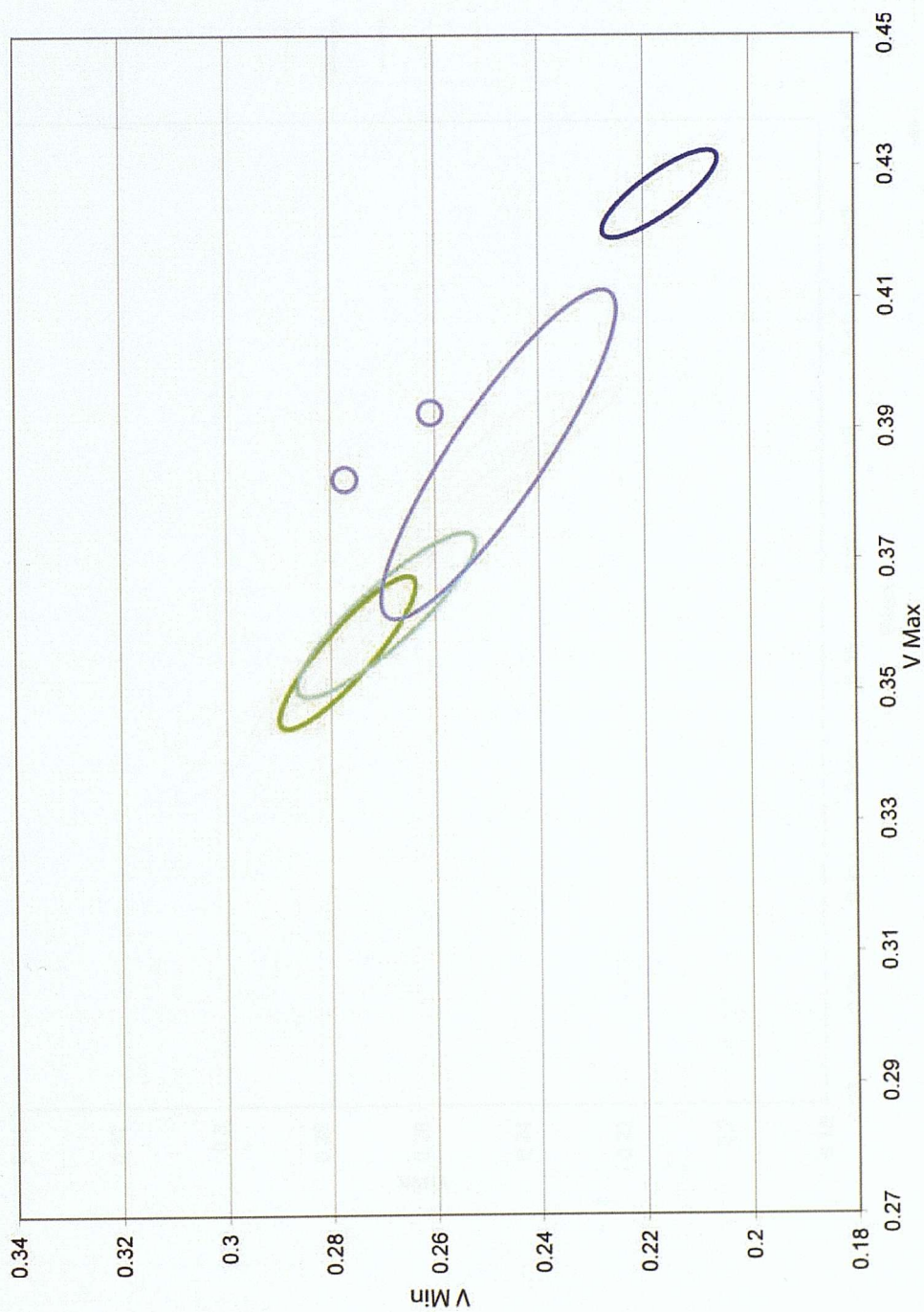


Figure 11.12 Distribution of orientation data for the Lias Clay

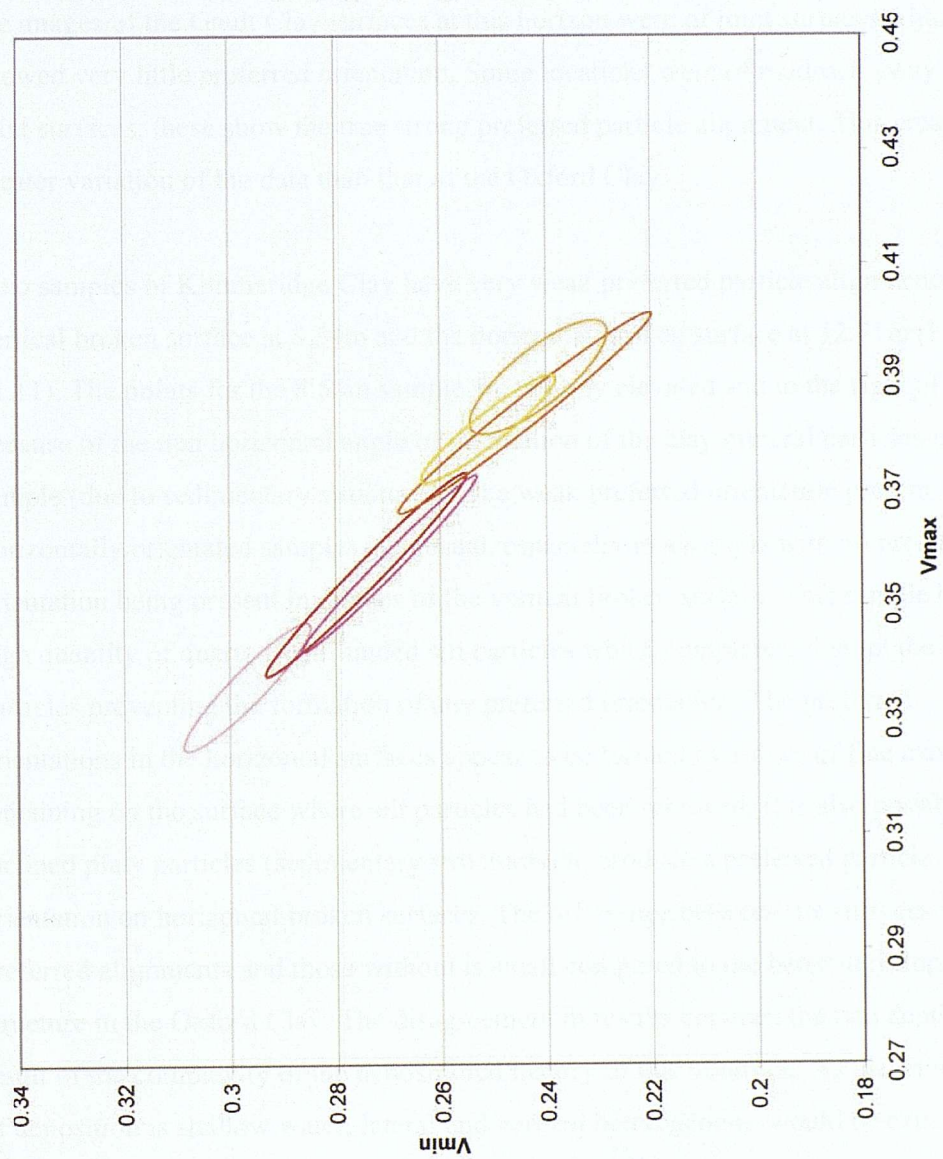


Figure 11.13 Distribution of orientation data for the London Clay

The Oxford Clay shows a clear difference in plot locations between the vertical and horizontal broken surfaces (Figure 11.9), showing a strong preferred particle alignment, even given the wide spread in the vertical broken surface data. Most of the Gault Clay data, whether from vertical or horizontal broken surfaces, plot in a similar location (indicating very little preferred orientation) on the graph (Figure 11.10). The vertical break in the Gault Clay at 6.82m clearly has a stronger preferred orientation than the other Gault Clay samples, plotting much higher than the majority of the other horizons. Images from the vertical breaks of the Gault Clay block samples vary widely; from absolutely no preferred orientation to a relatively strong preferred orientation. Most of the images of the Gault Clay surfaces at this horizon were of joint surfaces which showed very little preferred orientation. Some locations were of mudrock away from the joint surfaces; these show the true strong preferred particle alignment. This creates the greater variation of the data than that of the Oxford Clay.

Two samples of Kimmeridge Clay have very weak preferred particle alignments: the vertical broken surface at 8.54m and the horizontal broken surface at 12.71m (Figure 11.11). The points for the 8.54m sample are slightly elevated and to the right of the line because of the non horizontal angle of orientation of the clay mineral particles in this sample (due to sedimentary structures). The weak preferred orientation present in horizontally orientated samples is unusual, especially in a sample with no preferred orientation being present in images of the vertical broken surface. This sample had a high quantity of quartz-rich rounded silt particles which completely disrupt the platy particles preventing the formation of any preferred orientation. The preferred orientations in the horizontal surfaces appear to be formed by ridges of fine material remaining on the surface where silt particles had been removed. It is also possible for inclined platy particles (sedimentary structures) to produce a preferred particle orientation on horizontal broken surfaces. The difference between the surfaces with preferred alignments and those without is small compared to the better-developed structure in the Oxford Clay. The disagreement in results between the two depths is a result of the complexity of the depositional history of this mudrock. As the environment of deposition is shallow water, lateral and vertical heterogeneity would be expected over relatively small distances.

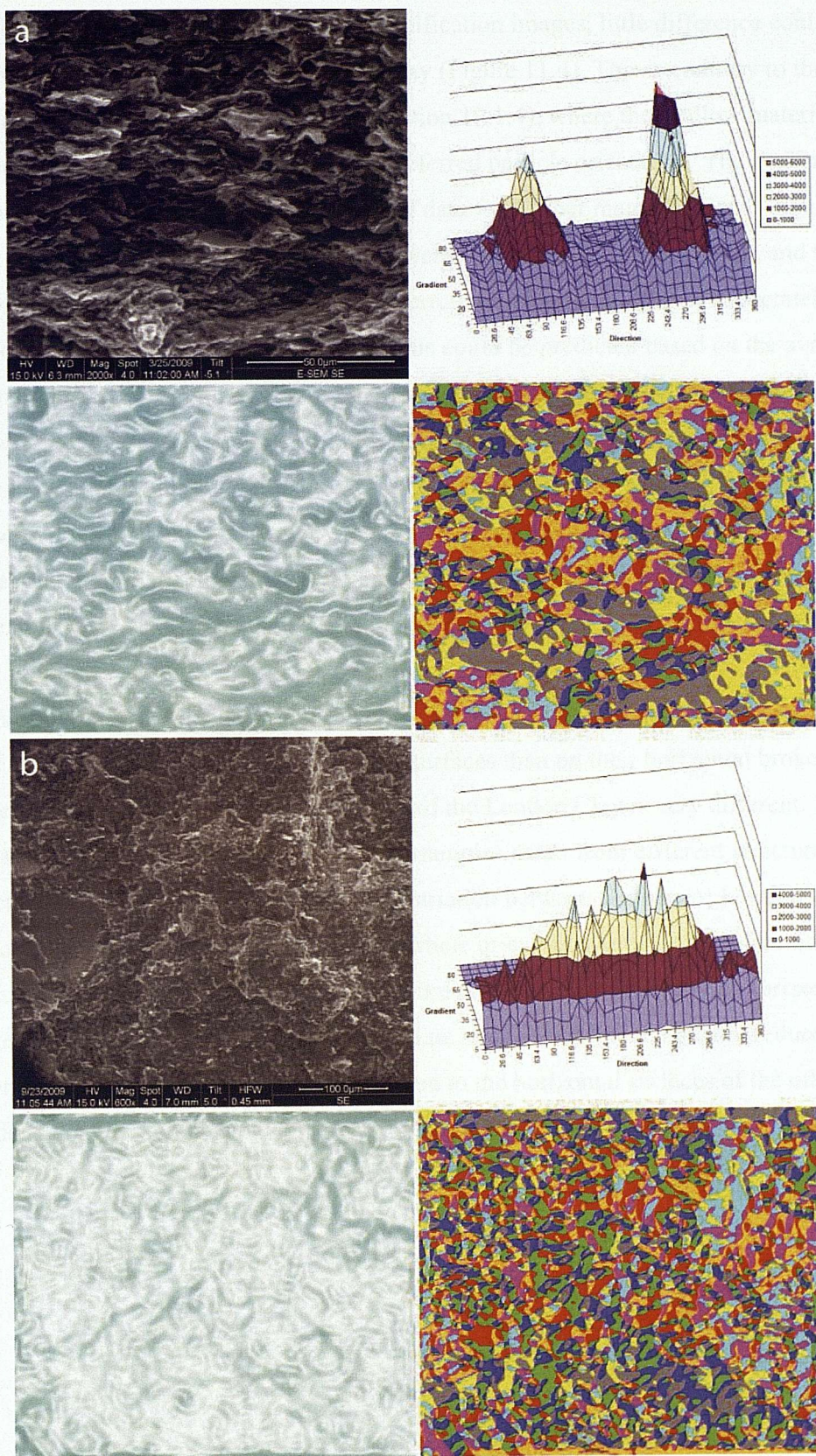


Figure 11.14 Oxford Clay block sample, vertical broken surface (a) horizontal broken surface (b), strong preferred particle orientation. See section 9.4 for explanation of images. Double peak indicates orientations of slopes of the linear peaks running across the surface. Linear slopes indicated by lines on gradient and aspect maps. This sort of structure is not present in the horizontal image (hence this indicates a preferred particle orientation).

Based on the rose diagrams for X1500 magnification images, little difference could be observed between the two depths of Lias Clay (Figure 11.4). This is contrary to the qualitative descriptions of the mudrock (Section 10.1.4), where the shallow material was described as having a much stronger preferred particle orientation. The qualitative interpretation is supported by the addition of data from other magnifications (Figure 11.12). The 11.6m bgl sample clearly has a very strong preferred orientation, and the 16.46m bgl sample has a wide range of preferred orientation data; some moderately developed, some very weakly developed. This could be predicted based on the available mineralogical data for the Lias materials. The Charmouth Mudstone has a greater proportion of clay minerals, and so is expected to have strong preferred orientation, while the Blue Lias has a greater proportion of calcite, much of which is in the form of microfossils, which allow a greater variability to the particle orientation. This combined with a reduction in the quantity of clay minerals in the Blue Lias results in a reduction in the preferred orientations observed.

All three depths investigated in the London Clay have a better developed preferred particle orientation on their vertical broken surfaces than on their horizontal broken surfaces (Figure 11.13). Each of the images of the London Clay is very different. The similar preferred particle orientations of the samples result from different structural aspects in each image set. This underlying variation between samples of London Clay is due to factors unrelated to their preferred particle orientation and it is therefore not measured by this method of analysis. The more pronounced topography of horizontal images of the 33.45-34.95m bgl sample, and its nodular appearance greatly reduces the preferred orientation on this surface compared to the horizontal surfaces of the other two depths in the London Clay (7.9-9.4m & 30.45-31.95m bgl).

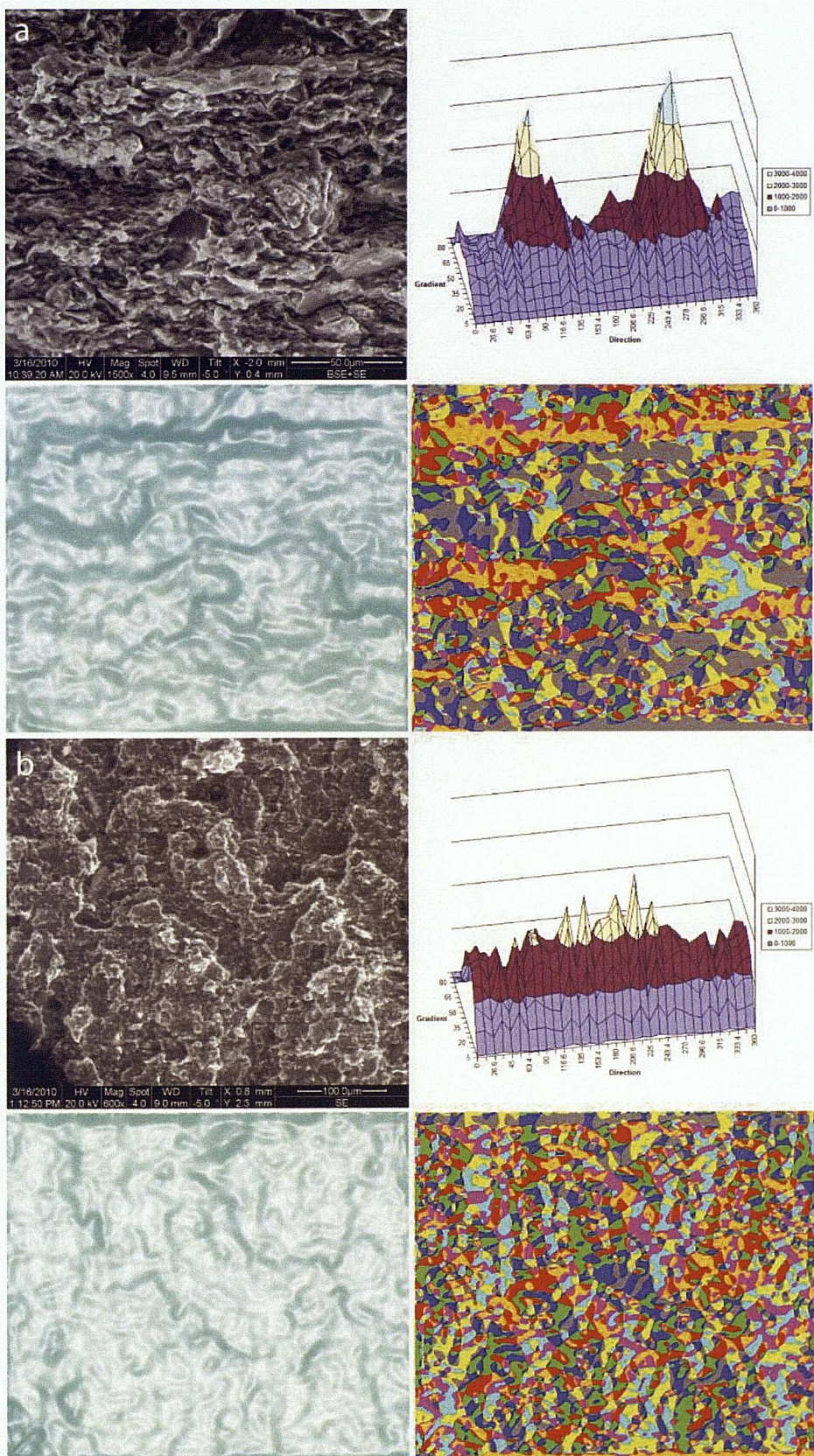


Figure 11.15 London Clay 30.45-31.95m, vertical broken surface (a) horizontal broken surface (b). See section 9.4 for explanation of images. Moderate preferred particle orientation similar to Figure 11.14, peaks in vertical surface analysis (a) possibly wider than in Figure 11.14

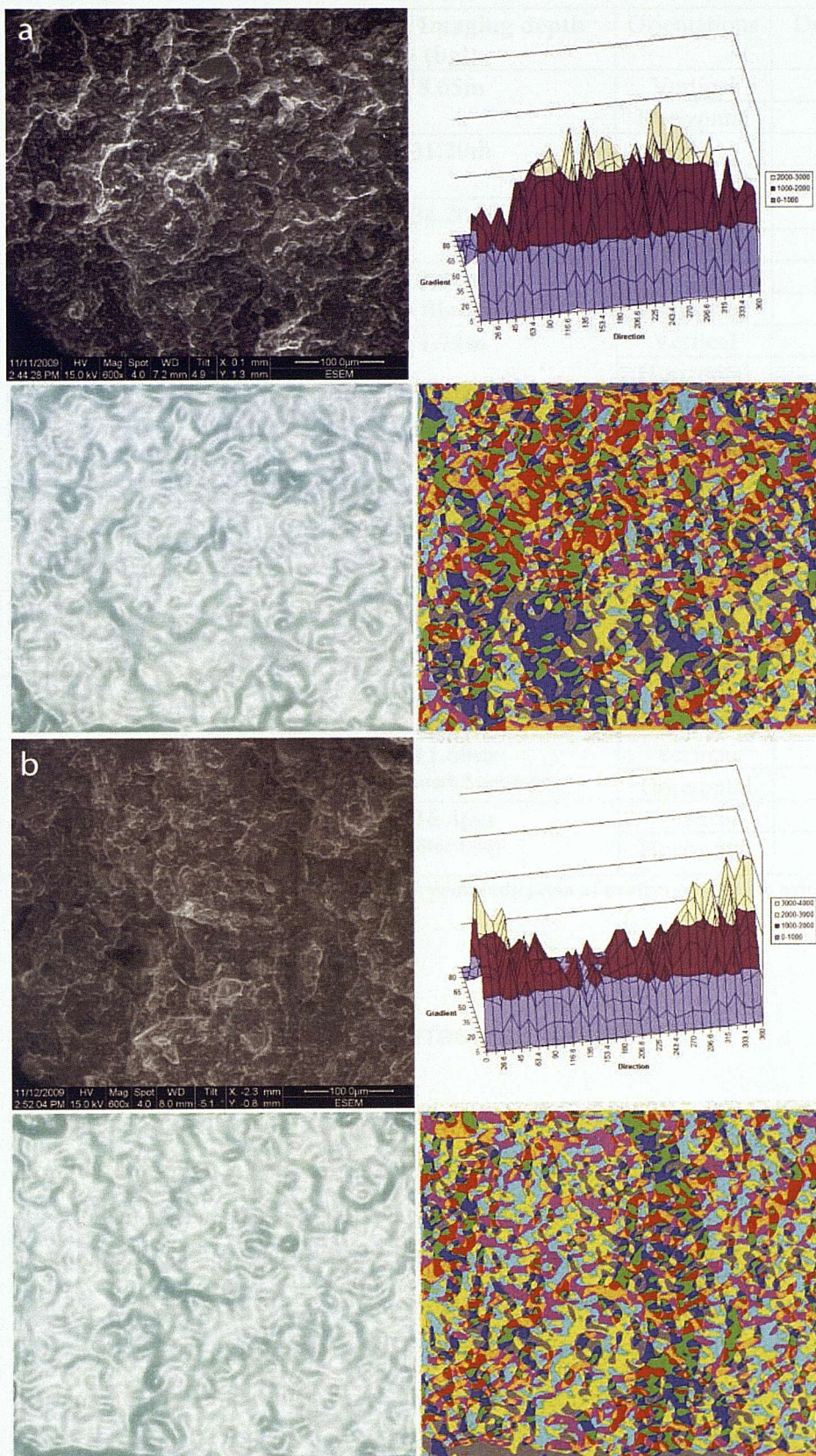


Figure 11.16 Gault Clay 1.72m, vertical broken surface (a) horizontal broken surface (b). See section 9.4 for explanation of images. No preferred particle orientation, both vertical (a) and horizontal (b) surface analysis is a single wide peak. Gradient and aspect map show circular rather than linear features. Colour blocks in the aspect map indicate the dominant direction of tilt of the surface.

Material	Average Imaging depth (bgl)	Orientations	Double Peak?
London Clay (Heathrow Terminal 5)	8.65m	Vertical	Yes
		Horizontal	No-Weak
	31.20m	Vertical	Yes
		Horizontal	No
	34.20m	Vertical	Yes-Weak
		Horizontal	No
Gault Clay (High Cross, Madingley)	3.00m Block	Vertical	No
		Horizontal	No
	1.72m	Vertical	No
		Horizontal	No
	6.82m	Vertical	Yes
		Horizontal	No
	13.46m	Vertical	No
		Horizontal	No
Kimmeridge Clay (Willowbrook Farm)	8.54m	Vertical	Yes-some places
		Horizontal	No
	12.71m	Vertical	No
		Horizontal	Yes
Oxford Clay (Elstow)	10.00m	Vertical	Yes
		Horizontal	No
Lias Clay (M5 Junction 13)	11.60m (Charmouth Mudstone)	Vertical	Yes
		Horizontal	No
	16.46m (Blue Lias)	Vertical	Yes-weak
		Horizontal	No

Table 11-2 Summary of whether the double peak indication of preferred particle orientation is seen in each material. See Appendix D.

11.2 Three-dimensional surface analysis

The ultimate aim of surface analysis is to quantify the variations in the surface in a meaningful fashion. Changes in surface topography should be described by magnitude, direction and scale. A method of quantification which encompasses all of these factors has not been achieved during this work. However the initial results of the surface analysis method (Section 9.4.1) which quantify magnitudes and direction of surface change at a specific scale (9x9 pixels) are a step towards this objective. The required measurement is similar to existing surface roughness measurements, but differs in that the surfaces are granular. They are composed of many loosely bonded particles and their presence is an artefact of the mechanism used to break the surface. The standard roughness measurements are included in Appendix D for comparison. There is great

variation in the surface topography. As no bulk sample analysis has been carried out, meaningful data can only be extracted from single images.

Therefore, the results may not be representative of the entire material. Idealised surfaces from three of the materials are presented here. The rest are presented in Appendix D. The main measurements taken from the surface are gradient and direction. Gradient is measured between 0 and 90°, where 0° is horizontal and 90° is vertical. Direction is measured between 0 and 360°, where 0° is on the positive x-axis, and anticlockwise increments are positive such that the positive y-axis is 90° (Section 9.4.1).

The three different types of surface (strong, moderate and weak preferred particle orientations) are: the Oxford Clay, which has a strong preferred particle orientation, the London Clay at 30.45-31.95m, which has a moderate preferred particle orientation and the Gault Clay at 1.72m, which has no preferred particle orientation. The surface analysis identifies the topographic expression caused by preferred particle orientation. The Oxford Clay vertical broken surface has a very strong binomial distribution of direction of maximum surface gradient (Figure 11.14 a). The maximum peak is 5346 pixels dipping at 70° towards 256°, the second peak is 3475 pixels dipping at 65° towards 90°. The asymmetry of the pixel distribution is caused by the tilt of the original surface. The horizontal surface of the Oxford Clay has a wide single peaked pixel distribution with its maximum dipping at 20° towards 217° (Figure 11.14 b). The gradient of pixels in this image is much lower than that of the vertical surface as the structure is not creating topography. This can be seen in the gradient maps, as the shade of blue is much lighter in the horizontal (Figure 11.14 b) than the vertical (Figure 11.14 a) image. The peak in the pixel distribution for the horizontal image is caused by a small tilt in the original surface. The morphology of features in the gradient and aspect maps also qualitatively reflects the structure of the surfaces. Vertical broken surfaces clearly contain linear features, whereas horizontal broken surfaces show more sub-rounded features. The granular aspect map shows a preference for blue and green, due to the surface tilt towards 217° (Figure 11.14 b).

The London Clay generally follows this same pattern. Vertical broken surfaces have a binomial distribution of pixel orientations (Figure 11.15 a). The peaks are less pronounced and wider than those for the Oxford Clay. This represents greater variation in the orientations on the surface. However there is great variation between different

images from a single mudrock so, without adding together distributions from a large number of images, little can be made of this with regards to the overall material structure. Preferred orientation is expressed as horizontal lines in the gradient and aspect plots for the vertical surfaces while horizontal surfaces appear more granular, the number of horizontal lines is fewer and they are more widely spaced. The distribution of pixels for the horizontal broken surface forms a single, low and very wide peak. The peaks for the vertical surface are 3977 pixels orientated at 60° to 270° and 3328 pixels orientated at 65° to 90°. This time the single peak for the horizontal surface is only slightly shorter than that of the vertical being 3257 pixels orientated at 30° towards 217°. Even so the overall amount of inclination of pixels is clearly less on the horizontal surfaces than on the vertical, indicating more subdued topography.

The Gault Clay at 1.72m bgl has no preferred particle orientation due to material changes resulting from weathering. Both the vertical (Figure 11.16 a) and the horizontal (Figure 11.16 b) broken surfaces have a single low relief, broad peak. The peak for the vertical surface is 2753 pixels with an orientation of 25° towards 143°. The peak of the horizontal surface is 3093 pixels with an orientation of 40° towards 323°. These measurements are affected by the tilt of the surface and in the horizontal broken surface by small step changes in the surface elevation. This has produced broad regions of similar character (Figure 11.16 b).

All images do not show these general features because of local variations (*e.g.*, fossils and minor structures). However, if all data from a large number of images were combined, then an overall character for a surface may emerge. The presence of a double peak is the main indicator of preferred particle orientation. The samples in which this double peak occurs match those which had strong preferred particle orientations in the 2D analysis (Table 11-2). This approach would only be useful once the method is perfected. The plot of direction against gradient currently uses a 9x9 grid of pixels allowing 32 directions. If an 11x11 grid were used, 40 directions would be possible. The effectiveness and efficiency of this approach has yet to be evaluated. At the moment there are a number of factors that remain to be evaluated including scale effects on roughness measurement and its quantification. The effect of a square shape, in particular on preferential partitioning has also not been investigated. For example would the use of a circular shape improve the analysis? These questions must be answered before the method can be employed successfully on a larger scale. The initial results presented

here however, are very positive and provide an independent corroboration of the preferred particle orientation measurements by other methods (Section 10.1, and 11.1).

11.3 Preliminary laboratory data

All of the laboratory data shall be presented and discussed elsewhere (Brosse, 2011; Hosseini-Kamal, 2011). Some preliminary findings can be presented here (Figure 11.17 to Figure 11.23). Stiffness data shows that the measured anisotropy of the Gault Clay (Figure 11.17) and the Kimmeridge Clay (Figure 11.18) are very similar being 2.00 and 2.01 respectively. The Oxford Clay (Figure 11.19) has an elevated anisotropy of 2.44. A similar effect occurs in the material stress paths. The Gault Clay (Figure 11.20) and the Kimmeridge Clay (Figure 11.21) show very little difference between their intact and reconstituted samples. The Oxford Clay (Figure 11.22) shows a clear difference (structure) between the two samples.

In the hollow cylinder, the Oxford Clay clearly has a strong anisotropy (Figure 11.23). Its strength at failure is greatest for the vertical samples and least at 70 degrees. The Gault and London Clay have a lesser amount of anisotropy and the Kimmeridge Clay has almost no anisotropy (Figure 11.23).

These preliminary results compare favourably with the image analysis results. The strong alignment of the Oxford Clay and the lack of alignment in the silty Kimmeridge Clay match the measurements of anisotropy. The deepest horizon of the Gault Clay is measured (9.4-11.9m bgl), showing little anisotropy, this correlates with the deepest imaging horizon (13.46m) which also showed little preferred particle alignment.

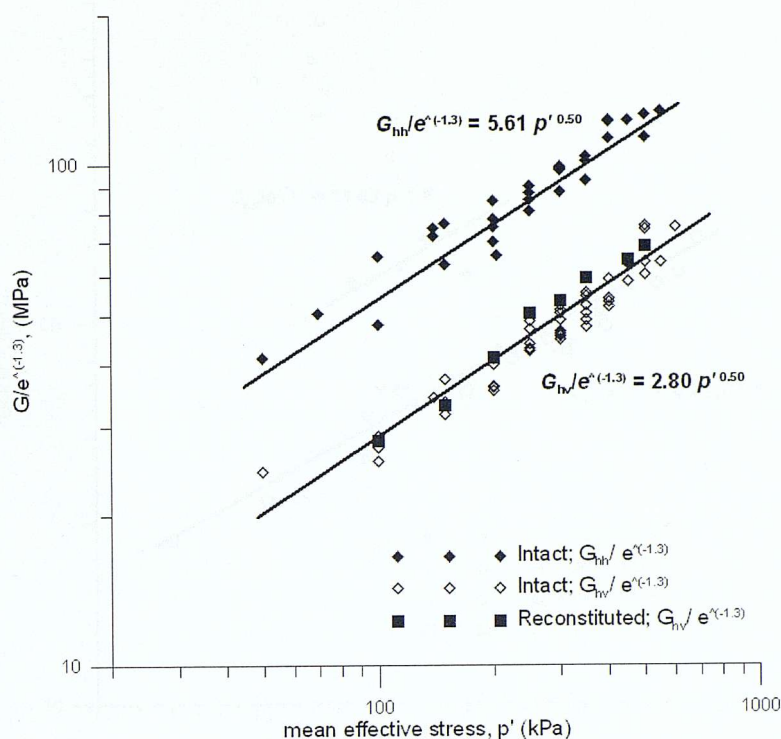


Figure 11.17 Stiffness data from the Gault Clay obtained from Bender elements normalised by void ratio from (Hosseini-Kamal, 2011). G_{hh} (horizontal), G_{hv} (vertical) $G_{hh}/G_{hv}=2.00$

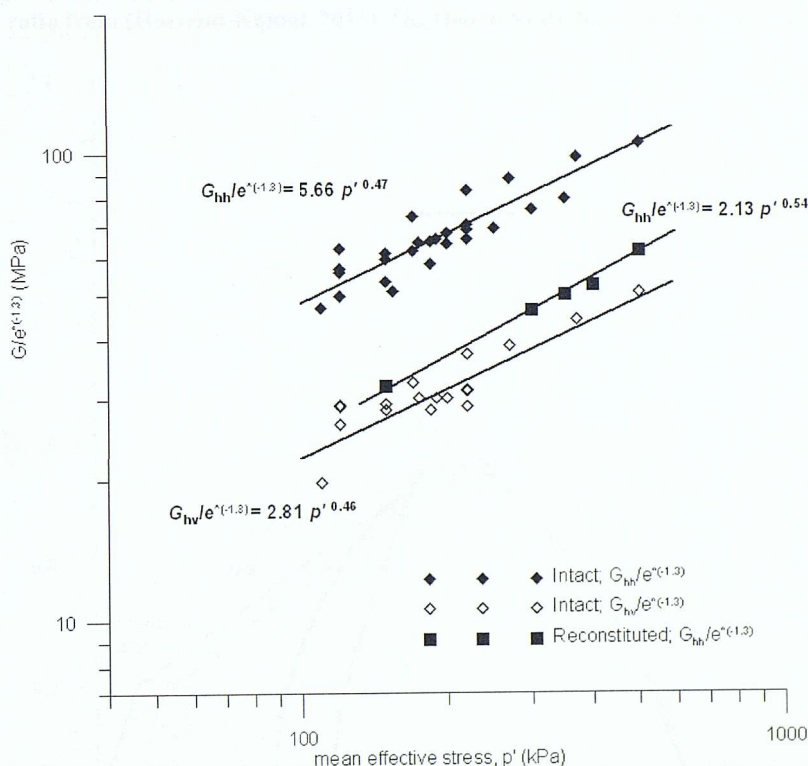


Figure 11.18 Stiffness data from the Kimmeridge Clay obtained from Bender elements normalised by void ratio from (Hosseini-Kamal, 2011). G_{hh} (horizontal), G_{hv} (vertical) $G_{hh}/G_{hv}=2.01$

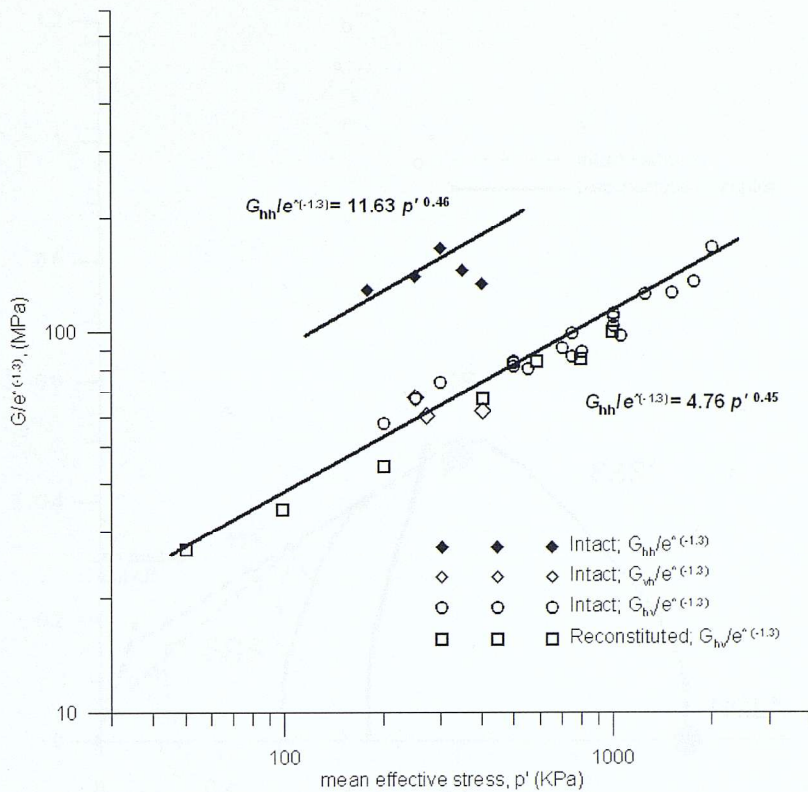


Figure 11.19 Stiffness data from the Oxford Clay obtained from bender elements, normalised by void ratio from (Hosseini-Kamal, 2011). G_{hh} (horizontal), G_{hv} (vertical) $G_{hh}/G_{hv}=2.44$

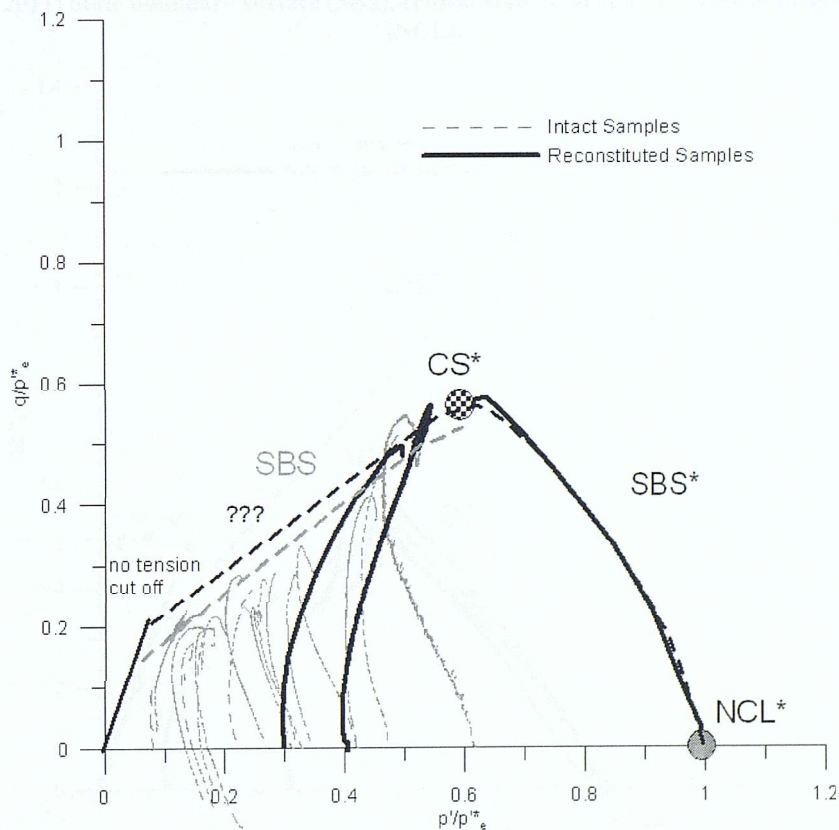


Figure 11.20 Stress path of the Gault Clay intact and reconstituted samples from (Hosseini-Kamal, 2011). State boundary surface (SBS), critical state (CS) and Normal Compression Line (NCL).

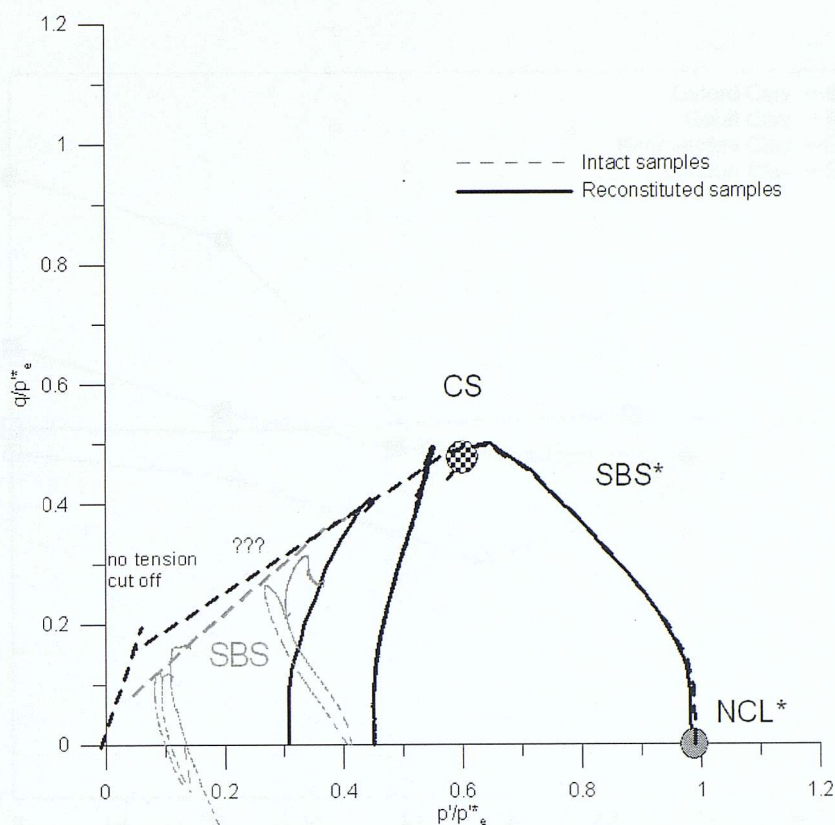


Figure 11.21 Stress path of the Kimmeridge Clay intact and reconstituted samples from (Hosseini-Kamal, 2011) State boundary surface (SBS), critical state (CS) and Normal Compression Line (NCL).

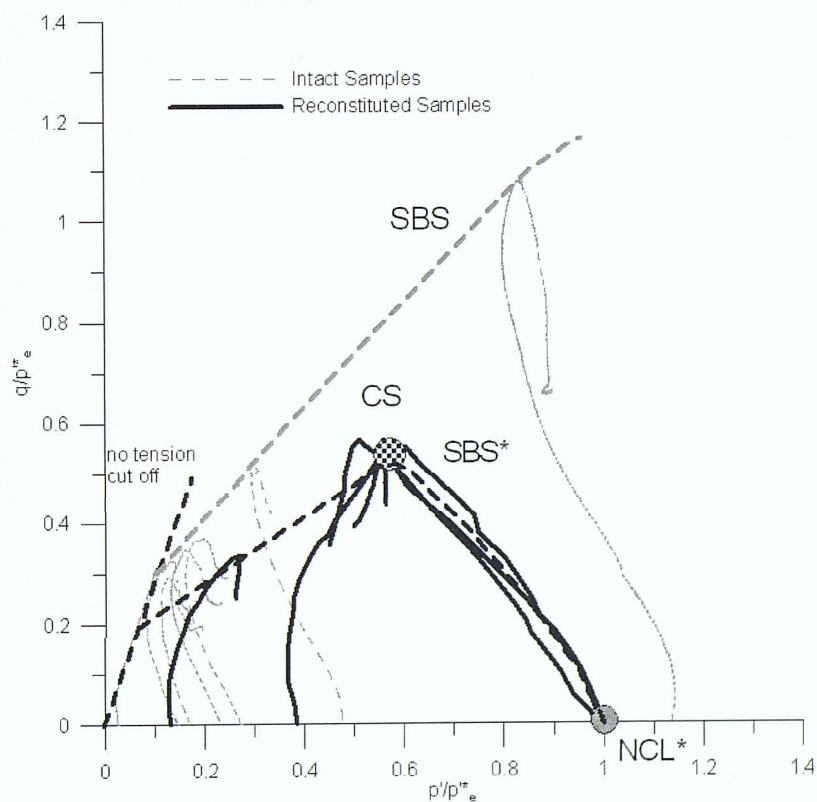


Figure 11.22 Stress path of the Oxford Clay intact and reconstituted samples from (Hosseini-Kamal, 2011). State boundary surface (SBS), critical state (CS) and Normal Compression Line (NCL).

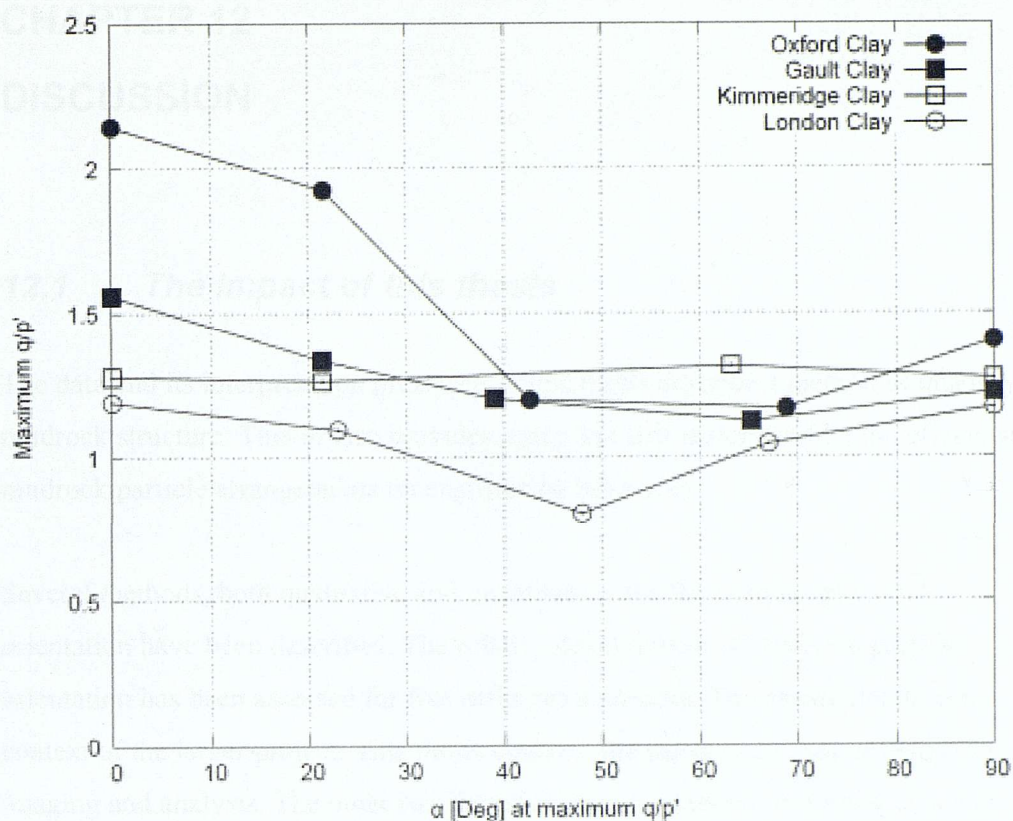


Figure 11.23 Anisotropy of samples measured using a hollow cylinder, Gault Clay 9.4-11.9m bgl. Kimmeridge Clay 11.04-12.06m bgl. From (Brosse, 2011).

CHAPTER 12

DISCUSSION

12.1 *The impact of this thesis*

The data and its interpretation presented in this thesis describe a method of quantifying mudrock structure. This in turn provides a step towards understanding the effects of mudrock particle arrangements on engineering behaviour.

Several methods, both qualitative and quantitative, for the analysis of particle orientation have been described. The relative development of preferred particle orientation has been assessed for five different mudrocks. This thesis stands in the context of the larger project. This thesis contains the background, site investigation data, imaging and analysis. The other two theses contain the laboratory testing data and analysis focusing on triaxial testing (Hosseini-Kamal, 2011) and hollow cylinder testing (Brosse, 2011). The most important outcomes of this work are dependent on the comparison of the image and particle orientation data with the laboratory testing data (Section 11.3).

The scope covered by the project as a whole will be published this year (Wilkinson et al., 2011). Initial methods and results from the image analysis part of the project have already been published (Wilkinson and Fenton, 2010). It is expected that several further aspects of this project will be published as journal articles once all aspects of the project have been completed. Prior to the completion of the larger project, the impact of this thesis can be described based on the completion of its initial aims. A statement of how this thesis addresses these aims is presented below.

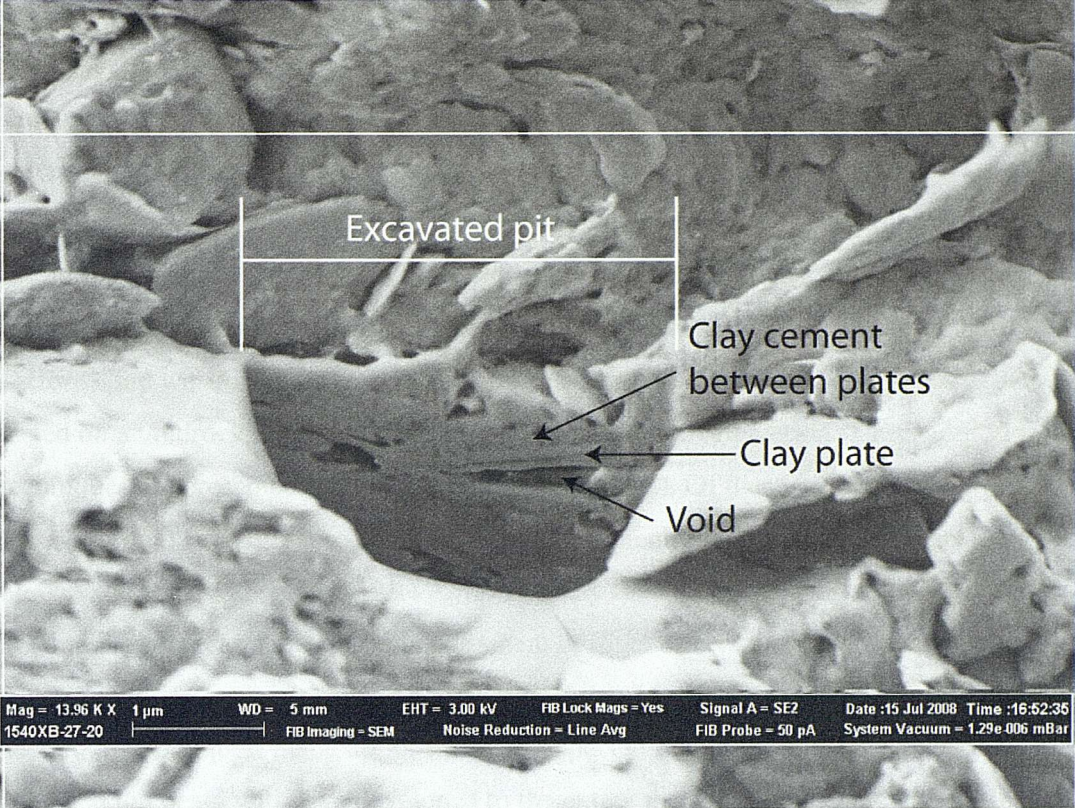


Figure 12.1 Focussed ion beam image of the Oxford Clay, showing clay particles with clay mineral cement between particles (Appendix C).

Figure 12.2 Relative amounts of burial for each material (Figure 3.11), depths of London Clay from (Hight et al., 2007). Upper estimate based on stratigraphy, lower estimate based on Apatite fission track analysis (Section 3.4).

Name	Depth of burial by stratigraphy, estimated depth, (depth range) (m)	Depth of burial by apatite fission track analysis estimated depth, (depth range) (m)
London Clay	200 (150-300)	-
Gault Clay	284 (277-375)	871 (595-1166)
Kimmeridge Clay	412 (389-512)	1081 (795-1366)
Oxford Clay	582 (536-704)	1126 (840-1411)
Lias Clay	788 (707-965)	1700 (1414-1986)

Figure 12.2 Relative amounts of burial for each material (Figure 3.11), depths of London Clay from (Hight et al., 2007). Upper estimate based on stratigraphy, lower estimate based on Apatite fission track analysis (Section 3.4).

12.1.1 Imaging of microstructure

The imaging methods in the initial aims of this thesis were environmental scanning electron microscopy (E-SEM), x-ray computed tomography (X-ray CT) and 3D imaging. X-ray CT was discounted after early investigation as at its maximum resolution it is not able to identify individual clay mineral particles. Several imaging techniques were tested to find a replacement for this method. The early sampling of the Oxford Clay was fortunate as it allowed a great number of imaging techniques to be tested on a mudrock with a very strong preferred particle orientation. These additional techniques are presented in the appendix (Appendix C). Of these techniques, only the focussed ion beam (FIB) produced images of sufficient quality for three dimensional models to be created (Figure 12.1). Unfortunately the high cost of this method was beyond the project budget. The abandonment of three dimensional block imaging made several of the later aims unattainable. However the good results obtained from the cross section of techniques, made up for this. It is expected that in the next few years the focussed ion beam will have developed to the extent that it becomes practical for the analysis of mudrocks.

E-SEM imaging, including stereo imaging to create three dimensional images of mudrock surfaces was identified as the best imaging method to be applied across all the mudrocks. The first four surfaces, vertical and horizontal broken surfaces from the block samples of the Oxford and Gault Clay were completed relatively early during the project. This allowed several methods for analysing the SEM images to be investigated and perfected. Fourteen surfaces had to be imaged from the other mudrocks, as they were taken from boreholes. To allow better comparisons with the London Clay a further six surfaces both horizontal and vertical from three depths were imaged and analysed using the same method. These images were obtained at the start of the third year of the project.

The E-SEM was a particularly good method for the analysis of mudrocks because moisture is not removed during imaging. It provides images of excellent resolution and it allowed the creation of three-dimensional surface models. Considering that relatively little sample preparation is necessary, it is an excellent imaging tool. One element of imaging which greatly enhanced the results was the selection and use of consistent imaging magnifications after the initial testing of the method. This allowed greater

numbers of images to be taken and compared, removing much of the noise in single unusual images from the results.

12.1.2 Quantification of microstructure

In Chapter 4 structure was defined as being the combination of particle arrangement and bonding (cementation). It was also defined as the property of the material which allows it to maintain a void ratio at higher stress levels than a reconstituted sample of the same material. Preferred particle orientation will be disrupted by the reconstitution of a soil. So in shallowly buried mudrocks the resulting preferred particle orientation will cause a response associated with a structured soil.

A full analysis of structure requires the quantification of stress concentrations which form between the particles of the mudrock as it is loaded. It must show how the macro application of pressure is translated to particles interactions within the mudrock. For this to be possible full 3D imaging of blocks of mudrock must be undertaken and the properties of each particle type must be measured. Until this is achieved, although the effects of structure can be measured and they can be related in a general way to the overall arrangement of particles, the relative effects of the different components of a mudrock's structure cannot be separated.

As mentioned above, the focused ion beam may in the future allow the creation of 3D block models of mudrocks at the particle scale. If a method of measuring the strength and stiffness (including anisotropy) of these particles was found then a complete micro-mechanical model could be created. Only then could the causes of the laboratory behaviour of mudrocks be fully investigated. In the absence of complete three-dimensional data, only preferred particle orientation and its relationship to anisotropy can be investigated quantitatively.

The three-dimensional surface analysis presented in the results is no replacement for a full three-dimensional model of the mudrock. It allows the topography of broken surfaces to be measured which relates to mudrock structure (as preferred particle orientation also does). Further work on this method will result in an improvement in the measurement of surface roughness. It is unfortunate that only the initial results of this method could be presented within this thesis.

12.1.3 Micro-mechanical models

With the lack of a three-dimensional model it is impossible for a micro-mechanical model to be created. When such a model becomes possible in the future it will revolutionise the modelling of mudrock behaviour as the prediction of unusual material behaviour will become possible. However, predictions of the engineering behaviour of mudrocks can be made based on qualitative image description (Collins and McGown, 1974). The creation of such a model would require further work.

12.1.4 Geological processes and micro-mechanical behaviour

With the absence of a micromechanical model, relationships cannot be drawn between geological processes and micro-mechanical behaviour. However links can be drawn between geological processes and the generation of fabric and cementation. The preferred particle orientation measurements in the results can be related to depositional and burial processes. This forms the majority of the remainder of this discussion. One important observation from this work is that platy particles (clay minerals) do not have to be present in a mudrock. Mudrocks with no platy particles do not form any part of previous categorisations of mudrock fabric (except possibly honeycomb fabric). Mudrocks containing a low proportion of clay minerals (*e.g.*, rock flour), will behave very differently to those with high quantities of clay minerals. Section 12.3 focuses on the effects of geological history on the creation of structure.

12.2 Trends in preferred particle orientation

Three materials have a strong preferred particle orientation: the Oxford Clay, 6.82m in the Gault Clay and 11.60m in the Lias Clay. These materials are spread through time from the Early Jurassic to the Middle Cretaceous. They are also spread through a large range of depths of maximum burial, from 300-870m for the Gault Clay to 800-1700m for the Lias Clay (Figure 12.2). Such a range of conditions for strong preferred particle orientations indicates that deep burial is not necessary to form a strong preferred particle orientation.

Moderate preferred particle orientations were observed in all depths of the London Clay, arguably all depths of the Kimmeridge Clay and 16.46m in the Lias Clay. The London Clay contains many silty horizons where a weaker preferred particle orientation might be expected, however the orientation measurements throughout are remarkably consistent. Even in horizons which appear rich in clay minerals a spread is observed in the orientations of the crystals present. The relatively shallow burial (200m) of London Clay (Figure 12.2) may possibly explain these observations, where clay mineral rich horizons have not been compressed enough for strong preferred particle orientations to form. As strong preferred particle orientations are observed within the Gault Clay, a lack of sufficient burial cannot be used as an explanation for materials buried deeper than the Gault Clay. Therefore other effects in addition to the depth of burial are important for determining the particle arrangement of mudrocks. Moderate preferred particle orientations are present throughout the material and depth of burial spectrum.

The Gault Clay is the only mudrock which has multiple samples with very weak or negligible preferred particle orientations. In the Gault at 1.72m the reduction in preferred orientation is probably the result of weathering (Section 10.1.2), whereas at 13.46m it is either a diagenetic effect or a result of the presence of fossils. As the Gault Clay at 6.82m has a clear strong preferred orientation, depth of burial is not the sole cause. If this was the case then other mudrocks which have experienced greater or lesser depths of burial may also have no preferred orientation.

The effects of depth of burial are initially to cause the realignment of platy particles (where they are present) to form a preferred orientation (Curtis, 1985). This realignment occurs by rotation, and can only occur until particles come into contact with each other such that they rotate no further. Thus this process is limited by the initial depositional conditions of the mudrock, both mineralogical and micro-structural. Further alignment of particles occurs through either the breaking of particles, pressure solution and reprecipitation, or reactions causing mineralogical changes.

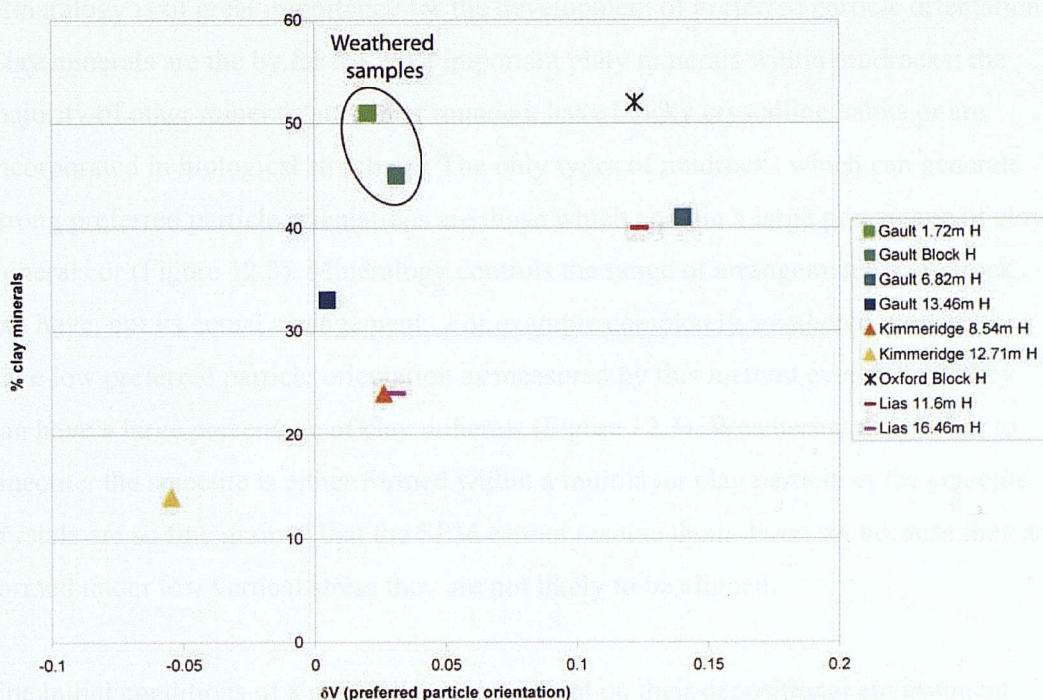


Figure 12.3 Plot of preferred particle orientation δV (Section 11.1.4) against percentage of clay minerals (Chapters 5-8), for the mudrocks sampled as part of this thesis.

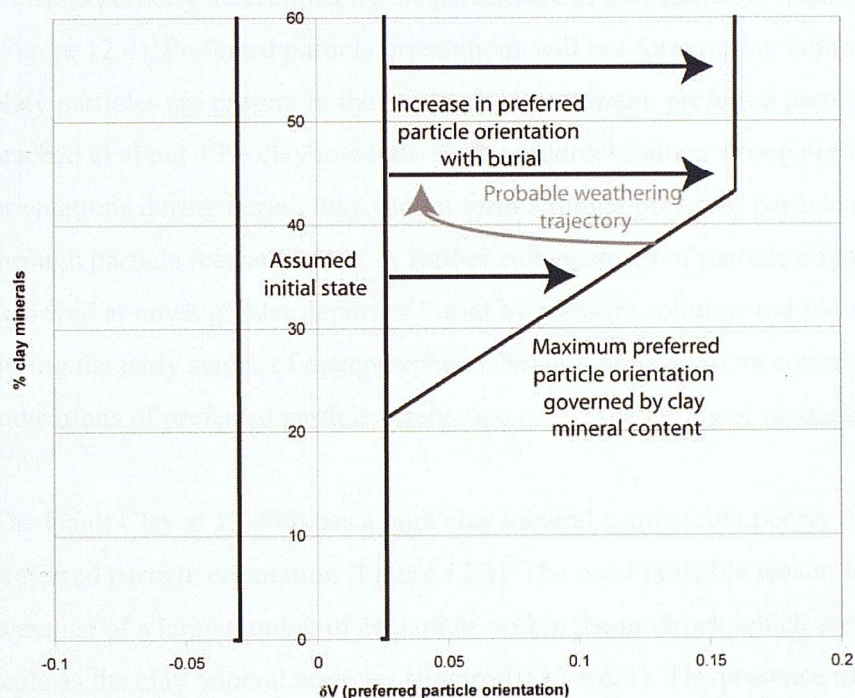


Figure 12.4 Interpretation of Figure 12.3, mudrocks are assumed to have an initial state of preferred orientation and an initial amount of clay minerals defined by depositional processes. The early stages of burial cause an increase in preferred particle alignment, up to a maximum which is defined by the initial amount of clay minerals within the mudrock. A maximum preferred orientation cut off occurs between 40% and 45% clay mineral content. Weathering decreases the preferred particle orientation of the mudrock and slightly increases the amount of clay minerals through chemical reactions.

Mineralogy is of great importance for the development of preferred particle orientation. Clay minerals are the by far the most important platy minerals within mudrocks; the majority of other minerals are either rounded, have blocky crystalline habits or are incorporated in biological structures. The only types of mudrocks which can generate strong preferred particle orientations are those which contain a large percentage of clay minerals or (Figure 12.3). Mineralogy controls the range of arrangements a mudrock can have, not its actual arrangement. For example completely weathered mudrocks have low preferred particle orientation as measured by this method even though they can have a large percentage of clay minerals (Figure 12.3). Weathering alters illite to smectite, the smectite is either formed within a multilayer clay particle or the smectite crystals are so fine grained that the SEM cannot resolve them. Even so, because they are formed under low vertical stress they are not likely to be aligned.

The initial conditions of a mudrock are dependent on their depositional environment. Normally their preferred particle orientations increase with burial up to a maximum which is partially determined by the percentage of clay minerals within the mudrock (Figure 12.4). Preferred particle orientations will not form during compression if few platy particles are present in the mudrock. A maximum preferred particle orientation is reached at about 40% clay minerals. These mudrocks attain strong preferred particle orientations during burial; they cannot form stronger preferred particle orientations through particle rearrangement. A further enhancement of particle alignment can be achieved at much greater depths of burial by pressure solution and re-precipitation and during the early stages of metamorphism. Several processes can completely remove all indications of preferred particle orientation (*e.g.*, weathering or reconstitution).

The Gault Clay at 13.46m has a high clay mineral content, but poorly developed preferred particle orientation (Figure 12.3). The most probable reason for this is the presence of a large number of coccoliths within the mudrock which are on the same scale as the clay mineral particles (Figure 10.12 e & f). The presence of the two different types of platy particle (clay minerals and coccoliths) appears to stop either from aligning. In general fossils can have a large impact on the behaviour of geological materials. Many flat shells are aligned to bedding in the Jurassic mudrocks. In the Gault Clay rounded microfossils and coccoliths are more common. So biological evolution, and the fauna present in the depositional environment of each mudrock is also having an

effect on the behaviour of the mudrocks in the laboratory at the present day. Calcite morphology is also an important factor for determining mudrock behaviour.

12.3 Geological history

The events in a mudrock's geological history are responsible for its structure. Ideally a study of mudrock structure would have either a consistent deposited material, or a consistent burial history. This is not possible in geological systems as the mineralogy and the geological history of deposits change with physical location (Section 3.3). By selecting one stratigraphic horizon of one mudrock and measuring its structure in different locations across the UK this can be improved. Even so, the effects of depositional and burial histories are entwined and cannot be separated, because the structure caused by burial overprints that caused by deposition and this in turn can be modified during uplift and by late-stage surficial or near surface processes. In cases where the depositional structure has been completely replaced, it may still be inferred from other data about the depositional environment.

12.3.1 Effects of depositional history

The depositional environment of a mudrock sets its initial conditions and from this point on the mudrock evolves. The initial mineralogy of each mudrock remains unchanged during early burial. It is controlled by the sediment sources for the depositional environment. Thus the initial mudrock composition is a function of weathering of the surrounding landmasses, transport and deposition mechanisms, biogenic materials from the fauna in the environment, and occasionally distal volcanic input.

As the input by weathering is generated through the erosion of the weathered material from the landmasses present at the time of deposition, it is important to determine the palaeogeography at the time for deposition of each mudrock. The landmasses which are being eroded are similar throughout the Jurassic (Section 3.3.1). The landmasses from which the Gault and London Clays are derived include much larger land areas from which materials are derived. Both of these mudrocks partially originate from the erosion of the Jurassic mudrocks (Section 3.1.1).

The clay mineralogy is moved towards a smectite dominant system if a significant volume of volcanic ash is mixed into the depositional environment. This can either occur by direct input of ash into the sea or the erosion and transportation of terrestrial ash deposits (Section 3.2.1).

The biogenic content of mudrocks changes depending on the nature of the fauna in the depositional environment. The Lias Clay has many flat shells ($\sim 10\text{-}15\mu\text{m}$), some diatoms, and few coccoliths. Flat shells are numerous in the Oxford Clay ($<5\mu\text{m}$) while some samples show coccoliths. Relatively few shell fragments are present in the Kimmeridge Clay, although some horizontal surfaces display coccoliths. The Gault Clay is full of the rounded shells of microfossils and numerous coccoliths. Shell fragments are rare in the London Clay. Those that are present are curved and no coccoliths have been identified. Flat shells in the Lias and Oxford Clay reinforce the anisotropy of these mudrocks. The change to rounded shells in the Gault Clay probably weakens this material (as it no longer has the horizontal reinforcement that flat shells provide). The increase in coccoliths in the Gault Clay can completely disrupt the preferred particle alignment of the material as the coccoliths are of similar size to the clay mineral particles (Section 10.1.2).

Previous investigations of mudrock microstructures focused solely on the arrangement of the clay minerals (Section 4.3). None of the mudrocks presented in this thesis is formed entirely of clay mineral particles. Mudrocks formed of 100% clay minerals are very rare. However, under initial depositional conditions clay minerals can sustain a very high void ratio. Therefore the initial volume of the mudrock containing clay minerals can be very high compared to the volume containing silt particles. This ratio will decrease with burial as the voids between clay minerals collapse. All of the diagrams of mudrocks structure shown in Section 4.3 are of depositional structures. Very few representations have been created for diagenetic structures from more deeply buried mudrocks.

If a mudrock has a high quantity of clay minerals then a relatively strong preferred particle orientation can exist upon deposition (Section 4.3). The dispersed fabric has a very strong preferred particle orientation. Turbostatic and stack fabrics create local preferred orientations which change from place to place. As clay particles have an electric charge on their surfaces they naturally repel each other. Energy is normally

required to create a strong alignment. Dispersed aligned fabrics are normally formed where organic matter is present in the inter particle space to overcome the electric charge. The Oxford Clay and the Kimmeridge Clay have the highest organic contents. The Oxford Clay has a very strong particle alignment, whereas the Kimmeridge Clay has a much weaker alignment. This is because the sampling location from which the Kimmeridge Clay was taken had limited clay mineral content, so the alignment could not form. As the London, Gault and Lias Clays contain much less organic material than the Oxford and Kimmeridge Clay, they probably initially had more open structures.

A possible exception to this is if the mudrock were initially deposited under turbulent conditions such as those suggested for parts of the Kimmeridge Clay (Macquaker and Bohacs, 2007; Wignall, 1989). Turbulent structures are produced in a high energy depositional environment; the energy of the environment is enough to overcome the electrostatic repulsion of the clay minerals. This environment creates colloids (silt to sand sized particles) of amalgamated clay minerals within the moving water column. When these colloids become large enough they are deposited in ripples the same way sand is deposited (Macquaker and Bohacs, 2007; Schieber et al., 2007). Each colloid has one direction, this direction is not necessarily shared by all the colloids. The result of this is the creation of local regions of alignment in an overall disordered material (similar to turbostatic fabric). This can evolve through burial into a strongly aligned material.

12.3.2 Effects of burial

During the initial stages of burial the void ratio of mudrocks is reduced through the rearrangement of particles. The particles in general move towards the most efficient arrangement: horizontally aligned particles if subject to a vertical (burial) load. This normally occurs through rotation. Rotation is limited however. If the initial deposited mudrock has a greater range of particle orientations (disordered) is the more likely it is that particles will be obstructed during their rotation resulting in low preferred particle alignments. Of course a sufficient depth must be reached for the rotational process to be completed.

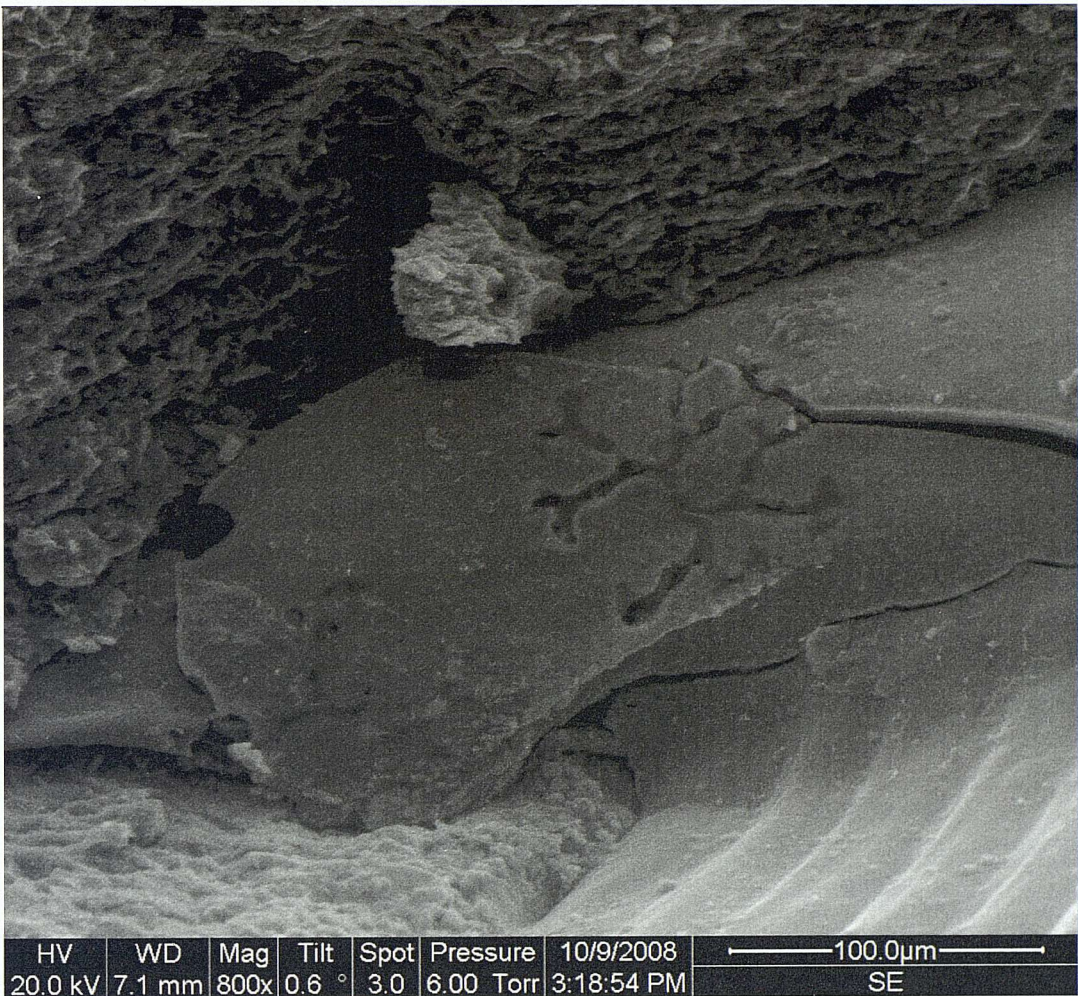


Figure 12.5 Probable indications of the dissolution of calcite within the Oxford Clay

Below a certain point pressure solution causes the dissolution of minerals within the mudrock. They are then re-deposited in areas with less stress (within the voids of the mudrock). This process results in the increasing of the area of contacts between particles as well as the reduction of the void space in the mudrock. This increases the mudrock's capacity to withstand the stress of burial across the long time scale (i.e. pressure solution and reprecipitation will have a lesser effect during instantaneous loading). Due to the growth mechanism, if there are voids in the material, new crystals are unlikely to be aligned with the existing structure (Figure 12.1). As the void space becomes filled the orientations of the larger particles become fixed as rearrangement becomes impossible. The Gault Clay and the Jurassic mudrocks were all buried to the point where further rearrangement could not occur, and diagenetic cements have started to form.

The cement which forms within a mudrock is partially dependent on its geochemical and hydrogeological environment. The major cements within these mudrocks are calcite and the secondary clay minerals. The deposition of cements is an equilibrium process. Mudrocks which contain organic material and have low permeability tend to have slightly acidic pore water. In these environments slow dissolution occurs, some evidence exists for this process in the Oxford Clay (Figure 12.5). If calcite is dissolving into the pore water, due to acidity not pressure solution, then it is unlikely that it will be re-deposited as calcite without a change in pore water pH. Thus clay minerals form the major cement in the Oxford Clay (Figure 12.1).

In Kimmeridge Clay some overgrowth cements (probably calcite) were present covering some of the rounded silt particles (Section 10.1.3). No evidence of cement was identified in the Gault or Lias Clays. The extent and variability of cementation can only be identified where a 3D block of mudrock has been analysed.

With even deeper burial the clay cements in the pore space start to align through pressure solution until a perfectly aligned material is formed. Chemical reactions occur altering the mineralogy of the mudrock (*e.g.*, smectite transformation to illite). With increased depth further reactions become possible as the conditions for low grade metamorphism are reached. None of the Jurassic mudrocks have been elevated to pressures and temperatures normally associated with metamorphism.

CHAPTER 13

CONCLUSIONS

The aims of this work were to investigate the correlation between the geological history, microstructure and engineering behaviour of UK mudrocks. The focus of this thesis was the quantification of the microstructure of five different mudrocks, namely the London, Gault, Kimmeridge, Oxford, and Lias Clays.

The original aims of the project were to:

- Quantify the effects of structure on the mudrocks' engineering properties, particularly the observed anisotropy in strength and stiffness;
- Determine explicitly the three-dimensional (3-D) microstructure of mudrocks, using environmental scanning electron microscopy (E-SEM), X-ray computed tomography (X-ray CT), 3-D imaging, and mechanical and chemical destructuring;
- Develop micro-mechanical structural models for the mudrocks;
- Generate a general unified framework linking together post-depositional geological processes, micro-mechanical structural models, and the mechanical properties.

Because it was not possible to create a detailed three dimensional image of the mudrocks' structure, a three dimensional micro-mechanical model was not developed. Thus, the objectives were altered to focus on the methods of quantification of mudrock structure from the best imaging methods available. The main property quantified in this study was preferred particle orientation. This correlates well with mechanical anisotropy measured in each of the mudrocks investigated.

The engineering behaviour of a geological material is primarily determined by both the chemical composition of the mineral particles and the particle shape and volume of the voids. Further variations in its behaviour are caused by the arrangement of the particles, including orientation of platy particles, development of cement and other micro-structural features.

Previous investigations of mudrock structure and its influence on mechanical behaviour have been limited to the London Clay. The London Clay is an Eocene mudrock which was deposited in the London Basin (Section 3.3.2). It experienced relatively shallow burial of approximately 200m (Hight et al., 2007). This study expanded these investigations to include older mudrocks exposed in the UK. These units, the Gault, Kimmeridge, Oxford and Lias Clays, have been subject to deeper burial and more complex geological histories. The Cretaceous Gault Clay was sampled from High Cross on the Midlands Platform (Section 3.3.2). The samples were acquired from a range of depths (Chapter 6). The Late Jurassic Kimmeridge Clay was obtained from Willowbrook Farm the Midlands Platform (Section 3.3.2). These samples were from a shallow water depositional environment which is not typical of the Kimmeridge Clay (Chapter 7). The Late Jurassic Oxford Clay was sampled at Elstow on the Midlands Platform. This material characteristically slips easily along horizontal layers (Chapter 5). The Early Jurassic Lias Clay was obtained from a location at Junction 13 of the M5 in the Worcester Basin (Chapter 8).

After evaluating the suitability of various imaging methods (Table 9-1), E-SEM was selected as the most effective method of imaging the microstructure of mudrocks (Chapter 9). In some cases pairs of stereoscopic images were taken allowing the development of three dimensional surface models. Each greyscale image was reduced to a black and white image using a grey level threshold. The particle shapes on these resultant images were analysed for long-axis orientations. The distribution of long-axis orientations was used as a representative measure of structure (fabric anisotropy) in each sample. Where the vast majority of particles have the same orientation the sample has a strong preferred particle orientation (Table 11-1). Preferred particle orientations were described qualitatively (Chapter 10) and quantitatively (Chapter 11). The Oxford Clay, Charmouth mudstone (Lias Clay, 11.6m sample), and the Gault Clay (at 6.82m) all showed strong preferred particle orientations. The London Clay and the Blue Lias showed moderate preferred particle orientations. The Kimmeridge Clay showed an inclined (to the horizontal) preferred orientation at 8.54m while at 12.71m particles show a vertical orientation. Other samples from the Gault Clay did not show any preferred orientation.

Several factors promote the formation of a preferred particle alignment. A high percentage of clay minerals and low silt content are required irrespective of the burial

history of the material. Mudrocks which contain few platy particles will never develop a preferred particle alignment. They may however display anisotropy through stratigraphic variation. A small amount of calcium carbonate predominantly in the form of platy nanofossil fragments, also seems to enhance preferred particle orientations. Particle alignment is enhanced in the depositional environment by a high organic content or by the formation of either turbstatic (high energy) or dispersed (low energy) fabrics. However in all cases the alignment of particles increased through burial. Mudrocks which are initially highly aligned attain the greatest preferred particle alignments during burial. Mudrocks with little initial alignment, become aligned through particle rotation during burial until they lock up and can rotate no further. The growth of calcite cements in the pores of these mudrocks during early burial may halt the reorientation of clay minerals. These mudrocks can develop moderate preferred particle orientations. Weathering results in the gradual destruction of preferred alignment as observed in the Gault Clay (Table 11-1).

All of the factors mentioned above result from the depositional, burial and uplift processes which have occurred during the geological history experienced by each of the mudrocks. The relative development of preferred particle orientation can be correlated with the measured anisotropy in engineering properties (Brosse, 2011; Hosseini-Kamal, 2011). Understanding the origins of the observed anisotropy in mudrock behaviour, may lead to early predication of expected engineering behaviour based on a simple understanding of the deposition and post deposition history of particular mudrocks.

Predictions

The Oxford Clay, the upper part of the unweathered Gault Clay and the upper part of the Lias Clay (Charmouth Mudstone) clearly have strong preferred particle orientations. They are expected to show high mechanical anisotropy. Anisotropic behaviour is also expected in the London Clay and the Lower part of the Lias Clay (Blue Lias).

In the lower part of the Gault Clay effects of the many joints in the mudrock are expected to become more important, and in the top of the Gault Clay weathering and desiccation cracks are expected to dominate the mudrock's behaviour. Greater anisotropy is expected in the upper unweathered part of the Gault Clay compared to the deeper borehole samples.

The behaviour of the Kimmeridge Clay should be much more variable due to the changes in silt and shell content through the cores caused by its higher energy depositional environment. It is expected that this variability will be represented in the testing results as it is in the CPT profile. Silty horizons are likely to display little or no anisotropy, whereas silt poor horizons are likely to show a greater level of anisotropy.

13.1 Future work

From the work performed during this study, several avenues of future research have been identified:

- **Focused ion beam analysis** would allow the creation of full three-dimensional models which would greatly enhance methods of modelling mudrock behaviour. At the moment this technique is prohibitively expensive because it is new. In the future the costs will come down.
- **Micromechanical modelling of mudrocks.** The creation of such a model would allow an opportunity for a three dimensional understanding of the relationships between individual particles. This would allow the better prediction of the behaviour of these materials during laboratory testing and in the field.
- **Three-dimensional surface analysis.** The completion of the surface analysis models will create a new method of quantifying the roughness of materials. The effect of scale on the measurement of roughness is one of the most important factors and requires a solution.
- **Focused studies.** To fully link microstructure to geological history a focused study is required for each mudrock to show how its behaviour and microstructure changes across the UK. By obtaining several samples from the same stratigraphic horizon in one mudrock across the UK the effects of environmental conditions can be analysed with more clarity.

The mineralogy of the Oxford Clay is very consistent across the UK. It would therefore be a good mudrock to use for the study of the effect of different burial histories on

mudrocks. The Gault Clay is very variable across the UK an intensive study of this mudrock would show the effects of depositional variability on mudrock microstructure.

- **Effects of greater burial.** A study of the microstructure of even older mudrocks (flaggy, fissile and massive mudstones), would reveal more information about the burial process. The Jurassic mudrocks are part way on the burial scale to these older materials. By comparing the microstructure of the very old mudstones to that of the Jurassic mudrocks it may be possible to identify what type of mudstone each material was evolving towards. This would allow a better understanding of the processes which were occurring in each of the Jurassic mudrocks while they were being buried.

- **Macro/outcrop-scale variability.** One of the most obvious examples of large scale variability in mudrocks is carbonate bands. The influence of such macroscopic heterogeneities on mudrock mechanical behaviour has yet to be investigated. Other examples of large scale variability which need investigations include shell beds, silt horizons, and joints.

- **Geological structures and tectonic history.** The response of microstructure of a mudrock to tectonic loading, in particular how it changes across the limbs of a fold or with proximity to a major fault have not been investigated in detail The Wessex and Cleveland basins were specifically avoided in this study due to the tectonic effects they have experienced in their histories. Perhaps an investigation in one of these basins may identify what the influences of these tectonic processes on mudrocks microstructure are.

- **Effect of joints.** Shallow samples of the Gault Clay showed much less preferred particle orientation on joints compared to within the material. It would be interesting to look at the changes in particle orientation with distance from a joint surface.

- **Quantification of further aspects of material structure.** This work has focused on the quantification of preferred particle orientations, quantifications of pore volume, shape and size, grain contacts, cementation and grain size distribution would aid the understanding of material structure.

- **Effect of different aspects of weathering on preferred orientation.** The images of the Gault Clay show a reduction in preferred orientation with weathering. But

it also shows a reduction at depth. It would be useful to study what the effect of different types of weathering is on particle orientation.

- **Effect of reconstitution.** The process of reconstitution does not reproduce the effects of sedimentation. It would be useful to look at the structure of reconstituted samples to measure the effects of reconstitution on particle orientation. It would also be interesting to compare the particle orientations in weathered and reconstituted samples to see if there are any similarities.

- **Effects of different bonding mechanisms and materials.** Clay cements have been observed in the Oxford Clay it would be interesting to quantify the effects of different types of bonding mechanism on the strength and compressibility of these materials.

- **A unified model of microstructure evolution.** A unified model will show the evolution of microstructure within geological events. It will include a wide range of initial mineralogies including multiple particle morphologies. It will also include a wide variety of depositional environments. In addition it will include a variety of burial histories considering the effects of fluid flow and tectonics. Such a model will require data from a great number of materials and a much focused research.

APPENDIX A

GLOSSARY OF TERMS

AFTA Apatite fission track analysis

Anisotropy Variation in properties with direction

Basin An area of deposition; where accommodation space is forming and sediment is being supplied.

Bonding A physical non frictional attachment between particles (cementation)

Burial history The depth of the material below ground level through time, also includes the effects of fluid flow through the material

Clay¹ A particle with a grain size less than 2µm

Clay² Part of the name of a lithostratigraphic unit the Gault Clay for example

Clay³ A general term of a mineral from the clay minerals group (*e.g.*, kaolin, smectite and Illite)

Clay⁴ An informal term for a geological unit predominantly composed of fine grained material

Concretion A highly cemented area within a geological unit normally ovoid in shape

CPT Cone Penetration Test

Cretaceous A period in earths history currently defined as 65.5-145.5Ma (Gradstein et al., 2004)

Cryo-SEM Cryo Scanning Electron Microscope

Cryoturbation disturbances to the structure of soil caused by geological processes in a cold environment

CT Computer tomography (See Appendix C)

Depositional history In the context of this project: the environment in which the material was deposited (*e.g.*, shallow sea), the minerals brought into the environment and the geological processes occurring in the environment.

Diagenesis The physical and chemical changes which occur within a material during shallow burial, prior to low grade metamorphism

Discontinuity a planar region of limited extent forming a break between two continuous areas of a material

Distal The furthest point affected by geological processes within an environment, for example the deepest part of the basin

E-SEM Environmental Scanning Electron Microscope

Environment All factors affecting a location and the material in that location. For example, water depth, sediment supply, fauns, ocean processes, temperature, stress, and fluid flow.

Exposure The area of a geological formation which is visible at ground level. Does not include the entire outcrop

Fabric The arrangement of the particles in a material. Normally the large scale, repetitive and persistent arrangement of particles.

Facies a volume of rock having similar characteristics, normally having been deposited in the same environment

FIB Focused Ion Beam

Flocculation The process of forming flocs (Figure 4.1), or the aggregation of particles into colloids

Formation See Lithostratigraphic hierarchy

Geomaterial a material which has been formed through geological processes

Group See Lithostratigraphic hierarchy

Horizon A stratigraphic layer within a geological unit

Jurassic A period in the earth's history currently defined as 145.5-199.6Ma (Gradstein et al., 2004)

Lithostratigraphic hierarchy in order of rank: Supergroup, Group, Formation, Member and Bed. Each rank can contain several geological units of the succeeding rank. The Lias Clay is a Group it contains twelve formations. All other mudrocks in this thesis are studied as Formations. The part of the Oxford Clay studied was identified as the Peterborough Member.

Material A general term for a substance which has a strength and stiffness, often used interchangeably with mudrock in this thesis.

Member See Lithostratigraphic hierarchy

Microstructure Structure¹ as observed via magnifying devices

Mineral A crystalline substance with a chemically defined and repeating unit cell

Mineralogy the percentage content of all minerals within a material

Mudrock a material containing more than 50% material with a grain size less than 0.63 μm , based on (Ingram, 1953; ISO 14689-1, 2003).

Nodule a region within a geomaterial which is chemically distinct from the material around it. Nodules are normally harder than the surrounding material

Outcrop The area of the ground under which the geological unit is present. In a few places it may be exposed.

Palaeogeography normally refers to the location of the coastline at some time in the past. Land masses will be eroding and many areas under water will be experiencing deposition

Particle¹ a coherent region of a material clearly unattached to its surroundings

Particle² an area of an image selected through the application of a threshold

Particle orientation The angle of the long axis of a particle in space relative to the horizontal or relative to north

Preferred particle orientation the statistical precedence of one particle orientation over all others

Reconstituted soil a soil which has been mixed at high water content and then recompressed, also known as a destructured soil (interestingly this material has a structure¹ but does not have a structure² as the reconstituted soil is the datum)

SDB Scanning Dual Beam

Sediment the constituent parts of a sedimentary rock prior to deposition

SEM Scanning electron microscope

Structure¹ An arrangement of particles within a material, often forming a repetitive pattern or clear configuration to the material's particles

Structure² The organisation of the material responsible for allowing it to maintain a void ratio to a higher stress than a reconstituted sample of the same material

TEM Transmission Electron Microscope

Uplift history the depth of the material below ground level through time while it is being returned to the surface.

Weathering The breakdown of geomaterials through physical, chemical and biological processes occurring on or near the earth's surface

XUM X-ray ultra microscope

APPENDIX B

GEOLOGICAL LOGS

B.1 Site identification

The identification of and liaising with appropriate sampling sites for all of the mudrocks tested were the authors' responsibility. The factors that were taken into consideration when identifying appropriate sites from which high quality samples could be obtained are discussed below:

Geological basin history, recent weathering, and presence of geotechnical site investigation data were all important influences on the selection of site locations. Of these the most important factor is that samples should have received as little disturbance as possible (*i.e.*, have a relatively simple geological history). Areas which have experienced tectonic compression such as the Wessex basin should be avoided (Section 3.3.2), as well as those that have complex depositional histories such as the Cleveland basin and the northern boundary of the East Midlands Platform. The largest land areas remaining are the Midlands Platform and the Worcester Basin. Due to the movements along the Vale of Moreton Axis during the Jurassic, the Midlands Platform was preferred. Ideally samples should be unweathered, as weathering can replace a mudrocks microstructure. This can be achieved by taking the samples from depth. The ideal site should have extensive prior site investigation data, so that the data obtained by laboratory tests can be correlated to data obtained from the mudrock in its natural state in the field. The target sites therefore were pre-existing, deep excavations into mudrocks within the Midlands, to which were attached a large amount of site investigation data.

To achieve this aim, sampling requests were sent out to several engineering companies in the hopes that they might have access to ideal sites. Due to the economic downturn, work in the construction industry came to a sudden halt near the start of the project and therefore relatively few active deep excavations were located. Of these, only one site allowed sampling to take place; Arup kindly provided access to a ten metre excavation in the Oxford Clay located near Elstow. Samples could not be taken from other deep excavations as site owners considered that the block sampling process would cause too

much of a delay to the works schedule. So other than the Oxford Clay no further samples could be obtained from pre-existing open excavations.

Cambridge, as one of the few cities situated on the Gault Clay on the Midlands Platform, was identified early in the project as an area to investigate for sampling opportunities. Several site investigations in the city were considered, however none allowed sampling. The site finally selected was High Cross (also known in the literature as Madingley, as it is located on Madingley Road (Houlsby and Withers, 1988; Lings et al., 2000)). Block sampling was carried out in an excavation supervised by Geotechnics Limited with the permission of Cambridge University. The samples from this excavation had a very high suction due to desiccation caused by the trees on site. A second investigation was undertaken on the site, collecting borehole samples. This investigation also allowed better comparisons between the mudrocks as it allowed testing to be undertaken at a consistent depth across all the mudrocks.

As the Kimmeridge Clay has a smaller outcrop than the other mudrocks, some effort was required to locate a sampling site. The Kimmeridge Clay is found at depth to the north of Cambridge beneath the fens, in this location it is inaccessible. Over much of the Midlands Platform the entire Kimmeridge Clay sequence has been removed by erosion. The only large patch of Kimmeridge Clay on the Midlands Platform runs from the west of Luton through to the South East of Swindon. No geotechnical activity could be identified on the Kimmeridge Clay in this area as there are no major cities there. Fortunately the old site investigation for the Upper Thames Reservoir is based on the Kimmeridge Clay. The British Geological Survey website shows this as an area relatively densely packed with boreholes. Land owners were identified, after several attempts, by sending applications to the Land Registry office in Gloucester. Mr Neil Walker kindly allowed a borehole investigation to be undertaken on his land. Unfortunately at the time of writing the site investigation data for the Upper Thames Reservoir was not available to the project due to its political sensitivity. This data will become available in the future, at which point useful comparisons can be made.

The Lias Group contains 12 different formations of which there are three mainly mudstone formations and two partly mudstone partly limestone formations. The remainder of the Lias Group is sandstones and limestones. No single mudstone formation is present in all the basins of the southern UK. The Whitby Mudstone

Formations at the top of the Lias Group directly precedes the limestones of the Middle Jurassic. These limestones form a high ridge across the country, and the mudrock sequence below them forms the slope of this escarpment. Thus these samples tend to have been greatly affected by slope processes, especially those that occurred during the Quaternary glaciations. The mudrocks in the south of the UK do not spread far onto the Midlands Platform, and no sites could be located on the Midlands Platform. Fortunately InterRoute kindly provided access to samples taken from a site on the M5, to the South of Gloucester within the Worcester Basin.

Oxford Clay sampling

Samples were obtained from the site from Thursday the twenty seventh of September to Wednesday the third of October 2007. The CPT works were completed on this site in August 2008 a year after the initial sampling, as this was when the CPT services were donated to the project

Gault Clay sampling

A recognisance survey of the site was carried out during March 2008. Cone penetration testing was undertaken a few days prior to the block sampling campaign during late June 2008. Trees were cleared from the site on the same day as the testing took place. Reference profiles were created while the excavation site was being cleared of trees. Cpt1, 2 & 3 profiles were created in the afternoon. The Reference CPT profiles were created using a piezocone. The other CPT profiles were created using a seismic cone. The excavation was undertaken by Geotechnics Limited during July 2008 the day after the CPT profiling. Samples were re-logged in detail in the laboratory where more time could be devoted to sample description. The re-logging occurred in June 2010

Kimmeridge Clay sampling

Sampling took place during July 2009. Very little detail of the cores of Kimmeridge Clay was visible in the field. The true detail of the core became apparent during relogging due to the use of a sculpting tool and a well lit environment. The CPT testing at Willowbrook Farm took place during April 2009.

B.2 Comparison sites

Because each mudrock is variable both with depth, and laterally across the country. Early during the investigation the author decided to visit sites other than the sampling locations to allow comparisons to be made. Most of the sites visited were areas where there is a greater exposure of the mudrock than was present at the sampling site. Most of the sites were also within a few kilometres of the sampling site. For the Oxford Clay, Stewartby Quarry in Bedfordshire and for the Gault Clay, Barrington Quarry in Cambridgeshire was visited. For the Kimmeridge Clay no suitable site was available near the sampling site, so Kimmeridge Bay was visited for comparison. For the Lias Clay, Hock Cliff, which is formed by the Severn bore, was within a few km of the site, providing an excellent exposure of the Blue Lias. A visit to the A421 site investigation was also undertaken to study the river gravels and alluvium above the Oxford Clay.

From each site visit valuable information, of confirmation/clarification of information from the sites was gained. Differences between the sampling sites and the larger exposures highlighted processes occurring at the sampling site. These are all explained in the appropriate chapter.

B.3 Sampling

Three different sampling methods were used to obtain mudrock samples. The Oxford and Gault Clay were obtained by block sampling. The Kimmeridge and Gault Clay were obtained using borehole sampling monitored by us. Samples of Lias Clay were obtained from borehole samples obtained by InterRoute.

B.3.1 Block sampling

Block sampling was carried out for both the Oxford Clay and the Gault Clay. Block samples experience much less disturbance than any other method of sampling. They are very useful for obtaining a large number of intact samples from a specific geological horizon. The procedure for block sampling is as follows:

1. An approximately half metre trench is dug into the mudrock using a mechanical excavator.
2. A pair of alcoves are cut into the side of the trench, either by hand or using a clay spade. The alcoves should be approximately 0.5m apart and going back approximately 0.7m
3. A trench is excavated by hand between the backs of the pair of alcoves, joining them together and creating a pillar of mudrock in the trench.
4. The square pillar is reduced in size using a spade until it is approximately 0.31m across from edge to edge in both directions.
5. Using wall paper scrapers all four sides of the block are shaved down until they are smooth and the block is the right size to fit inside its transport box
6. A layer of Clingfilm, followed by a layer of wax is applied to the sample such that as little air as possible is trapped between the Clingfilm and the sample.
7. A second layer of Clingfilm then wax is applied to the sample in the same way as above
8. The sample is wrapped in aluminium foil.
9. The sides of the box are placed over the top of the sample.
10. Expanding polystyrene is sprayed into the gap between the sample and the box such that all the space is filled. More is sprayed across the top of the sample
11. The wooden lid of the box is placed on top and screwed into place.
12. Box lid is labelled with: top, sample number and direction of north. A side is also labelled for convenience.
13. Sample is left over night for the expanded polystyrene to harden
14. Base of the pillar is cut through and sample is rotated onto its top.
15. Excess expanded polystyrene is removed from the base.
16. The sample is cut down using wallpaper scrapers until it is 0.02m below the lip of the box.
17. Base of sample is covered in a double layer of Clingfilm and wax and a layer of foil (as steps 6-8)
18. Expanding polystyrene is sprayed across the base of the sample and its wooden base is screwed into place. Excess polystyrene which has been squeezed out of the sample is removed.
19. The sample is then returned to the laboratory for testing.

B.3.2 High quality rotary coring

High quality rotary coring was used to obtain samples of Gault and Kimmeridge Clay. High quality rotary coring causes more disturbance to samples than block sampling, it is still high quality compared to other commonly used sampling methods. The coring was undertaken by Emerson-Moore Drilling under the supervision of Bill Robshaw with his crew Nathan Jones and Graham Harper. The Imperial College team was also on site to ensure that the samples were properly sealed preserving their moisture content. The drilling method was directed by Emerson-Moore Drilling, the basic procedure is as follows. An inspection pit was dug out to approximately 1-1.5m and drilling commenced at the base of the pit. The drilling rig was a Comacchio 450P. The core was drilled using the triple barrelled wireline Geobore-S method recovering 100mm samples to a depth of around 14m. The drillers used a PH balanced natural polymer mud flush.

A table was converted, by the addition of two pieces of wood, screwed into place, between which a gutter pipe was placed. Samples were returned in their tubes, and extruded slowly onto the guttering using a plunger. The recovered samples were preserved using the following method:

1. The top of the sample is cleaned using a knife blade so that the surface of the sample is visible.
2. A quick field log of the prominent features is created.
3. The sample is divided up into manageable lengths (from which testing samples can be formed) using a knife or hand saw.
4. The outer mm of material is removed from each length so that neither drillers' fluid nor hydrated outer material is preserved.
5. The sample is wrapped in Clingfilm
6. The Clingfilm is labelled with: top, bottom and sample number
7. The sample is covered in a layer of wax
8. A further layer of Clingfilm and wax is applied.
9. The sample is covered with aluminium foil.
10. The foil is labelled with top, bottom and sample number
11. A sticky label is affixed to the sample giving details of sampling location, sample number, depth, student name, and sampling date.
12. The original sample tube is cut in half using a hook blade knife

13. The half sample tubes are placed into core boxes
14. Samples are placed in order into the tubes
15. The other half of the tube was placed on top and the remainder of the box is packed with bubble wrap
16. The samples, protected in their core boxes are then returned to the laboratory for testing.

B.3.3 Sample comparison

As sampling is part of the history of a material prior to testing, it is expected that the method of sampling used will affect the testing behaviour of the material. The mud flush used to keep the drill bit cool during coring contains a large amount of water this water remains around the sample right up until the point the sample is cleaned and waxed. This flush can easily get into joints especially given the elevation head which is maintained throughout the drilling process. If the moisture content of the sample is affected it is likely to be slightly wetter than in its natural state. In block sampling no water is used. The blocks are left overnight waxed and protected on 5 sides with the sixth side in direct contact with the ground. Due to the excavation of the trench it is expected that water would migrate down out of the sample very slowly during the night. If there is any change to the samples moisture content it will be dryer than it is in the ground. During block sampling the compressive stresses applied to the sample are very small. Coring can potentially apply large stresses to the sample, these are minimised by keeping the rate of advance as low as possible. For example at the Kimmeridge and Gault Clay sites each 14m run was completed across a full day. These differences may affect the laboratory behaviour of the samples, however they are unlikely to be of high enough magnitude to have an effect on the imaging of the samples, which is the subject of this thesis.

B.3.4 Sample logging

The borehole samples were re-logged during June 2010. The re-logging allowed high resolution logging and photographic documentation of the core in a well lit environment. The method used for re-logging was as follows:

1. A label is written for the sample giving its full details
2. The sample is orientated on the table with its top to the left
3. A strip of the wax and Clingfilm covering is removed from the core using a craft knife.
4. The surface of the core is scraped with a wire hoop clay sculpting tool.
5. A detailed log of the sample features is written
6. A photograph is taken of the sample
7. The sample is resealed with Clingfilm and wax

Bagged samples could not be orientated, excluding orientation they were logged in the same manner. The key advantages of logging in the laboratory are: the well lit environment, the lack of time pressure, and the sculpting tool which creates a good smooth surface revealing features of the mudrock which could not been seen or described in detail in the field. Several of the features of the samples had changed between the two logging dates (due to the presence of drillers' fluid ingress into joints in the mudrock for example).

B.4 Cone penetration testing (CPT)

The CPT was donated to the project by InsituSI under the instruction of Darren Ward. The CPT work included both soil moisture and seismic cone investigations. On some sites dilatometer measurements were also taken. In this thesis the geological implications of this data are discussed. The engineering properties and measurements will be discussed along with the laboratory results in other researcher's work (Brosse, 2011; Hosseini-Kamal, 2011). Members of the Imperial College team including the author were present at all sites.

The cone penetration test involves pushing a steel tipped metal pole into the ground. The rate of advance is usually kept constant. The fount of the advancing pole can contain various devices for the analysis of the soil. The standard measurements are, end resistance, and sleeve friction. These measurements can be interpreted to identify variations in the geological materials in the ground:

Cone end resistance

The end resistance is one component of the energy required to advance the cone into the soil. It is proportional to the strength of the material which the cone is pushing through. Clay tends to have a lower end resistance than sand. Loose, or high void ratio material requires less energy to push through than denser or heavily cemented material. The weight of the CPT truck determines the hardest/densest material that can be advanced through.

Sleeve friction

Friction along the full length of the inserted rod is the second factor affecting the energy required to advance the cone into the soil. Sleeve friction measures the frictional force applied to a section of a given length on the cone which is advancing into the soil. The amount of friction measured depends on a number of factors: *e.g.*, mineralogy and contact area. Fine grained materials tend to have high sleeve friction values due to higher contact area.

B.5 Microfossil analysis

The stratigraphic origin of many of the samples could not be identified without reference to their fossil content. No macrofossils were identified in the Gault Clay or in the borehole samples. Thus the stratigraphy of the samples was analysed based on microfossils. These were extracted from the mudrock by the author, the identification of fossils was undertaken by experts on the relevant geological systems. The Gault Clay microfossils were identified by Ian Wilkinson of the BGS and the Lias Clay microfossils were identified by Philip Copestake from Merlin Energy Resources Limited.

The procedure for the extraction of microfossils from their host clay is outlined here:

1. A small cube (~50g) of the mudrock is collected from the appropriate horizon in the material.
2. The cube is broken up without applying excess stress to the mudrock. This can be achieved by placing the mudrock into a plastic bag, which is then placed into a vice. Smaller pieces of the mudrock can be generated by slowly closing the vice until the sample yields. This process can be repeated for any large intact

pieces. At no point should the samples be completely crushed (microfossils must be preserved).

3. A bowl is half filled with water, a small amount of bicarbonate of soda (deflocculating agent) is added (~2 teaspoons full), followed by the sample.
4. The water in the bowl is brought to boil, and boiled for about 5 minutes.
5. The resultant liquid and sediment is washed through a sieve stack, a 250 μ m sieve above a 63 μ m sieve removing all finer material. The material in the 250 μ m sieve is discarded.
6. The material in the 63 μ m sieve is allowed to dry overnight in a cool location.
7. This fraction is analysed using a reflected light stereoscopic microscope. Microfossils are identified and separated from other grains.
8. The microfossils are placed onto a slide and imaged using extended focus light microscope imaging.
9. Images and slides are forwarded to experts for identification.

Only one sample of microfossils was identified from each site to pin each profile at one point to stratigraphy. Full analysis of mudrock stratigraphy would require microfossils to be identified at regular intervals in the core. This process is very time intensive, requiring lots of expert input. Thus for the most part stratigraphy is identified by geographical location and depth.

B.6 Borehole logs

Several limiting factors apply to the field logs of the mudrock samples outlined below. These limitations required the relogging of all core samples in greater detail a year after the initial sampling.

During the field work 12.5m of mudrock were obtained on each day. This material was normally divided into samples of ~25cm, sometimes the length varied to take into account the stratigraphic changes and discontinuities in the material. Normally 50-60 samples were recovered. The core must be cleaned and logged, it must be divided into samples with a knife or a saw. Each sample must be cleaned and preserved in a double layer of Clingfilm and wax, then labelled with sample number and core orientation. On some occasions only two members of the Imperial College team were on-site. This was

not enough, and so corners had to be cut, even with team members remaining on site until 23:00 hours

As the samples moisture content must be preserved it is bad for them to lie in their sampling tubes in drilling fluid for an extended period of time. In the worst case samples had to remain in their tubes for five hours. Quite rightly there was an emphasis on sample preservation during field work. Only limited time could be spent on logging. All the work was done inside an unlighted steel container. Logging in these poor lighting conditions especially after sunset was near impossible. Subtle changes in colouration could not be accounted for. The tool used for cleaning the sample prior to logging was a steel knife blade. The blade cleaned drilling fluid from the surface, it also smeared the surface of the material confusing its detailed features of the material.

In summary the field logs were constructed in poor lighting using the wrong tools by an individual (the author) whose time had to be focused on other tasks. The field logs are therefore low resolution. They were constructed on the assumption of a continuous sample with all core loss being applied across the core as a whole.

Ideally where samples must be preserved, the onsite team should comprise 4-5 members:

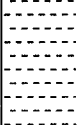
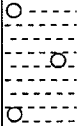
1. Geologist, to be with the drillers at all times to record all anomalies and experiences during the drilling process
2. Geologist, to log the core immediately as it arrives
3. Sample tester, to divide the core into suitable lengths for testing in consultation with geologists' log. And to clean the outside of the core prior to preservation.
4. Technician to preserve the core in wax and to label it with number and orientation
5. Extra optional technician to aid in the wax preservation.

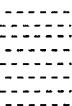
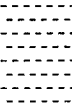
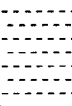
This would allow more details during the sampling process to be recorded, and ensure that the samples are completely and rapidly preserved.

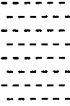
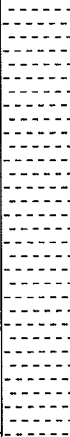
Due to the low resolution of the field logs the samples had to be relogged. Relogging is a very time consuming process. The four boreholes for this project took three and a half weeks to cut open, clean, describe, photograph and re-wax. The relogging was done in a well lighted laboratory, with a clay sculpting loop, without any time pressures on the

process. The logs produced are patchy, as inevitably samples had already been used. They are also incomplete as ends of samples are flattened during the sampling process. Normally discontinuities, nodules and cemented bands are selected as sample ends as they are not ideal within samples. They are therefore sometimes not present in material for a secondary log. Areas with multiple discontinuities are broken samples and are normally stored in bags not in waxed and labelled cores. These discontinuities are also lost. Some nodules present in only one location around the core. In these circumstances some nodules are not identified during the relogging.

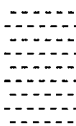
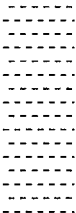
In these secondary logs, core loss is placed within the log based on where it is most likely to have occurred due to changes in the amount of core loss/gain with each core run, based on the driller's observations. Both sets of logs are presented in the following pages.

Equipment and methods Hand dug inspection pit to 1.34m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 13.80m using natural polymer mud flush.		Location Cambridge, High Cross			Borehole HC1
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON		Coordinates: TL 42205 58862		Date: 21/07/09	
Sample Discription	Legend	depth (thick)	Samples Taken	Notes	
Reinforced concrete		(0.29) 0.29			
Soft light greyish yellow CLAY, containing some rootlets (Weathered Gault Clay)		(1.05) 1.34			
Firm-Stiff light grey mottled yellow CLAY, some broken shells (Weathered Gault Clay)		(0.65) 2.01	HC1-1 21cm HC1-2 22cm	1.34-7.80 100% Flush Return 1.56m joint 20°	
Stiff light grey slightly sandy CLAY some areas of red brown sand interpreted as weathered pyrite nodules (Gault Clay)		(1.16) 3.17 (0.29) 3.46	HC1-3 24cm HC1-4 25cm HC1-5 24cm HC1-6 24cm HC1-7 24cm HC1-8 22cm HC1-9 18cm HC1-10 11cm	2.59m weathered pyrite nodules 2.70m weathered pyrite nodules 2.81m weathered pyrite nodules Some coarse sand possibly shell fragments	
Very Stiff light grey CLAY with sandy pockets		(3.77)	HC1-11 30cm HC1-12 29cm HC1-13 9cm HC1-14 28cm HC1-15 29cm HC1-16 3cm HC1-17 25cm HC1-18 25cm HC1-19 25cm HC1-20 25cm HC1-21 26cm HC1-22 16cm HC1-23 14cm HC1-24 6cm HC1-25 32cm HC1-26 15cm HC1-27 7.5cm HC1-28 7.5cm HC1-29 29cm	4.17-4.80 dropped from core barrel	
Very Stiff light grey slightly sandy CLAY (Gault Clay)		7.23 (0.08) 7.31	HC1-30 35cm	6.86m Joint 0°, 6.90m Joint 0° 7.41m Joint 25° Holes later identified as the intersection of joints meeting the surface of the core	
Very stiff light grey with yellow pockets slightly sandy CLAY					
Very Stiff light grey CLAYwith regularly spaced 0.5cm holes					
Notes: Piezometer installed, tip 13.20-13.50m Sand 12.64-13.80m					(Continued on next sheet)
					Logged by SW
					Scale 1:50
					p 1 of 2

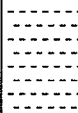
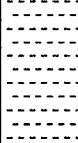
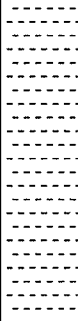
Equipment and methods Hand dug inspection pit to 1.34m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 13.80m using natural polymer mud flush.		Location Cambridge, High Cross Borehole HC1		
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON		Coordinates: TL 42205 58862		Date: 21/07/09
Sample Discription	Legend	depth (thick)	Samples Taken	Notes
Very Stiff light grey CLAY several 0.5 cm holes on core surface (Gault Clay)		(1.92)	HC1-31 3cm HC1-32 2cm HC1-33 27cm HC1-34 28cm HC1-35 30cm	7.80-9.30m 70% Flush return 7.91m Joint 20° 8.00m Joint 70° 8.16m Joint 70° 8.29m Joint 40° 8.52m Joint 0°
			HC1-36 10cm HC1-37 30cm HC1-38 10cm HC1-39 4cm	930-10.80m 80% Flush return
			HC1-40 14cm HC1-41 18cm HC1-42 10cm HC1-43 19cm	
			HC1-44 25cm HC1-45 25cm HC1-46 23cm HC1-47 16cm	
			HC1-48 6cm HC1-49 5cm	
Very Stiff grey CLAY several 1 cm holes on core surface (Gault Clay)		(1.62)	HC1-50 24cm HC1-51 22cm	10.23m Joint 0° 10.48-10.73 core thinned possible rotation 10.96m Joint 0° 11.08m Joint 45°
Very Stiff grey CLAY some 0.5cm bands (Gault Clay)		10.85 (0.34)	HC1-52 19cm HC1-53 25cm HC1-54 25cm HC1-55 25cm HC1-56 5cm HC1-57 6cm HC1-58 21cm HC1-59 4cm HC1-60 10cm HC1-61 25cm HC1-62 26cm HC1-63 26cm	Broken zone drilling error run too long 10.80-12.30m 80% Flush return 11.47m Joint 60°
Broken zone		11.19 11.29 (0.10)		
Very Stiff grey CLAY many small ~0.5 cm holes on core surface (Gault Clay)		(2.03)	HC1-64 25cm HC1-65 2cm	11.85m Joint 45° 12.58m Joint 10° 12.30-13.80m 90% Flush return 12.80m Joint 10°
Very Stiff grey CLAY smooth surface, few small indentations no holes (Gault Clay)		13.32 (0.30) 13.62		13.36m Joint 0°
Notes: Piezometer installed, tip 13.20-13.50m Sand 12.64-13.80m				Logged by SW Scale 1:50 p 2 of 2

Equipment and methods Hand dug inspection pit to 1.07m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.09m using natural polymer mud flush.	Location Cambridge, High Cross Borehole HC2			
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: TL 42201 58864 Date: 22/07/09			
Sample Discription	Legend	depth (thick)	Samples Taken	Notes
Reinforced concrete		(0.20) 0.20		
Soft light greyish yellow CLAY, containing some rootlets (Weathered Gault Clay)		(0.87) 1.07		100% Flush returns
Firm-Stiff light grey mottled yellow slightly sandy CLAY (Weathered Gault Clay)		(1.16) 2.23	HC2-66 17cm HC2-67 16cm HC2-68 17cm HC2-69 23cm HC2-70 23cm HC2-71 23cm	Signs of core rotation 1.61m 0° shear 1.84m 10° shear
Stiff light grey CLAY containing yellow clay inclusions yellow inclusions decrease with depth (Weathered Gault Clay)		(1.83) 4.06	HC2-72 26cm HC2-73 3cm HC2-74 27cm HC2-75 28cm HC2-76 28cm HC2-77 28cm HC2-78 32cm	2.38m 30° Joint 3.66m 70° Joint 3.76m 30° Joint
Very Stiff light grey CLAY containing rare sand particles (microfossils?) Sand often associated with areas of <1cm yellow discolourations on the core surface (Gault Clay)		(3.16) 7.22	HC2-79 9m HC2-80 3cm HC2-81 25cm HC2-82 28cm HC2-83 28cm HC2-84 28cm HC2-85 29cm HC2-86 3cm HC2-87 5cm HC2-88 27cm HC2-89 26cm HC2-90 27cm HC2-91 26cm HC2-92 30cm HC2-93 10cm HC2-94 2cm HC2-95 26cm	4.89m 0° Joint 5.05m 45° Joint 5.32m 50° Joint 5.88m 10° Joint 6.18m 80° Joint 6.65m 60° Joint 6.69m 0° Joint 7.01m 45° Joint 65% Flush Returns, water returns much darker, mud breaking down
Very Stiff grey slightly silty CLAY (Gault Clay)				
Notes: On several occasions on this core, multiple attempts were required to retrieve samples				(Continued on next sheet) Logged by SW Scale 1:50 p 1 of 2

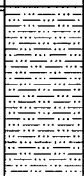
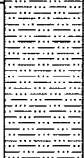
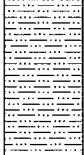
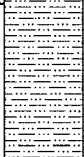
Equipment and methods Hand dug inspection pit to 1.07m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.09m using natural polymer mud flush.	Location Cambridge, High Cross				Borehole HC2
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: TL 42201 58864			Date: 22/07/09	
Sample Discription	Legend	depth (thick)	Samples Taken	Notes	
Very stiff grey slightly silty CLAY (Gault Clay)		(1.43)	HC2-96 26cm HC2-97 26cm HC2-98 23cm HC2-99 26cm HC2-100 24cm HC2-101 16cm	65% Flush Returns, water returns much darker, mud breaking down	
Very stiff grey slightly silty CLAY some holes present in the core max =4cm (joints)		8.65	HC2-102 5cm HC2-103 10cm HC2-104 25cm HC2-105 26cm HC2-106 27cm HC2-107 3cm HC2-108 1cm HC2-109 24cm HC2-110 25cm HC2-111 25cm	20% Flush Returns 8.81m 4 cm Hole 9.15m 2cm Hole 30° Joint 9.22m 2cm Hole	
		(3.58)	HC2-112 26cm HC2-113 25cm HC2-114 10cm HC2-115 4cm HC2-116 23cm HC2-117 15cm HC2-118 12cm HC2-119 11cm HC2-120 24cm HC2-121 8cm	9.74 30° 9.86 40° 10.03 70° 10.33 60° 10.74 0° 10.89 20° 11.25 0° 11.34 43° Possible core rotation 11.57 0° 11.70 0° 11.94 0°	
			HC2-122 27cm HC2-123 33cm HC2-124 3cm		
			HC2-125 25cm HC2-126 20cm HC2-127 26cm HC2-128 2cm	100% Flush Returns	
Very stiff grey CLAY some coarse sand			12.23 (0.42)		
Very stiff light grey CLAY some holes on the core surface (joints)		12.65			
		(0.75)			
		13.40			
Notes: On several occasions on this core, multiple attempts were required to retrieve samples					Logged by SW
					Scale 1:50
					p 2 of 2

Equipment and methods Hand dug inspection pit to 1.35m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 13.80m using natural polymer mud flush.	Location Cambridge, High Cross (relogged) Borehole HC1		
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: TL 42205 58862		Date: 21/07/09
Sample Discription	Legend	depth m (thick)	Notes
Reinforced concrete		(0.29) 0.29	
Soft Lt G Y CLAY, containing some rootlets (Weathered Gault Clay)		(1.06) 1.35	
Firm, slightly moist Lt Br G, 50% mottled Y Br at top reducing to 10% Y Br and 15% Lt G mottling at base, slightly silty slightly sandy CLAY, some broken shells some ~1mm holes may be root pores Y Br areas follow horizontal layers (Weathered Gault Clay)		(0.67) 2.02 (0.25) 2.27	1.35-7.80 100% Flush Return 1.45m Possible closed joint at 60° 1.88m Possible closed joint at 15°
Used			
Firm, slightly moist Lt Br G, 5% mottled Y Br, slightly silty slightly sandy CLAY, sand particles appear to follow bands, becoming more aligned to planes (discontinuities?) with depth, may be selenite (Weathered Gault Clay)	 	(0.94) 3.21 (0.18) 3.39	sand grains in linear arrangement possible joints, 2.81m 30°, 2.88m 30°, 2.92m 10°, 2.98m 15°
Used			
Firm, slightly moist Lt Br G, slightly silty slightly sandy CLAY, some 10mm sand patches, some sand particles aligned to planes few R 1mm points on core (Gault Clay)		(0.70) 4.09 4.18 (0.09)	3.50m 30mm phosphatic nodule 3.75m 10mm Lt G band
Used			
Firm, slightly moist Lt Br G, slightly silty slightly sandy CLAY, some 10 mm sand patches (2% of core), some sand particles aligned to planes (Gault Clay)		(1.77) 5.95	4.32m 10mm sand band 4.17-4.80 dropped from core barrel 4.80 5mm black phosphatic nodule 5.28m 45° line of black shiny sand 5.66m 10mm black phosphatic nodule 5.74m 5mm sand rich patch
Firm, slightly moist Lt Br G, slightly silty slightly sandy CLAY, some 4mm Y Br patches, few Lt Br areas discolouring core surface 20% becoming 40% with depth, few R 1mm points on core (Gault Clay)		(1.40) 7.35	7.20-7.35 2mm Lt Br sandy pockets
Firm, slightly moist G, slightly silty CLAY, horizontally aligned Dk Gr G patches			
Notes: Piezometer installed, tip 13.20-13.50m Sand 12.64-13.80m			(Continued on next sheet)
Lt G Y Light Greyish Yellow Lt Gr G Light Greenish Grey Y Br Yellowish Brown Lt G Light Grey Lt Br G Light Brownish Grey R Red Lt Br Light Brown G Grey Dk Gr G Dark Greenish Grey			Logged by SW
			Scale 1:50
			p 1 of 2

Equipment and methods Hand dug inspection pit to 1.35m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 13.80m using natural polymer mud flush.	Location Cambridge, High Cross (relogged) Borehole HC1		
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: TL 42205 58862 Date: 21/07/09		
Sample Discription	Legend	depth m (thick)	Notes
Firm-stiff, slightly moist G, slightly silty CLAY, some horizontally aligned <10mm Dk Gr G patches. Some ~10mm moist soft patches (indents in surface). Some Lt Br discoloration on core surface (Gault Clay)	-----	(0.80)	7.80-9.30m 70% Flush return
Bands of firm, slightly moist Lt Br G, slightly silty CLAY, mixed with bands of firm-stiff, slightly moist Dk Gr G, slightly silty CLAY. Bands vary from 3-10cm size of Dk Gr G bands increases with depth, surface starts to break along horizontal planes (Gault Clay)	-----	8.15 (0.40)	8.19m 10mm Lt Y W phosphatic nodule
Stiff, slightly moist Dk Gr G, slightly silty CLAY, some Lt Br G motteling (30%)	-----	8.55 (0.40)	
Some ~10mm moist soft patches (indents in surface). (Gault Clay)	-----	8.95 (0.04)	9.06m possible joint 0°
Used	-----	8.99	9.17m band of Lt Y W phosphatic nodules 5mm
Stiff, slightly moist Dk Gr G, slightly silty CLAY, 10mm patched of Lt Br. some Lt Br G motteling (30%) (Gault Clay)	-----	(0.42)	
Used	-----	9.41 (0.92)	9.30-10.80m 80% Flush return
Stiff, slightly moist Lt Br G, slightly silty CLAY, 10mm patches of Lt Gr G, some ~10mm moist soft patches (Gault Clay)	-----	10.33 (0.16)	10.34m 10mm Lt Y W phosphatic nodule
Stiff, slightly moist Dk Gr G, slightly silty CLAY, up to 30mm moist soft patches forming holes in core surface (Gault Clay)	-----	10.49 (0.57)	10.52m possible joint 30°
Used	-----	11.06 (0.19)	10.48-10.73 core thinned possible rotation
Stiff, slightly moist Dk Gr G, slightly silty CLAY, some 2mm Bk flecks up to 30mm moist soft patches forming holes in core surface (Gault Clay) Lt Br G, band 11.50-11.67m	-----	11.25 (0.75)	10.80-12.30m 80% Flush return 11.87m 10mm Lt Y W phosphatic nodule 11.90m possible joint 10°
Used	-----	12.00 (0.05)	
Stiff, slightly moist Dk Gr G, slightly silty CLAY (Gault Clay)	-----	12.05 (0.21)	12.07m 10mm Lt Y W phosphatic nodule
Used	-----	12.11 (0.39)	12.30-13.80m 90% Flush return
Very stiff, slightly moist Dk Gr G, slightly silty CLAY, some 5mm holes in surface few ~10mm moist soft patches (Gault Clay)	-----	12.32 (0.77)	12.41m possible joint 80°
Used	-----	12.71	12.56m band of Lt Y W phosphatic nodules 5mm
Used	-----	(0.77)	12.66m possible joint 15°
Very stiff, slightly moist Dk Gr G, slightly silty CLAY (Gault Clay)	-----	13.48 (0.02)	
Core Loss	-----	13.50 (0.30)	
	-----	13.80	
Notes: Piezometer installed, tip 13.20-13.50m Sand 12.64-13.80m Lt Br Light Brown Lt Br G Light Brownish Grey Lt Gr G Light Greyish Green G Grey Lt Y W Lt Yellowish White Dk Gr G Dark Greenish Grey Bk Black "Used" marks samples used for laboratory tests prior to logging			Logged by SW Scale 1:50 p 2 of 2

Equipment and methods Hand dug inspection pit to 1.07m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.09m using natural polymer mud flush.	Location Cambridge, High Cross (relogged) Borehole HC2		
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: TL 42201 58864		Date: 22/07/09
Sample Discription	Legend	depth m (thick)	Notes
Reinforced concrete		(0.20) 0.20	
Soft Lt G Y CLAY, containing some rootlets (Weathered Gault Clay)		(0.87)	
Soft-Firm, moist Lt G, 40% mottled Lt Br picking out 45° planes, slightly silty CLAY, ~1mm holes may be root pores (Weathered Gault Clay)		1.07 (0.33)	100% Flush returns
Used		1.40 (0.17)	1.30m possible joint 70°
Firm, slightly moist Lt G, 40% mottled Lt Br picking out horizontal layers, slightly silty CLAY, ~1mm holes may be root pores (Weathered Gault Clay)		1.57 (0.63)	
Firm, slightly moist Lt Br G, mottled Lt Br, some W near vertical veins slightly silty slightly sandy CLAY, several sandy pockets up to 30mm, some sand follows planes(Weathered Gault Clay)		2.20 (0.32)	2.40m possible joint 30°
Core Loss		2.52 (0.20)	
Firm, slightly moist Lt Br G, mottled Lt Br, some W near vertical veins slightly silty slightly sandy CLAY, several sandy pockets up to 30mm, some sand follows planes(Weathered Gault Clay)		2.72 (1.24)	2.94m 30mm sandy pocket 3.02m 10mm Lt Y W phosphatic nodule
Stiff, slightly moist Lt Br G, slightly silty slightly sandy CLAY, sand follows planes, some 3mm Lt Br patches, some 1mm R holes (possible root pores) (Weathered Gault Clay)		3.96 (2.30)	3.75m 5mm Lt Y W phosphatic nodules 3.77m possible joint 30° 4.17m 10mm Lt Y W phosphatic nodules 4.30m 30mm sandy weathered pyritic nodule 4.72m possibl joint 60° 5.07m 5mm Lt Y W phosphatic nodule 5.24m 10mm Bk phosphatic nodule 5.46m 15mm Lt Y W phosphatic nodule 5.68m 5mm Lt Y W phosphatic nodule 6.19m possible joint 65°
Stiff, slightly moist Lt Br G, slightly silty CLAY, mottled Lt G some horizontal alignment some Lt Br patches, few 1mm R spots (Weathered Gault Clay)		6.26 (1.24)	6.97m 4mm Lt Y W phosphatic nodule 65% Flush Returns, water returns much darker, mud breaking down
Notes: On several occasions on this core, multiple attempts were required to retrieve samples			(Continued on next sheet)
Lt G Y Light Greyish Yellow Lt G Light Grey Lt Br Light Brown Lt Gr G Light Greenish Grey Lt Br G Light Brownish Grey W White Y Br Yellowish Brown R Red Lt Y W Light Yellowish White			Logged by SW
			Scale 1:50
			p 1 of 2

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush.	Location Oxfordshire Willow Brook Farm				Borehole WBF2
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44120 92958		Date: 16/07/09		
Sample Discription	Legend	depth m (thick)	Samples Taken	Notes	
Topsoil		0.10 (0.10)		5.67m Ammonite 5.83m joint 0°	
Firm yellow mottled grey silty CLAY containing rootlets flint & brick Iron nodules at top reduces to flecks with depth WEATHERED KIMMERIDGE CLAY		(1.18)			
		1.28	WBF2-40 11cm WBF2-41 16cm WBF2-42 13cm WBF2-43 14cm WBF2-44 15cm		
Stiff grey mottled yellow slightly sandy silty CLAY, sand appears to be crystalline calcite? WEATHERED KIMMERIDGE CLAY		(0.91)	WBF2-45 13cm WBF2-46 28cm WBF2-47 28cm WBF2-48 28cm WBF2-49 31cm		
		2.19			
Stiff grey mottled yellow slightly sandy silty CLAY, mottling decreases with depth possible calcite crystals WEATHERED KIMMERIDGE CLAY		(1.77)	WBF2-50 12cm WBF2-51 9 cm WBF2-52 25cm WBF2-53 27cm WBF2-54 25cm WBF2-55 26cm WBF2-56 26cm WBF2-57 10cm		
		3.96			
Stiff grey slightly sandy silty CLAY, some small yellow nodules WEATHERED KIMMERIDGE CLAY		(0.71)	WBF2-58 6 cm WBF2-59 33cm WBF2-60 6 cm WBF2-61 17cm WBF2-62 26cm WBF2-63 28cm WBF2-64 25cm WBF2-65 7 cm		
		4.67			
Stiff dark grey CLAY, KIMMERIDGE CLAY		(0.65)	WBF2-66 5 cm WBF2-67 30cm WBF2-68 14cm WBF2-69 30cm WBF2-70 29cm WBF2-71 8 cm		
		5.32			
Hard shelly light grey slightly clayey SAND separated by thin layers of dark grey slightly sandy slightly silty clay shells are broken KIMMERIDGE CLAY		(1.14)			
		6.46			
Stiff yellowish grey slightly sandy silty CLAY, containing some broken shells KIMMERIDGE CLAY		(1.06)			
Notes:					(Continued on next sheet)
					Logged by SW
					Scale 1:50
					p 1 of 2

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Cornacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush.	Location Oxfordshire Willow Brook Farm				Borehole WBF2
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44120 92958			Date: 16/07/09	
Sample Discription	Legend	depth (thick)	Samples Taken	Notes	
Very stiff grey slightly sandy silty CLAY, variation in colour (banding?) many shelly horizons, several hard bands, some yellowish grey clay bands (varying from 20-100mm in size KIMMERIDGE CLAY.		7.52	WBF2-72 22cm		
		(1.19)	WBF2-73 13cm WBF2-74 13cm WBF2-75 10cm WBF2-76 21cm WBF2-77 13cm	8.16m 60mm hard band 8.57m 20mm hard band	
Very stiff dark grey slightly sandy silty CLAY, a few shell fragments		8.71	WBF2-78 23cm WBF2-79 21cm WBF2-80 29cm WBF2-81 6 cm	8.75m 45° joint	
		(2.28)	WBF2-82 22cm WBF2-83 11cm WBF2-84 27cm WBF2-85 10cm WBF2-86 2 cm	9.45m shell band	
			WBF2-87 28cm WBF2-88 28cm WBF2-89 20cm	10.67m 20mm shell band	
		10.99			
Very stiff dark grey slightly sandy silty CLAY, a few shell fragments forming rare bands ~10mm thick			WBF2-90 16cm WBF2-91 25cm WBF2-92 25cm WBF2-93 24cm WBF2-94 25cm WBF2-95 25cm WBF2-96 1 cm	11.11m broken shell frgments 11.47m 40° joint 11.75m shell band 11.94m shell band 12.17m shell band	
		(1.72)			
			WBF2-97 11cm WBF2-98 24cm WBF2-99 25cm WBF2-100 26cm	13.27m shell band	
		12.71			
Very stiff dark grey slightly sandy silty CLAY, a few shell fragments		(1.35)	WBF2-101 6 cm WBF2-102 25cm WBF2-103 15cm		
		14.06			
Notes:					(Continued on next sheet)
					Logged by SW
					Scale 1:50
					p 2 of 2

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush.	Location Oxfordshire Willow Brook Farm				Borehole WBF2
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44120 92958		Date: 16/07/09		
Sample Discription	Legend	depth m (thick)	Samples Taken	Notes	
Topsoil		0.10 (0.10)		5.67m Ammonite 5.83m joint 0°	
Firm yellow mottled grey silty CLAY containing rootlets flint & brick Iron nodules at top reduces to flecks with depth WEATHERED KIMMERIDGE CLAY		(1.18)			
		1.28	WBF2-40 11cm WBF2-41 16cm WBF2-42 13cm WBF2-43 14cm WBF2-44 15cm		
Stiff grey mottled yellow slightly sandy silty CLAY, sand appears to be crystalline calcite? WEATHERED KIMMERIDGE CLAY		(0.91)	WBF2-45 13cm WBF2-46 28cm WBF2-47 28cm WBF2-48 28cm WBF2-49 31cm		
		2.19			
Stiff grey mottled yellow slightly sandy silty CLAY, mottling decreases with depth possible calcite crystals WEATHERED KIMMERIDGE CLAY		(1.77)	WBF2-50 12cm WBF2-51 9 cm WBF2-52 25cm WBF2-53 27cm WBF2-54 25cm WBF2-55 26cm WBF2-56 26cm WBF2-57 10cm		
		3.96			
Stiff grey slightly sandy silty CLAY, some small yellow nodules WEATHERED KIMMERIDGE CLAY		(0.71)	WBF2-58 6 cm WBF2-59 33cm WBF2-60 6 cm WBF2-61 17cm WBF2-62 26cm WBF2-63 28cm WBF2-64 25cm WBF2-65 7 cm		
		4.67			
Stiff dark grey CLAY, KIMMERIDGE CLAY		(0.65)	WBF2-66 5 cm WBF2-67 30cm WBF2-68 14cm WBF2-69 30cm WBF2-70 29cm WBF2-71 8 cm		
		5.32			
Hard shelly light grey slightly clayey SAND separated by thin layers of dark grey slightly sandy slightly silty clay shells are broken KIMMERIDGE CLAY		(1.14)			
		6.46			
Stiff yellowish grey slightly sandy silty CLAY, containing some broken shells KIMMERIDGE CLAY		(1.06)			
Notes:					(Continued on next sheet)
					Logged by SW
					Scale 1:50
					p 1 of 2

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Cornacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush.	Location Oxfordshire Willow Brook Farm				Borehole WBF2	
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44120 92958			Date: 16/07/09		
Sample Discription	Legend	depth (thick)	Samples Taken	Notes		
Very stiff grey slightly sandy silty CLAY, variation in colour (banding?) many shelly horizons, several hard bands, some yellowish grey clay bands (varying from 20-100mm in size KIMMERIDGE CLAY		7.52	WBF2-72 22cm	8.16m 60mm hard band		
		(1.19)	WBF2-73 13cm			
			WBF2-74 13cm			
			WBF2-75 10cm			
			WBF2-76 21cm	8.57m 20mm hard band		
			WBF2-77 13cm			
		8.71	WBF2-78 23cm	8.75m 45° joint		
			WBF2-79 21cm			
			WBF2-80 29cm	9.45m shell band		
			WBF2-81 6 cm			
		(2.28)	WBF2-82 22cm			
			WBF2-83 11cm			
			WBF2-84 27cm			
			WBF2-85 10cm			
			WBF2-86 2 cm			
			WBF2-87 28cm	10.67m 20mm shell band		
			WBF2-88 28cm			
		10.99	WBF2-89 20cm	11.11m broken shell frgments		
			WBF2-90 16cm			
			WBF2-91 25cm	11.47m 40° joint		
			WBF2-92 25cm			
		(1.72)	WBF2-93 24cm	11.75m shell band		
			WBF2-94 25cm			
			WBF2-95 25cm	11.94m shell band		
			WBF2-96 1 cm			
			WBF2-97 11cm	12.17m shell band		
			WBF2-98 24cm			
		12.71	WBF2-99 25cm			
			WBF2-100 26cm			
		(1.35)	WBF2-101 6 cm	13.27m shell band		
			WBF2-102 25cm			
			WBF2-103 15cm			
		14.06				
Notes:						
(Continued on next sheet)					Logged by SW	
					Scale 1:50	
					p 2 of 2	

Equipment and methods Hand dug inspection pit to 1.35m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.37m using natural polymer mud flush	Location Oxfordshire, Willowbrook Farm (relogged) Borehole WBF1			
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44129 92974 Date: 14/07/09 - 15/07/09			
Sample Discription	Legend	depth m (thick)	Notes	
Very stiff dark brown slightly sandy silty CLAY containing some gravel: flint & brick some roots		(0.34)		
Very stiff brownish grey slightly sandy silty CLAY some sandy nodules, calcareous inclusions, rootles surrounded by iron staining		0.34 (0.29)		
Firm light grey slightly sandy silty CLAY mottled dark grey calcareous inclusions, dark red and yellow speckles		0.63 (0.72)		
Firm, slightly moist 40% Lt Br G mottled with 40% Lt Br and 20% Bk, slightly sandy slightly silty CLAY some Lt Y W sandy patches 2mm some bedding parallel sand areas, possible bedding (WEATHERED KIMMERIDGE CLAY)		1.35 (0.48)	1.35-7.80 100% Flush Return	
Firm, slightly moist G, slightly sandy slightly silty CLAY 30% mottled Lt Br. mottling follows bedding, sand appears concentrated in G areas cross bedding like terminations observed (WEATHERED KIMMERIDGE CLAY)		1.83 (0.94)	1.93m 10mm Y Br sandy nodule 2.12m 20mm Y Br sandy nodule 2.32m 20mm R nodule	
Stiff, slightly moist Lt Br slightly sandy slightly silty CLAY, G 2mm thick subhorizontal lines every ~20mm containing sand. Some 20mm lenses of Lt Br G clay, few 10mm sandy pockets (WEATHERED KIMMERIDGE CLAY)		2.77 (0.62)	2.58m 80mm burrow 2.77m Joint 70°	
Stiff, slightly moist G slightly sandy slightly silty CLAY, some Lt Br stained areas some 10mm sandy layers (WEATHERED KIMMERIDGE CLAY)		3.39 (0.30)	4.19m possible joint 45°	
Stiff, slightly moist G sandy silty CLAY, some Lt Gr mottling, some Lt Y W lenses app. 40mm width, 5mm height, becomes banding with depth some Dk Gr G lenses (WEATHERED KIMMERIDGE CLAY)		3.69 (0.51)		
Stiff, slightly moist G slightly silty CLAY, some Dk Gr G lenses 10mm thick 30mm long some 7mm shelly pockets. 20mm sandy layer at 5.12m (KIMMERIDGE CLAY)		4.20 (1.15)		
Hard, slightly moist Dk G slighly silty slightly sandy CLAY, many W flecks of broken shell fragments, aligned to horizontal in rough layers some slight colour variation forming 5mm bands. 10mm sandy layers towards base (KIMMERIDGE CLAY)		5.35 (0.77)	5.22m 10mm W nodule 5.51m possible joint 30°	
Stiff, slightly moist G slighly silty slightly sandy CLAY, few W flecks = broken shell fragments, (KIMMERIDGE CLAY)		6.12 (0.25) 6.37 (0.09) 6.46	6.86m 30mm silty band 6.99m 30mm hard sandy band	
Hard, slightly moist Dk G slighly silty slightly sandy CLAY, several shells (KIMMERIDGE CLAY)		(0.83)		
Stiff, slightly moist Dk G slighly silty slightly sandy CLAY, few W flecks = broken shell fragments, some forming layers (KIMMERIDGE CLAY)		7.29 (0.29)		
Used				
Notes: Lt G Y Light Greyish Yellow Lt G Light Grey Lt Br Light Brown Lt Gr G Light Greenish Grey Lt Br G Light Brownish Grey G Grey Y Br Yellowish Brown R Red Dk Gr G Dark Greenish Grey W White			(Continued on next sheet)	
	Logged by SW			
	Scale 1:50			
	p 1 of 2			

Equipment and methods Hand dug inspection pit to 1.35m Rotary drilling using Comacchio 450P rig wireline Geobore S (102mm diameter) to 14.37m using natural polymer mud flush	Location Oxfordshire, Willowbrook Farm (relogged)		Borehole WB1
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44129 92974	Date: 14/07/09 - 15/07/09	
Sample Discription	Legend	depth m (thick)	Notes
Used		7.58 (0.20)	7.69m 3mm thick shell
Hard, slightly moist G slighly silty slightly sandy CLAY, some micaceous sandy pockets 5mm thich 15mm width (KIMMERIDGE CLAY)		7.78 (0.23)	
Very stiff, slightly moist Dk G slighly silty slightly sandy CLAY, few shells some silty lenses 5mm thick (KIMMERIDGE CLAY)		8.01 (0.10) 8.11 (0.30)	
Hard, dense silty clayey SAND many broken shells (KIMMERIDGE CLAY)		8.41 (0.19) 8.60	
Used			9.18m and 9.21m 6mm shell bands possible ammonite 9.30m boundary feathery
Very stiff, slightly moist Dk G slighly silty slightly sandy CLAY, some sandy pockets some shells (KIMMERIDGE CLAY)		(0.70)	
Very stiff, slightly moist Dk G slighly silty CLAY, broken shells rarer with depth some slightly darker lenses, variation G-DK G (KIMMERIDGE CLAY)		9.30	
Very stiff, slightly moist Dk G slighly silty CLAY, some rare shells (KIMMERIDGE CLAY)		(0.52)	
Core Loss		9.82 (0.60)	10.78m 5mm shell fragment 10.84m 10mm thick silty band 10.96m 10mm shell, possible ammonite
Very stiff, slightly moist Dk G slighly silty CLAY, some rare shells (KIMMERIDGE CLAY)		10.42 (0.62)	
Very stiff, slightly moist Dk G slighly silty CLAY, some rare shells some silty lenses, some thick silty layers 11.42-11.49m, 11.64-11.67m silty areas contain more shells, some indications of terminations/crossbedding (KIMMERIDGE CLAY)		11.04 (1.49)	
Very stiff, slightly moist layered Dk G, G slighly sandy slighly silty CLAY, dark areas contain ~10% more sand lowere boundary very ragged/interfingered (KIMMERIDGE CLAY)		12.53 (0.88)	
Very stiff, slightly moist layered Dk G, slightly sandy slighly silty CLAY, layers formed by variations in silt and sand, shell fragments very small mainly in sandy layers (KIMMERIDGE CLAY)		13.41 (0.66)	13.05-13.14m silty band
Core Loss		14.07 (0.27) 14.34	13.55-3.59m silty band
Notes:			Logged by SW
G Grey Dk G Dark Grey			Scale 1:50
			p 2 of 2

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Cornacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush		Location Oxfordshire, Willowbrook Farm (relogged)		Borehole WBF2	
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON		Coordinates: SU 44120 92958		Date: 16/07/09	
Sample Discription		Legend	depth m (thick)	Notes	
TOPSOIL			0.10 (0.10)		
Very stiff grey slightly sandy CLAY some rootlets surrounded by iron staining other iron rich nodules			(0.31)		
Firm light grey slightly sandy silty CLAY mottled yellow containing bricks, flint and rootlets some red iron flecks			0.41 (0.34)		
Not recovered			0.75 (0.28)		
Firm, moist Lt Br slightly silty slightly sandy CLAY holes, possible root pores, some rootlets no structure (WEATHERED KIMMERIDGE CLAY)			1.03 (0.14)		
Firm, moist Lt Br G slightly silty slightly sandy CLAY motteled Lt Br 45% motteled Bk 20% moteling decreases with depth, some 10mm sand rich horizons (WEATHERED KIMMERIDGE CLAY)			1.17 (0.40)		
Firm, slightly moist Lt Br 50% slightly silty slightly sandy CLAY motteled Lt Br G 10% several horizontal wigley lines with vertical conections surounded by G areas some sandy inclusions. G areas and lines becoming more spaced out with depth (WEATHERED KIMMERIDGE CLAY)			1.57 (0.68)	1.90m 20mm R sand pocket weathered pyrite nodule	
Stiff G slightly silty slightly sandy CLAY motteled Lt Br, 10mm pockets of coarse sand/clear crystals (WEATHERED KIMMERIDGE CLAY)			2.25 (0.34)		
Stiff Lt Br G slightly silty slightly sandy CLAY G areas surrounding sand lines 20% some lines forming ovals sand becomes more common with depth (WEATHERED KIMMERIDGE CLAY)			2.59 (1.31)		
Very Stiff Dk G slightly silty CLAY small lenses of slightly darker material becoming larger some forming 100mm bands at depth some bands of shells (KIMMERIDGE CLAY)			3.90 (1.33)	3.81m 10mm sandy band 4.02m 3mm shell band 4.47m 10mm sandy band 4.61m Possible joint 80°	
Hard G Sandy Clay shell rich band (KIMMERIDGE CLAY)			5.23 5.29 (0.06)	5.37m 10mm broken shell band Possible amonite	
Very Stiff Dk G slightly silty slightly sandy CLAY lots of shells mostly fragments grain size variation in layers (KIMMERIDGE CLAY)			(0.45)		
Used			5.74 (0.25)		
Very Stiff Dk G slightly silty slightly sandy CLAY lots of shells mostly fragments grain size variation in layers (KIMMERIDGE CLAY)			5.99 (0.42)		
Very Stiff Dk G slightly silty slightly sandy CLAY containing some sandy horizons, 6.69-6.77m & 6.96-7.05m (~25% sand), few shells (KIMMERIDGE CLAY)			6.41 (0.81)	6.40m 10mm sandy hard band	
Core Loss			7.22 (0.15)		
See next sheet			7.37		
Notes:			(Continued on next sheet)		
Lt G Y Light Greyish Yellow Lt Gr G Light Greenish Grey Y Br Yellowish Brown			Lt G Light Grey Lt Br G Light Brownish Grey R Red		
			Lt Br Light Brown G Grey Dk Gr G Dark Greenish Grey		
			Logged by SW Scale 1:50 p 1 of 2		

Equipment and methods Hand dug inspection pit to 1.03m Rotary drilling using Cornacchio 450P rig wireline Geobore S (102mm diameter) to 14.30m using natural polymer mud flush	Location Oxfordshire, Willowbrook Farm (relogged)		Borehole WBF2
Carried out by EMMERSON AND MOORE & IMPERIAL COLLEGE LONDON	Coordinates: SU 44120 92958	Date: 16/07/09	
Sample Discription	Legend	depth m (thick)	Notes
Very Stiff Dk G slightly silty slightly sandy CLAY no structures, few shells Sand incresing towards base (KIMMERIDGE CLAY)		(0.92)	7.98m 5mm band of broken shells 8.44m 10mm shell possible ammonite 8.51m 10mm band of Y Br hard nodules 8.58m 20mm hard nodule 8.65m 40mm hard band 8.90m 30mm hard band
Very Stiff- Hard bands Dk G slightly silty slightly sandy CLAY harder bands and hard nodules, containing slightly more sand (KIMMERIDGE CLAY)		8.29 (0.23)	
Hard Dk G slightly garvelly slightly sandy silty CLAY several hard nodules		8.52 (0.21)	
Very Stiff slightly moist G slightly silty slightly sandy CLAY some Dk G lenses possible terminations (KIMMERIDGE CLAY)		8.73 (0.16)	
Very Stiff slightly moist Dk G slightly silty slightly sandy CLAY some shells some silty bands (KIMMERIDGE CLAY)		8.89 (0.89)	
Used		9.78 (0.78)	
Core Loss		10.56 (0.35)	
Used		10.91 (0.41)	
Very Stiff slightly moist Dk G slightly silty slightly sandy CLAY some shells many aligned to the horizontal some colour shade variation (KIMMERIDGE CLAY)		11.32 (0.74)	11.42m 5mm nodule band 11.71m 5mm ammonite shells 11.91m 10mm ammonite shells 12.00m 3mm ammonite shells 12.29m 20mm amonite shells 13.10m 20mm ammonite shells 13.47m 2mm thick broken shell
Very Stiff slightly moist Dk G silty CLAY silt concentrated as lenses some bands several shells some forming bands, amonite cross sections (KIMMERIDGE CLAY)		12.06 (0.26)	
Core Loss		12.32 (0.30)	
Very Stiff slightly moist Dk G slightly sandy slightly silty CLAY sand and silt form bands (KIMMERIDGE CLAY)		12.62 (0.39)	
Very Stiff slightly moist Dk G silty CLAY silt concentrated as lenses some bands several shells some forming bands, amonite cross sections (KIMMERIDGE CLAY)		13.01 (0.93)	
Core Loss		13.94 (0.36)	
		14.30	
Notes:			
Lt G Y Light Greyish Yellow Lt Gr G Light Greenish Grey Y Br Yellowish Brown Lt G Light Grey Lt Br G Light Brownish Grey R Red Lt Br Light Brown G Grey Dk Gr G Dark Greenish Grey			

APPENDIX C

IMAGING TECHNIQUES

During the early stages of the project, the Oxford Clay was the only mudrock available for sampling. Most of the initial work of evaluating different imaging techniques was carried out using this material. The imaging methods suggested in the project brief were environmental scanning electron microscopy and CT imaging. Of these CT imaging was discounted early on as it does not have the resolution to image individual clay mineral particles. Thus several imaging methods were reviewed to find the most appropriate to use for the imaging of mudrocks. These methods, how they work and their application to mudrocks are summarised in this appendix. Many of the references point to expositions of much greater detail.

C.1 Optical microscope

The use of lenses for magnification can be traced back to ancient Egypt ~1000BC, some lenses surviving in museums today. The compound microscope was invented in 1590 by Hans and Zacharias Janssen using multiple lenses separated by a tube (Clay and Court, 1932). The most important early work on the microscope was *Micrographia* written by Robert Hooke on the instruction of the Royal Society (Hooke, 1665). The publication of this book popularised the compound microscope among the upper class of the time. The scientific community continued to use simple microscopes (many different styles, most only using a single lens) (Hartley, 1993). One of the earliest recorded uses of the microscope in the subject of geology is in 1705. Anthony van Leuwenhoek used his simple microscope to observe the shape of sand grains (van Leuwenhoek A., 1705a), and pumice, coral, and sponges (van Leuwenhoek A., 1705b). The polarisation of light was discovered by Etienne-Louis Malus in 1808 this was a big step in the field of optics (Kahr and Claborn, 2008). However in order to use polarised light to identify minerals a method of creating slices of rock thin enough for light to pass through was required. The precise origins of the thin sectioning technique is lost to history, its invention is commonly attributed to William Nicol in 1831 (Young, 2003).

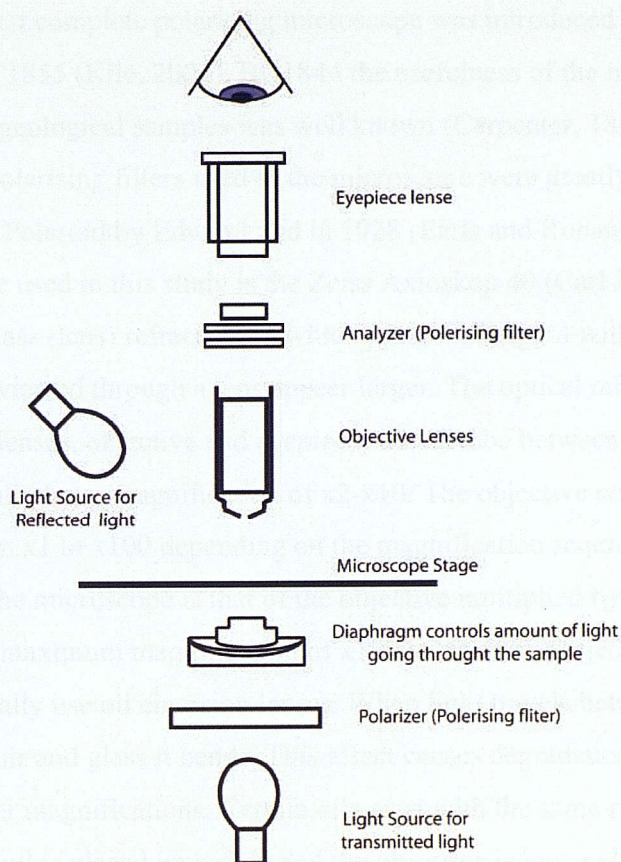


Figure C.1 A schematic diagram of the important components of a petrological optical microscope based on (Gribble and Hall, 1992)

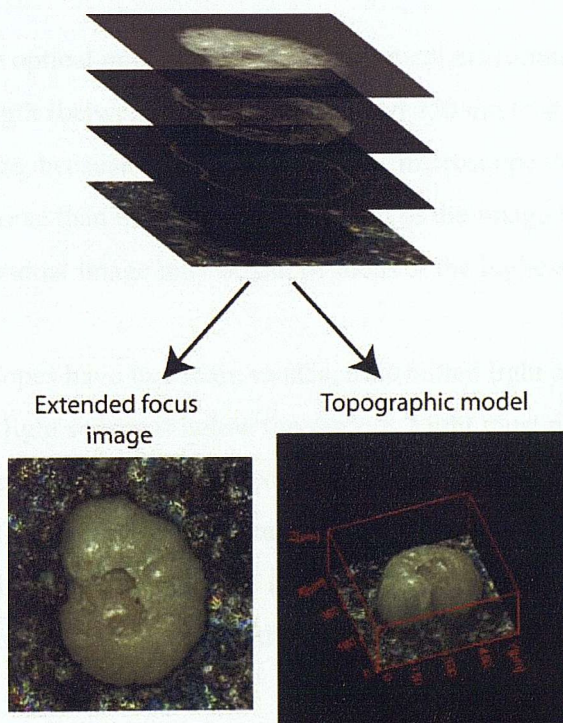


Figure C.2 The use of a z-stack to form both extended focus images and topographic models. Four representative slices are shown out of the 20 used for these reconstructions. Image analysis using AxioVision (Carl Zeiss, 2008).

Probably the earliest complete polarising microscope was introduced by Natchet between 1838 and 1855 (Kile, 2003). By 1844 the usefulness of the microscope in reflected light for geological samples was well known (Carpenter, 1844). The efficiency of the polarising filters used in the microscope were greatly improved by the invention of sheet Polaroid by Edwin Land in 1928 (Earls and Rohani, 2005). The optical microscope used in this study is the Zeiss Axioskop 40 (Carl Zeiss, 2008). A convex piece of glass (lens) refracts light which passes through it with the effect of making an object viewed through a lens appear larger. The optical microscope is composed of two lenses, objective and eyepiece, with a tube between them (Figure C.1). The eyepiece usually has a magnification of $\times 2$ - $\times 10$. The objective commonly ranges in magnification from $\times 1$ to $\times 100$ depending on the magnification required. The total magnification of the microscope is that of the objective multiplied by that of the eyepiece giving a maximum magnification of $\times 1000$ (100×10). Objectives with high magnification usually use oil emersion lenses. When light travels between two different materials such as air and glass it bends. This affect causes degradation of the image especially at higher magnifications. Certain oils exist with the same refractive index as glass. If a drop of oil is placed on a slide and the objective is lowered until it comes into contact with the oil, image distortion is avoided.

The resolution of the optical microscope has a theoretical maximum limit of the same order as the wavelength (between 380 nm (violet) and 750 nm (red)) of the light due to diffraction. In practice, because of other errors in the microscope the maximum resolution may be worse than this. The depth of field of the image becomes very small and much of an individual image may be out of focus at the highest magnifications.

Petrological microscopes have two main modes, transmitted light and reflected light. In transmitted light the light source is below the sample. Light must pass through the sample in order to be observed. In reflected light the light source is above the sample. Light is reflected off the surface of the sample before entering the objective. Reflected light can be used to image the topography and nature of a samples surface. Using transmitted light some information about the internal structure of the material can be obtained.

In transmitted light the petrological microscope allows the use of two optional polarising filters, the polariser below the sample and the analyser above the sample

(Figure C.1). The polariser ensures that the light entering the sample is polarised into a single plane (plane polarised light). The analyser polarises at 90 degrees (cross polarised light) to the polariser meaning that in the absence of the sample no light will pass. Minerals split the polarised beam of light into component beams. These components may travel through the mineral at different velocities. On exiting the mineral they may recombine out of phase. The insertion of the analyser allows the interference colour of these recombined beams to be seen. While the majority of minerals have some level of anisotropy, some minerals are isotropic. They have the same properties in all directions and so appear black in cross polarised light.

With rotation of the microscope stage the interference colours of the minerals will go from a maximum intensity to completely black (extinction) four times during a 360° rotation. This occurs when either one of the two mineral vibration directions is parallel with the polariser. Meaning that the light passing through the mineral will have no component in the same orientation as the analyser, and so no light can pass. These optical properties of minerals are used in petrology for the analysis of the mineral content of geological thin sections.

Most modern microscopes are combined with a digital camera which allows images to be saved. An individual image taken using the optical microscope will not be completely in focus due to the limited focal plane of the optical technique. A completely in focus image can be digitally constructed. If the camera is focused on a focal plane below the sample and sequential images are taken from this position to a focal plane above the sample a stack of images (z-stack) is formed (Figure C.2). By taking only the in-focus data from each of these images an extended focus image where the entire sample is in focus can be created. If a stepping motor is used to specify the magnitude of the change in height in microns between each image, a topographic representation of a sample can be created from the in focus information. This process is automated by the use of computer software (Carl Zeiss, 2008).

To use the optical microscope in transmitted light on mudrocks a thin section 30µm in thickness must be made (Reed and Mergner, 1953). To create a thin section one side of the mudrock is ground and polished flat and then glued to a piece of glass. The other side would then be ground down until it is the correct thickness. The grinding process is most commonly lubricated by water which is undesirable for mudrock specimens. It

may be lubricated by oil if the machine owner will allow (Cetin and Söylemez, 2004), or air, unfortunately this causes excessive heating of the apparatus. On attempting this process using air, the samples started to pull apart at about 500µm thickness. Another method of making thin sections involves embedding samples in a resin prior to sectioning (Dudoignon et al., 2001). However it is uncertain what affect the embedding process has on the structure of the mudrock the resin is being imbibed into.

Assuming that a reasonable thin section can be made then rough estimates of the alignment or non alignment of minerals can be made (Dudoignon and Pantet, 1998). The amount of light travelling through the microscope using cross polarised light will be dependent on the orientation of clay mineral crystals within the sample (Bai and Smart, 1997; Morgenstern and Tchalenko, 1967). If all of the crystals are perfectly aligned then as the stage is rotated all of the crystals will all go into extinction at the same time and will be perfectly out of extinction at the same time (Bai et al., 1994). If all of the crystals are in random orientations then there will be no change in the amount of light travelling through the slide with rotation of the stage (Dudoignon et al., 2001). The optical microscope has been used to study the preferred orientation of several of the materials studied in this thesis (Tchalenko, 1968). In order of weak preferred alignment to strong preferred alignment: Gault Clay, Lias Clay, London Clay, Oxford Clay.

C.2 Confocal microscope

The confocal microscope gains accuracy of measurement over the optical microscope as it uses a limited wavelength of light. The improvement in resolution is by a factor of root 2 (1.414) (Amos and White, 2003). The lower the wavelength used the higher the resolution. The extent of this resolution improvement has been questioned as being not as significant as the theoretical improvement suggests (Cox and Sheppard, 2004). The major improvement to imaging provided by the confocal microscope is the ability to focus on individual focal planes of material and cutting out all out of focus information without complex computations.

The idea of the confocal microscope was first conceived in 1955 (Minsky, 1988). Even though a working model was made and a patent was written, this invention was overlooked for many years, being known only within optics/physics papers (Carlsson et al., 1985). Eventually the confocal microscope was reintroduced as a commercial

product in 1986 and began a rapid revolution of biological microscopy (White and Amos, 1987). The main advantage the confocal microscope had was it could create stacks of images in space and time. So a z-stack of images could be taken, then ten minutes later another stack could be taken. After days of observation a video of the three dimensional movements of the sample could be made.

Within the geological sciences, the first interest in confocal microscopy was for the observation of fission tracks (Petford and Miller, 1990). A three dimensional perspective is particularly useful as the fission tracks run in all directions. Later the same technique was used to image the pore structure of geological materials (Fredrich et al., 1995). This application has been expanded for work on fluid flow. The first time the confocal microscope was applied to the study of mudrock microstructure was in 2003 (Nix and Feist-Burkhardt, 2003)

The Confocal microscope is designed to only allow light from the focal plane to reach the camera for imaging. This is achieved by using a laser light source and a pin hole in front of the detector (Figure C.3). All light from outside of the focal plane cannot reach the detector. The laser used in the microscope commonly uses a limited wavelength of light which is normally the wavelength which produces the best florescence from the material. Normally wavelengths range within the optical range 380 nm (violet) and 750 nm (red). Outside of the visible range, ultra violet light down to 340 nm wavelength can also be applied and displayed in false colour (Müller, 2006).

Theoretically this technique can be used to image the internal structure of mudrocks. However clear observations of focal planes below the surface of the material are limited. When looking inside the material minerals in the upper layers partially absorb the laser light so the intensity of light reaching the focal plane is not uniform (Figure C.4). Imaging the near surface can, and has in the past been effective (Nix and Feist-Burkhardt, 2003). Due to the limitations explained above the confocal microscope could never be used to image the internal structure of semi opaque materials. It could be used to image topography of geological materials however as the optical microscope can do this in true colour the small gain in resolution does not produce a significant improvement. In general for each advantage the confocal microscope gives there is another available method which can do it better for mudrocks.

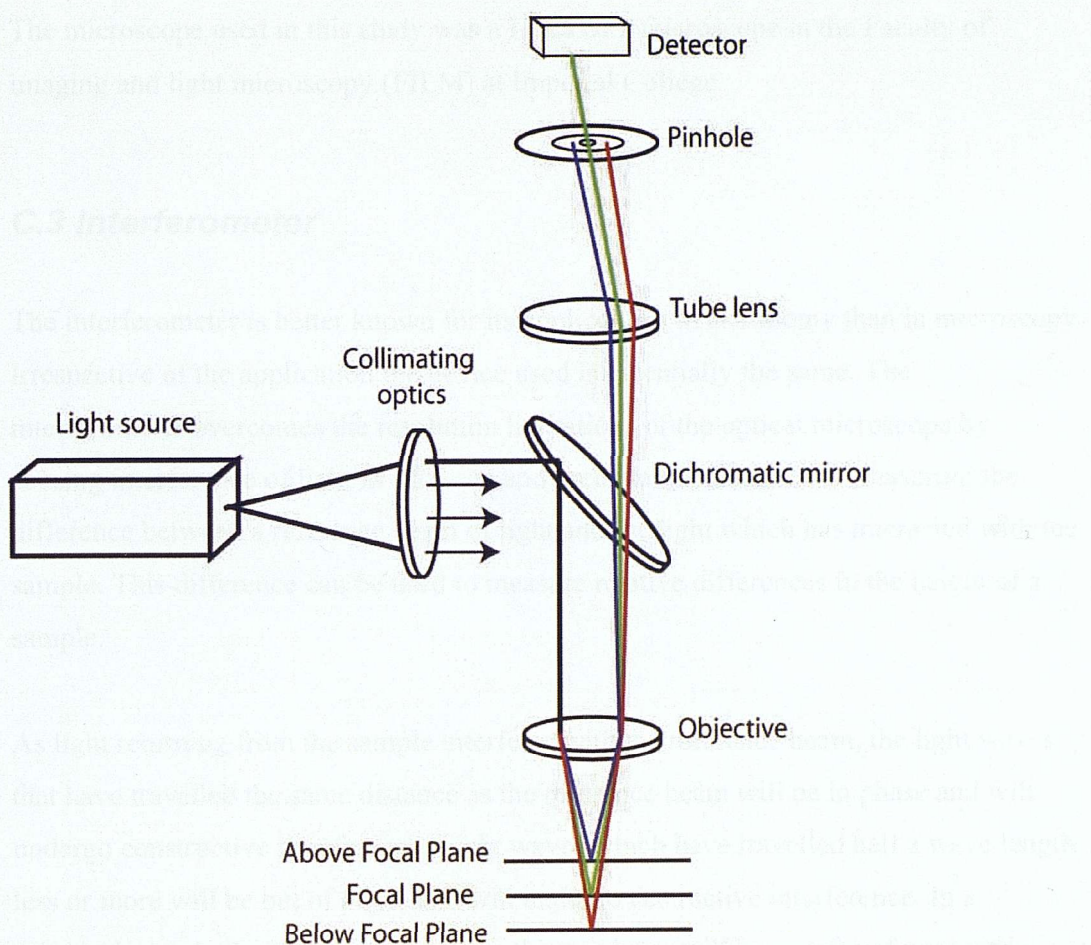


Figure C.3: The components of the confocal microscope, colours indicate light paths not colours of laser light. Only light from the focal plane can pass through the pin hole to the detector. By changing the focal plane a z-stack of images can be taken and the internal structure of a material can be reconstructed.

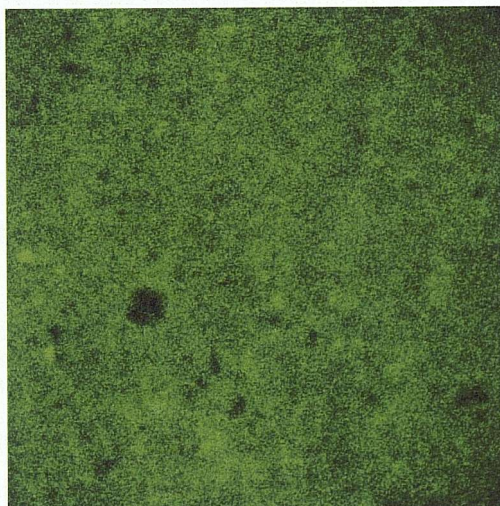


Figure C.4: An example confocal microscope image. Large crystals can be easily seen, and a slight variation in the intensity of colour possibly indicates the layering of the material. This is not very clearly illustrated. Image taken at the Faculty of Imaging and Light Microscopy (FILM) at Imperial College London.

The microscope used in this study was a Leica SP2 microscope in the Faculty of imaging and light microscopy (FILM) at Imperial College.

C.3 Interferometer

The interferometer is better known for its applications in astronomy than in microscopy. Irrespective of the application the device used is essentially the same. The interferometer overcomes the resolution limitations of the optical microscope by causing interference of light in a known and predictable manner and measuring the difference between a reference beam of light and the light which has interacted with the sample. This difference can be used to measure relative differences in the height of a sample.

As light returning from the sample interferes with the reference beam, the light waves that have travelled the same distance as the reference beam will be in phase and will undergo constructive interference. Light waves which have travelled half a wave length less or more will be out of phase and will undergo destructive interference. In a spherical sample the interference pattern this produces will be a series of concentric rings of light and darkness. The difference in height between two adjacent rings will be the same as the wavelength of the light used. Thus the elevation of the sample can be reconstructed to a much greater degree of accuracy than by the use of z-stacked images in the optical microscope.

C.4 Atomic force microscopy

The atomic force microscope (AFM) has a very sharp tip made of silicon. The microscope can measure the force applied to the tip very precisely. This is achieved using a cantilever and a laser (Figure C.5). The AFM can be used to measure the topography of materials, this can be achieved at the molecular scale. However due to the geometry of the microscope extremes of topography, such as cavities or overhangs, cannot be measured. The atomic force microscope was introduced to the scientific community in 1986 (Binnig et al., 1986). Within four years it had been used for the purpose of imaging the surface of minerals (Hochella et al., 1990) and a short while later a clay particle at the molecular scale (Hartman et al., 1990).

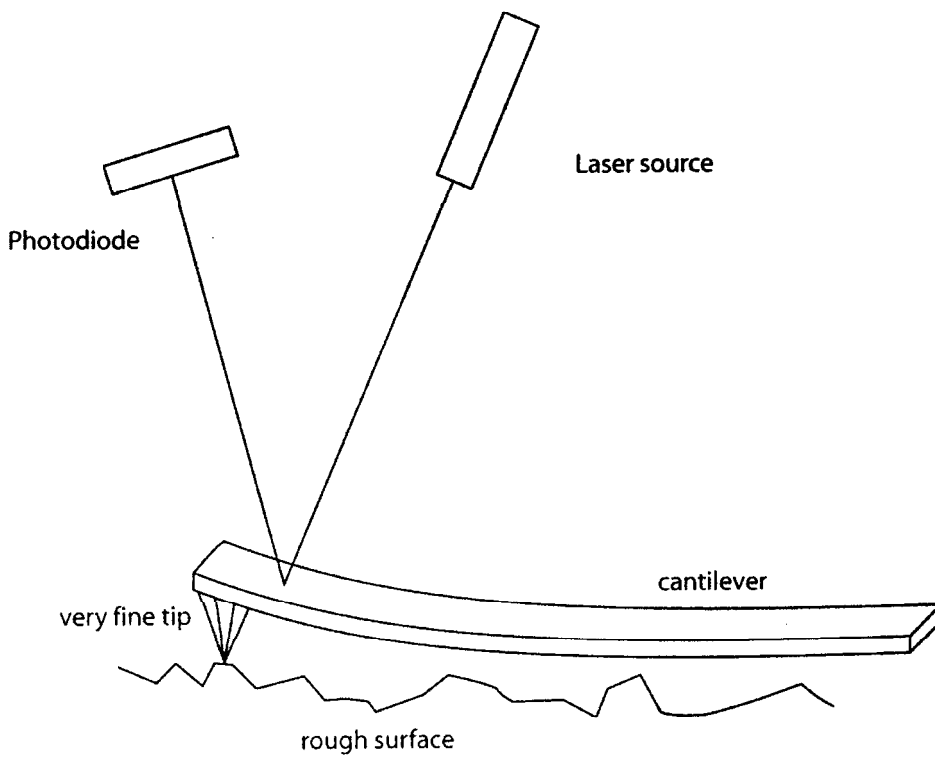


Figure C.5 Basic components of an atomic force microscope

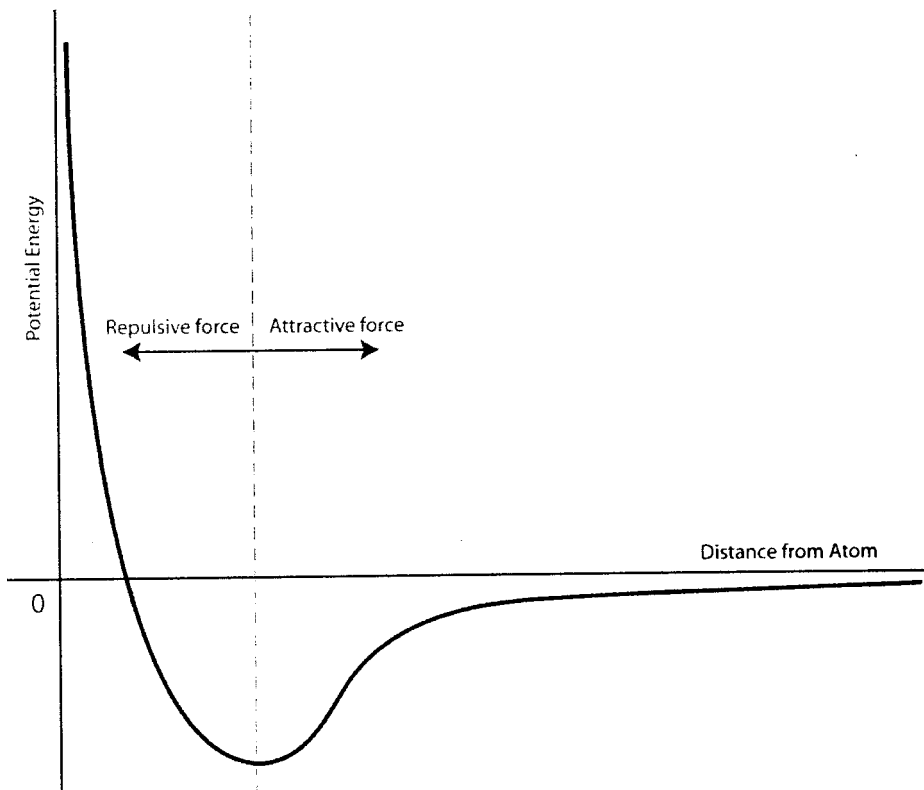


Figure C.6 The potential energy of a system of two atoms varying with the distance between the atoms (Based on (Young et al., 2004)). In the atomic force microscope, contact mode is in the repulsive regime, non-contact mode is in the attractive regime, and tapping mode varies between both regimes.

The cantilever and tip arrangement of the atomic force microscope means that a very small force applied to the tip of the microscope will produce a deflection of the cantilever. This deflection can be easily sensed by the altered path of a laser bounced off the cantilever onto a photodiode.

There are many different methods by which the atomic force microscope can be used. As the tip approaches the surface it experiences an attractive force. At a certain point, close to the material this force becomes repulsive (Figure C.6). The tip can be moved across the surface of the material in the repulsive regime (contact mode). The height of the probe required to keep a constant deflection of the cantilever (constant force on the sample) is measured. This mode is used for the highest resolution images of the topography of surfaces. With delicate samples, contact with the sample is undesirable. It is possible to work at a small distance from the sample, in the attractive force regime (non contact mode). Instead of having a static probe, it is also possible to use a vibrating cantilever. The vibrating cantilever can vary within attractive (non contact), repulsive (contact), or between both regimes (dynamic (tapping) mode). Having an oscillating probe allows many more variables to be measured: amplitude, frequency, phase shift and deflection all of which can be used to gather information about the material surface. This mode has the advantage that it can image very rough surfaces with less risk of collisions between the side of the probe and the particles sitting proud of the surface.

For samples with high moisture contents AFM will not be possible. Scanning ion conductance microscopy follows a similar principle (Hansma et al., 1989). It works either on submerged materials or those where a bead of water can be moved across their surface without having a major impact on the sample. A split tipped microscope should be considered for materials approaching their liquid limit and a single tip instrument for completely submerged materials.

The atomic force microscope is underused within the geological sciences. It is used broadly for chemical geology, for mineralogy and to measure mineral strength and stiffness (Prasad, 2002). However it has rarely been used to image the structure of geological samples. One of the main reasons for this is the idea that the probe may disturb the structure of the sample. This is not the case; in fact the force applied to the sample by the probe is less than that applied by the vacuum used in most scanning electron microscopes.

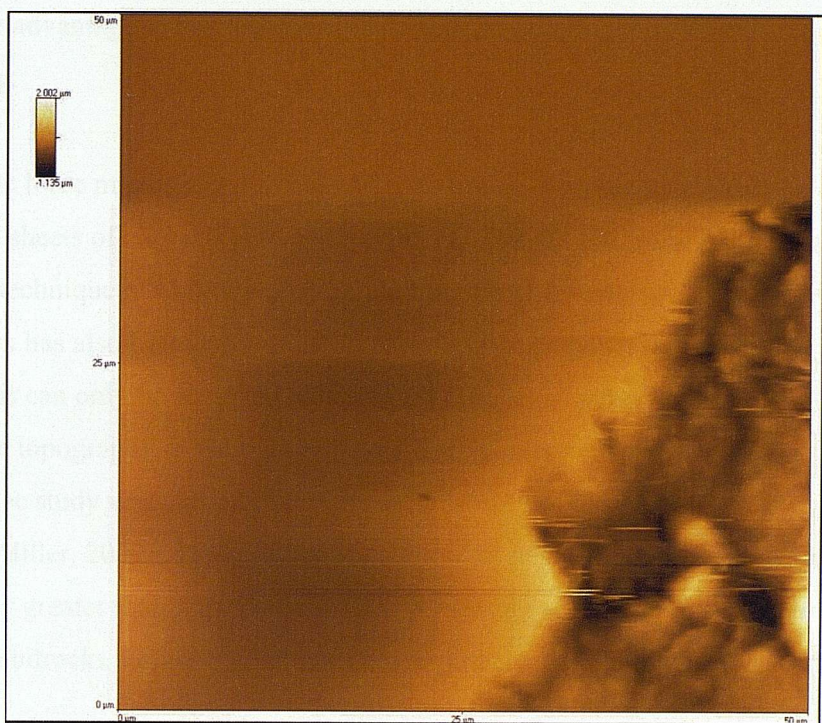


Figure C.7 The surface of the oxford clay images using AFM. The area with no detail is where the surface is lower than the osculation of the AFM. From (Miller, 2009).

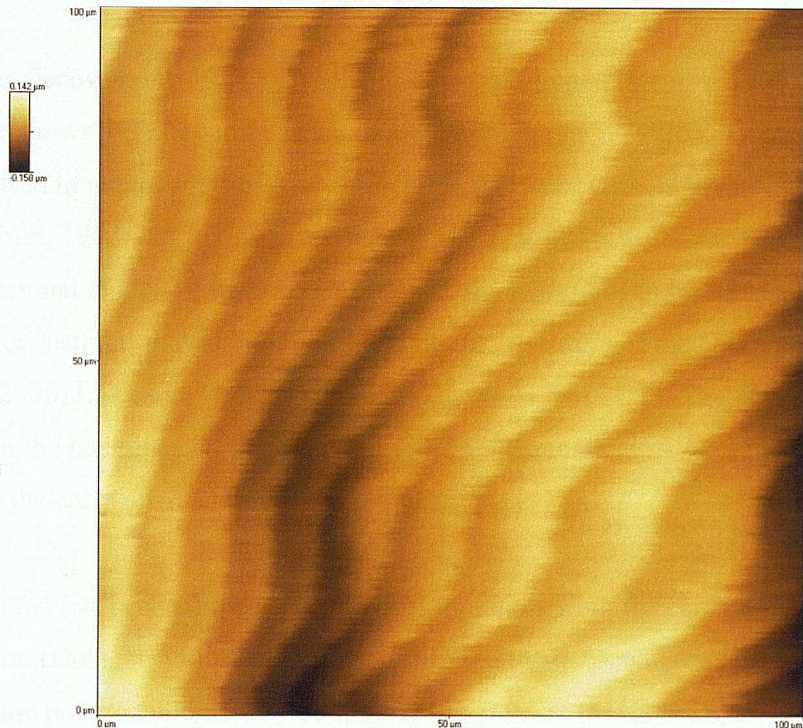


Figure C.8 A image taken where a particle has become attached to the tip of the AFM increasing the wave length of its oscillations. A common occurrence when imaging samples with topography of a greater extent than the AFM can measure. From (Miller, 2009).

The major advantage to this technique is no special sample preparation is required prior to imaging.

The atomic force microscope has been used to image the molecular structure of individual sheets of clay minerals (Hartman et al., 1990). Since the publication of this paper the technique has been used multiple times for the imaging of mineral surfaces. Some work has also been carried out on particle arrangements (Frydman and Frydman, 2005). This can only be achieved with a small number of particles, as at a larger scale the surface topography of the material extends beyond the maximum range of the AFM. A short Msc study was carried out of the applicability of the AFM to broken mudrocks surfaces (Miller, 2009). The results were somewhat negative. Samples often displayed topography greater than what the AFM could measure (Figure C.7) and loose particles from the mudrocks surface sometimes became attached to the microscopes tip (Figure C.8).

C.5 Micro computed tomography (CT)

X-rays were discovered in 1895 by Wilhelm Conrad Röntgen (Dennis, 1995). A single x-ray image shows the x-ray impedance of a material in one direction. These images have been used to identify sedimentary structures for many years (Calvert and Veevers, 1962; Hamblin, 1962). By taking x-ray images from multiple directions (Figure C.9) a three dimensional model of the x-ray impedance of a material can be constructed. This process of reconstruction was only feasible with the invention of the computer (Kalendar, 2006) it became known as computed tomography (CT). The idea of CT was conceived in the late nineteen sixties by Sir Godfrey Hounsfield. A working CT device was built in the early nineteen seventies (Hounsfield, 1973).

The CT scanner has been applied to geological materials from the earliest days of its first invention (Duliu, 1999). Its major application in the geosciences is the study of porosity as the pore to solid density contrast is the greatest (Mees et al., 2003). It is also a non destructive method which can provide three dimensional images. Its limitations are resolution and long imaging time (making it expensive if used more than a few times).

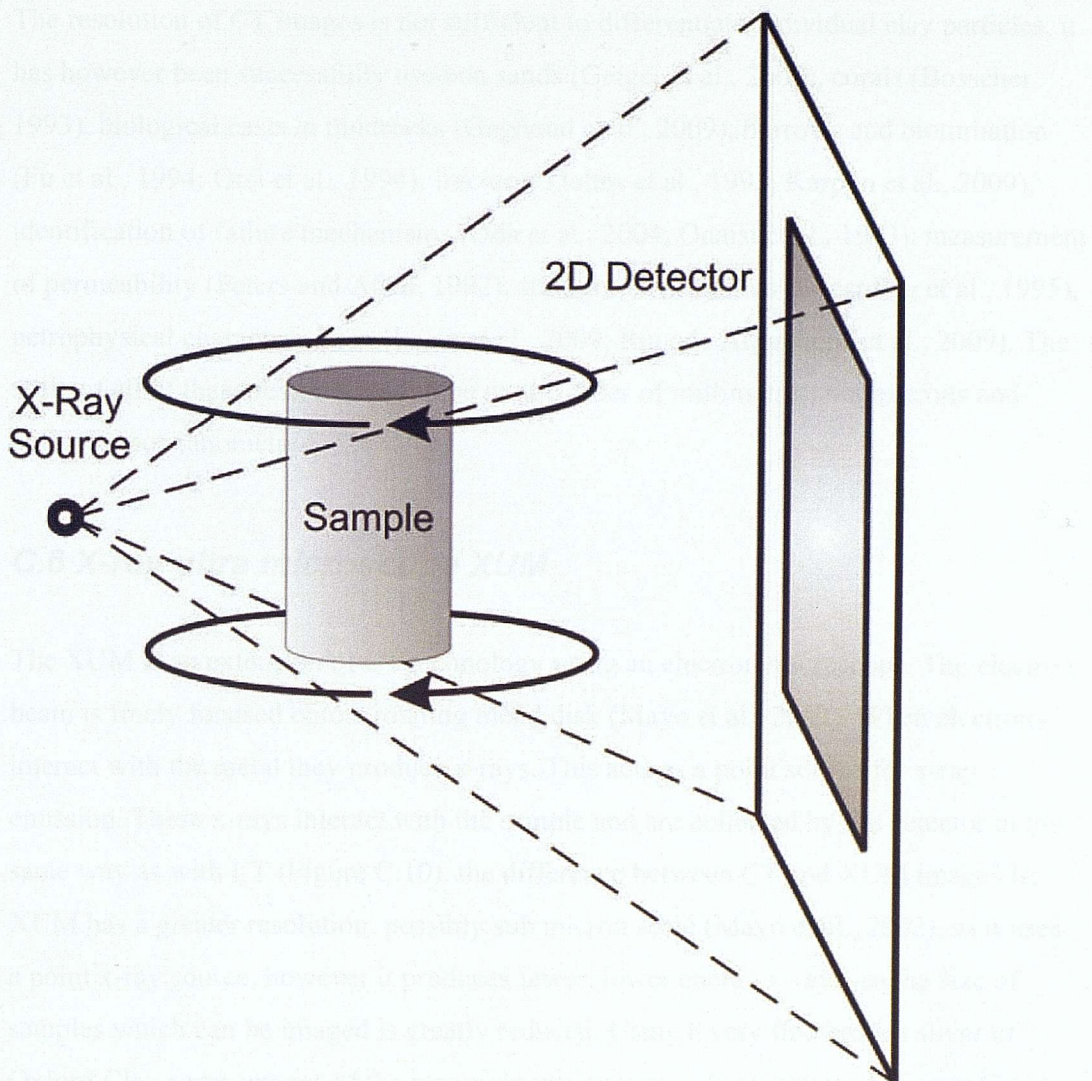


Figure C.9 Basic diagram of a micro CT scanner

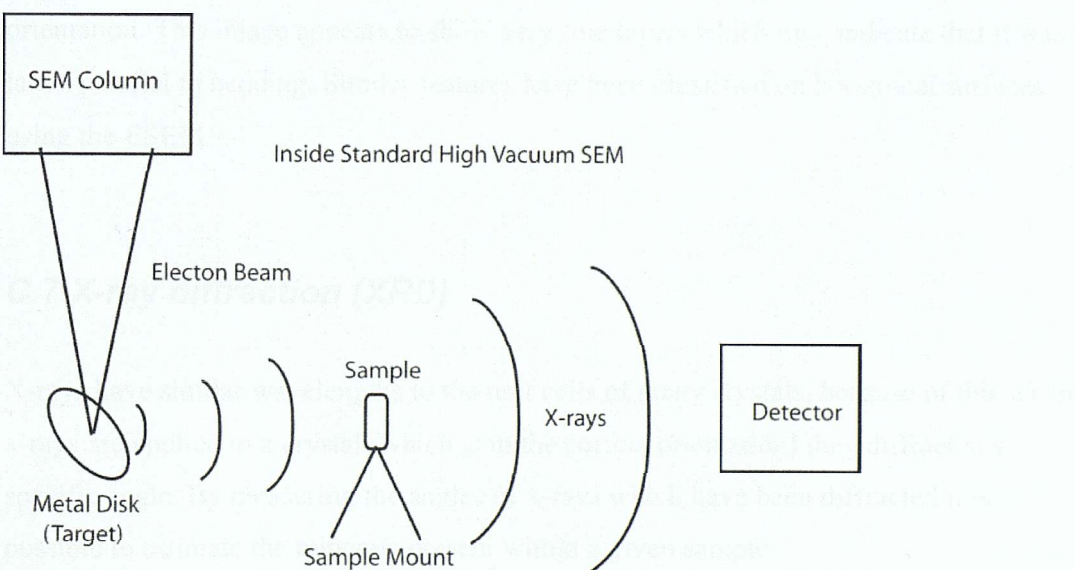


Figure C.10 Basic diagram of the x-ray ultra microscope

The resolution of CT images is not sufficient to differentiate individual clay particles. it has however been successfully used on sands (Geiger et al., 2009), corals (Bosscher, 1993), biological casts in mudrocks (Gagnoud et al., 2009), burrows and bioturbation (Fu et al., 1994; Orsi et al., 1994), fractures (Johns et al., 1993; Karpyn et al., 2009), identification of failure mechanisms (Oda et al., 2004; Onaisi et al., 1993), measurement of permeability (Peters and Afzal, 1992), stratigraphic rhythms (Boespflug et al., 1995), petrophysical characterisation (Ilovea et al., 2009; Ruiz de Argandoña et al., 2009). The scale of all of these features tend to be on the order of millimetres, not microns and defiantly not nanometres.

C.6 X-ray ultra microscope XUM

The XUM is an extension of CT technology using an electron microscope. The electron beam is finely focused onto a rotating metal disk (Mayo et al., 2003). When electrons interact with the metal they produce x-rays. This acts as a point source for x-ray emission. These x-rays interact with the sample and are collected by the detector in the same way as with CT (Figure C.10). the difference between CT and XUM images is; XUM has a greater resolution, possibly sub micron scale (Mayo et al., 2002), as it uses a point x-ray source, however it produces fewer, lower energy x-rays, so the size of samples which can be imaged is greatly reduced. Using a very fine broken sliver of Oxford Clay some images of the materials structure have been obtained (Figure C.11). The size of sample used was so small that is was not possible to keep track of its orientation. This image appears to show very fine layers which may indicate that it was taken parallel to bedding. Similar features have been identified on horizontal surfaces using the ESEM

C.7 X-ray diffraction (XRD)

X-rays have similar wavelengths to the unit cells of many crystals, because of this when x-rays are applied to a crystal (which is in the correct orientation) they diffract at a specific angle. By measuring the angles of x-rays which have been diffracted it is possible to estimate the minerals present within a given sample.

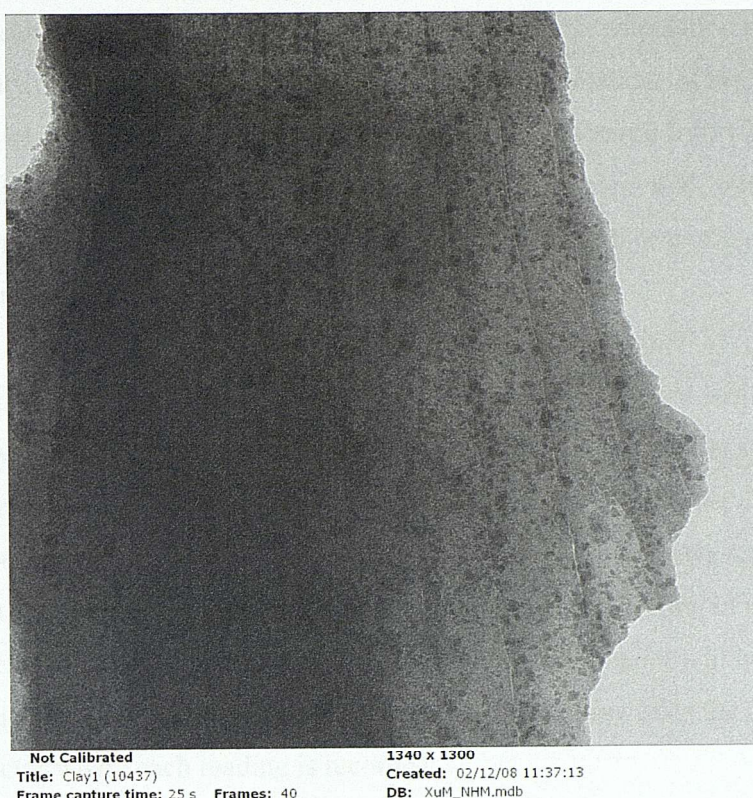


Figure C.11 Image of the Oxford Clay taken using the x-ray ultra microscope, image taken by Lauren Howard

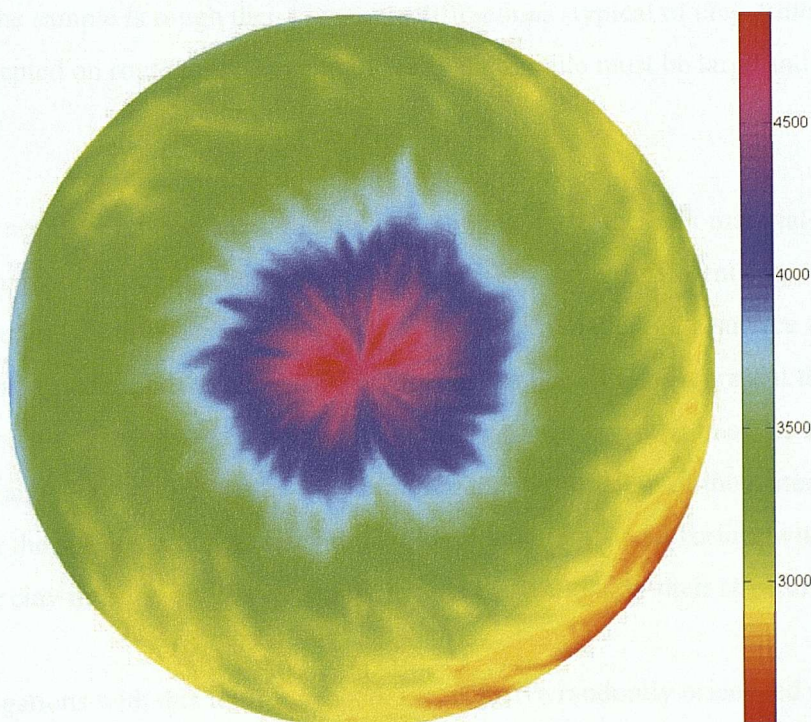


Figure C.12 XRG result showing count of x-rays in each orientation (upper hemispherical projection) background noise ~3000 counts, central peak of ~5000 counts. The slight asymmetry is due to the sample not being perfectly flat. Analysis carried out by Richard Sweeney. Results plotted in Matlab.

Given the assumption that the number of crystals in a specific orientation is proportional to the intensity of diffracted x-rays, an estimate of bulk orientations of minerals in the material can be made. This process was attempted in the 1960s (Gillott, 1970) and again more recently (Sachan and Penumadu, 2007). The major problem with x-ray diffraction results is extracting meaningful information about the specific fabric of the material from a bulk measurement.

The diffractometer fired a beam of x-rays onto the sample. This beam will hit an area of the sample which is proportional to the angle of the beam to the material surface. The beam will penetrate into the sample by an amount which is related to the density of the material tested (generally on the order of a few hundred microns, However this may be much less in dense samples such as those containing a high proportion of metal). If an x-ray interacts with a crystal structure in the right orientation then it will be diffracted at an angle which is specific for every material. This diffracted ray exits the material and hits the detector where each reading is recorded.

If the angle of incidence between the beam and the sample is too shallow, then x-rays will overshoot the sample causing a very high reading in the low angle end of the spectrum. If the sample is rough then low angle diffractions (typical of clay minerals) may be intercepted on route to the detector. Ideally the sample must be large and have a flat surface.

Mudrocks do not have flat surfaces which are representative of the bulk materials. They have joints, many of which are nearly flat. However joints have a different structure to that of the interior of the mudrock. The only way to create a flat enough surface for XRD is to sand a surface of the material. This sort of surface will not represent the structure of the material, however, it makes it possible for the orientation of the surface to be defined and as x-rays penetrate into the material a perturbation of the materials structure near the surface probably has little effect. In order to sand a surface without smearing, the clay must be dry. Drying clays is well known to alter their structure.

Initial investigations with this technique were inconclusive randomly orientated powder samples producing more variation than the orientated material. However further work with an X-ray goniometer provided a much better idea of the true preferred orientation of the material.

C.8 X-ray goniometry (XRG)

XRG is essentially an automated version of the above XRD method (Ho et al., 2001). The XRD angle of interest (mineral peak, *e.g.*, illite) is selected prior to the test. The goniometer measures the number of x-rays returned at that angle to the incidence beam. This measurement is repeated across short time intervals as the sample is rotated and inclined. The relative x-ray response in each orientation is related to the number of crystals arranged in that alignment (Van Der Pluijm B. A., 1994). This method has previously been used to measure the preferred orientation of clay minerals (Curtis et al., 1980). The Oxford Clay showed a weak preferred orientation using XRG (Figure C.12). That is weak compared to all crystals in perfect alignment. This is due to the relatively high diffraction background compared to the height of the illite peak.

C.9 The history of the electron microscope

In 1897 the electron (cathode ray) was observed by J.J. Thomson (Thomson, 1897). In his paper he expresses surprise at finding a ray that follows a curved path within a magnetic field. The ability of magnetic fields to alter the course of electrons was later used to develop a way of focusing a beam of electrons, in effect a lens (1926-1931) (Haguenau et al., 2003). This was put to practical use with the development of the first electron microscope by Ernst Ruska in 1931 (Freundlich, 1963; Ruska, 1986). This instrument had a maximum magnification of $\times 17.4$. However, the instrument developed quickly. Within two years he had developed a microscope with a maximum magnification of $\times 12000$. The first scanning electron microscope was reported in 1938 in the laboratory of Manfred von Ardenne (Mulvey, 1997).

His work on electron microscopy was unfortunately cut short as his laboratory and all of its microscopes were bombed in 1944 by the Royal Air Force (RAF). The facility was rebuilt the next year after the end of the war, unfortunately the work of the institute was turned away from electron microscopy and towards the production of uranium 235 isotopes (due to new interest in nuclear weapons after their use during World War 2).

The main problem with transmission electron microscopy is the creation of samples thin enough to be electron transparent. The development of the ultra-microtome between 1948 and 1953 made the creation of very thin sections of material possible. This was

greatly improved by the development of embedding media 1956-1960 (Haguenau et al., 2003). Even with these improvements some samples still could not be sliced finely enough for the transmission electron microscope (TEM) to image. The SEM had the advantage that it could be used on a wider range of materials and with much less preparation being required. In 1960 Charles Oatley and his PhD students work came to full fruition, the Cambridge Instrument Company launches the first commercial SEM (Breton et al., 2004). This SEM greatly outstripped its sales expectation, and led to further commercial ventures. More details of the SEM are presented in the body of the thesis (Section 9.1)

C.10 Transmission electron microscope (TEM)

The Transmission electron microscope fires electrons at a much greater voltage than the scanning electron microscope. These electrons have enough energy to traverse a short distance through an ultra thin slice of a material (maximum 10 μm normally much smaller). The interaction of the electrons produces an image of the internal structure of this thin slice. To construct a full 3D representation of the internal structure of the material the thin slice must be rotated and re images several times. In order to create such tin slices of material resin impregnation is normally required. One resin regularly used to study mudrocks maintains the water content of smectite, preventing its collapse (Kim et al., 1995). The TEM was not used due to a reluctance to use a resin due to the possible disruption the resin might have on the mudrock structure. Also the very thin nature of the slices makes the study of the particle scale structure of materials difficult.

C.11 Focused ion beam (FIB)

The focused ion beam microscope used in this work is the Carl Zeiss XB1540 “Cross-Beam” apparatus at The London Centre for Nanotechnology. This device has a focused ion beam for cutting and a electron beam for imaging. The focused ion beam fires a beam of high energy gallium ions at a small area of the sample (Volkert and Minor, 2007). This beam cuts into the sample; if the beam is scanned in a line across the sample then a line of material will be removed. This allows a section of the mudrock to be imaged with the electron beam. The focused ion beam can then be used to remove another line of material.



Figure C.13 Focussed ion beam image of the Oxford Clay, showing clay particles with clay mineral cement between particles.

By building up a series of images a full block of material can be imaged. This method can be easily used to image up to 10 μ m length of section to a depth of 5 μ m for example. This is a good scale for observing particle-particle contacts and cement within the sample. However, as this method looks at such a small area, many such pits would be required to quantify the variability of the material.

This technique is relatively new compared to most of the techniques presented in this appendix. It has however been applied to both granular geomaterials (Holzer and Münch, 2009; Uchic et al., 2007), and mudrocks (Desbois et al., 2008). Although destructive and expensive this method provides high resolution 3D data which cannot be obtained through CT scanning. The back surface of an experimental pit in the Oxford Clay showed individual clay mineral crystals and a clay mineral cement between the crystals (Figure C.13)

APPENDIX D

INTRODUCTION TO THE ELECTRONIC APPENDIX

The electronic appendix DVD contains both the original data used for this thesis, in the form of electron microscope image files in tif format and the processed data sheets for both the two dimensional and three dimensional analyses as files in pdf format. Each image has been used to generate a single page two dimensional analysis sheet, and each image pair has been used to generate a two page three dimensional analysis sheet.

The file structure of the DVD is displayed in this appendix. The tables show the division of the DVD into two sections: images (Table D-1), and processed data files (Table D-2).

The first page of each of the processed data files is the title page. This page contains details relating to the sampling site, the date shown is that of imaging. The remainder of each file is divided up into several sections. The first is a summary of the two dimensional data, the remaining sections divide the data up by the magnification used for imaging.

The summary section contains a series of rose diagrams which show the preferred orientation of all of the particles in all of the images at each scale (upper and lower quartile given separately). Within each of the other sections there are two distinct types of data sheet, two dimensional and three dimensional analyses.

The two dimensional sheets each consist of a single page (Figure D.1). The page shows from top to bottom: the original SEM image and the image after contrast enhancement, binary images created using the upper and lower quartile thresholds, ellipse models of the particles in the binary images, rose diagrams of particle orientation, and a data bar at the base of the image.

The three dimensional data are divided into two sheets. The first (Figure D.2) is the three dimensional analysis of the materials surface. The second (Figure D.3) contains

data related to the roughness of the imaged surfaces. The images on the first page from top to bottom are: the original two SEM images taken from two different orientations, an anaglyph and 3D elevation model of the surface, height map and gradient map, and aspect map and cumulative frequency plot of gradient and direction. The second page of the three dimensional data sheet shows the original SEM image. The line across the image is the line of the profile below the image. A table of roughness measurements for the line and for the surface as a whole is presented below the profile (Table D-3). All measurements are taken using AxioVision (Carl Zeiss, 2008).

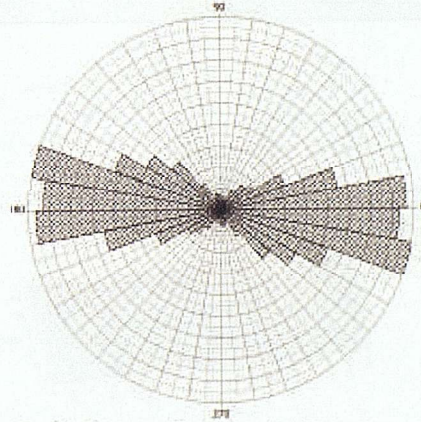
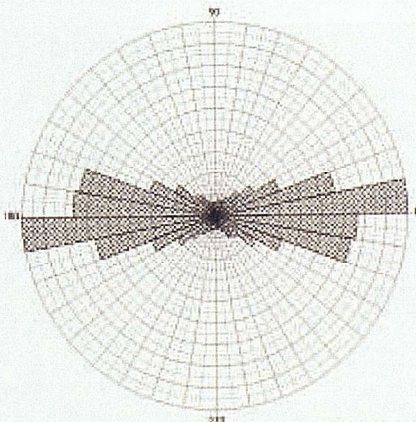
Folder Level 1	Folder Level 2	Folder Level 3	Folder Level 4	Folder Level 5
A-SEM images	Gault Clay	08-10-08	Block Sample Vertical	97 tif images
		09-10-08	Block Sample Vertical	76 tif images
		11-11-09	HC1 6.82m Horizontal	48 tif images
			HC1 6.82m Vertical	41 tif images
			HC2 1.72m Vertical	52 tif images
		12-11-09	HC2 1.72m Horizontal	55 tif images
		23-09-09	Block Sample Horizontal	47 tif images
			HC1 13.46-13.48m Horizontal	38 tif images
			HC1 13.46-13.48m Vertical	37 tif images
		25-03-09	Block Sample Vertical	39 tif images
		26-03-09	Block Sample Vertical	34 tif images
	Kimmeridge Clay	13-11-09	WBF2 8.54-8.59m Horizontal	75 tif images
			WBF2 8.54-8.59m Vertical	55 tif images
		21-09-09	WBF1 12.71m Horizontal 1	25 tif images
			WBF1 12.71m Horizontal 2	26 tif images
			WBF1 12.71m Vertical 1	24 tif images
			WBF1 12.71m Vertical 2	26 tif images
	Lias Clay	12-11-09	BH4 11.60-11.65m Horizontal	50 tif images
			BH4 11.60-11.65m Vertical	44 tif images
		22-09-09	BH4 16.46m Horizontal	42 tif images
			BH4 16.46m Vertical	77 tif images
	London Clay	16-03-10	30.45-31.95m Horizontal	51 tif images
			30.45-31.95m Vertical	57 tif images
		17-03-10	7.9-9.4m Horizontal	112 tif images
			7.9-9.4m Vertical	74 tif images
		18-03-10	33.45-34.95m Horizontal	146 tif images
			33.45-34.95m Vertical	114 tif images
	Oxford Clay	09-10-08	Block Sample Vertical E-W	45 tif images
			Block Sample Vertical N-S	50 tif images
		23-09-09	Block Sample Horizontal	41 tif images
		25-03-09	Block Sample Vertical	101 tif images

Table D-1 DVD folder structure part 1: images. Folder level two divides the data by mudrock. Folder level three divides the data by imaging date. Folder level four divides the data by imaging sample, and image orientation. All of the images are stored in the fifth folder level. The number of images in each folder is given.

Folder Level 1	Folder Level 2	Folder Level 3	Folder Level 4
B-Processed data	Gault Clay	Block Sample Horizontal	GaultClay 23-09-09A.pdf
		Block Sample Vertical	GaultClay 08-10-08.pdf
			GaultClay 09-10-08.pdf
			GaultClay 25-03-09.pdf
			GaultClay 26-03-09.pdf
		HC1 6.82m Horizontal	GaultClay 11-11-09A.pdf
		HC1 6.82m Vertical	GaultClay 11-11-09B.pdf
		HC1 13.46m Horizontal	GaultClay 23-09-09B.pdf
		HC1 13.46m Vertical	GaultClay 23-09-09C.pdf
		HC2 1.72m Horizontal	GaultClay 12-11-09.pdf
		HC2 1.72m Vertical	GaultClay 11-11-09C.pdf
	Kimmeridge Clay	WBF2 8.54m Horizontal	KimmeridgeClay 13-11-09A.pdf
		WBF2 8.54m Vertical	Kimmeridge Clay 13-11-09B.pdf
		WBF2 12.71m Horizontal	KimmeridgeClay 21-09-09A.pdf
			KimmeridgeClay 21-09-09B.pdf
		WBF2 12.71m Vertical	KimmeridgeClay 21-09-09C.pdf
	Lias Clay	BH4 11.60m Horizontal	LiasClay 12-11-09A.pdf
		BH4 11.60m Vertical	LiasClay 12-11-09B.pdf
		BH4 16.46m Horizontal	LiasClay 22-09-09A.pdf
		BH4 16.46m Vertical	LiasClay 22-09-09B.pdf
	London Clay	7.9-9.4m Horizontal	LondonClay 17-03-10A.pdf
		7.9-9.4m Vertical	LondonClay 17-03-10B.pdf
		30.45-31.95m Horizontal	LondonClay 16-03-10A.pdf
		30.45-31.95m Vertical	LondonClay 16-03-10B.pdf
		33.45-34.95m Horizontal	LondonClay 18-03-10A.pdf
	Oxford Clay	33.45-34.95m Vertical	LondonClay 18-03-10B.pdf
		Block Sample Horizontal	OxfordClay 23-09-09.pdf
		Block Sample Vertical	OxfordClay 25-03-09.pdf
		Block Sample Vertical E-W	OxfordClay 09-10-08A.pdf
		Block Sample Vertical N-S	OxfordClay 09-10-08B.pdf

Table D-2 DVD folder structure part 2: processed data. Folder level two divides the data by mudrock. Folder level three divides the data by sample depth, and image orientation. All of the pdf files are stored in the fourth folder level. The pdf file names contain the mudrock name and date of imaging. .

A black and white photograph of a rocky, uneven terrain, possibly a beach or a rocky shore. The foreground is dominated by dark, jagged rocks and pebbles. In the background, the ocean is visible, with white foam from breaking waves. The sky is dark and overcast.



H ₂ O	Wt%	Mg	Snd	Gravel	Tot		2000gms
8.0	96.0	0.0	4.0	0.0	0.0		G-SEM SE

EXAMPLE SHEET 2 PAGE 1

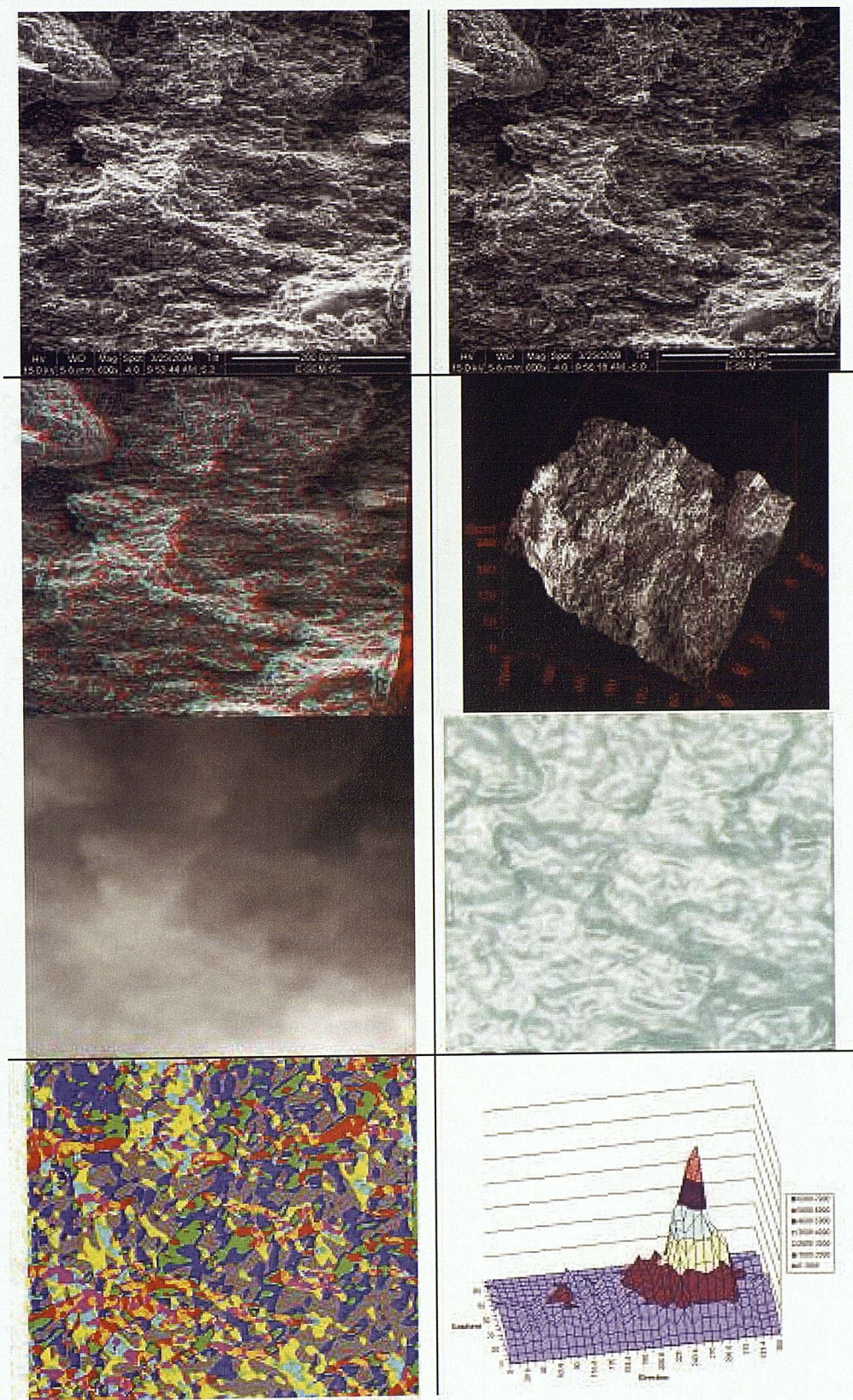


Figure D.2 Example 3D analysis sheet part 1. Anaglyph image, surface elevation model and height map created using AxioVision (Carl Zeiss, 2008).

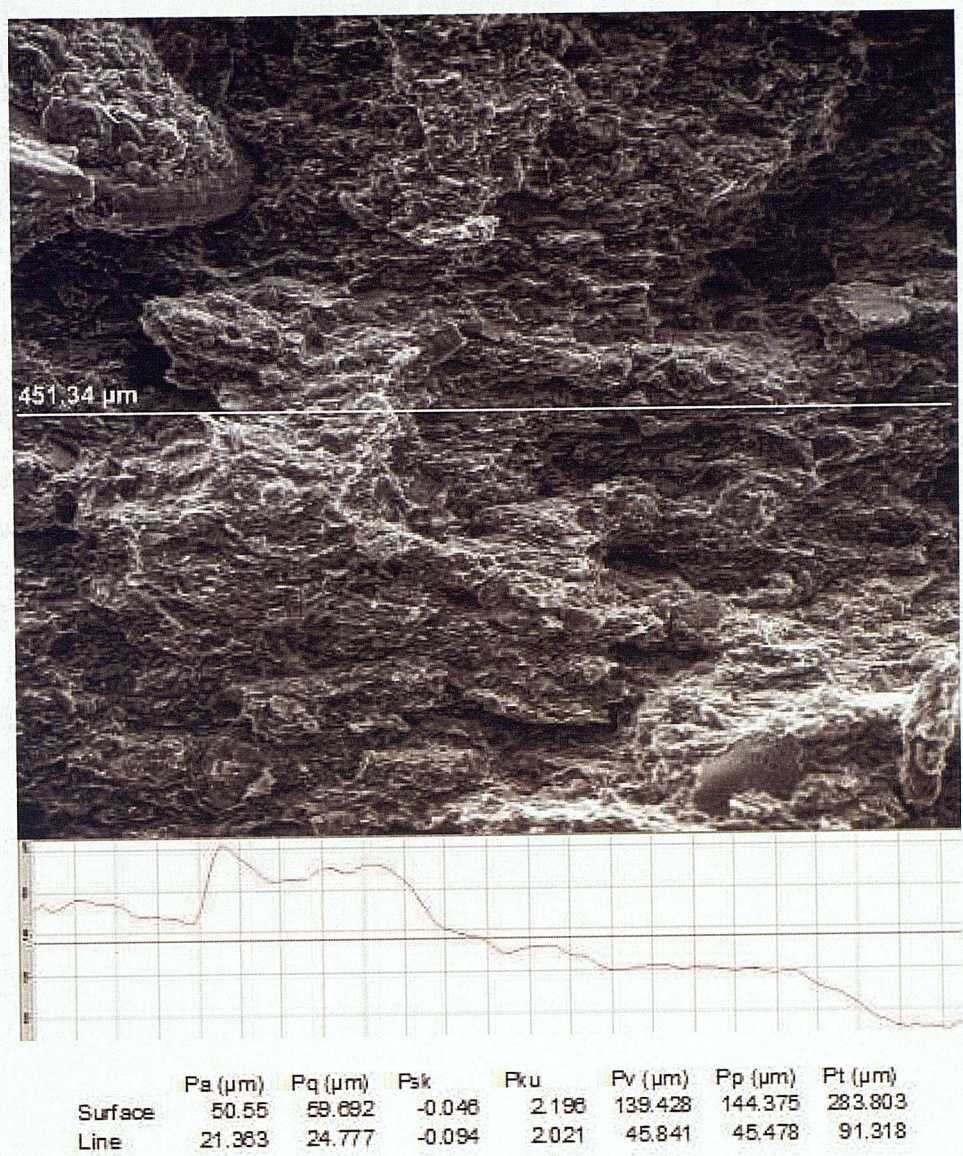


Figure D.3 Example 3D analysis sheet part 2, image, profile and roughness data created by AxioVision (Carl Zeiss, 2008)

Parameter	Summary	Equation
Pa	arithmetic mean of the profile ordinates	$Pa = \frac{1}{l} \int_0^l z(x) - \bar{z} dx$
Pq	quadratic mean of the profile ordinates	$Pq = \sqrt{\frac{1}{l} \int_0^l (z(x) - \bar{z})^2 dx}$
Psk	skewness of the profile	$Psk = \frac{1}{Pq^3} \left[\frac{1}{l} \int_0^l (z(x) - \bar{z})^3 dx \right]$
Pku	steepness of the profile	$Pku = \frac{1}{Pq^4} \left[\frac{1}{l} \int_0^l (z(x) - \bar{z})^4 dx \right]$
Pv	depth of the greatest profile valley	$Pv = \bar{z} - z_{\min}$
Pp	height of the greatest profile peak	$Pp = z_{\max} - \bar{z}$
Pt	total height of the profile	$Pt = z_{\max} - z_{\min}$

Table D-3 Roughness parameters as used in three dimensional analyses (Figure D.3). Data from AxioVision (Carl Zeiss, 2008).

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