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Fatigue in Gamma Titanium Aluminide

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I hereby declare that this thesis and the work reported herein was composed by and originated entirely from me. Information derived from the published and unpublished work of others has been acknowledged in the text and references are given in the list of sources.

Vitae

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Abstract

Despite the limited ductility of Ti-45Al-2Mn-2Nb-0.8 vol.% TiB₂-XD, it is relatively tough, but with poor fatigue strength and high crack growth rates. It is therefore imperative that cracks should not be allowed to initiate and propagate, making the fatigue crack growth threshold critically important. The dependence of this threshold on the initial defect size has been investigated in this alloy using a machined notch. When the machined notch was larger than 70 μm , the scale of the underlying microstructure, the threshold was found to be independent of the notch size. When the machined notch was smaller than the microstructural scale, crack initiation occurred away from the machined notch and from the underlying microstructure.

At 400°C in air the fatigue crack growth threshold for the machined starter notches was $4.03 \pm 0.11 \text{ MPa}\sqrt{\text{m}}$ for twelve specimens in trans-lamellar fracture mode through the randomly oriented colonies local to the notch. The initiation site on specimens that initiated away from the machined notch was determined, and this threshold could be calculated as approximately $4 \text{ MPa}\sqrt{\text{m}}$. Multiple notches were machined in a single specimen, and a notch chosen to measure the fatigue crack growth threshold that would initiate in inter-lamellar fracture mode. This measured threshold in inter-lamellar fracture mode at 400°C in air was $2.7 \text{ MPa}\sqrt{\text{m}}$.

The fatigue crack growth threshold is strongly microstructure and temperature dependent. This increased from $4.0 \text{ MPa}\sqrt{\text{m}}$ at 400°C to $7.1 \text{ MPa}\sqrt{\text{m}}$ at 750°C in air in trans-lamellar fracture mode. A step increase in the temperature at low values of the stress intensity factor range, ΔK , determined the inter-lamellar fracture mode fatigue crack growth threshold of $5.8 \text{ MPa}\sqrt{\text{m}}$ at 750°C in air. Step increasing the temperature to 750°C at larger values of ΔK caused the crack growth rate to initially increase, then quickly retard. This is due to the initial inter-lamellar fracture mode, until reaching a colony boundary and crack propagation being retarded by this boundary. Step decreasing the temperature back to 400°C caused crack growth rate retardation due to the larger plastic zone previously formed at the higher temperature.

The resulting fracture surface can be categorised into regions of fatigue and overload. There is also a visible roughness change attributed to the plastic zone size reaching the scale of the microstructure and therefore a change in cracking mechanism from trans-lamellar to mixed trans-, inter- and intra- lamellar. An oxide scale also formed giving a clear visible indication of temperature application.

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Chapter 1

Introduction

The civil jet engine market is dominated by three competitors: Rolls-Royce, General Electric and Pratt and Whitney. These suppliers compete on product price, service and fuel efficiency. Since fuel accounts for around a third of airline costs, small fuel efficiency improvements (and hence CO₂ emissions) are critical to the competitiveness of an engine programme. Efficiency in turn is determined both by the design of the turbomachinery, by airplane and rotating mass, and by engine temperatures, which drive the thermodynamics of the heat engine cycle. Therefore material behaviour and properties are a key determinant of gas turbine technology.

The jet engine in its purest form is a method of accelerating a gas stream to impart a thrust. The size of this thrust is dependent on both the mass of air pushed through the engine and the increase in velocity achieved. The jet turbine engine can provide thrust at low speeds due to the turbine driven compressor. Figure 1.1 shows a Rolls-Royce Trent XWB jet engine. The cycle of the turbine engine is to draw in air, from the left of Figure 1.1, with the fan. This continuous flow of air is then compressed by the spinning compressor blades. An injection of fuel is then mixed with the incoming pressurised air and ignited by the combustor. This is then forced through the turbine blades generating mechanical power, to be expelled out the rear end to generate the thrust. These rotating turbine blades use their energy gained to power the compressor and fan.

The temperature, pressure and axial velocity evolution through the engine are shown in Figure 1.2 [1]. Material choice throughout the engine is the lowest density material that fulfills

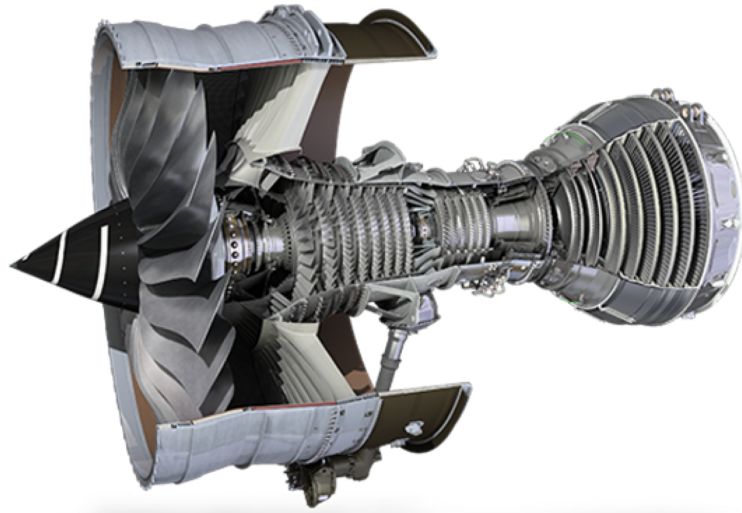


Figure 1.1: A cut-through animated image of a Rolls-Royce Trent XWB jet engine. The air is sucked in through the fan on the left, through the compressor, then mixed with fuel and ignited in the combustor. The air is expelled through the turbine blades generating thrust. Image courtesy of Rolls-Royce plc..

the properties required for the specific component, Figure 1.3. The temperature requirements are initially low through the fan and compressor, so this is predominately made of titanium. As the pressure increases, there is a transition from titanium to nickel superalloys. The fuel ignition in the combustor causes a sharp temperature increase, requiring the continued use of nickel superalloys.

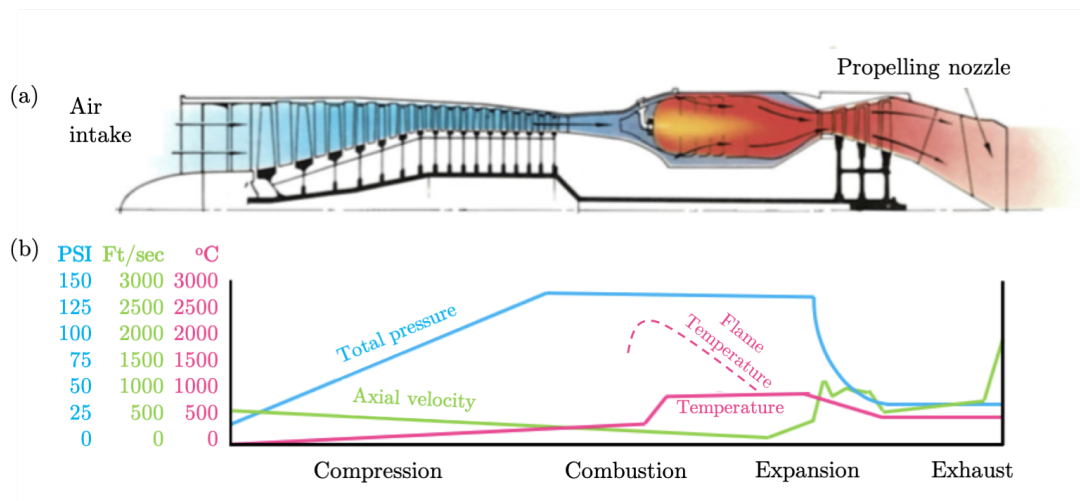


Figure 1.2: Temperature, axial velocity and pressure through the various components in a typical gas turbine engine. These are shown by (a) colour depicting change in temperature through the various components and (b) the temperature, axial velocity and pressure at that position in the engine, in relation to the above engine schematic [1].

Titanium and its alloys currently have a wide range of applications. Titanium alloys are commonly used for two reasons: their corrosion resistance and their high fatigue-allowable specific strength, especially at elevated temperatures [2]. Their good strength and creep properties make them attractive at temperatures up to 650°C [3]. Alloying titanium with aluminium improves the resistance to oxidation and resistance to creep, whilst also reducing the overall density. This enables titanium alloys to compete with common high temperature steels and nickel superalloys [4]. Titanium alloys make up approximately 33% of the weight in the modern gas turbine engine in parts such as fan blades and compressor discs and blades, with other materials such as nickel superalloys and steels comprising the remainder.

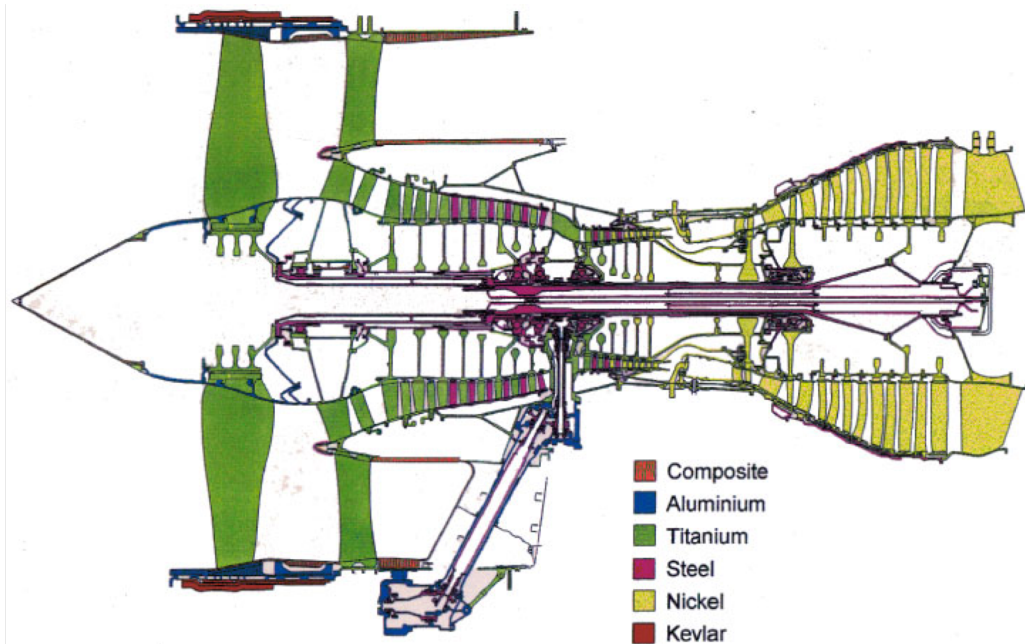


Figure 1.3: Materials chosen for the components through a conventional gas turbine engine [1].

The LP (low pressure) turbine blades towards the rear of the engine are required to operate at regular temperatures of 400°C, up to a maximum of 750°C during take off or landing, Figure 1.2. Titanium aluminide is being investigated due to its high strength to weight ratio and its corrosion resistance [5]. Gamma titanium aluminide, γ -(TiAl), has the highest melting point of the titanium aluminides [4], and was announced by General Electric in 2006 to be used in their GENx-1B (General Electric Next-generation) engine in the LP turbine blades [6; 7]. It is also being developed as a potential material for Rolls-Royce LP turbine blades in future engine architectures.

Should the material properties of a new part of an engine be satisfied, it is not only the prospective life of this part that is a cause for investigation. Elongation of the life of a part using various techniques could also be explored.

1.1 Motivation

In recent years, increased use of aviation in the private and public sectors has fuelled research into developing new alloys in gas turbine engines. Currently about 10% of all UK CO₂ emissions are from aviation (37.8 MtCO₂ / 364.1 MtCO₂, 2018), and globally around 2% (895 MtCO₂ / 42 000 MtCO₂), so improving the efficiency in terms of fuel consumption and emissions is incredibly important for long term sustainability [8–10].

γ -(TiAl) based alloys are intended for use in the low pressure turbine blades of the modern gas turbine engine, replacing nickel superalloys. This alloy will give a decrease in blade mass and increase efficiency. The main concern with the introduction of this material is fatigue: crack nucleation and crack propagation.

1.2 Thesis structure

This thesis first surveys the relevant literature covering a overview of the alloy, its processing route, deformation, fatigue fundamentals, use of the alloy and similar alloys in industry and comparing fatigue performance of this alloy with similar alloys. Two methodology chapters, Chapters 3 and 4, describe the microscopy techniques used and crack propagation techniques used and developed throughout. The results are then split into four chapters, Chapters 5 to 8: Effect of Notch Size, Characterising the Fracture Surface, Effect of Colony Orientation on Threshold and Effect of Temperature Excursions on Crack Growth. An overview of the first two results chapters is given by the conference proceedings at Fatigue 2018 Congress and World Titanium 2019. The fourth results chapter is intended to become a paper.

1.3 Statement of originality

The work presented here has been carried out at Imperial College London under the supervision of Professor David Dye and Professor Trevor Lindley between October 2015 and September 2019. The thesis and associated research are my own work and if other sources have been used they are indicated and acknowledged by referencing.

1.4 Copyright

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Chapter 2

Literature Review

2.1 γ -(TiAl) and jet engine technology

γ -(TiAl) based alloys have a unique blend of properties that have provoked prolonged research, dating back to 1953 [11]. These properties include a high melting point, low density, high specific strength and modulus, good resistance against oxidation and corrosion, and good creep resistance at intermediate temperatures, 500 to 700°C [2].

Due to the material being an intermetallic, it has a relatively large Burgers vector and therefore is strong and has diffusion and creep resistance, but is not especially ductile. This material, being an aluminide, has a low density and is oxidation resistant. γ -(TiAl) has a density of around 4 g cm⁻³, half that of a nickel based superalloy around 9 g cm⁻³. These combined factors give rise to an attractive high temperature material, if protected from impact and defects. These properties, on a density corrected basis, can be found to be superior to that of superalloys, and gives cause for this alloy to be attractive for use in jet engines [2; 3]. However, only recently have this class of alloys begun to see service in civil jet engines, owing to the challenge involved in engineering with brittle materials in safety-critical structural applications.

Figure 2.1 compares the density corrected strength of various materials including γ -(TiAl) based alloys with operating temperature. It can be seen to perform favourably when compared to superalloys, which have a lower strength per unit mass when operating at a wide range of temperatures, from 250 to 1000°C [12]. γ -(TiAl) based alloys can also be seen to have a higher

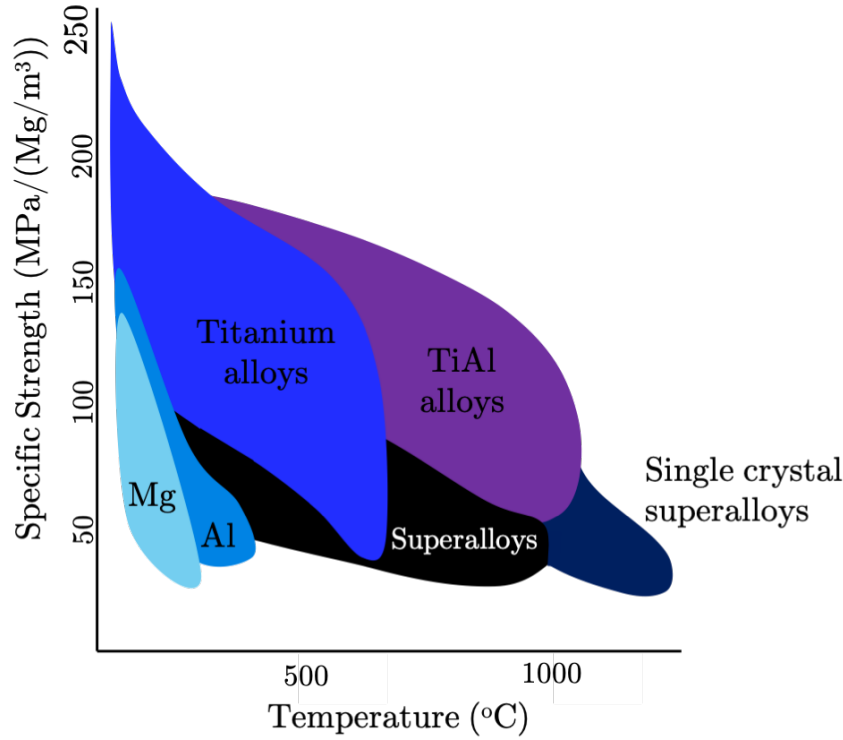


Figure 2.1: Specific strength of γ -(TiAl) in relation to other aerospace materials. On a density corrected basis, γ -(TiAl) shows a superior strength at intermediate temperatures. Figure adapted from [12].

specific strength than titanium alloys and retain this up to higher operating temperatures of around 1000°C as opposed to approximately 750°C for titanium alloys [2].

Table 2.1 summarises the relative properties of titanium based alloys γ -(TiAl), α_2 -(Ti₃Al) and nickel superalloys [13]. γ -(TiAl) based alloys have the highest melting point of all the titanium aluminides, yet also has the lowest ductility with 1 - 4 % room temperature ductility, as opposed to 10 - 25 % for titanium based alloys, and 3 - 25 % for nickel based alloys, Table 2.1. γ -(TiAl) is used in aerospace applications due to its resistance to high temperatures, *i.e.* high melting temperature and oxidation resistance. It suffers from oxidation at temperatures above 800°C, whereas titanium and α_2 based alloys suffer above 650°C. γ -(TiAl) is also used due to its lower density and therefore the weight reduction in comparison to nickel alloys. γ -(TiAl) based alloys have a room temperature fracture toughness 12 - 35 MPa $\sqrt{m}$, in comparison to 12 - 50 MPa $\sqrt{m}$ for titanium alloys. Traditional tough metallic alloys such as steels are often lifed presupposing the presence of defects, either arising during processing and manufacturing such as machining marks or inclusions, or those arising in service due to *e.g.* hard particles

Property	Ti-based	Ti ₃ Al-based	TiAl-based	Ni-based
Structure	HCP/BCC	D0 ₁₉ (HCP)	L1 ₀ (FCT)	FCC/L1 ₂
Density (g cm ⁻³)	4.5	4.1 - 4.7	3.7 - 3.9	7.9 - 8.5
Modulus (GPa)	95 - 115	110 - 145	160 - 180	206
Yield Strength (MPa)	380 - 1150	700 - 990	350 - 600	800 - 1200
Tensile Strength (MPa)	480 - 1200	800 - 1140	440 - 700	1250 - 1450
RT Ductility (%)	10 - 25	2 - 10	1 - 4	3 - 25
RT Fracture Toughness (MPa√m)	12 - 50	13 - 30	12 - 35	30 - 100
Creep Limit (°C)	750	750	750 ^a - 950 ^b	800 ^a - 1090 ^e
Oxidation (°C)	650	650	800 ^c - 950 ^d	870 ^c - 1090 ^d
Melting Temperature (°C)	~ 1690	~ 1650	~ 1480	~ 1300

Table 2.1: Table comparing properties of titanium based alloys, α_2 -(Ti₃Al) based alloys, γ -(TiAl) based alloys and nickel based alloys. Where notation *a* is duplex microstructure, *b* is fully lamellar, *c* is uncoated, *d* is coated or actively cooled and *e* is single crystal [13].

entrained in the gas stream. However, these approaches drastically limit the stresses that can be allowed in a brittle material, and lead to concerns around residual stresses (again, *e.g.* from machining). Therefore it has been difficult conceptually to introduce γ -(TiAl) into civil jet engines. This limitation in their ductility and toughness is in common with most intermetallics. An overview to the relative benefits of using γ -(TiAl) can be found in [12].

2.2 γ -(TiAl) phase and process metallurgy

Alloying leads to the formation of several intermetallic phases in γ -(TiAl) based alloys. These include, among others: α -(Ti), β -(Ti), α_2 -(Ti₃Al) and γ -(TiAl). α_2 -(Ti₃Al) and γ -(TiAl) have enough ductility to be used in engineering applications [2]. The titanium aluminium binary phase diagram has been in discussion for many years, with numerous suggestions, for example by Okamoto and Chen *et al.* [14; 15]. The uncertainty in the phase diagram has been thought to be due to the high sensitivity of titanium aluminide to chemical composition, non-metallic impurities, and phase equilibria imbalances due to slow phase transformations [16]. The binary phase diagram proposed in 2006 by Schuster and Palm [17] in Figure 2.2 is preferred, especially in the central section for aluminium concentrations of 40 - 55 at.% [4]. However, of these intermetallic phases, only alloys with the majority γ and small amounts of α_2 have emerged as possible structural materials. From the phase diagram, the atomic percentage of aluminium in

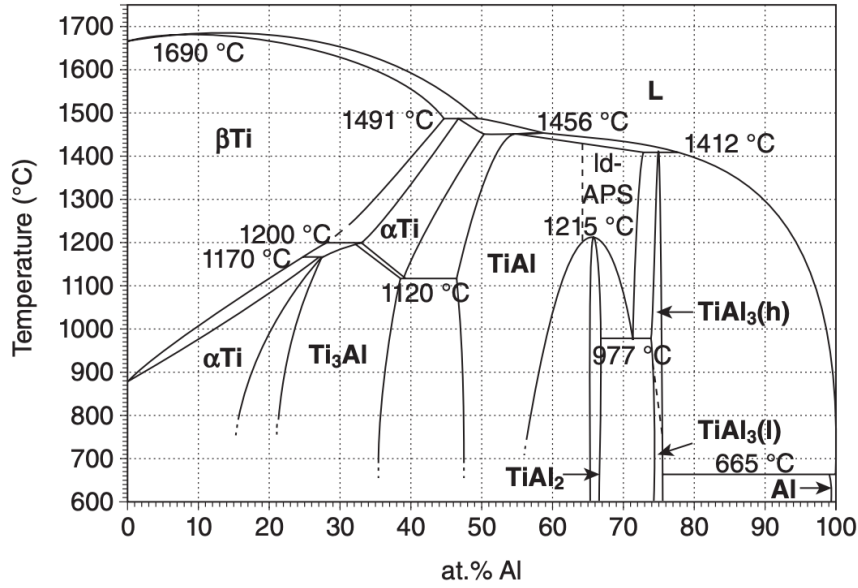


Figure 2.2: Binary titanium aluminium phase diagram as proposed by [17]. The region between the α_2 -(Ti_3Al) and γ -(TiAl) is the dual phase region under investigation.

this alloy can therefore range between 37 and 49 at.% to form the dual phase α_2 -(Ti_3Al) and γ -(TiAl).

α -(Ti) is the disordered hexagonally close-packed (HCP) microstructure, whereas α_2 -(Ti_3Al) is the ordered HCP microstructure, Figure 2.3(a), with lattice parameters of $a = 0.5782$ nm and $c = 0.4629$ nm [2], with Strukturbericht designation D0_{19} . β -(Ti) is the disordered body-centred cubic (BCC) microstructure, with Strukturbericht designation A2, with the ordered variant with Strukturbericht designation B2, Figure 2.3(c). γ -(TiAl) has a face centred tetragonal

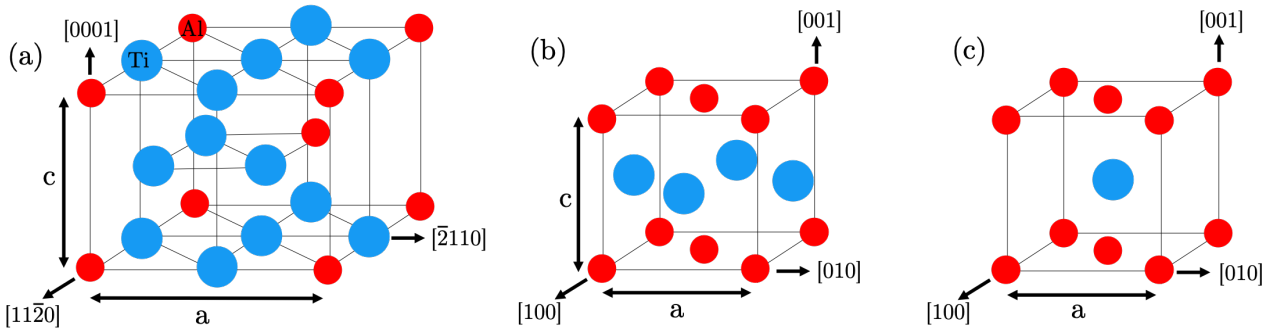


Figure 2.3: Crystal structures of phases in titanium aluminide alloys, where blue hard spheres are titanium and red hard spheres are aluminium. (a) Hexagonally close-packed α_2 -(Ti_3Al), Strukturbericht designation D0_{19} (N.B. not full α_2 -(Ti_3Al) cell), (b) face centred tetragonal γ -(TiAl), Strukturbericht designation L1_0 , (c) body-centred cubic β , Strukturbericht designation B2. Schematic adapted from [2].

(FCT) microstructure, Figure 2.3(b) [2], with Strukturbericht designation L1₀. It has lattice parameters differing marginally across the literature: $a = 0.3997$ nm, $c = 0.4062$ nm [18]; $a = 0.4000$ nm, $c = 0.4075$ nm [19], or $a = 0.4016$ nm, $c = 0.4068$ nm [20]. However, each of these lattice parameters shows the FCT microstructure being only marginally tetragonal, with lattice parameter c being 1.6%, 1.9% and 1.3% respectively greater than lattice parameter a in all of the literature.

With the addition of other alloying elements, a phase can be stable at an elevated or depressed temperature relative to the temperature at which it is usually stable, or stable at a different percentage ratio of the main alloying elements [4]. Known α stabilisers in titanium aluminide are oxygen, nitrogen and carbon [21]. Known β stabilisers are molybdenum, niobium and vanadium [5; 22]. Iron and manganese have also been seen to exhibit β stabilisation [4; 21]. γ -(TiAl) is sensitive to small changes in chemistry, especially when changing the aluminium concentration [23]. The relationship between phase and microstructure is closely linked to mechanical properties. Introducing molybdenum, niobium or chromium allows γ -(TiAl) to operate at higher temperatures and become less brittle. Tantalum has also been seen to improve high temperature strength [24]. Boride additions of 0.1 - 2.0 % can act as a grain refining agent and are used to stabilise the microstructure, especially at higher temperatures [25; 26]. Borides are introduced as TiB₂ into the microstructure and act to nucleate the prior- β grains [27].

Single phase γ -(TiAl) has limited ductility, causing fracture at room temperature. Despite the application of various alloying techniques, little improvement has been made. This has led to development of two phase titanium aluminide alloys using the α_2 -(Ti₃Al) and γ -(TiAl) phases [2]. In thermodynamic equilibrium the fraction of α_2 -(Ti₃Al) is controlled by the aluminium content and is usually in the range of 5 - 20 at.% [28; 29]. Despite α_2 -(Ti₃Al) actually being more brittle than the γ -(TiAl), it has a significant effect on the deformation behaviour and mechanical properties. α_2 -(Ti₃Al) has an increased solubility for impurities in comparison to γ -(TiAl), and therefore acts sacrificially to purify the γ -(TiAl) and improve its ability to deform plastically, even at lower temperatures [4]. Introduction of α_2 -(Ti₃Al) to γ -(TiAl) also causes the introduction of small amounts of the β phase, Figure 2.3(c) [30]. Alloying elements can be found to segregate differently into the γ -(TiAl) phase and α_2 -(Ti₃Al)

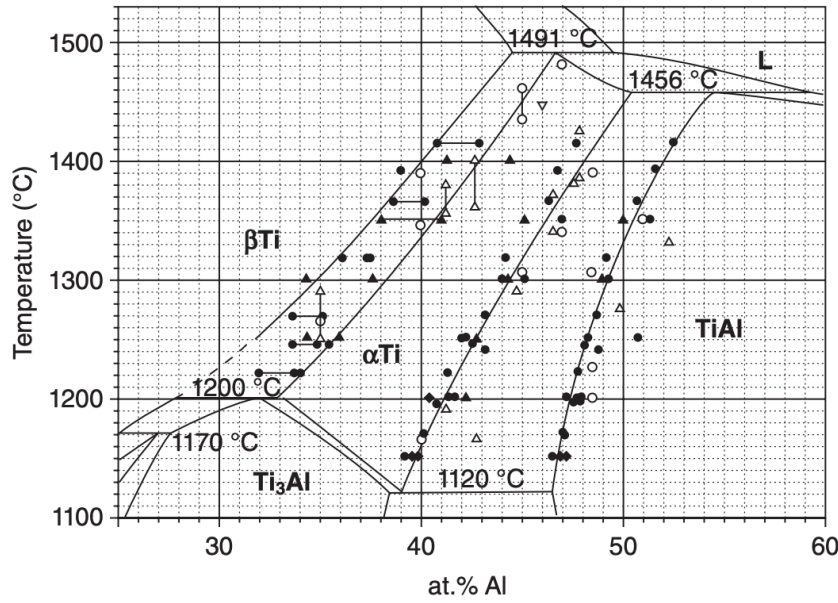


Figure 2.4: Binary titanium aluminide phase diagram from Figure 2.2, focussing on the central region, between temperatures of 1100 and 1520°C, and between 25 and 60 at.% aluminium [17].

phases; chromium, tantalum and molybdenum are enriched in the α_2 -(Ti₃Al) phase, manganese and niobium are evenly distributed in the two phases, and zirconium is predominantly in the γ -(TiAl) phase [31]. Oxygen was found to be more soluble in α_2 -(Ti₃Al) than γ -(TiAl) [32; 33]. O in γ -(TiAl) has a solubility of 300 at. ppm, whereas in α_2 -(Ti₃Al) the solubility ranges from 8000 - 22 000 at. ppm depending on the aluminium content. The authors speculate that this may be due to the α_2 -(Ti₃Al) phase having octahedral sites surrounded by six titanium atoms, which acts to prevent the formation of oxides [34].

The binary phase Ti-45Al-2Mn-2Nb-XD alloy is formed by following the phase diagram down with 45 at.% aluminium, Figure 2.4. β initially solidifies out of the liquid. This then solidifies through the small window to form α grains around the β grains, then becoming completely α . γ plates then grow into the α grains. As these γ platelets widen, the α structure transforms into α_2 . A schematic of the stages of this transformation is shown in Figure 2.5, to give the binary microstructure of platelets of γ and α_2 . Small changes in the aluminium content of γ -(TiAl) based alloys can cause large changes in the microstructure.

Single phase γ -(TiAl) exhibits limited ductility and therefore the worst crack growth properties, whereas according to current knowledge the most advantageous microstructure is that of coarse lamellae of the dual phase alloy [4; 24; 35].

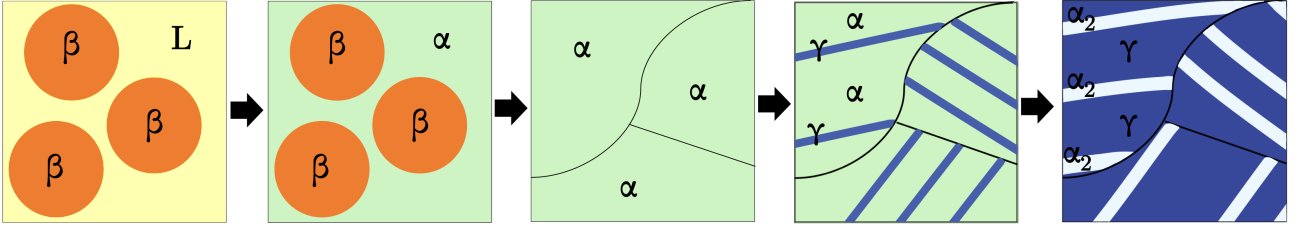


Figure 2.5: Schematic of phase transformation sequence of two-phase γ and α_2 from the liquid through to the final microstructure.

Over recent years many γ -(TiAl) based alloys have gone through alloy development programs, leading to a variation in the alloys produced. Each of these is a variant due to different processing routes and applications. The general composition of these alloys is:

$$\text{Ti-(42-49)Al-(0.1-10)X(at.\%)}$$

with X designating the elements: chromium, niobium, vanadium, manganese, tantalum, molybdenum, zirconium, tungsten, silicon, yttrium, or boron [13; 36–38].

2.3 Plasticity and crystallography of deformation

The deformation behaviour of titanium aluminide alloys suffers from the typical characteristics of other intermetallic compounds, such as low ductility and toughness. This is due to their large unit cells and hence dislocation Burgers vector lengths. There is also strong dependence of yield stress on temperature and orientation [39]. As γ -(TiAl) can be seen to be brittle, it therefore shows low plasticity when deformed. This behaviour can be related to the crystallographic defects, or dislocations, in the material. Brittle behaviour of a material is attributed to dislocations being too few or too immobile to accommodate the superimposed strain rate [4], and is seen to persist up to high temperatures [35]. The anisotropic nature of the microstructure can also affect the plastic deformation. The mechanisms at play within this microstructure include: slip and twinning systems, dislocation multiplication, twin nucleation, glide resistance and dislocation mobility [4].

The movement of many dislocations causes slip within a material, which is the micromechanism of plastic deformation. The easiest dislocations for a crystal structure to undergo are those with the shortest Burgers vectors on the closely packed plane of the crystal structure. This is

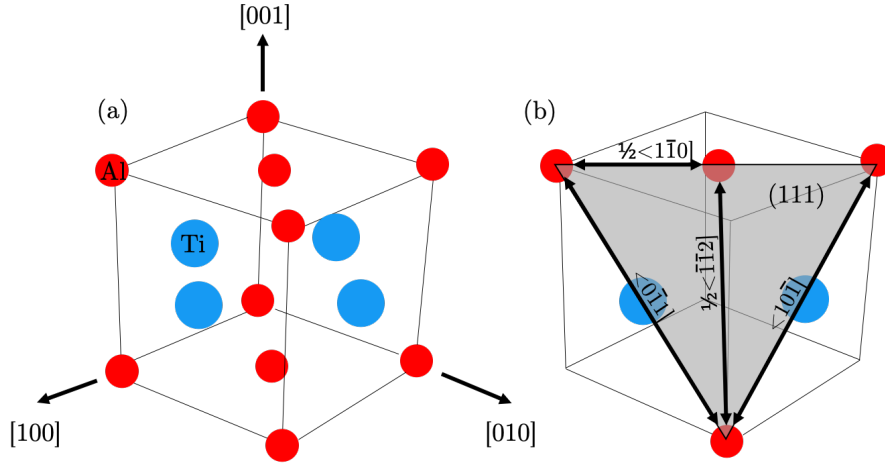


Figure 2.6: Available slip systems in γ -(TiAl). (a) Atomic arrangement of L1₀ structure of γ -(TiAl), showing one of the four possible octahedral $\{111\}$ planes. (b) Five Burgers vectors on the (111) plane. Image adapted from [2].

due to the strain energy per unit length being a function of the shear modulus and the square of the Burgers vector.

γ -(TiAl) has an FCC microstructure. However, due to γ -(TiAl) having lattice parameter a a maximum of 1.9% less than c , modified face centred cubic (FCC) notation is used [40]. This notation uses mixed parentheses $\langle uvw \rangle$ and $\{hkl\}$. This indicates that the last of the three indices in the brackets is fixed whereas the first two can have any permutation. Similar to FCC crystal structures, γ -(TiAl) prefers dislocations on $\{111\}$ planes. This is the close packed or relatively close packed plane. However, due to its tetragonal nature, together with the fact that the dislocation Burgers vector must be a lattice translation vector, imposes several restrictions on dislocation mechanisms. Figure 2.6(a) shows the crystal structure, and Figure 2.6(b) shows the possible Burgers vectors [2].

Due to a and c being marginally different, the close-packed $\langle 110 \rangle$ directions are crystallographically not equivalent. In this group of directions the shortest lattice translation vector is $\mathbf{b}_{\langle 110 \rangle} = \frac{1}{2}\langle 1\bar{1}0 \rangle$, on the $\{111\}$, $\{110\}$ and (001) planes. Due to this being the same as the Burgers vector for disordered FCC crystals this is often referred to as a unit or ordinary dislocation [41]. The displacement $\frac{1}{2}\langle 0\bar{1}1 \rangle$ includes a c component, and therefore is not a lattice translation vector in this structure. This would leave behind an interface with a packing error formed, an antiphase boundary, and therefore must be followed by a second displacement of the same magnitude and vector. This leaves the perfect dislocation in the c direction to have the

Burgers vector $\mathbf{b}_{\langle 011 \rangle} = \langle 0\bar{1}1 \rangle$, with two identical dislocations bounding an antiphase domain [42; 43]. Two partial dislocations making a perfect dislocation are often called a superpartial dislocation. Another possible superpartial dislocation in this structure is $\mathbf{b}_{\langle 112 \rangle} = 1/2 \langle 11\bar{2} \rangle$ with the superpartials $1/2 \langle 10\bar{1} \rangle$ and $1/2 \langle 01\bar{1} \rangle$, again bounding an antiphase domain [44].

So glide in γ -(TiAl) can occur on $\{111\}$ planes with unit dislocations $\mathbf{b}_{\langle 110 \rangle} = 1/2 \langle 1\bar{1}0 \rangle$, superdislocations $\mathbf{b}_{\langle 011 \rangle} = \langle 0\bar{1}1 \rangle$ with two identical dislocations $1/2 \langle 0\bar{1}1 \rangle$ and $\mathbf{b}_{\langle 112 \rangle} = 1/2 \langle 11\bar{2} \rangle$ with two different dislocations $1/2 \langle 10\bar{1} \rangle$ and $1/2 \langle 01\bar{1} \rangle$. c being greater than a , $a/2 \langle 1\bar{1}0 \rangle$ dislocations have a lower energy, but these cannot produce shear in the c -axis. This leads to the c -axis deformation being accommodated by $\langle 0\bar{1}1 \rangle$ superdislocation pairs.

Cross slip plays an important part in deformation in γ -(TiAl). This is the change in slip plane from one to another where the two planes share a common slip direction. Potential slip planes therefore must cross the close packed (111) plane of the FCT crystal structure. The unit dislocation may cross glide onto the $(\bar{1}\bar{1}1)$, (110) and (001) planes, Figure 2.7(a). The first of the two mentioned superpartial dislocations may cross slip from (111) onto the glide planes $(1\bar{1}\bar{1})$, (100) and (011), Figure 2.7(b). The choice of slip plane for these dislocations depends on temperature [45]. The second of the two mentioned superpartial dislocations can only glide on

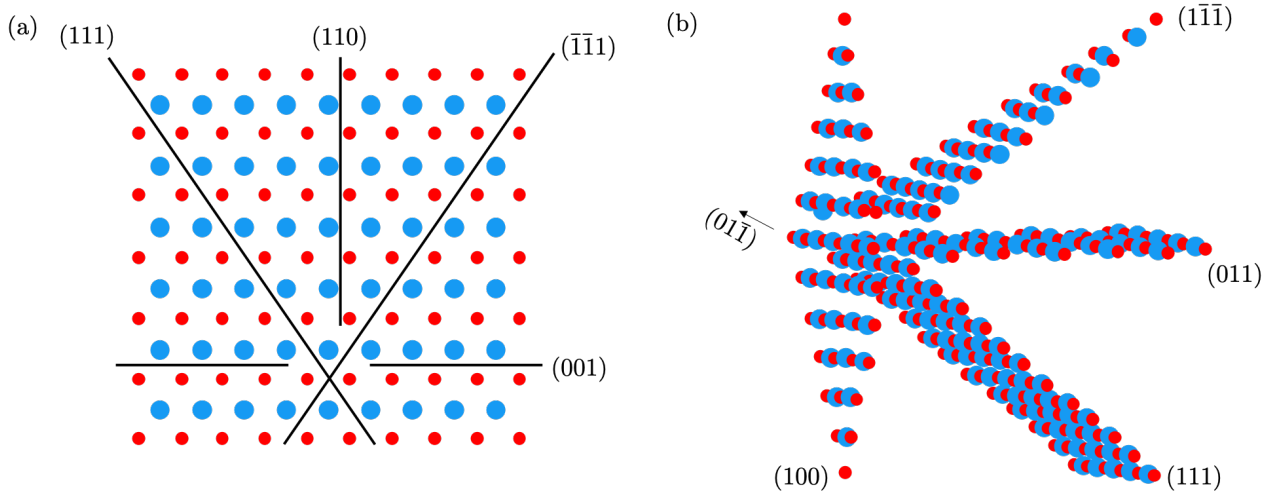


Figure 2.7: Potential cross slip systems in γ -(TiAl) for dislocations gliding on the primary (111) plane where the blue hard spheres are titanium and the red hard spheres are aluminium. (a) A unit dislocation with Burgers vector $\mathbf{b} = \langle 110 \rangle$ may cross slip on planes (110), $(1\bar{1}\bar{1})$ and (001), indicated by the lines. The lattice is drawn so that the relevant Burgers vector is perpendicular to the plane of the schematic. (b) Possible cross slip planes of $\langle 011 \rangle$ superdislocation. Schematic adapted from [2].

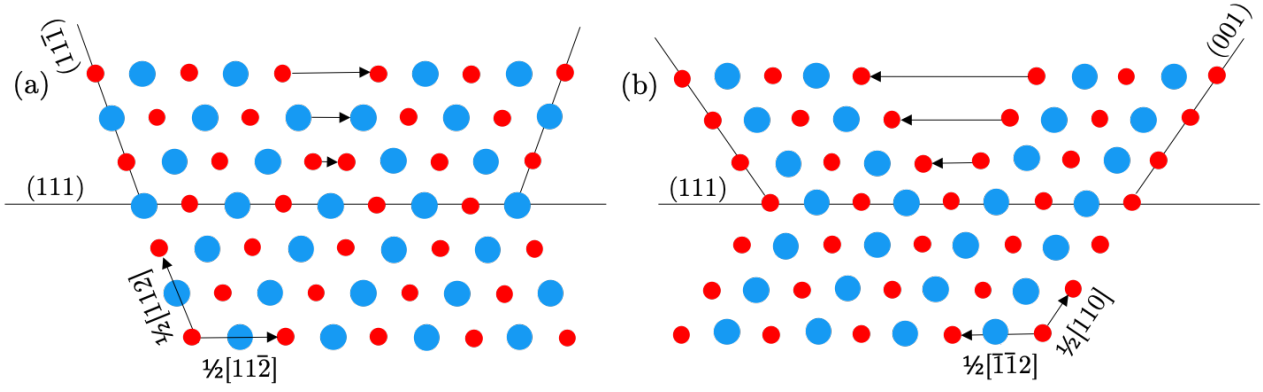


Figure 2.8: Structural model of mechanical twinning in γ -(TiAl), where blue hard spheres are titanium, and red hard spheres are aluminium. (a) Primary twinning defined by elements (111) , $(11\bar{1})$, $\frac{1}{2}[11\bar{2}]$ and $\frac{1}{2}[112]$. (b) Complementary twinning with (111) , (001) , $\frac{1}{2}[\bar{1}\bar{1}2]$, $\frac{1}{2}[110]$. Schematic adapted from [2].

the low index plane $\{1\bar{1}0\}$.

Deformation in γ -(TiAl) can also occur by mechanical twinning. This is different to dislocation glide and has implications for plasticity. Like slip, mechanical twinning leads to permanent shape change of a crystal with little change to the volume. However, mechanical twinning shears the structure, leaving a mirror plane at the interface. Often, twin propagation is dislocation mediated. There is a primary twinning and reverse or complementary twinning mode which can be seen in Figure 2.8. The complementary twinning mode has a shear in the reverse direction, approximately double that of primary twinning [46]. A narrow twin that propagated at room temperature in Ti-48.5Al-0.37C at.% can be seen in Figure 2.9. Due to the six rotation variants of the γ phase when grown out from the parent α_2 phase, the $\gamma\|\gamma$ boundaries can be pseudo-twins with a 60° misorientation, 120° rotational faults or true twins with 180° rotation [2], ordered from the highest to lowest interfacial energy variant respectively.

Figure 2.10 [48] summarises the potential slip and twinning systems in the γ -(TiAl) phase. Figure 2.10(a) shows the dislocation of Burgers vectors $\frac{1}{2}\langle 110 \rangle$, $\frac{1}{2}\langle 11\bar{2} \rangle$ and $\langle 011 \rangle$ glide on $\{111\}$. Plastic deformation of γ -(TiAl) can also be achieved by the mechanical $\frac{1}{6}\langle 11\bar{2} \rangle \{111\}$ twin, shown in Figure 2.10(b). Increased slip can be seen at elevated temperatures, therefore also occurring in the $\langle 100 \rangle$ directions [4; 28]. Twin nucleation is thought to occur at the $\gamma-\gamma_T$ phase interface [4]. The mobility of dislocations in γ -(TiAl) is determined by the number of lattice defects within the material.

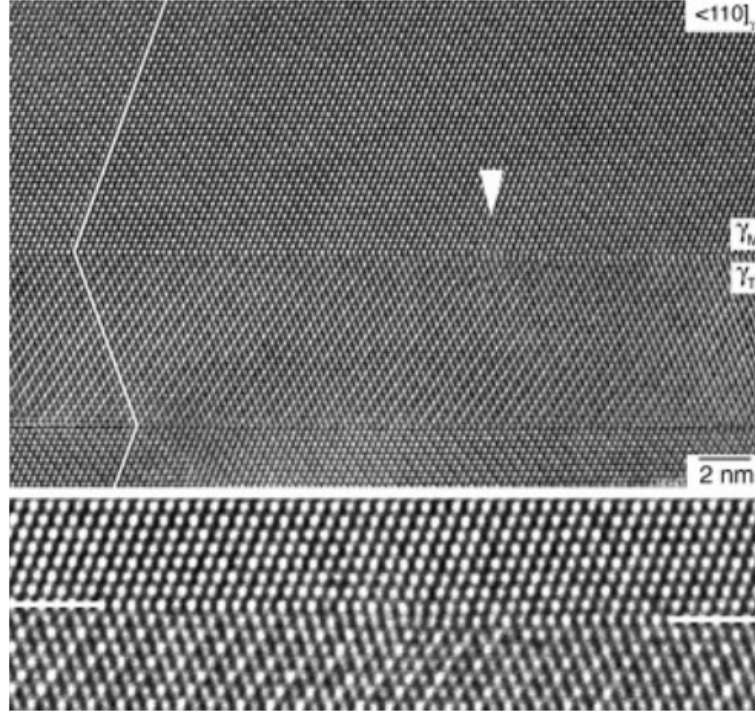


Figure 2.9: Atomic structure of a deformation twin observed in Ti-48.5Al-0.37C at.% alloy after compression at room temperature. High resolution TEM image along $\langle 110 \rangle$. γ_M designates the matrix, white lines indicate the trace of the $(\bar{1}\bar{1}1)$ planes. The arrow indicates the one plane step on the (111) due to partial dislocation. The core of the dislocation is indicated in the lower micrograph at a higher magnification. Micrograph from [47].

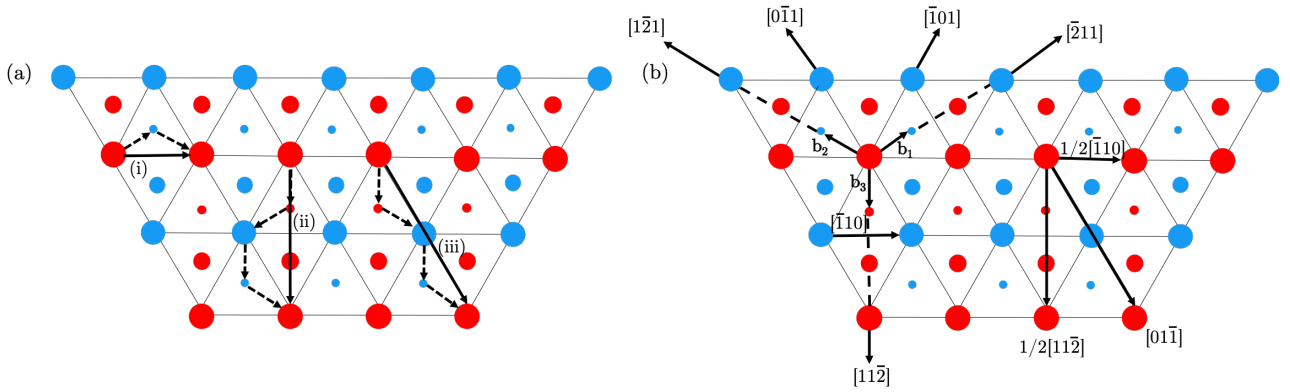


Figure 2.10: Potential slip and twinning systems of the γ , $L1_0$ structure, schematic drawing of three-layer sequence of atom stacking on the (111) plane shown by small, medium and larger circles, where blue hard spheres are titanium, and red hard spheres are aluminium. (a) Core configurations of dislocations with Burgers vectors $\mathbf{b} = 1/2[\bar{1}10]$, $\mathbf{b} = 1/2[11\bar{2}]$ and $\mathbf{b} = [01\bar{1}]$. (b) $\mathbf{b}_1 = 1/6[\bar{2}11]$, $\mathbf{b}_2 = 1/6[1\bar{2}1]$ and $\mathbf{b}_3 = 1/6[11\bar{2}]$ are the Burgers vectors of partial dislocations. $\mathbf{b}_3$ is perpendicular to the Burgers vector $\mathbf{b} = 1/6[\bar{1}10]$ for ordinary slip and represent the Shockley partial dislocation for true twinning, where $\mathbf{b}_1$ and $\mathbf{b}_2$ represent pseudo-twinning. Schematic adapted from [47].

In γ -(TiAl) at room temperature it is found that dislocations from $\mathbf{b}=1/2\langle 101 \rangle$ occur with some contribution from twinning, with dislocations $\mathbf{b}=1/2\langle 110 \rangle$ making up a small, nearly negligible fraction. However, at temperatures above 600°C, dislocations $\mathbf{b}=1/2\langle 110 \rangle$ dominate. The lack of mobility of the $\mathbf{b}=1/2\langle 110 \rangle$ dislocations at room temperature is attributed to covalency, and the already active $\mathbf{b}=1/2\langle 101 \rangle$ dislocations. This gives rise to the increased ductility at higher temperatures, due to the operation of thermally activated slip systems $1/2\langle 110 \rangle\{111\}$, with more glide from $\mathbf{b}=1/2\langle 110 \rangle$ dislocations, and apparent increase in mechanical twinning above 600°C [49].

α_2 -(Ti₃Al) is HCP as shown in Figure 2.11. The standard notation here is again amended to have a fourth index, a third index added to the basal plane to simplify vector notation, Figure 2.11. Hexagonal materials are most likely to slip on basal planes $\{0001\}$, prismatic planes $\{10\bar{1}0\}$ and on pyramidal planes $\{1\bar{2}11\}$, Figure 2.12. Deformation occurs with two types of superdislocations, with the $\langle \mathbf{a} \rangle$ -type dislocations with $\mathbf{b}=1/3\langle 11\bar{2}0 \rangle$ and the $\langle 2\mathbf{c}+\mathbf{a} \rangle$ -type $\mathbf{b}=1/3\langle \bar{1}\bar{1}26 \rangle$. Deformation is largely carried out on the prismatic plane by the $1/3\langle 11\bar{2}0 \rangle$ superpartial dislocations on the $\{10\bar{1}0\}$ prism planes [50; 51]. Basal slip is the second most likely form of deformation, but much less likely than prismatic, and often occurs in conjunction with prismatic. Dislocation activity on the pyramidal planes is less frequent [52]. Slip is found to be activated at a grain boundary, due to it being a stress raiser.

Increasing temperature increases the activation of prismatic, basal and pyramidal slip in

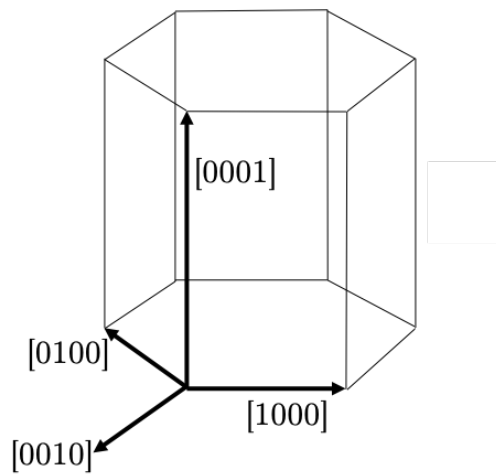


Figure 2.11: Notation for HCP, the addition of a third index on the basal plane.

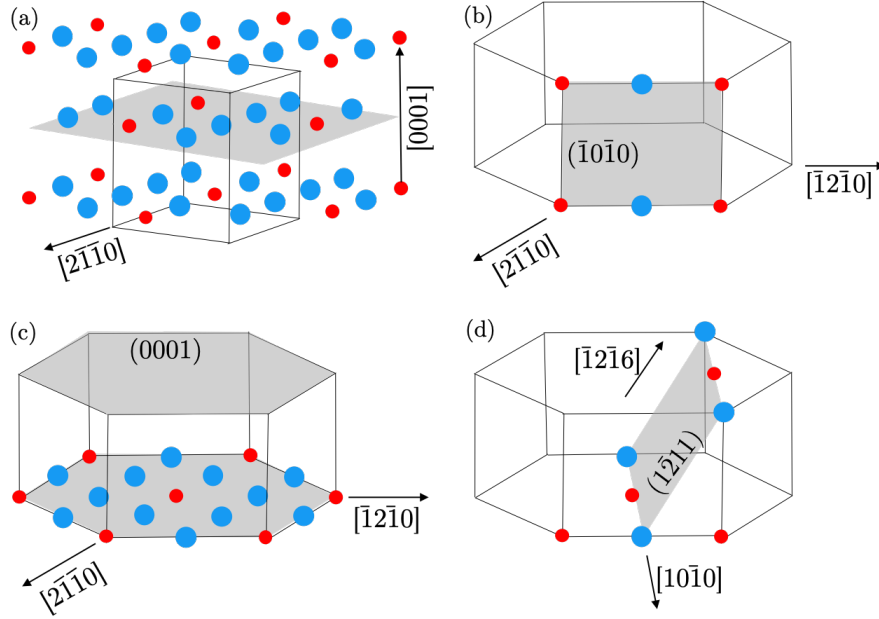


Figure 2.12: α_2 where the blue hard spheres are titanium and the red hard spheres are aluminium. (a) DO₁₉ structure with unit cell indicated. (b) Prismatic slip system $[1\bar{2}\bar{1}0](10\bar{1}0)$. (c) Basal slip system $[1\bar{2}\bar{1}0](0001)$. (d) Pyramidal slip system $[1\bar{2}\bar{1}6](1\bar{2}11)$. Schematic adapted from [2].

α_2 , with ease of activation in that order, Figure 2.13(a) [58]. Pyramidal slip can be seen to decrease at intermediate temperatures, Figure 2.13(b). Due to pyramidal slip being the only

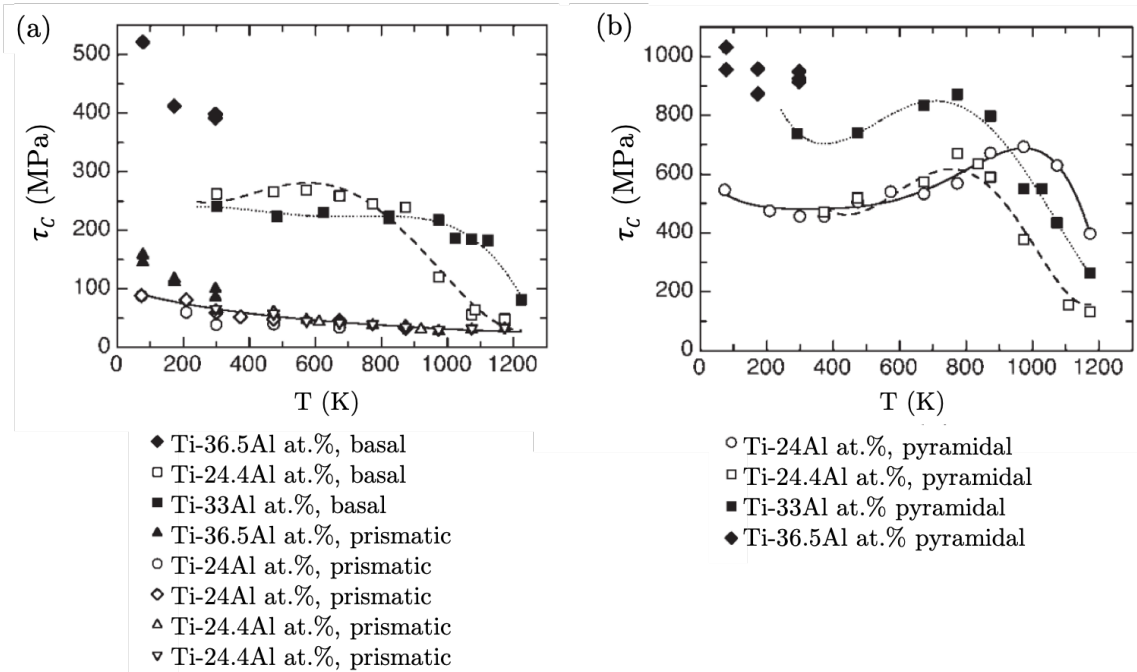


Figure 2.13: Deformation behaviour of single phase α_2 . Variation of the critically resolved stress with temperature [53–57]. (a) Basal slip $1/3\langle 11\bar{2}0 \rangle(0001)$ and prismatic slip $1/3\langle 11\bar{2}0 \rangle\{10\bar{1}0\}$. (b) Pyramidal slip $1/3\langle 1\bar{1}\bar{2}6 \rangle\{1\bar{2}11\}$. Data compiled by [2].

glide plane to have a c component, it is required for general plasticity of the material. However, due to prismatic slip increasing at higher temperatures, Figure 2.13(a), and pyramidal slip decreasing at intermediate temperatures, this causes a strong preference for prismatic slip, and can contribute to a decrease in the ductility of the α_2 phase at increased temperatures [53–57].

Two phase γ -(TiAl) and α_2 -(Ti₃Al) alloys exhibit much better mechanical properties than either of the individual constituents if the phase distribution and grain size are suitably controlled.

As the interfaces take a preference for the lowest interfacial energy, there is gives a tendency for directions or planes with high atomic densities to align. There is a common orientation relationship between the phases at the lamellar boundaries. The matching close packed planes and directions are $\{111\}_\gamma \parallel (0001)_{\alpha_2}$ and $\langle 1\bar{1}0 \rangle_\gamma \parallel \langle 11\bar{2}0 \rangle_{\alpha_2}$. The basal plane of the hexagonal α_2 -(Ti₃Al) lies parallel to the $\langle 111 \rangle$ of the tetragonal γ -(TiAl), Figure 2.14. This allows six rotations of γ -(TiAl) around the vector $\langle 111 \rangle$ to create the α_2 -(Ti₃Al) and γ -(TiAl) interfaces. Figure 2.15(b) shows this interface. However, not only are there interfaces between

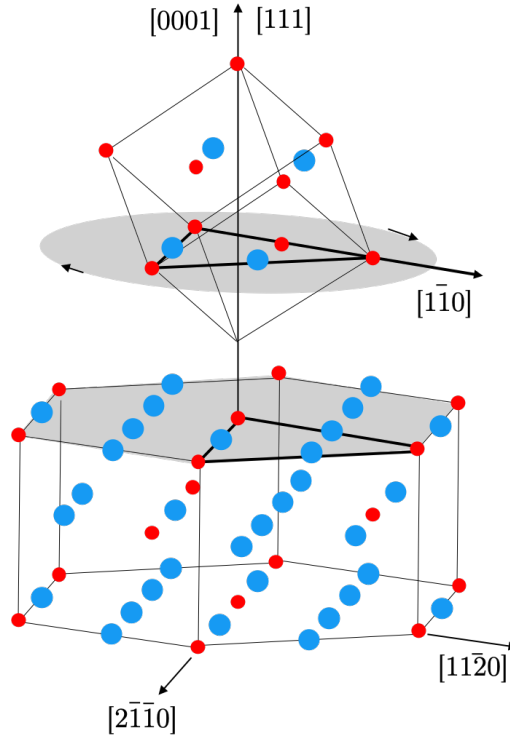


Figure 2.14: Crystallographic orientation relationship of α_2 -(Ti₃Al) and γ -(TiAl). The basal plane of the hexagonal α_2 , the bottom structure, lies parallel to the $\langle 111 \rangle$ plane of the γ -(TiAl), the top structure. The blue hard spheres are titanium and the red hard spheres are aluminium. Schematic adapted from [2].

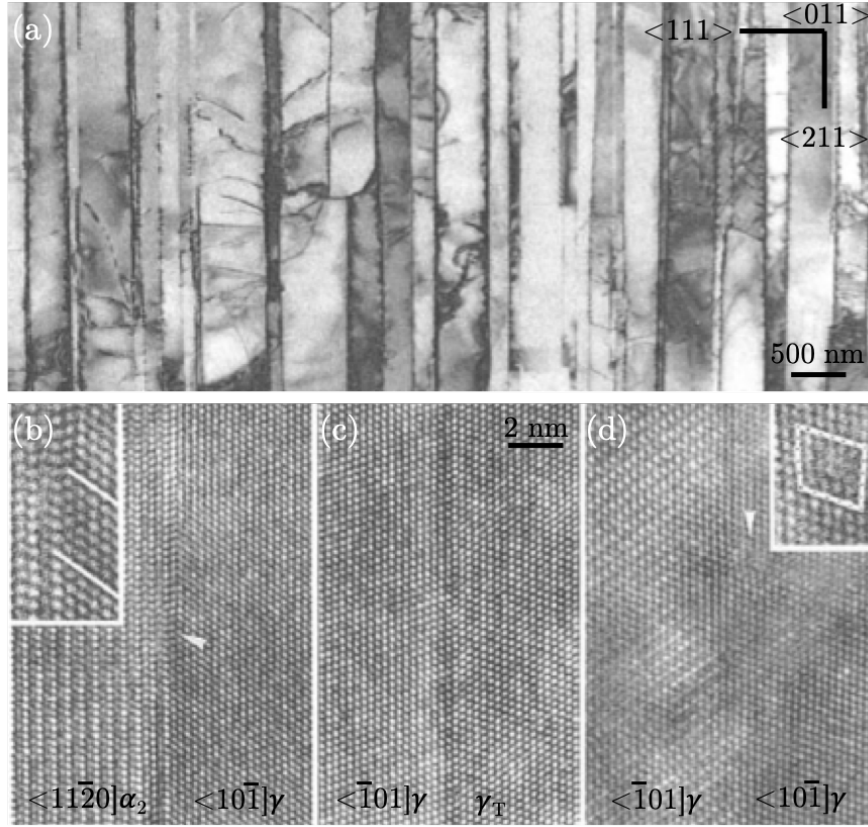


Figure 2.15: Structure of lamellar interfaces of dual phase alloy of composition Ti-48Al-2Cr. (a) TEM image of lamellar microstructure, (b) High resolution electron micrograph of a α_2 — γ interface (c) coherent γ — γ_T interface, (d) a semi-coherent γ — γ_T interface with 120° matrix rotational variant. Micrographs from [4].

α_2 -(Ti₃Al) and γ -(TiAl) phases, there are also interfaces between lamellae of γ -(TiAl). The γ — γ_T interfaces are most usually true twins, rotating 180° around $\langle 111 \rangle$, as this has the lowest interfacial energy, leaving a coherent interface as can be seen in the TEM image in Figure 2.15(c) [4]. Other orientations between the lamellae of the γ — γ_T interface can be seen to occur. These are pseudo-twins, rotating 60° or 300° around $\langle 111 \rangle$, leaving an incoherent interface, Figure 2.15(d). The α_2 lamellae within one colony will all be of the same orientation, as it will have originated from the same α or α_2 grain, but this is not the case for γ , which can be one of its six rotational variants within one colony.

Due to purification of the γ -(TiAl) by the preferential partitioning of interstitial hardeners such as oxygen, nitrogen and carbon to α_2 -(Ti₃Al), the mobility of dislocations in γ -(TiAl) within the dual phase structure is increased in comparison to single phase γ -(TiAl) [4]. This, and the lack of pyramidal slip in α_2 -(Ti₃Al) at room temperature giving rise to plastic anisotropy

[59], leads to deformation mainly occurring in the two phase alloy in the γ -(TiAl) at room temperature regardless of the microstructure [60–66]. The deformation in the γ -(TiAl) phase is mainly carried out by the three Burgers vectors and mechanical twinning along $1/6\langle 11\bar{2} \rangle\{111\}$, where ordinary dislocations are the primary dislocation mode followed by mechanical twinning, unlike the single phase γ alloy, which shows a reluctance to twin [67–69]. The α_2 is more difficult to deform, so remains free of defects. However, for strain continuity, both phases must deform, and α_2 mainly deforms by prismatic glide of $1/3\langle 11\bar{2}0 \rangle$ superpartial dislocations.

Between 700 - 800 °C, deformation is again mainly carried out by the γ -(TiAl). The glide modes are the same as those at room temperature, but there is more strain accommodation due to climb of ordinary dislocations [62; 70; 71]. There is also increased mechanical twinning at increased temperatures, though superpartial dislocations are not especially enhanced. Elevated temperatures above 700°C show activation of the pyramidal slip in the α_2 and therefore shows a decrease in the plastic anisotropy and therefore an increase in ductility. This decrease in the difference in plasticity of the two phases at elevated temperatures is beneficial for the material's overall plastic deformation behaviour. The combination of the changes in slip system activation in the two phases contributes to there being a brittle to ductile transition in the region of 700°C.

Interfaces of $\alpha_2 \parallel \gamma$ and $\gamma \parallel \gamma$ give rise to glide resistance and plastic anisotropy, impeding slip transfer through lamellae. Overcoming these interfaces involves all the above discussed processes, and the barrier strength is dependent on the interface, be it $\gamma \parallel \gamma$ (any of its twin variants) or $\gamma \parallel \alpha_2$. It is also dependent on the incoming dislocation and the slip plane relative to the interface. For example, slip transfer through a true twin boundary would simply be the activation of a conjugate deformation system, where the incoming and outgoing slip systems are in mirror symmetry. Similarly, unit dislocations with a Burgers vector parallel to a twin interface could easily overcome the γ true twin interface by cross-slip. This gives rise to a low barrier strength for true twin boundaries [72]. Strain is found to elastically transfer across these interfaces to activate dislocation sources at its exit side. In general, the slip transfer through an interface occurs by: overcoming long range coherency stresses, elastic interaction and intersection of incoming dislocations with interfacial dislocations, reaction of incoming dislocations with dislocations at the interface to incorporate incoming dislocations into interfacial dislocations,

decomposition and dissociation of incoming dislocations onto new slip planes, elastic shear transfer through the lamellae, or activation of dislocation sources at the exit side of the interface [2, p. 173]. The $\gamma\|\alpha_2$ interface is found to have the higher barrier interface when compared to the $\gamma\|\gamma$ interface [61; 62]. This is due to it being associated with the energy dissipating processes, often with strain being elastically transferred. These processes are not thought to be supported by thermal activation, and are therefore independent of temperature [2, p. 176].

Fracture behaviour of the γ and α_2 phases is temperature dependent, and therefore fatigue crack growth characteristics can be seen to altered with a change in temperature. Crack paths are seen to be less tortuous, and more straight at elevated temperatures. This is believed to be due to activation of a higher number of slip systems. Change in temperature results in homogenisation of slip, which is assisted by diffusion and cross glide. The change in deformation modes with temperature causes variations in crack growth rates in comparison to those seen at room temperature. At elevated temperature the tendency for planar slip is no longer observed, in contrast to room temperature [73]. Overall, dominant fracture mechanisms present in lamellar alloys are interfacial delamination, trans-lamellar fracture and decohesion of lamellar colonies [4].

2.4 Fatigue fundamentals

Fatigue of a material is the change in its properties due to the application of cyclic loading. Most often, this refers to a lowering of the usable strength, but may also include loss of functional properties. Microstructural changes due to cyclic fatigue will eventually lead to failure of a material when under cyclic stress, which may be below the yield stress (σ_y) [74]. Knowledge of a material's fatigue performance allows materials and components to be designed against cyclic deformation and fatigue failure. In the 1860's the life of a material or component varying with applied stress was first documented and compiled, which gave rise to the stress-life curve, or as it is more commonly referred, an S-N curve, Figure 2.16. This curve is unique to a material or component. At a large stress, the material or component cannot survive even one cycle until failure. At a low stress, the material can endure a large number of load cycles, tending to an infinite number. This stress is called the endurance limit. Fatigue can be categorised as low

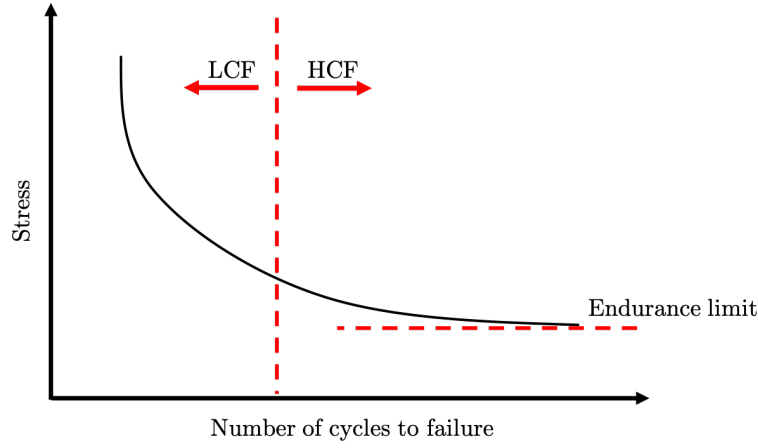


Figure 2.16: Schematic of a stress *vs.* life, commonly known as an S-N curve.

cycle fatigue (LCF), which is dominated by crack growth, or high cycle fatigue (HCF), which is dominated by the number of cycles required to initiate the crack, defined as typically 10^5 cycles or above. This is a total fatigue life approach, where the material life at a certain stress is determined until final failure.

The ability to carry out fatigue failure characterisation and comparison between materials and components remained limited until late 1950's. Irwin then discovered the amplitude of the stress ahead of the crack tip could be described as the stress intensity factor K . This is a function of loading, crack length and specimen geometry [75, p. 7]. Paris, Gomez and Anderson then furthered this idea, comparing the range of the stress intensity factor, $\Delta K = K_{max} - K_{min}$, to the increment in fatigue crack per cycle, da/dN , during constant amplitude loading [75, p. 159]. This method allows characterisation of growth of fatigue cracks undergoing plastic deformation ahead of the crack tip in a material. With an increase in the stress intensity range, ΔK , applied under cyclic loading, the fatigue induced crack can be seen to advance and grow until final fracture. The relationship between these two variables on a log-log plot can be seen in Figure 2.17, with three distinct regions seen to occur [76, p. 341].

Region I

Below the application of a certain ΔK , there is no, or undetectable, crack growth. For a crack to propagate in the material there is an initial stress intensity threshold, ΔK_{th} , that must be overcome. This is when a material is loaded beyond its plastic limit, causing permanent

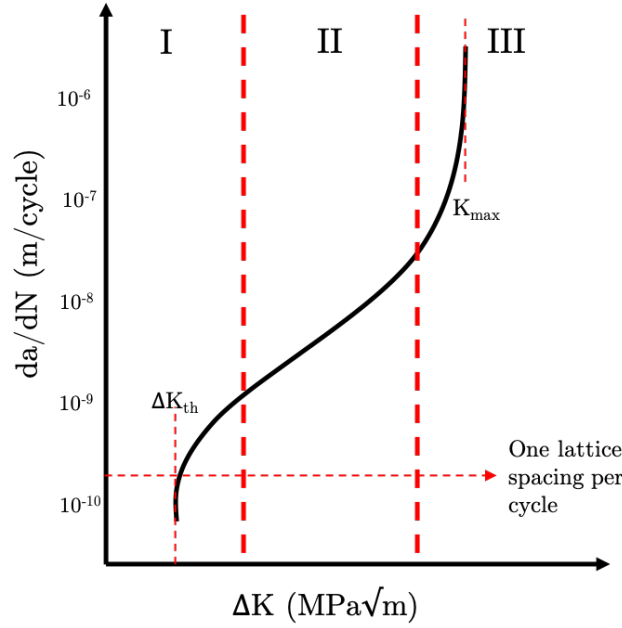


Figure 2.17: Schematic of the fatigue crack growth curve with the three regions of fatigue crack propagation, I, II and III, stress intensity threshold ΔK_{th} , maximum stress intensity value for the fatigue cycle K_{max} and the growth of one lattice spacing per cycle. Adapted from [77].

plastic deformation. The crack growth threshold is essentially a limit for a defect to grow a crack, thus the initial stress intensity required for the crack to propagate in the material. Early crack growth, approximately less than 2×10^{-10} m/cycle, grows at less than one lattice spacing per cycle [77]. In a dual phase material, cracks are preferably formed in the ductile phase. Therefore, cracks should be seen to initiate in the γ -(TiAl) due to the preferential partitioning of interstitial hardeners to the α_2 phase, causing α_2 to become more brittle than γ . Initial crack growth from this threshold is rapid and non-linear on the log-log fatigue crack growth curve, as can be seen in Figure 2.17. The growth in this region can be seen to be microstructurally dependent, relating to grain size and to slip characteristics of the material [78].

Region II

The shift from region I to II corresponds to the transition from microstructure-sensitive to microstructure-insensitive crack growth. At intermediate stress intensity ranges, the crack growth curve is linear on a log-log plot, in relation to the ΔK applied. This relationship can be

demonstrated by the Paris Law and rearranged for the log-log plot [79]:

$$\frac{da}{dN} = C(\Delta K^m) \quad (2.1)$$

$$\log\left(\frac{da}{dN}\right) = \log(C) + m\log(\Delta K) \quad (2.2)$$

where $\log C$ is the intercept and m is the gradient of the Paris regime on a log-log plot. m is often called the Paris exponent and can be used to characterise the material. The waveform of this alternating stress intensity can take on many forms, including sinusoidal or trapezoidal, to imitate working conditions. The rates of fatigue crack growth are influenced by many variables including microstructure, mechanical loading and environment. Despite many attempts at modelling to predict the crack propagation rate for materials or component design in different testing environments, the large number of variables has inhibited success [76, p. 356] [80; 81]. A crack may deviate from an anticipated crack growth plane for many reasons, including inhomogeneities such as grain boundaries and interfaces. This may lead to a slower fatigue crack growth than initially anticipated, particularly in the low ΔK regime where da/dN is on the scale of the microstructure. Experimental observations for the Paris exponent for metals are around 4, and is common for most metals [82].

Region III

The application of large ΔK values causes final crack growth to increase rapidly in a non-linear fashion, until failure. Failure occurs in this region. The stress intensity at which this occurs is $K_{\max}$, Figure 2.17. In this region there is substantial sensitivity of crack growth to microstructure, load ratio and stress state, whereas test environment is no longer a factor due to increased crack propagation rates [76, p. 357].

This log-log plot is characteristic to a specific mean load and R ratio applied to a material or component. R ratio is the ratio of $K_{\min}$ to $K_{\max}$. The size of each region is not constant in relation to each other, and can therefore vary depending on the material, mean load or R ratio. Higher R ratios result in a higher mean stress, and therefore lower ΔK_{th} and $K_{\max}$ values [83]. This shifts the whole crack growth curve to lower ΔK values. The application of low R ratios

increases the value of K_{max} . K_{max} is related to the fracture toughness K_c by the equation:

$$K_{max} = \frac{\Delta K}{1 - R} \rightarrow K_c \quad (2.3)$$

which shows that when the R ratio is low, K_{max} tends to K_c , the fracture toughness of the material [76, p. 358]. Fracture toughness is a quantitative measure of a materials ability to resist fracture once a crack is present.

There are three modes of crack growth: mode I is crack tip opening or tensile mode, mode II is in-plane shear, and mode III is anti-plane shear, Figure 2.18. This causes a stress field ahead of the crack tip. Mode I loading occurs most commonly, and is the mode of concern throughout this thesis [84]. Fracture toughness under mode I crack growth is denoted K_{Ic} .

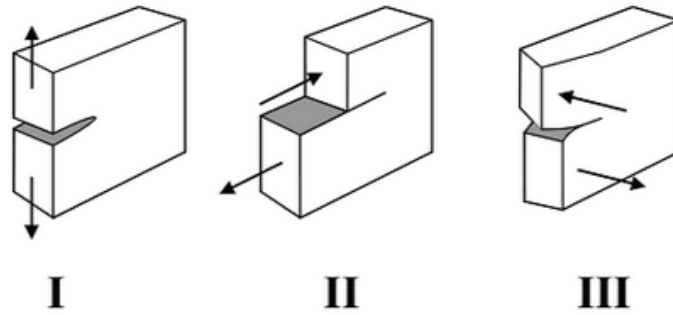


Figure 2.18: Three modes of crack growth: mode I is crack tip opening or tensile mode, mode II is in-plane shear, and mode III is anti-plane shear [84].

Stress intensity is a measure of the intensity distribution of the near-notch stress field. This near-notch stress field introduces dislocations and therefore deformation. An application of a stress intensity above a certain value causes this deformation to no longer be elastic, causing local plastic deformation, labelled the plastic zone. The material at the crack tip yields when near-tip stresses exceed the flow strength.

Ahead of a crack tip there is a region of reversed plastic flow [85], which is created by the application of a far-field tensile load, P , applied to a material with a crack tip. The material yields at the crack tip due to the load application creating a monotonic plastic zone, r_p , Figure 2.19(a). Partially unloading the tensile load changes the stress distribution at the crack tip and causes reverse plastic flow. This region of reversed flow is the cyclic plastic zone, r_c , Figure

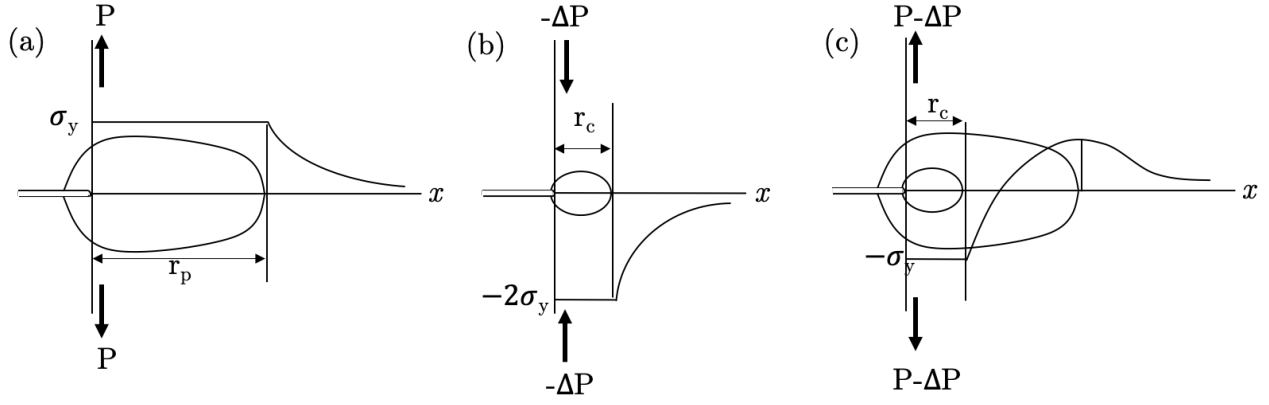


Figure 2.19: Development of the cyclic plastic zone. (a) Monotonic plastic zone, r_p , created far-field load application, P . (b) Reduction in load by ΔP changes the near-notch stress distribution to give region of compressive stresses, r_c . (c) Superposition of both (a) and (b). Schematic adapted from [76, p. 305].

2.19(b). This zone has residual compressive stresses induced and is embedded in the monotonic plastic zone, Figure 2.19(c) [76, p. 305].

The change in stress intensity, ΔK , relates the stress state at the crack tip with the crack length. This is related by the following equation:

$$\Delta K = Y \Delta \sigma \sqrt{\pi a} \quad (2.4)$$

where Y is a geometry factor which can be found in the standards [86], $\Delta \sigma$ is the change of applied stress during cyclic loading and a is the crack length. Determination of the geometry factor is discussed in Chapter 4. The elastic stress exceeds the flow strength at a distance $a = r_p$ ahead of the crack. At the elastic-plastic boundary ahead of the crack, let $\Delta \sigma = \sigma_y$. A common approximation for the geometry factor, $Y = \sqrt{2}$. The above equations can therefore be rearranged to:

$$2r_p = \frac{\Delta K^2}{\pi \sigma_y^2} \quad (2.5)$$

The shape of the plastic zone should also be considered, as the plastic zone diameter may vary with angle θ from the crack plane, x , Figure 2.19 [84].

The cyclic application of loading and unloading causes alternating plastic flow. This plasticity introduced at the crack tip causes dislocations to move back and forth on the slip planes each

cycle, causing tearing at the crack tip and crack advance, Figure 2.20. Loading a crack tip causes crack extension, but can be seen to cause crack plastic blunting, Figure 2.20(a)(b)(c). Unloading this crack tip cannot negate the crack growth and blunting, Figure 2.20(d)(e). Reloading the crack in the next cycle will cause the crack to advance further, Figure 2.20(f) [87]. For crack advance, the size of the plastic zone in relation to the microstructure is going to be of importance.

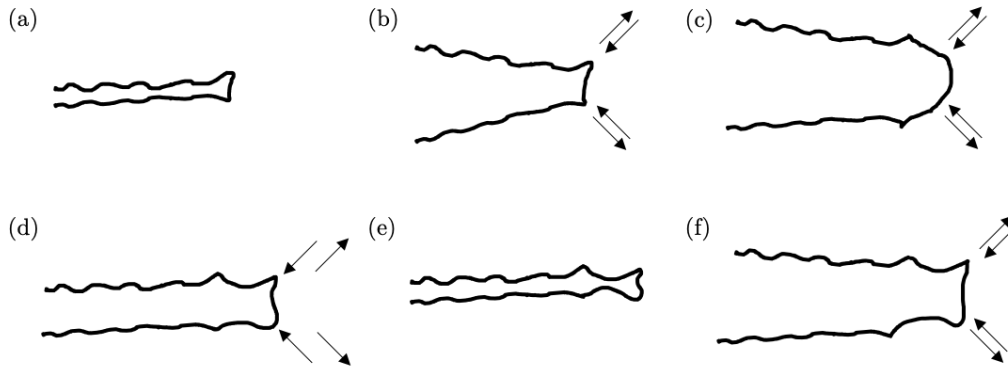


Figure 2.20: Cyclic loading at a crack tip causes dislocation movement on the slip planes every cycle. The crack front at (a) zero load, (b) small tensile load, (c) peak tensile load, (d) onset of load reversal, (e) peak compressive load, (f) start of next fatigue cycle with small tensile load. Schematic adapted from [76, p. 338].

Lifing using da/dN vs. ΔK curves is called the defect tolerant approach, accepting the hypothesis of an initial flaw. A material can be analysed for flaws using a non-destructive characterisation approach. Should no flaws be detected, this can be tested using a slightly higher stress than used in service. Should there be no crack initiated during this testing, the smallest flaw size can be assumed to be the resolution of the non-destructive analysis technique. The material life can then be determined then from this initial flaw size by testing this component until failure [75, p. 12]. There are many factors which may affect the fatigue performance, these include: mean stress, temperature, microstructure, component architecture, defect size and distribution, and environment, to list but a few [88; 89].

2.5 Alloy production and microstructure

Almost all processing routes for metallic alloys involve melting and subsequent solidification of the material. Thus, solidification not only determines the microstructure, texture and distribution of alloying elements in cast materials but also plays a significant role in processing. The γ -(TiAl) based alloy Ti4522-XD can be produced through many processing routes. These provide different microstructures and therefore properties, including fatigue performance.

The as-received material used in this thesis was near-net shape investment cast bars from Rolls-Royce plc.. Investment casting has the ability to create component shapes with high dimensional fidelity and therefore require minimal post casting machining. The process of investment casting is to make an exact component replica with wax and spray this with multiple layers of ceramic to create a shell around the wax replica. The wax replica is then melted out to create a hollow mold. The alloy is melted and poured into this mold, to create a near-net shape casting of the alloy. The XDTM process adds fine dispersions of ceramics to the melted alloy, in this case TiB₂, to act as a grain refiner and therefore to control the size of the colonies [90]. These remain stable throughout casting, enabling a homogeneous and equiaxed microstructure. The solidification process for this alloy has been discussed previously, as shown in the schematic in Figure 2.5. This creates a lamellar microstructure of γ and α_2 in the prior β grains as shown in Figure 2.21, with a colony size of approximately 60 μm . The darker phase is the γ and the lighter phase is the α_2 .

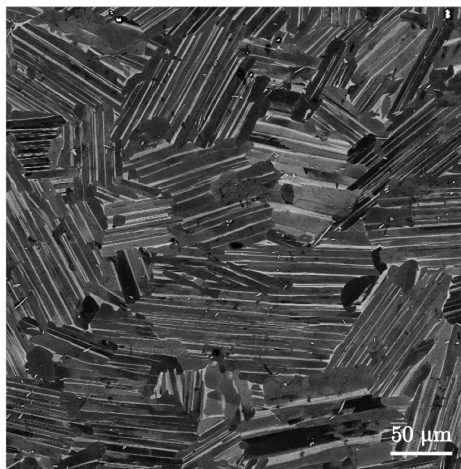


Figure 2.21: SEM backscatter image of the microstructure of the as-received near-net shape investment cast γ -(TiAl) based alloy.

Figure 2.22 shows the cast microstructure for Ti-48Al-2Nb-2Mn without grain refinement, Figure 2.22(a), and with the inclusion of 7 vol.% TiB₂ as a refining agent, Figure 2.22(b). The grains without the TiB₂ have a substantial variation in size, between 200 and 6000 μm , whereas including the refining agent reduces this range to 50 to 150 μm , nucleating from the walls of the casting [90].

Turbine blades currently being manufactured by General Electric are also cast. However, not near-net shape cast, but instead, are investment cast over-sized, and then machined back. This does not require the solidification route to be as good as for near-net cast materials. This is carried out with an alumina shell. GE has discussed building γ -(TiAl) based turbine blades through 3D printing with an electron-beam freeform powder additive manufacturing route. The surface finish is dependent on the feed direction, and in some directions is quite poor. This would therefore require secondary finishing of the component [91].

Pratt and Whitney in conjunction with MTU have made forged blades and discs for their Geared TurboFanTM. This turbofan allows the low pressure turbine to run at its optimum rotational velocity, while the fan runs slower, at its optimum rotational velocity, which increases the bypass ratio and decreases fuel consumption. The alloy in use here for the fast running, low pressure turbine blade is the β stabilised TNM alloy, used in the PW1000G-JM engine [22]. This alloy is based on γ -(TiAl), but has β stabilising additions of niobium and molybdenum. The aluminium content is below 45 at.%. This ensures solidification through the β /B2 phase in the phase diagram, Figure 2.23 [92]. Solidification through the β is said to result in a smaller grain

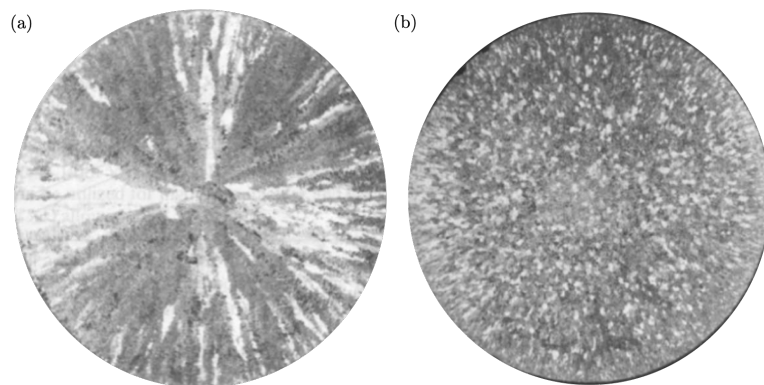


Figure 2.22: From [90], cast microstructure for γ -(TiAl) based alloy Ti-48Al-2Nb-2Mn. (a) Contains no borides showing large grains between 200 and 6000 μm , (b) contains 7 vol.% borides and therefore shows smaller grains between 50 and 150 μm .

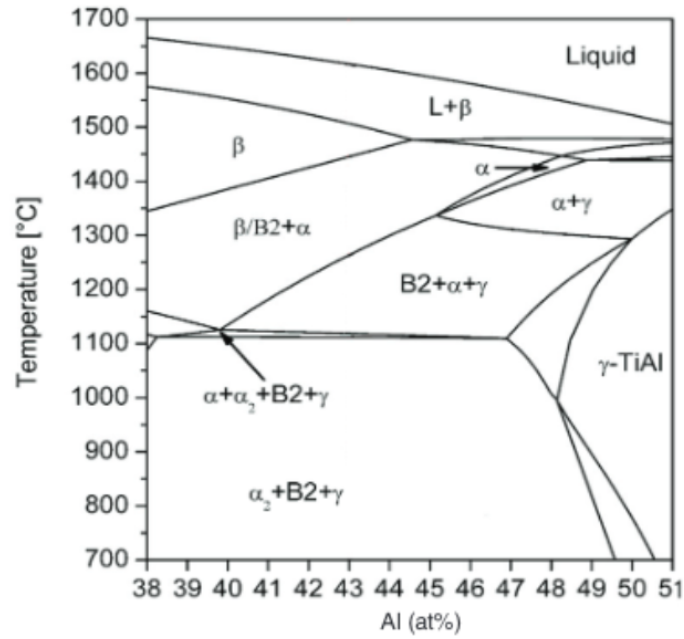


Figure 2.23: Pseudo-binary phase diagram of the TNM alloy from [92].

size [92]. Small amounts of borides are also included for further grain refinement [93]. Large amounts of the disordered β phase improves hot workability [94]. This allows good processing ability and mechanical properties, with variation of microstructural properties possible by variation in heat treatment. Due to the absence of the α phase there is a fine equiaxed structure, which leaves the three phase microstructure of α_2 , γ and β . This microstructure can be varied by the heat treatment later used. Figure 2.24 shows two heat treatment processes applied to this microstructure. Figure 2.24(a) is a triplex microstructure, with globular γ , β , and some α_2 and γ lamellae. Figure 2.24(b) shows a nearly fully lamellar microstructure of α_2 and γ .

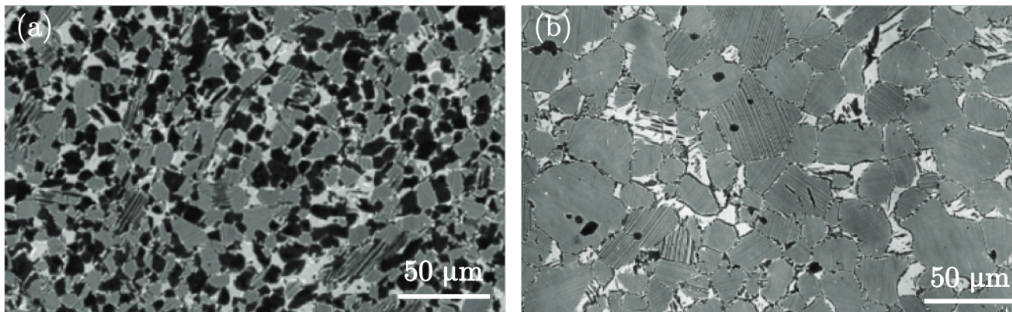


Figure 2.24: Microstructure of TNM with two different heat treatment processes carried out to generate two different microstructures. (a) A triplex microstructure, with globular γ , β , and some α_2 and γ lamellae, (b) nearly fully lamellar microstructure of α_2 and γ . Micrographs from [22].

These blades and discs are created using die forging at a high temperature between 980 and 1300 °C. Due to the brittle nature of γ -(TiAl), this must be carried out isothermally at low strain rates to limit the risk of brittle fracture. As molybdenum has a high melting point, it is used for the dies. However, molybdenum quickly reacts with the oxygen to form molybdenum oxide, which can evaporate or cause interstitial contamination. To remove this problem, the die forging is done in a vacuum or an inert environment to eliminate the oxidation of the molybdenum, causing the process to become more expensive and slower. A previous generation of cast γ -(TiAl) based alloys were also used by Pratt and Whitney in the low pressure turbine blades.

2.6 Fatigue performance and behaviour

A great deal of work has been carried out on titanium aluminide alloys since 1953 [95]. However, due to their brittle nature, they have undergone several microstructural iterations and are still considered a development alloy. As previously discussed, small changes in composition or cooling rate cause large changes to the microstructure, thus have a large impact on fatigue performance and behaviour [11]. α_2 and γ alloys with 45 - 48 at.% Al can have a lamellar microstructure of varying lath widths, equiaxed microstructure [96; 97], or bi-modal equiaxed and lamellar microstructure [98]. Different types of microstructure also commonly have different scales of colony size. The addition of alloying elements can promote one of these microstructures, but must not affect the stability of the surface oxide, so as to be able to use γ -(TiAl) based alloys in the uncoated condition. Fully lamellar microstructures have lower room temperature strength and lower ductility, but good creep resistance. Conversely, bi-modal equiaxed and lamellar microstructures have poorer creep resistance but better strength and ductility [99]. A comparison of the fatigue strength in γ -(TiAl) bi-modal and a fully lamellar microstructure can be seen in Figure 2.25(a). The lamellar microstructure can be seen to require a lower stress to reach its endurance limit than the bi-modal microstructure. However, when these strength are compared normalised to the ultimate tensile strength, the results for the two microstructures are similar, Figure 2.25(b) [11].

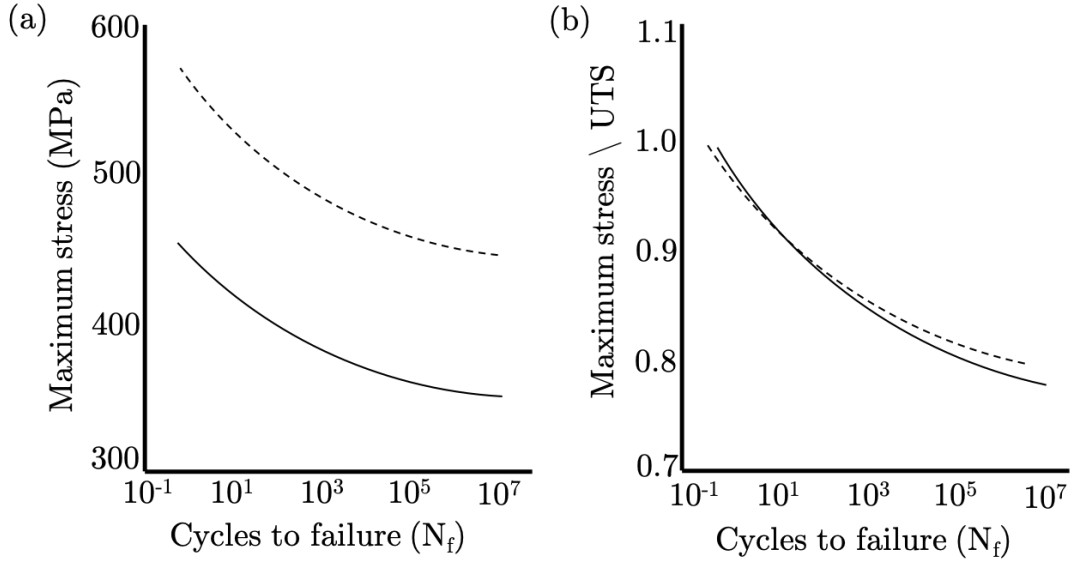


Figure 2.25: S-N curves for a γ -(TiAl) based alloy with a lamellar microstructure as a dashed line, and bi-modal equiaxed microstructure as a solid line, at room temperature in air with R ratio 0.1. (a) S-N curve for the two microstructures. (b) Normalised S-N curve with the ultimate tensile strength for the two microstructures. Figure adapted from [11].

γ -(TiAl) based alloys do not always follow the typical S-N curve shape. For example, Figure 2.26 shows five γ -(TiAl) based alloys with different elemental additions and microstructures, showing three different shapes to their S-N curve. For the γ -(TiAl) based alloy Ti-47Al-2Nb-2Mn-0.8B XD, the S-N curve does not approach an endurance limit at larger numbers of cycles, but the curve descends indefinitely [100; 102–106]. This dictates that the material failure will inevitably occur once enough cycles have been applied.

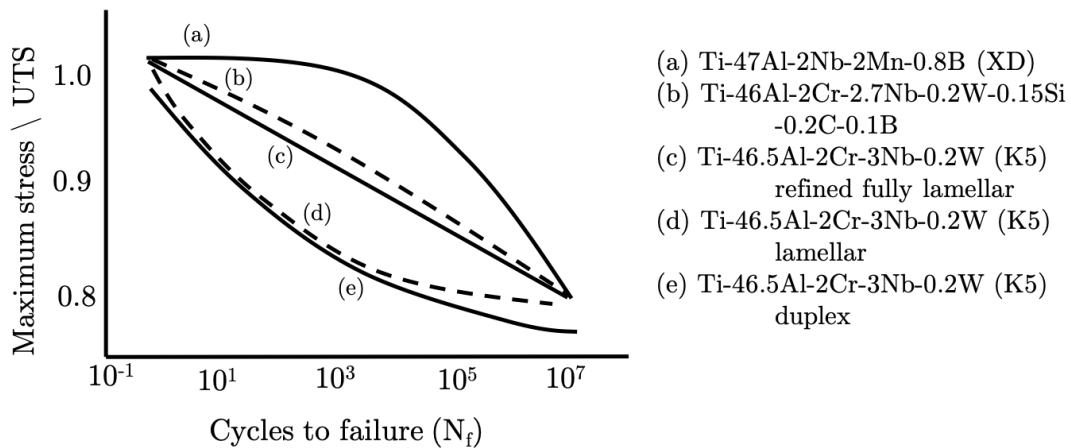


Figure 2.26: Room temperature S-N curves in air for different microstructures in γ -(TiAl) based alloys. Schematic adapted from [100].

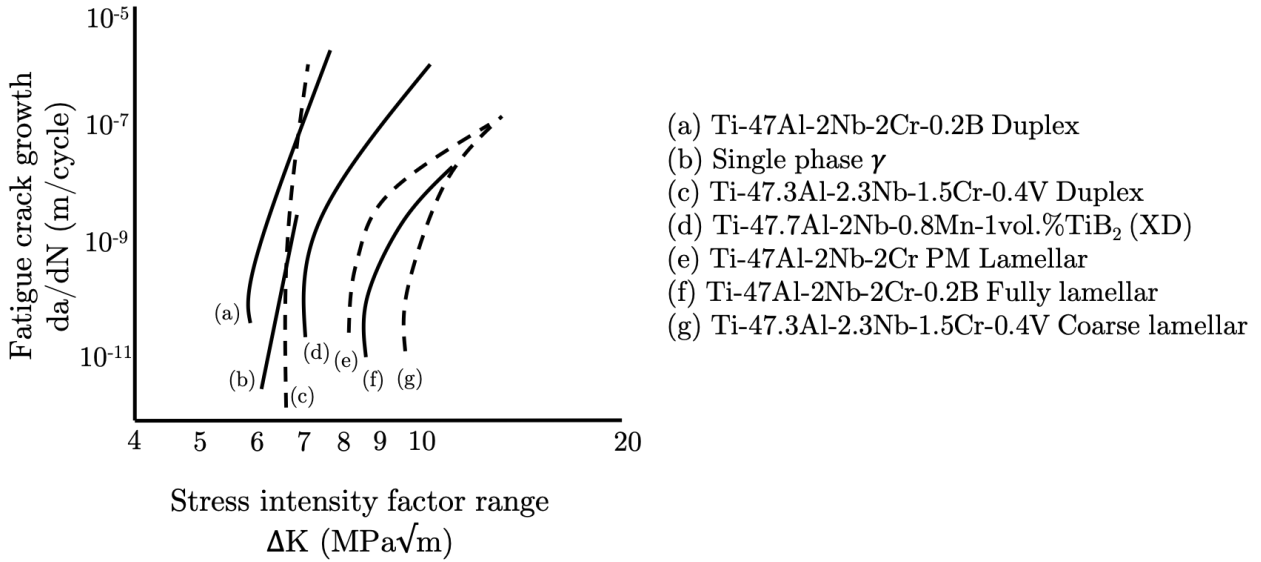


Figure 2.27: Fatigue crack growth propagation data for various γ -(TiAl) based alloys, at room temperature in air with R ratio 0.1 at 25 Hz. Figure adapted from [101].

Fatigue crack threshold and propagation is found to be strongly dependent on microstructure. Figure 2.27 shows the fatigue crack propagation rate is fast for all γ -(TiAl) based alloys, more-so in duplex alloys than lamellar alloys [101; 107; 108]. Due to the crack propagation rate being fairly high in these alloys, the fatigue design is mainly focussed on the stress intensity threshold. Lamellar microstructures were found to have better crack propagation resistance than duplex or equiaxed microstructures [109; 110]. Figure 2.27 shows the stress intensity threshold for the duplex microstructures is less than that of the lamellar microstructures, with duplex at approximately 6 and 7 $\text{MPa}\sqrt{\text{m}}$ for (a) and (c) respectively, whereas lamellar microstructures range from 8 to 10 $\text{MPa}\sqrt{\text{m}}$. This is because duplex microstructures have a smaller colony size, so tend to have a lower crack growth threshold. The crack propagation rates in the two microstructures are similar, but slightly slower in the lamellar microstructure compared to the duplex microstructure [111]. However, refining both microstructures, the fracture toughness is still larger in a lamellar microstructure than duplex microstructure [112]. Comparing these microstructures at room temperature *in vacuo*, Figure 2.28, yields similar results to the fatigue curves generated in air; lamellar microstructures have the higher threshold and duplex microstructures have the higher crack propagation rate [113]. Similar results were also found at elevated temperatures [114–116].

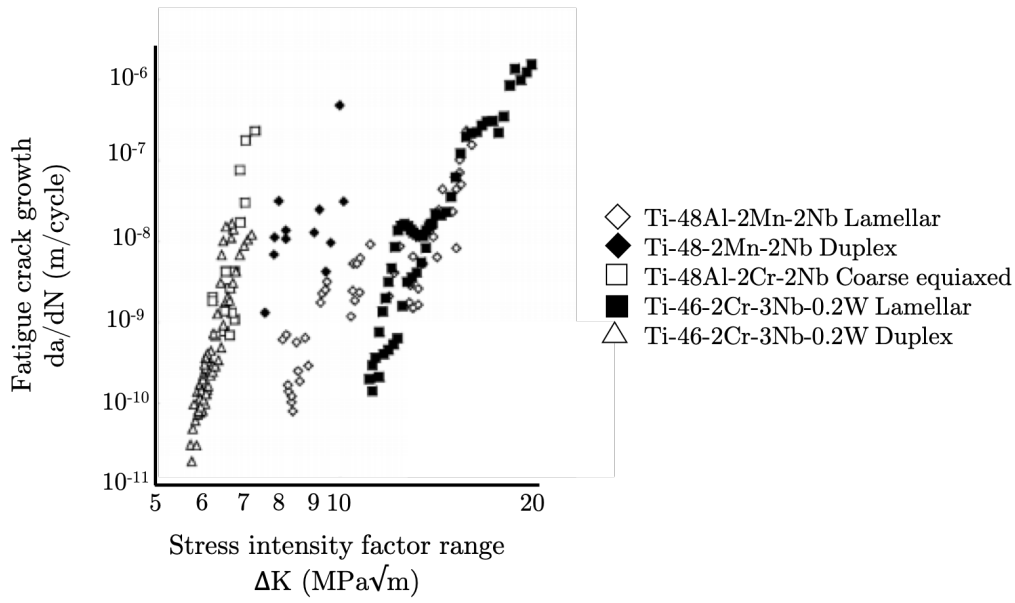


Figure 2.28: Crack growth behaviour *in vacuo* at room temperature of various microstructures of γ -(TiAl). Figure adapted from [113].

Reducing the percentage of the microstructure that is equiaxed increases the stress intensity threshold. This is highlighted by Figure 2.29, which shows the variation of the fatigue crack growth threshold on the volume fraction of equiaxed γ grains for several γ -(TiAl) based alloys [101].

The lath width of lamellar microstructures can also be seen to have an effect on the fatigue threshold and the crack propagation. Ti-48Al-2Mn-2Nb at.% alloy with different lamellar

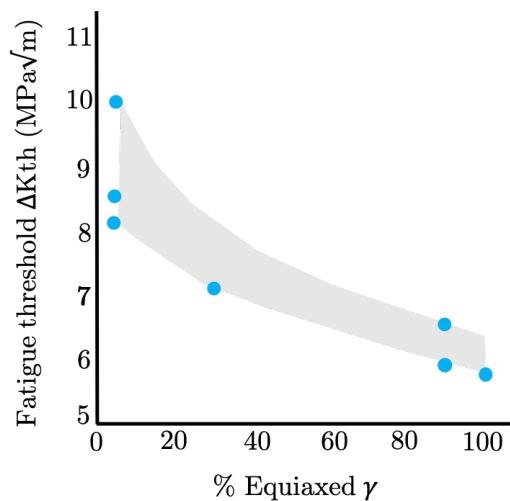


Figure 2.29: Stress intensity threshold depends on volume fraction of equiaxed grains. Figure adapted from [101].

spacings was investigated at room temperature by [117], Figure 2.30. The smallest lamellar spacing of $\sim 0.90 \mu\text{m}$ was found to have the highest threshold, when compared to larger lamellar lath widths of up to $1.47 \mu\text{m}$. The increase in resistance to fatigue crack growth is attributed to the number of lamellae per unit distance on the crack path. However, it can be seen that there is no direct correlation between fatigue crack threshold and lamellae width. For similar lamellae spacing, furnace cooled thresholds are lower than air cooled thresholds, and there is also evidence given of crack growth outside of the mode I crack growth direction [118]. Crack propagation remain similarly fast regardless of lamellae widths.

The colony size affects the fatigue crack growth characteristics, Figure 2.31 [119]. The smaller colony sizes, 90, 290 and $400 \mu\text{m}$ show similar fatigue crack growth curves. They have a similar threshold of approximately $8 - 9 \text{ MPa}\sqrt{\text{m}}$. Colony sizes over $1400 \mu\text{m}$ show a much lower threshold of $6.5 \text{ MPa}\sqrt{\text{m}}$, but a slower crack propagation rate.

Due to the lamellar microstructure providing anisotropy, the orientation of the lamellar colonies relative to the crack propagation plane will affect the crack growth behaviour. This relationship was investigated in a polysynthetically twinning γ -(TiAl) based alloy [120–123].

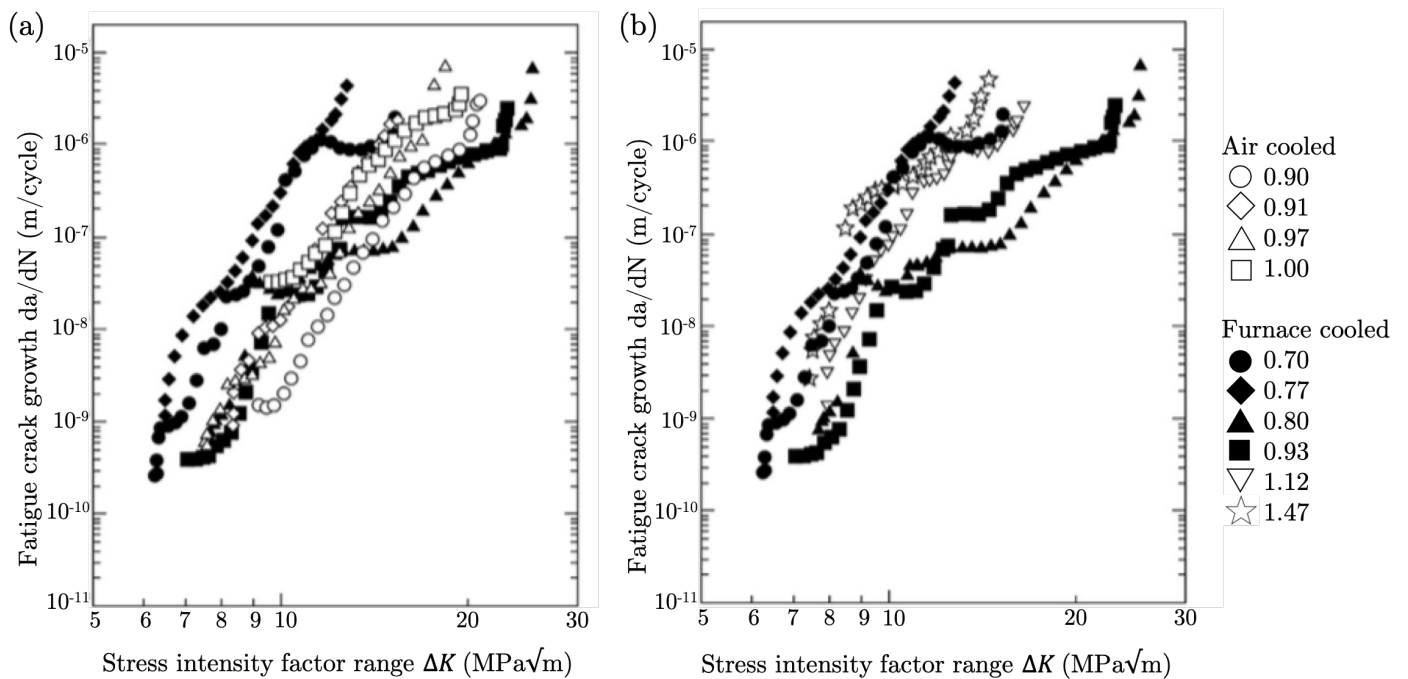


Figure 2.30: Fatigue crack growth curves in air cooled and furnace cooled specimens with differing lamellae widths. (a) Air cooled with lamellae widths 0.90 to $1.00 \mu\text{m}$. (b) Furnace cooled with lamellae widths 0.70 to $1.47 \mu\text{m}$. Figure adapted from [117].

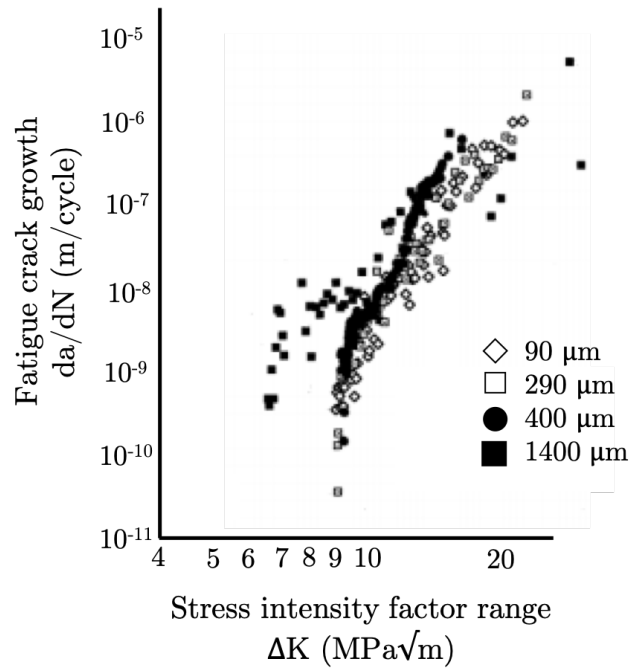


Figure 2.31: Fatigue crack propagation curves for four colony sizes, 90, 290 400 and 1400 μm , at room temperature in air with R ratio 0.1 at 10 Hz. Figure adapted from [119].

The fracture toughness was seen to be the highest when the lamellar interface was perpendicular to the crack growth direction. Conversely, lower fracture toughness is achieved with the lamellar interface being parallel to the crack growth direction. It has been noted that the scatter in fatigue crack growth data that has been generated for γ -(TiAl) may be due to the effect of orientation on the fatigue crack growth threshold, which is larger than for other microstructures [124].

The temperature which the crack tip is exposed to will affect the the fatigue crack behaviour. Figure 2.32 shows that the threshold is highest at 800°C, and lower at 600 and 25 °C [125].

Fatigue crack threshold and propagation can be found to be strongly dependent on microstructure. Lamellar microstructures can be seen to have better crack propagation resistance than duplex or equiaxed microstructures, leading to the microstructural preference in γ -(TiAl) based alloys to be lamellar. However, crack propagation rate in γ -(TiAl) based alloys is so high that once a crack has nucleated it has no meaningful life despite the microstructure, so threshold is the key consideration.

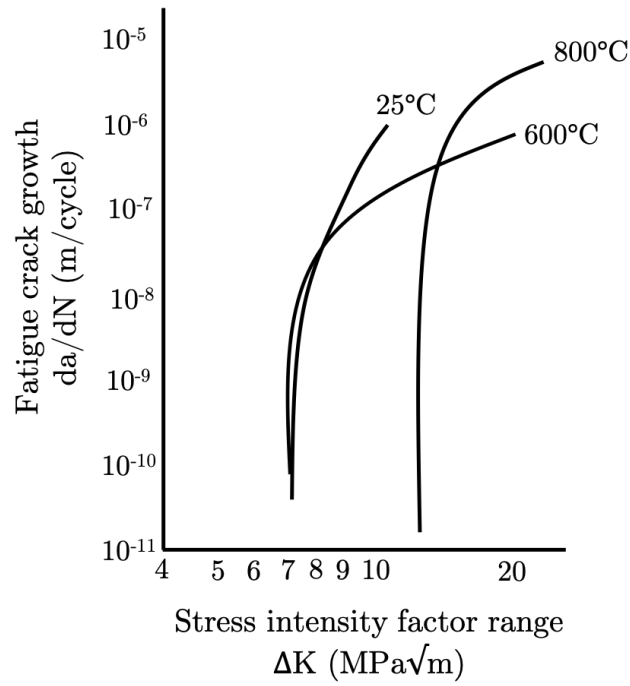


Figure 2.32: Fatigue crack growth curves for the same γ -(TiAl) based alloy at 25°C, 600°C and 800°C. Figure adapted from [125]

2.7 Summary

In this literature review, the rationale for the insertion of γ -(TiAl) into jet engines was first examined, followed by a discussion of solidification processing. The phase metallurgy, crystallography and deformation mechanisms were then reviewed. Next, the topic of fatigue was introduced. Alloy production, contrasting cast, near-net shape cast and forged blade production routes were then discussed. Finally, the fatigue performance of these different microstructures were reviewed. The over-riding finding is that despite their very limited ductility, γ -(TiAl) alloys can be relatively tough. However, their fatigue strengths are relatively poor and their fatigue crack growth rates are very high. Therefore it is imperative that cracks not be allowed to initiate and propagate, making the fatigue crack growth threshold of critical importance. This threshold is strongly microstructure dependent and suffers a minima at temperatures of around 400 - 600 °C in air. Because it is difficult to make long crack specimens in microstructures representative of γ -(TiAl) near-net shape castings, sub-size corner crack specimen designs will be utilised in this project. These can make pre-cracking difficult for normal fatigue threshold determination. Therefore the first part of this work will examine the effect of the initial notching

procedure and size on threshold, followed by examining the effect of colony orientation, and of temperature excursions. The sorts of microstructural features observed on fracture surfaces will be surveyed for the benefit of, *e.g.* the failure investigation of ex-service components.

Chapter 3

Characterisation Methodology and Material

3.1 Material

Cast bars of alloy Ti4522-XD were received from Rolls-Royce plc., Figure 3.1. These bars were near-net shape cast and underwent hot isostatic pressing (HIP) to reduce porosity. The composition, Table 3.1, was measured by IncoTest using ICP-OES; the gases oxygen and hydrogen were measured by combustion analysis. The alloy contains approximately 44 at.% aluminium, 2 at.% manganese, 2 at.% niobium and traces of silicon, iron, oxygen and hydrogen. The boron content is approximately 1 at.% and is found in this alloy as TiB_2 , which is equivalent to 0.8% volume fraction TiB_2 . Silicon in solution enhances creep resistance in Ti_3Al [126], restricting easy deformation by primary creep [127]. Manganese is added to improve ductility [128].

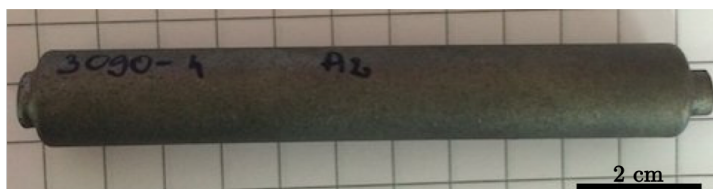


Figure 3.1: The as-received near-net shape cast bars of γ -(TiAl) from Rolls-Royce plc..

Element	Ti	Al	Mn	Nb	B	Si	Fe	O	H
Weight %	62.4	30.2	2.5	4.5	0.3	0.01	0.06	0.06	<10ppm
Atomic %	51.4	43.9	1.8	1.9	0.9	0.19	0.04	0.14	

Table 3.1: The alloy composition of the as-received material in weight and atomic percent measured by IncoTest.

3.2 Microscopy techniques

Optical microscopy can reach a maximum resolution of approximately 250 nm. In order to characterise surface of materials at an increased resolution, electron microscopy must be used. Electron microscopy exploits the decreased wavelength of electrons, 1.23 nm, as opposed to the wavelength of light, 550 nm. Scanning electron microscopy is the rastering of the electron beam across the surface to produce a signal and generate an image.

3.2.1 Secondary and backscatter electron microscopy

Scanning electron microscopy can utilise the interaction of the incoming electrons with the material surface in different ways to generate images. Incoming electrons from the beam can excite the electrons in the material, providing enough energy for the electrons to escape the material. This is secondary electron imaging, Figure 3.2(a). This enables a user to more precisely resolve a material surface, both polished flat and with surface topography, such as a fracture surface. Opposed to the excitation and liberation of electrons, incoming electrons from the beam may be deflected, which is backscatter electron microscopy, Figure 3.2(b). The incoming electrons deflect varying amounts, depending on the interaction atom, with heavier nuclei causing a brighter signal. Due to backscatter requiring the incoming electron beam to penetrate deeper into the material to reach the nucleus, there is decreased resolution with this technique. However, this technique enables the user to easily differentiate between phases with varying masses due to the brightness. This was carried out on the LEO Gemini 1525 FEGSEM.

3.2.2 Electron backscatter diffraction

Electron backscatter diffraction (EBSD) is another method of analysing the signal from the backscattered electrons. Most materials are crystalline in nature in the solid state. This crystal

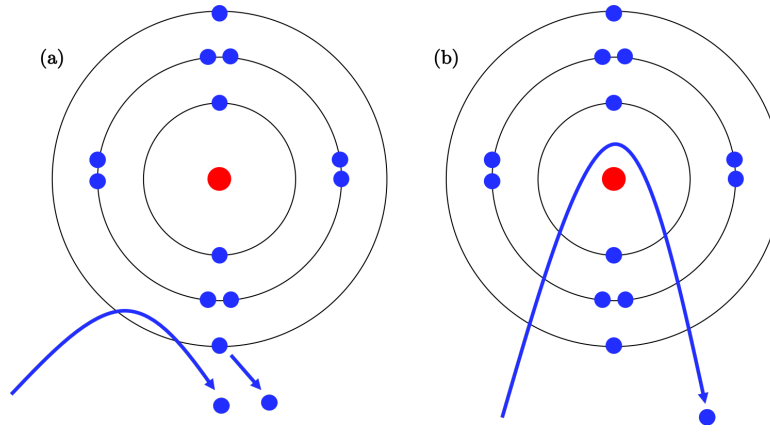


Figure 3.2: Schematic of electrons interacting with material; (a) inelastically as secondary electron emission, (b) elastically as backscattered electron emission.

can be rotated in relation to its lattice, which is described as its orientation. When an incoming electron beam scans the surface of the material, the backscattered electrons can be collected to determine the orientation of the crystalline material. A small area is analysed for this each time, creating a pixellated image of the orientations of the structure of the material. The resolution of this technique is therefore determined by this pixel size, as well as the aperture and the applied accelerating voltage. The sample is tilted to 70° to optimise outgoing electron collection, due to decreased path length. The electrons are backscattered in the sample, and scatter relative to the angle of the relevant crystal plane. When the electrons are at the crystal plane less than that of the Bragg angle, they scatter elastically. The diffraction of these electrons occurs in different directions depending on the channelling conditions, causing them to spread to the surface of a cone. These are then collected by a scintillator and lens onto a charge coupled device (CCD), Figure 3.3. The generated patterns are called Kikuchi patterns, which can be further analysed to generate an image. When the coordinates of the pattern centre, the specimen-to-screen distance and the relationship between the microscope stage and camera are known, the EBSD pattern can be indexed. Fast algorithms are able to resolve patterns which allows automatic orientation measurements within a fraction of a second. In this thesis, EBSD was also carried out on unprepared fracture surfaces. This was possible when the features were flat and with no blocking of the electron beam. The microscopes used for this process were the Zeiss Auriga Cross Beam and Zeiss Gemini Sigma 300.

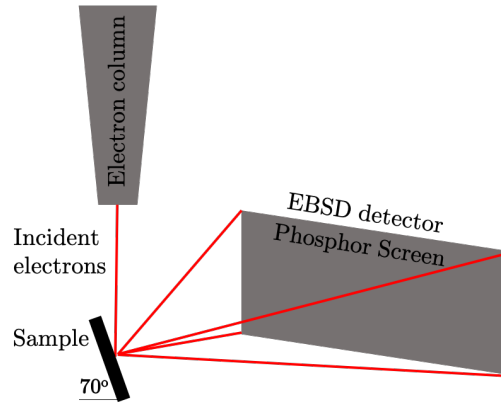


Figure 3.3: Schematic of EBSD microscopy setup and detection with sample at 70° tilt to the incident electron beam.

3.2.3 Transmission electron microscopy

Transmission electron microscopy (TEM) is a microscopy technique which allows the incoming electron beam to pass through very thin samples as opposed to excitation and emission or deflection. It permits a user to resolve a material to atom scale by generating diffraction patterns from the transmitted electron beams. The microscope used for this was a JEOL 2100Plus. Bright-field TEM imaging is the selection of the direct electron beam for imaging, and the exclusion of the diffracted electron beam, whereas in dark-field imaging the direct electron beam is excluded from the image, and the diffracted beam is selected.

3.2.4 Focussed ion beam milling

A focussed ion beam (FIB) is a stream of ionised atoms, most commonly gallium or xenon, which can be used to mill away a material surface. These ionised atoms scan the surface much like an SEM stream of electrons. The microscope used for this was a FEI Helios Nanolab 600 DualBeam system.

3.2.5 Slice-and-view technique

Slice-and-view technique combines the SEM and FIB to remove a layer of material, and then image the sample repeatedly to generate a 3D characterisation of the microstructure. This was carried out on a Zeiss Auriga Cross Beam FEGSEM fitted with gallium ion FIB.

3.3 Specimen preparation

To be able to image the material with the techniques described above, the samples must be prepared. The preparation method varies depending on the technique being used.

3.3.1 Scanning electron microscopy

For imaging of the microstructure with the scanning electron microscope (SEM), the samples needed to be ground with a series of grinding papers and then polished. The grinding and polishing was carried out on a RotoPol-15 or Tegramin-20. For the grinding step a series of SiC papers with increasing roughness from P500 to P4000 were used. After each grinding step, lasting about 1 minute, with a minimal force of 5 or 10 N, the samples were cleaned with water and washing liquid to remove grinding particles or debris. The final polishing step was carried out with OP-S, which had been neutralised (70 ml colloidal silica, 30 ml H_2O_2 and 100 ml H_2O), for two hours. When the final polishing step was complete, the samples were etched with Kroll's reagent (92% H_2O , 6% HNO_3 , 2% HF), then cleaned in the ultrasonic bath with butanol for ten minutes, then distilled water for ten minutes to reveal the microstructure under an optical microscope.

When viewing a fracture surface in the SEM, the sample was cleaned in the ultrasonic bath with butanol and distilled water as above, then left to dry in the desiccator for 24 hours.

3.3.2 EBSD

In order to analyse the samples with EBSD they were ground and polished as above for the SEM. EBSD requires a good surface polish due to the backscattered electrons being generated in the surface layer.

To target specific sites perpendicular to a fracture surface, prior preparation is required. The fracture surface was protected by coating with several layers of Microstop and left for 24 hours to dry. The sample was then cut on the Struers Accutom-5 or Secotom using a diamond saw with a low feed speed of 0.005 mm/s, a known distance from the target site, which would then be the depth the polishing process would remove. Fracture surfaces were polished perpendicular

to the fracture surface, again using OP-S, in preparation for SEM and EBSD. Once polishing was complete the Microstop could be removed using methyl ethyl ketone (MEK) for 24 hours, and then cleaned as before in the ultrasonic bath with butanol and distilled water. This process can be seen in Figure 3.4.



Figure 3.4: Fracture surfaces were dipped in Microstop and left to dry before being sectioned and polished. The Microstop was then removed and the sample fixed to a stub for microscopy. Images courtesy of Tom Zalewski, Rolls-Royce plc..

3.3.3 TEM

TEM requires the transmission of electrons through the sample. Due to the absorption of electrons in metals being very high, the sample is required to be thin, and therefore more electron transparent. These samples can either be obtained to target a specific site, or on a random sample of material. For TEM on a random sample of the material, thin slices of material were cut using Struers Accutom-5 or Secotom. The thicknesses of the slices were below 500 μm but still needed to be ground further to a thickness of around 250 - 300 μm . A polishing paper with fine particle size was used, to reduce the damage introduced into the surface. 3 mm holes were then punched through this by EDM spark erosion. The discs were then further thinned to a thickness of 120 - 150 μm by polishing. After careful cleaning with methanol, the final preparation step was electropolishing with a TenuPol-5. A mixture of 3% perchloric acid, 57% methanol and 40% butanol, cooled with liquid nitrogen to a temperature of -50°C was used for etching. The potential at the sample was set to 20 V. During the polishing, the centre of the disc experienced the most material removal, slowly creating a hole. An exposure meter measured the amount of light that passed through the sample and stopped the process at a set threshold. The material around the hole is sufficiently thin for transmission electron microscopy.

When the polishing stopped, the samples were washed repeatedly with deionised water to stop any further etching.

3.4 Characterisation

The microstructure of the as-received cast bars is shown in Figure 3.5. This shows a duplex microstructure with high volume fraction of coarse lamellar, prior β grains. Within the prior β grains randomly oriented colonies of γ -(TiAl) and α_2 -(Ti₃Al) were obtained, 40 - 100 μm in size, averaging 69 μm , consisting of 92% area fraction of γ -(TiAl) lamellae. In Figure 3.5, γ is darker in colour and 0.5 - 3.6 μm in lath width, averaging 1.4 μm , often with several laths together. The γ is interleaved with thin ligaments of α_2 , lighter in colour, 0.2 - 0.5 μm in plate width, averaging 0.3 μm [129]. TiB₂ appears as ribbons usually on colony boundaries, but also sometimes within colonies.

For microstructural characterisation, a metallographic sample was prepared using standard procedures, then polished with neutralised OP-S and etched using Kroll's reagent to expose the microstructure. The dual phase nature of the as-received material can also be seen in the bright-field TEM image in Figure 3.6. This shows the layered lamellae of the two phases, γ — α_2 — γ . On the right side of the TEM image, the γ -(TiAl) lamellae can be seen, with vertical dislocations. This is due to crystal alignment at lamellae interfaces, where the opposing crystal lattices find the lowest energy state which often causes serration [130].

This TEM sample was created by standard mechanical polishing and twin jet electropolishing.

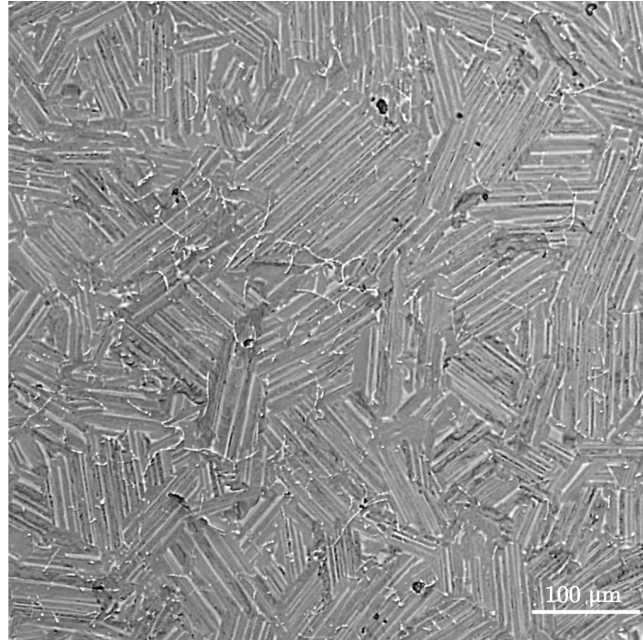


Figure 3.5: An optical micrograph of the microstructure of the as received γ -(TiAl) showing the coarse lamellar microstructure and the colony size in the range of 40 - 100 μm .

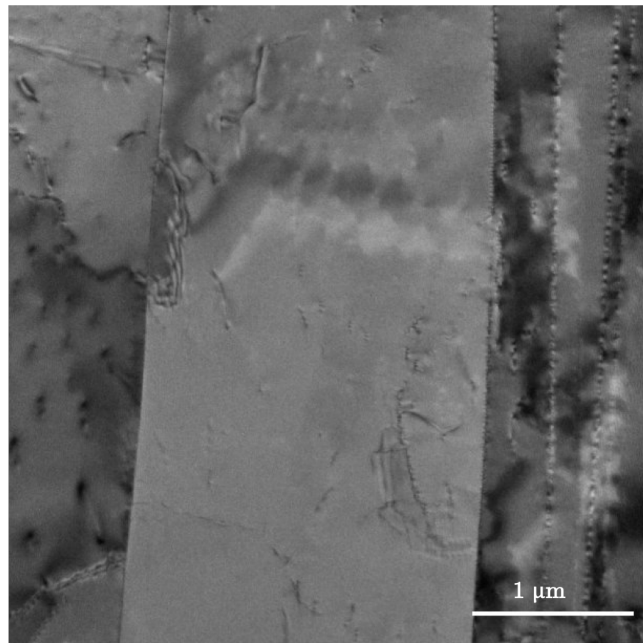


Figure 3.6: Bright field TEM image of the dual phase structure of α_2 -(Ti₃Al) and γ -(TiAl) is visible, and vertical dislocations between lamellae interfaces.

Chapter 4

Fatigue Testing Methodology

4.1 Fatigue samples

The cast bars were machined to form M12 threaded, 5 x 5 mm square section specimens with a gauge length of 15 mm and a machined corner notch, Figure 4.1. The specimen gauge lengths were then hand polished using neutralised OP-S. In the corner crack specimens, notches were created in the central 1 mm region of the gauge with a diamond saw. The specimens were then cleaned in an ultrasonic bath with butanol, and then with distilled water.

Fatigue in brittle materials is investigated under cyclic loading. With an increase in the stress intensity, K , the fatigue induced crack can be seen to advance. Below a given stress intensity threshold, ΔK_{th} , there is no progression of crack growth. In a dual phase material, cracks are more likely to form first in the less brittle, softer, material [131]. The crack after this can be seen to grow linearly on a log-log plot, before rapid failure. Fatigue crack propagation is commonly investigated by monitoring the crack growth rate per cycle, da/dN , against the change of stress intensity, ΔK .

4.2 Crack propagation measurement

Crack growth can be measured in multiple different ways, grouped into two categories. This can be either measuring the crack directly, or indirect measurement, through measuring material

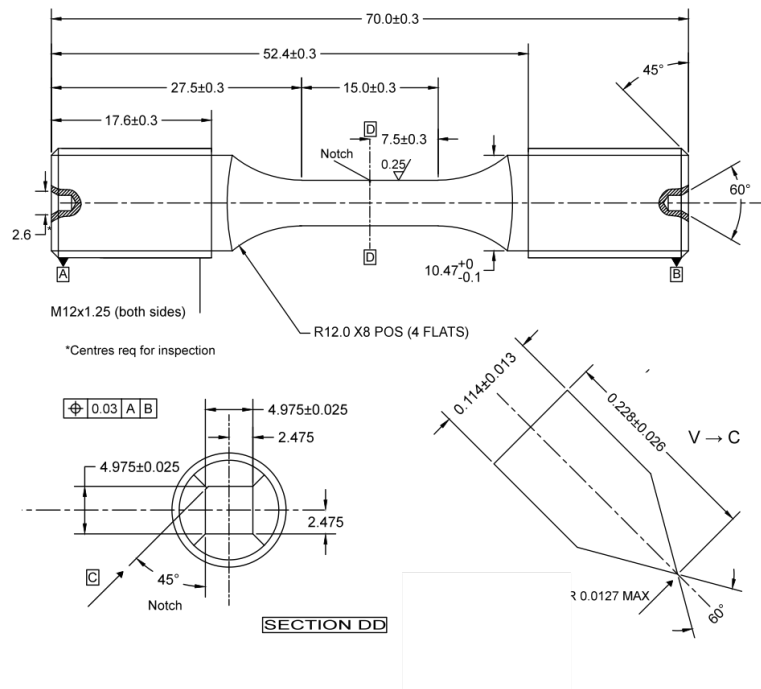


Figure 4.1: M12 Corner Crack Specimen with central 5 x 5 mm central gauge and notch size 0.25 mm, all dimensions are in mm.

properties to determine the crack length. Direct measurement of the crack requires visibility of the crack throughout the experiment, *e.g.* using a travelling microscope. This may be improved by performing a test within a SEM. However, the measured side crack length may not be directly relatable to the actual crack length at the crack front, or the side of the specimen may not be accessible to view directly.

Direct methods of measuring crack growth include *in situ* methods: visual, replicas, photographic or strain gauge measurement. *Ex situ* methods include: beach marks, striations, ink

Direct	Indirect
<i>In situ</i>	Resistivity
Visual	AC, DC potential drop
Replicas	Compliance
Photographic	Displacement
Strain gauge filament	Strains
<i>Ex situ</i>	Others
Beach marks	Acoustic emission, microscope
Ink stains	Ultrasonic
Heat tinting	Laser interferometry
Striations	Eddy current

Table 4.1: Methods of measuring the crack growth by direct and indirect methods [132].

stains and heat tinting. Indirect methods of crack growth measurement include resistivity, compliance, laser interferometry, and others, Table 4.1. Beach marking is the method of producing artificial marks on the fracture surface during testing by altering the crack tip plastic zone size, including introducing overloads or changing the R ratio. Marks may also be created on the fracture surface by heat tinting, changing the test temperature and holding the test at intervals to create a thicker oxide scale. Striations may also be used to measure local crack propagation rates. It may be assumed that in the Paris regime one striation is equal to one cycle [75]. Direct measurement of crack length is usually difficult.

Potential drop is usually used to monitor crack length in long crack specimens when fatigue loaded. This method is better than monitoring the crack progressions optically, as optically only allows the measurement on the material surface and solely in one dimension. However, a camera was set up in order to investigate whether measuring crack length optically was a possibility. This, however, is not possible when the experiment is carried out above room temperature due to the furnace being wrapped around the specimen. Currents can be either direct current (DC) or alternating current (AC). For DC, large currents are passed through the sample and only small potentials of a few microvolts are measured. For AC, currents are generated by induction below the sample surface, increasing the resistance of the material. With a larger initial resistance the applied currents are generally smaller when using alternating current. The most common method of potential drop is putting a constant current through the sample, and measuring the potential change as the crack grows due to the increased path of measurement. Other direct techniques involving acoustic emission or ultrasound are emerging but are not used routinely.

4.3 Potential drop experiment

Throughout all testing, crack growth was measured indirectly using direct current potential drop (DCPD). A constant current is placed over the sample, and the potential drop across the crack is measured. Potential (V), current (I) and resistance (R) are related by Ohm's law. The resistance is dependent on the materials resistivity (ρ), length of the conductor (l) and

conducting area (A):

$$\Delta V = I.R = \rho \frac{l}{A}.I \quad (4.1)$$

The current (I), resistivity (ρ) and length of the conductor (l) remain constant. The above equation shows that as the crack grows, and therefore the conducting area (A) of the sample decreases, the potential must increase. Although the method is based on simple physical principles, the instruments must be capable of measuring a very small potential of several tens of micro volts. Potential drop is an established method for crack growth measurements and described in standards [86]. The voltage can be related to a crack length with aid of calibration curves, a relationship between an individual material's potential drop across a crack and the crack length.

4.3.1 Set up

The sample used for potential drop was a corner crack specimen as specified above. Platinum wires with diameter 0.063 mm were spot welded either side of the notch within a range of 550 - 800 μm . Another pair of Pt wires of the same diameter were spot welded 1 mm below to provide a reference voltage. These were connected to a sensitive voltmeter. Thicker Pt wires of 0.5 mm diameter were spot welded to the top shoulder and bottom shoulder in order to supply the current of 5 A across the sample. The current was provided and the voltage was measured by a Matelect DCM-2. A furnace was wrapped around the specimen and the spot welded wires. The specimen was connected to a servo hydraulic mechanical test frame, MAYES 100 kN, with an Instron 8800 controller. The measuring equipment is able to resolve small changes of 0.05 to 0.50 μV . The stability of the current supply should be equal to the accuracy of the voltage measurement system. Moreover, all electrical equipment should be grounded. The metal grips and test frame offer an alternate way for the current to flow. It should therefore be ensured that the resistance through the grips is several orders of magnitude larger than the resistance through the sample. The current input wires can be mechanically fastened or welded to the sample. For the voltage reading, the wires should be as fine as possible to allow positioning

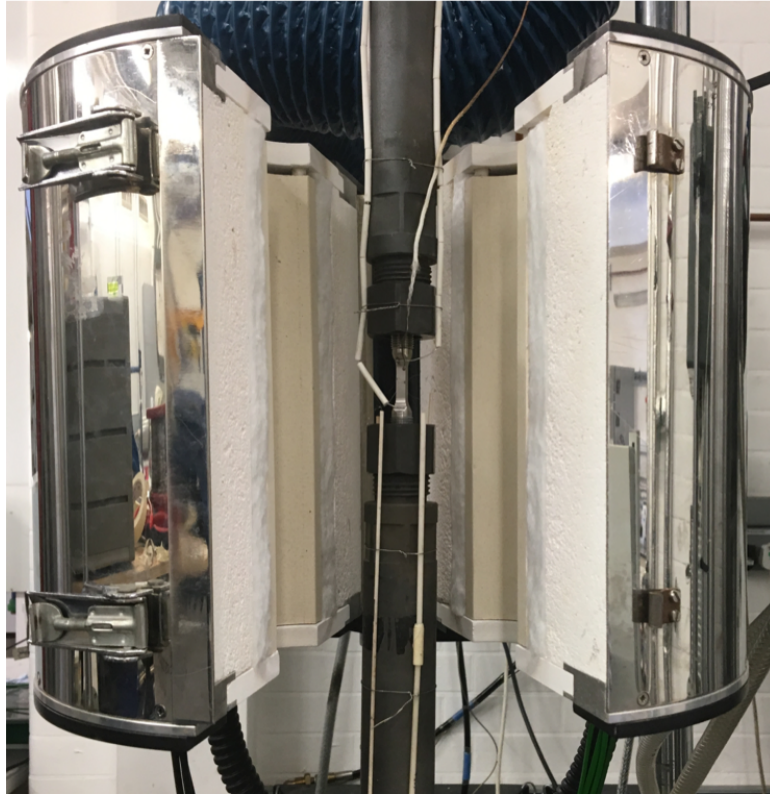


Figure 4.2: Set up of M12 corner crack specimen with 0.063 mm Pt wires spot welded either side of the notch, and as a reference 1 mm below the notch.

close to the crack, Figure 4.2.

All tests were carried out at a frequency of 5 Hz and R ratio of 0.1. Threshold was determined as later described and the test then continued under this load until final failure. Each data point is an average value of readings taken over 30 seconds taken at the mean stress, so as to measure with the crack tip open. A LabView script was written for the data acquisition. The PD readings were recorded via serial data from the potential drop meter, Figure 4.3. In addition, the analogue force and displacement signal from the test frame were logged and used to synchronise the data acquisition with the mechanical test. The data were collected after set intervals of fatigue cycling, kept constant throughout. To relate the potential drop to crack length calibration needs to be carried out.

The standard [133] recommends two methods of plotting the crack growth data, secant or polynomial. The secant method manually bins the data. The polynomial method uses minimal manual filtering, but fits a polynomial function to seven points in Fortran script. Both methods were tried, but the secant method was found to give a better representation of the data.

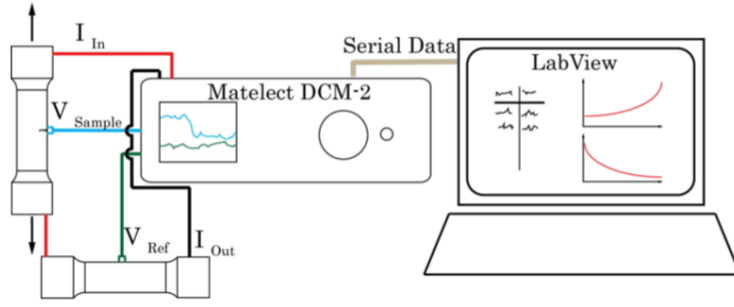


Figure 4.3: Sketch of principle test setup showing the voltage reading of the test and reference sample using a Matelect DCM-2. A LabView program controlled and recorded the PD meter. Image courtesy of Peter Tympel.

4.3.2 Threshold measurement

Threshold is defined as the point at which crack growth exceeds 1×10^{-9} m/cycle [75]. Methods for finding threshold include the K -decreasing technique (or load-shedding), step increase of load amplitude on specimens pre-cracked in cyclic compression and step increase of load amplitude on specimens with an initiating feature [134].

The conventional K -decreasing method is carried out to generate a naturally sharp crack from a machined starter notch, by (I) starting at ΔK above threshold, *e.g.* $10 \text{ MPa}\sqrt{\text{m}}$. The crack is then grown for a set number for cycles to a known ΔK , *e.g.* $12 \text{ MPa}\sqrt{\text{m}}$. Load is then shed incrementally (II) until the crack growth is measured to be less than threshold, 1×10^{-9} m/cycle. Once threshold has been found, the crack can then be grown until failure (III), Figure 4.4.

Measuring the fatigue crack growth threshold ΔK_{th} is difficult using the conventional K -decreasing method, due to this method requiring too much crack growth during pre-cracking, also crack growth is too fast in γ -(TiAl) for this method to be possible. Therefore, gradual increases in load were instead applied until threshold was found. This meant that sensitivity to the initial notch conditions needed to be investigated, Chapter 5.

Threshold was determined using incremental increases in ΔK until a crack growth rate of between 10^{-10} and 10^{-9} m / cycle was observed. Starting with a stress intensity factor range of $3 \text{ MPa}\sqrt{\text{m}}$, 2000 cycles were first applied at an R ratio of 0.1. The stress intensity factor was then increased by $0.1 \text{ MPa}\sqrt{\text{m}}$ should the potential drop not increase more than $3 \mu\text{V}$ within these cycles. This was continued until this potential drop was observed. The stress intensity

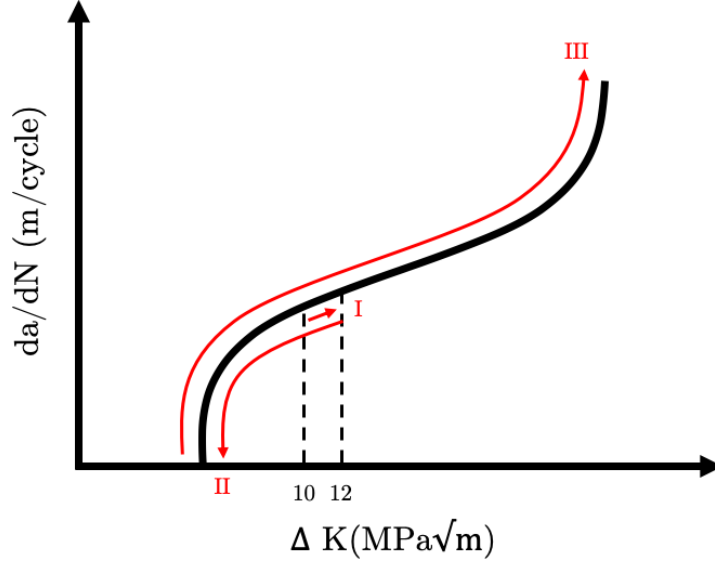


Figure 4.4: Stages of the K -decreasing method on a schematic fatigue crack growth curve. The crack is initially grown at ΔK above threshold, *e.g.* $10 \text{ MPa}\sqrt{\text{m}}$ for a set number of cycles. The load is then shed to find threshold, and the crack is grown out to failure.

applied at this ΔK was then applied throughout the test to finish, to grow the crack out to final failure.

4.3.3 Calibration

In order to relate the potential drop measurement to the crack length, calibration needs to be carried out. Calibration curves can be generated with analytical solutions or calibration samples. Analytical solutions are available for a few simple sample shapes [135].

Carrying out calibration on samples requires creating a feature in the fracture surface at a known potential drop several times. This then allows a plot of crack length *vs.* potential drop to be drawn and therefore calibration of the relationship between the crack length and potential drop. Conventional methods to create features on the fracture surface are the same as those used to measure crack growth, as shown in Table 4.1.

Due to γ -(TiAl) not forming striations when undergoing an overload, this could not be used as a method to calibrate the crack length to the potential drop measured, and therefore another less common method had to be used. The calibration was therefore carried out using heat tinting of the fracture surface. This was carried out at time intervals in order to allow

Test no.	No. of data points	Method	Held at
1	1	400°C for 3 hours after 29 μ V growth, 400°C for 1hr 20min after 40 μ V growth, 400°C for 1hr after 40 μ V growth.	Mean load
2	3	650°C for 4 hours after 60 μ V growth, 550°C for 4 hours after 170 μ V growth, Overload after 70 μ V.	Max load
3	3	650°C for 4 hours after 78 μ V growth, 550°C for 4 hours after 79 μ V growth, Overload after 80 μ V.	Max load

Table 4.2: The methods used to generate features on the fracture surface to create a calibration curve.

the sample to oxidise, leaving ‘beach marks’ of decreasing oxidation thickness. The specimen was held at mean load so to hold open the crack to allow oxidation. The fracture surface then has concentric rings of varying colour due to oxidation to relate crack length to potential drop. Testing was carried out at 400°C (as for the fatigue tests) with a standard machined notch size of 0.5 mm, therefore heat tinting was first carried out by holding the crack open at mean load after a pre-determined crack growth. This was done on one sample. Due to the lack in variation of oxidation colour, only one oxide concentric ring could be determined. In two further samples heat tints were therefore carried out at varying temperatures in order to obtain more data points from each sample, and held at maximum load in order to open the crack enough for the oxide film to form. These following two samples were heat tinted at 650°C and then 550°C, Table 4.2. Attempts were also made to try to obtain data points by overloading the sample towards the end of the test. Seven data points were obtained from these varying methods, this was enough to generate a calibration curve.

The fracture surfaces of the calibration samples from heat tinting can be seen in Figure 4.5, showing the blue concentric crack fronts of oxide films. The standard [1] dictates measuring at 10° intervals starting at the edge, to give 10 lengths at 10° intervals to be averaged to find the average crack length around the crack front. The datum point for zero potential drop can also be included, with the crack length of the starter notch to give an additional datum point. The calibration curve can then be fitted to relate crack length to the potential drop, Figure 4.6. The coefficient of determination, R^2 , was used as a measure of statistical variance and was found to

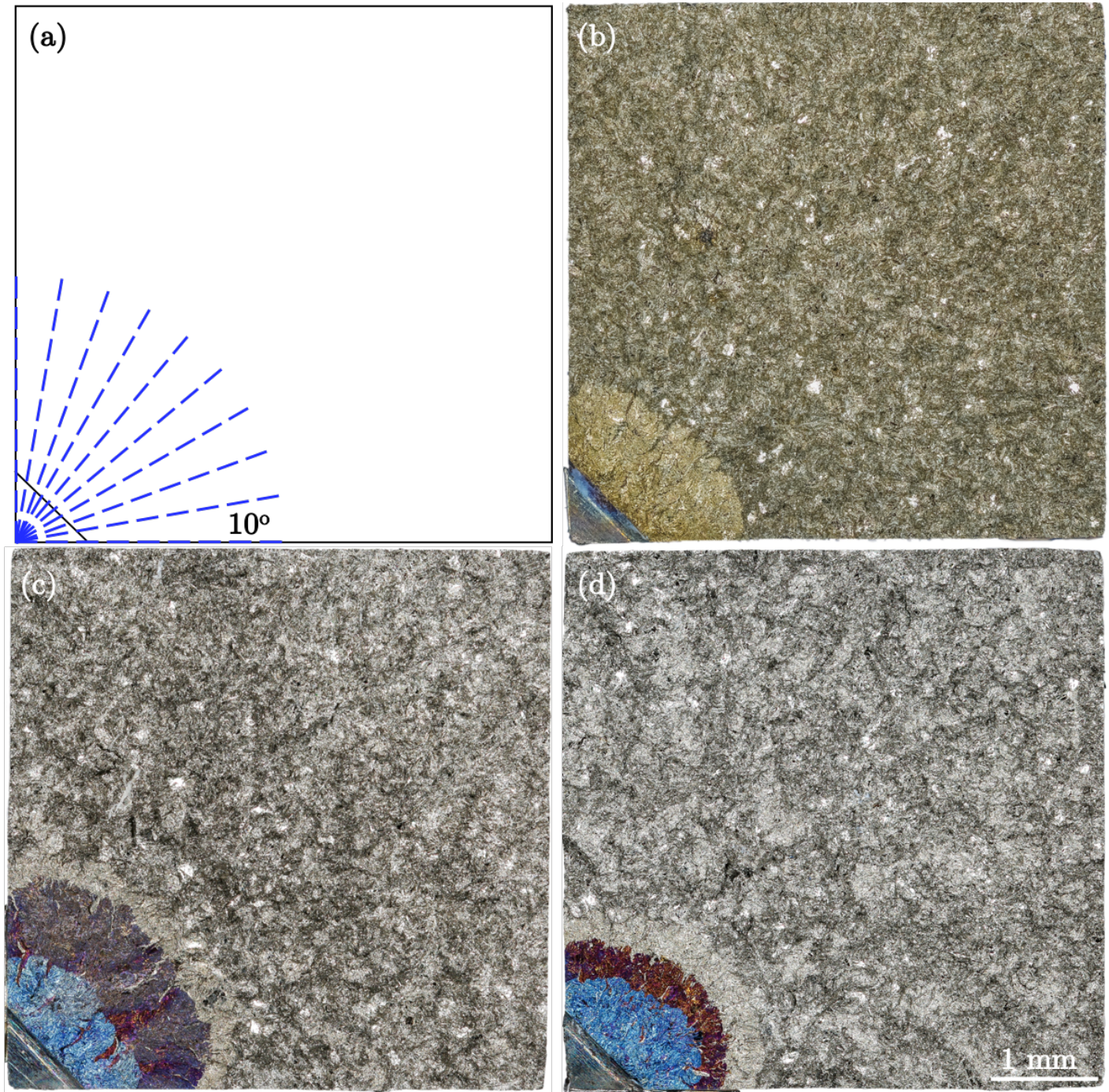


Figure 4.5: (a) Measurements of crack front taken every 10° and averaged. Fracture surfaces of (b) calibration 1 with only one data point (c) calibration 2 with three crack fronts (d) calibration 3 with three crack fronts. Photography courtesy of Failure Investigation, Derby, Rolls-Royce plc..

be 87%. The equation for the calibration curve is:

$$\text{Crack length(mm)} = 5.5035 \times 10^{-3} \times \Delta V(\mu\text{V}) + \text{Initial notch size(mm)} \quad (4.2)$$

This calibration curve was carried out at 400°C, but throughout the testing higher temperatures of 750°C were also tested. The same calibration curve has been applied. Application of the calibration curve to different notch sizes requires adjustment of the y -intercept to the notch depth. The calibration curve was assumed to be valid across all tests.

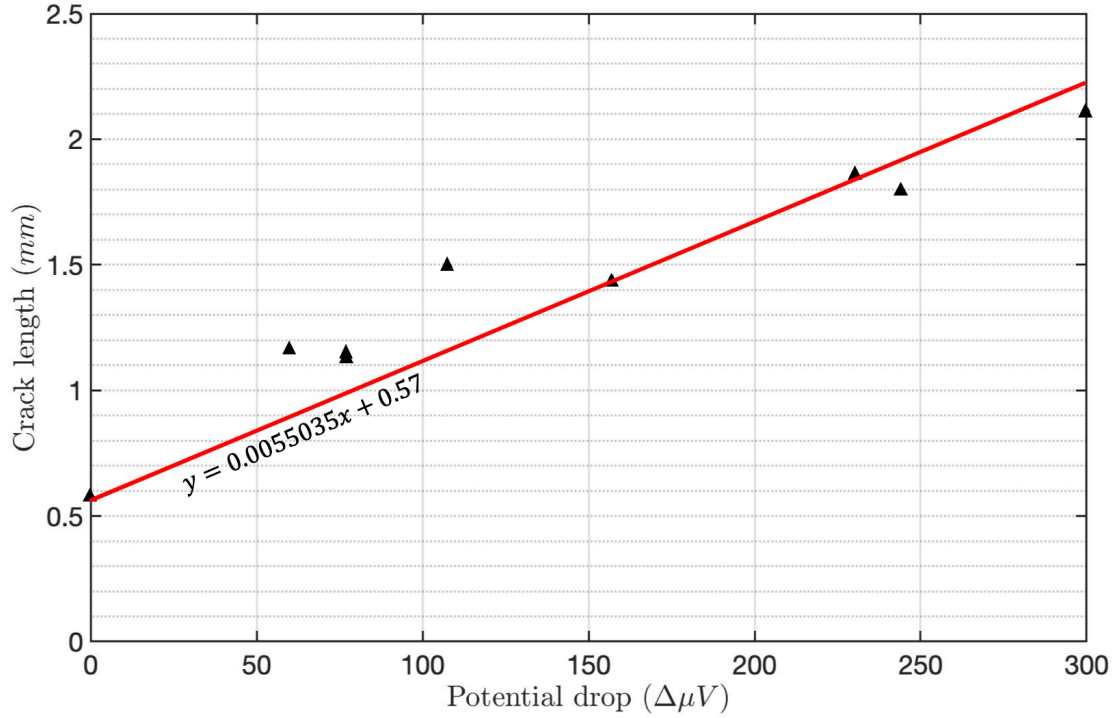


Figure 4.6: Data points from calibration samples and the zero datum point, to give equation for calibration curve.

4.3.4 Experimental errors

The factors affecting the resolution of DCPD include: current used, Pt wire spacing and specimen geometry. Other factors affecting the DCPD include: material sensitivity, thermally induced electrical potential and crack closure. Platinum wires were used as the probes to measure the potential drop in an attempt to minimise the issue with differences in the materials. Crack closure was minimised as tests were carried out at temperature in the presence of air and therefore an oxide film being formed, preventing crack closure, and the use of a positive R ratio.

Keeping the temperature throughout the test constant, and allowing temperature to stabilise before testing reduces the impact of the thermal effects on the DCPD. This removes effects from

thermal gradients in the sample. Measuring the reference voltage across the specimen without a notch also allows any fluctuations in voltage occurring to be detected. The reference should be located somewhere not influenced by the growing crack, but at a similar location on the gauge, so that it is under similar conditions, *i.e.* temperature.

The potential drop is measured in between intervals of fatigue where the sample is held at mean load. The number of cycles in each interval, or wavepacket, can be changed. However, it is not possible for the servo-hydraulic frame to reach minimum and maximum load on the first wave in that packet, and therefore there is a taper applied to the beginning of that wave packet. Should the number of sinusoidal waves in the packet applied change throughout testing, then errors to the cycle count would be introduced.

4.3.5 Stress intensity calculations

The calculations for the change in stress intensity factor ΔK for the corner crack specimen used throughout this thesis can be found in standard [86]. ΔK for the corner crack specimen is a function of the relevant compliance function y_{45} , crack length a and the change in stress applied $\Delta\sigma$:

$$\Delta K = \Delta\sigma \times y_{45} \times \sqrt{\pi a} \quad (4.3)$$

The geometry factor for the general position y_n can be calculated by:

$$y_n = M_G \times M_B \times M_S \times \frac{2}{\pi} \quad (4.4)$$

where M_G is the general correction factor, M_B is the back face correction factor and M_S is the side face correction factor. It follows from standard [86], that when the crack length a is less than 20% of the total specimen width W , *i.e.* $a \leq 0.2W$:

$$M_G = 1.143 \quad (4.5)$$

$$M_B = 1 + 0.06 \frac{a}{W} \quad (4.6)$$

$$M_s = 1 + 0.07 \frac{a}{W} \quad (4.7)$$

whereas for $a \geq 0.2W$:

$$M_G = 0.1 \left(\frac{a}{W} \right)^2 + 0.29 \left(\frac{a}{W} \right) + 1.081 \quad (4.8)$$

$$M_B = 0.75 \left(\frac{a}{W} \right)^2 - 0.185 \left(\frac{a}{W} \right) + 1.019 \quad (4.9)$$

$$M_s = 0.9 \left(\frac{a}{W} \right)^2 - 0.21 \left(\frac{a}{W} \right) + 1.02 \quad (4.10)$$

The compliance at the 45° position y_{45} for corner crack specimen is calculated according to the standard [86]:

$$y_{45} = y_n \left[0.9335 - 0.0045 \left(\frac{a}{W} \right) + 0.1295 \left(\frac{a}{W} \right)^2 - 0.4845 \left(\frac{a}{W} \right)^3 \right] \quad (4.11)$$

Throughout DCPD the variables measured are the number of cycles N , and the potential drop ΔV . The potential drop can be converted to the crack length via the calibration curve determined above, therefore the crack growth per cycle, da/dN , can be calculated. The change in stress intensity ΔK can be calculated as shown above, with the change in applied stress, $\Delta\sigma$, being a input determined in finding threshold. This enables N *vs.* ΔV to be converted into the familiar fatigue crack growth curve da/dN *vs.* ΔK , Figure 4.7.

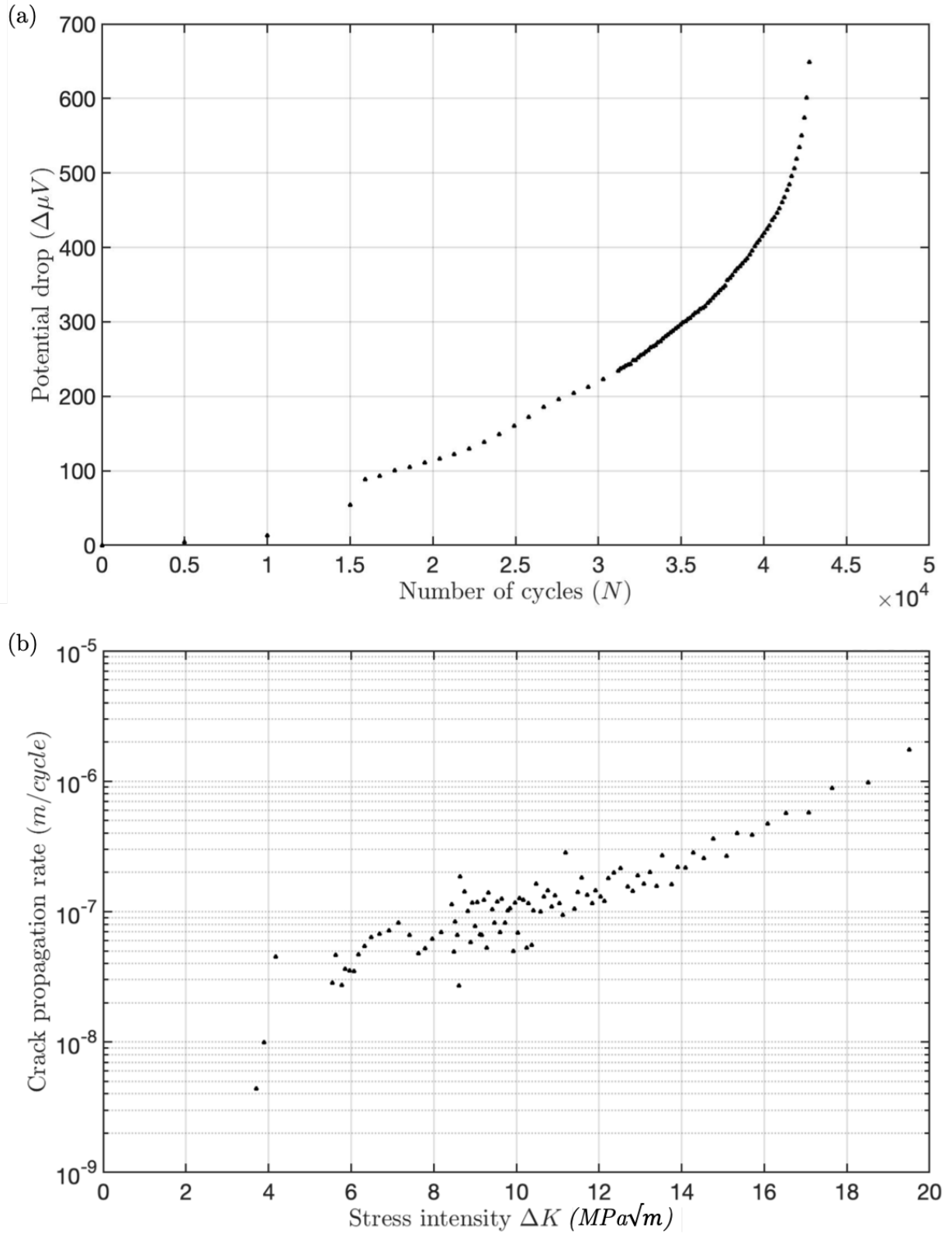


Figure 4.7: (a) Measured variables, number of cycles N and the potential drop ΔV can be converted to the fatigue crack growth curve (b) da/dN vs. ΔK .

Chapter 5

Effect of Notch Size

5.1 Introduction

γ -(TiAl) based alloys have long been attractive for use in jet engines due to their low density of around 4 g cm^{-3} , strength, oxidation resistance, and creep resistance at intermediate temperatures in the range of 450 - 700 °C [3]. This has prompted development efforts stretching back as far as 1953 [11], but only recently have this class of alloys begun to see service in civil jet engines. The limited ductility and relatively fast crack propagation of γ -(TiAl) make its use as a material for highly stressed components particularly challenging.

Investigation into fatigue crack growth, and therefore life determination, falls into three main categories: remaining below crack growth threshold, defect survival within one cycle at a given crack propagation rate, or remaining below the stress-life curve, as shown by (a), (b) and (c) respectively in Figure 5.1.

The fatigue crack growth threshold has been identified as a key material property for design, supported by an improved understanding of the deformation mechanisms associated with a crack growing near this threshold. For small initial defects, less than the plastic zone size, it is interesting to ask whether the initiating defect may affect the crack growth threshold. Many other factors also affect the stress intensity factor range at which a crack initiates, including temperature, residual stresses, local microstructure such as crystallographic orientation or initiating microstructural features, or any effect of environment on the crack, such as oxidation-

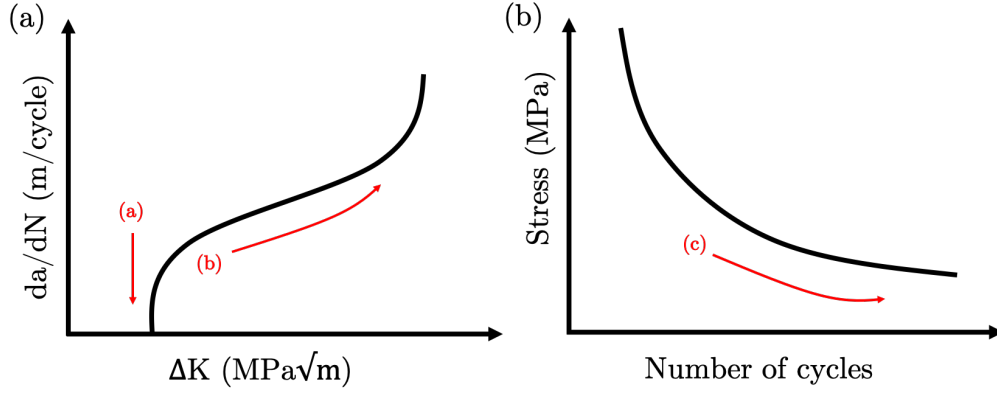


Figure 5.1: Life determination falls into three main categories: (a) remaining below threshold, (b) survival assuming an above-threshold starter crack, (c) remaining below the S-N curve on component-level tests.

induced closure [136][39]. The rate of crack growth, da/dN , may be affected by the initial crack size. There is the observation that small cracks, defined as those smaller than a few millimetres in length, often show crack growth rates much faster than those in longer cracks when the same ΔK is applied. Furthermore, cracks that are similar size to the microstructure exhibit a rate of crack growth which diminishes with an increase in crack length [75]. This is due to short cracks being more microstructurally dependent, as statistically a short crack is more likely to fracture through a homogeneous microstructure. The critical flaw size in γ -(TiAl) is typically small, and so its further investigation is crucial to component design [12].

5.2 Experimental methods

The material condition and preparation are detailed in Chapter 4. Corner crack specimens were notched with varying sized notches: 0.5, 0.25, 0.1, 0.06, 0.03 mm. The following notch sizes were machined by diamond saw: 0.5, 0.25, 0.1 and 0.06 mm. This was carried out external to Imperial College London, by PS Marsden Precision Engineering Ltd. The shape of the machined notches can be seen to form a U-shape, with a crack notch radius of $\sim 50 \mu\text{m}$, Figure 5.2(a). This was considered to be a sharp notch [134][137]. The smaller notch was made using a gallium Focussed Ion Beam (FIB) on FEI Helios Nanolab 600 Dual Beam system, as can be seen in Figure 5.2(b). The FIB notch was milled to replicate the geometry of the machined notches. However, the shape of the notch created using the Ga FIB can be seen to form a sharper V-shape, with

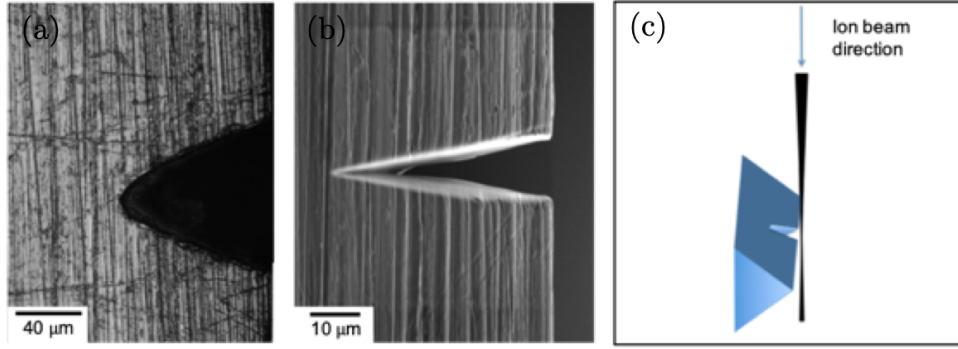


Figure 5.2: Notches machined in corner crack specimens: a) optical micrograph of 0.1 mm notch machined by diamond blade with U-shape, b) 0.03 mm notch milled using gallium FIB with V-shape, c) the geometry used to FIB the notch to limit gallium deposition in the specimen.

the crack notch radius only measuring a few microns. This must be taken into consideration when the influence of crack size on the fatigue crack growth threshold is investigated due to residual stresses at the crack tip. Ga ion FIB can also be seen to deposit gallium in the sample when milling. When the FIB notch is milled using the Ga ion beam, the geometry was taken into consideration in order to minimise the elemental gallium traces deposited in the specimen, Figure 5.2(c), as it is thought that Ga deposition in the notch may have an effect on the results of the study.

Fatigue testing was carried out on these notched corner crack specimens at R ratio 0.1, 5 Hz and 400°C, and the crack length measured using the DCPD method.

Due to the imprecision of diamond blade notching, notches machined to the same specification gave variation. The notch length on each specimen was therefore measured using an optical microscope after being polished with OP-S and cleaned in the ultrasonic bath before each fatigue test in order to accurately apply the correct load and therefore stress intensity. This was carried out by measuring the notch length on the two sides of the corner crack specimen x and y , and using basic geometry, *i.e.* Pythagoras' theorem, to determine a , Figure 5.3.

Crack growth threshold is defined as the point at which crack growth exceeds 10^{-9} m/cycle [75]. This was not always possible to measure and so the fatigue crack growth curve was extrapolated down from the data to this growth rate. In Figure 5.4, the crack growth rate has a minimum of 4.4×10^{-9} m/cycle. The vertical dashed red line shows the extrapolation down of this data to 10^{-9} m/cycle.

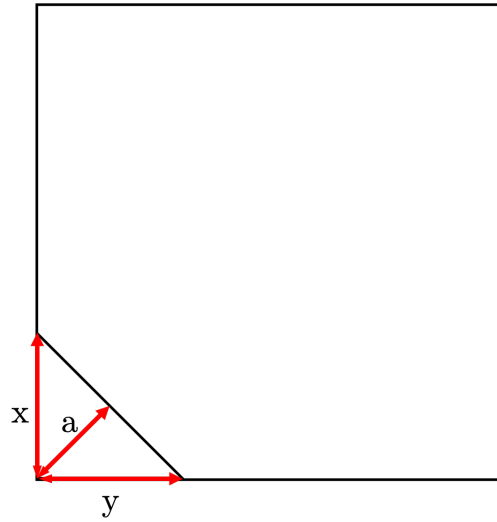


Figure 5.3: Schematic of cross section of central 5 x 5 mm gauge of corner crack specimen, measuring notch length, a , using distances x and y , measured on an optical microscope.

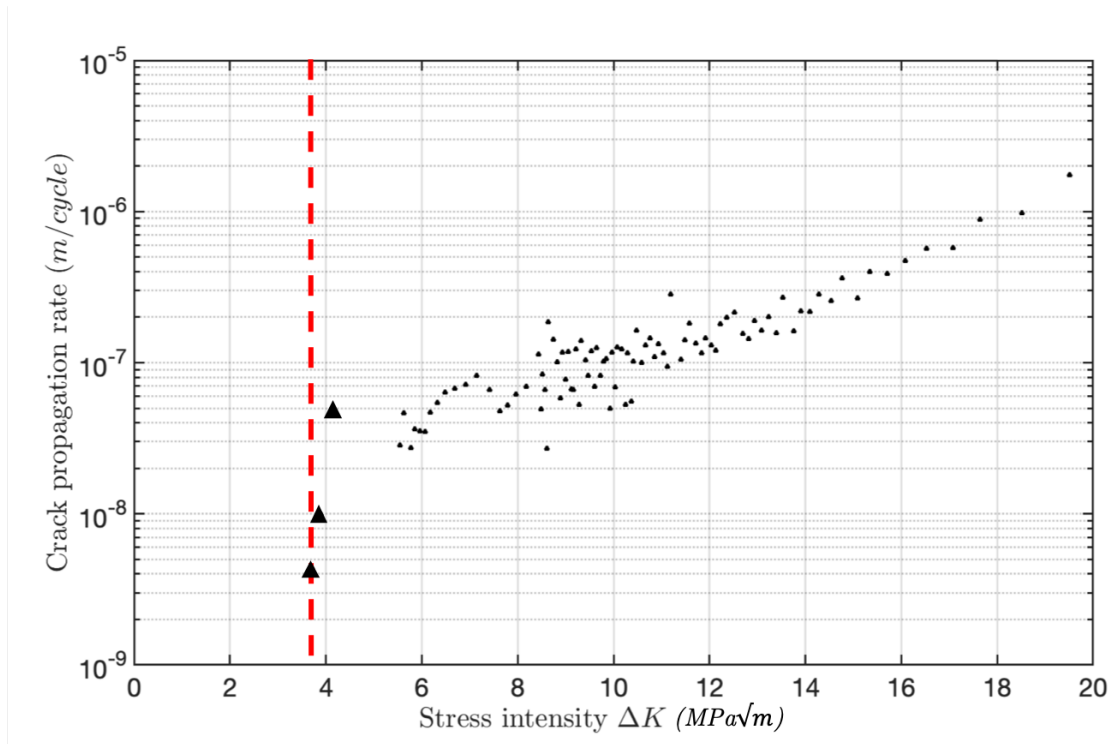


Figure 5.4: Measuring crack growth threshold by extrapolation of the data to 10^{-9} m/cycle.

5.3 Results

Specimens with longer 0.1, 0.25 and 0.5 mm notches initiated from the notch through randomly oriented colonies, and show no effect from initial notch size on either crack growth threshold or propagation. Specimens with shorter 0.06 and 0.03 mm notches initiated naturally, cracking

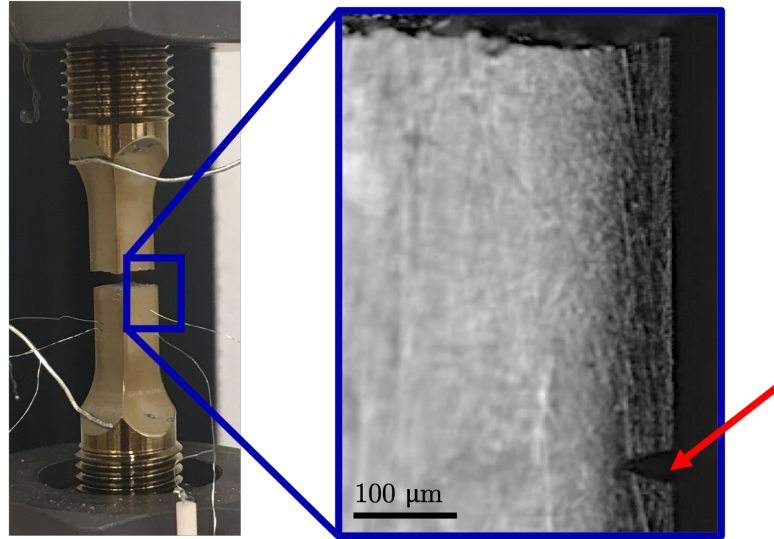


Figure 5.5: Specimen with notches 0.03 and 0.06 mm initiated away from the notch from the underlying microstructure. The red arrow shows the 0.06 mm notch, with the fracture surface above.

from microstructural features away from the notch, marked by the red arrow in Figure 5.5. This chapter compares and discusses the crack threshold and growth curves of the specimens that initiated from the machined notch. The specimens where cracks initiated from the underlying microstructure, 0.06 and 0.03 mm, are discussed further in Chapter 6.

Crack growth threshold was measured for nine specimens with a 0.5 mm machined starter notch, averaging at $4.02 \text{ MPa}\sqrt{\text{m}}$ with a standard deviation of $0.12 \text{ MPa}\sqrt{\text{m}}$. Crack growth threshold was measured for two specimens with a 0.1 mm machined starter notch, measuring 4.0 and $4.1 \text{ MPa}\sqrt{\text{m}}$. Crack growth threshold was measured for one specimen with a 0.25 mm machined starter notch, measuring $4.0 \text{ MPa}\sqrt{\text{m}}$. The threshold for the 0.1, 0.25 and 0.5 mm notches is therefore similar around $4 \text{ MPa}\sqrt{\text{m}}$, therefore showing no dependence on starter notch size on crack growth threshold.

The rate of fatigue crack growth, m , was measured in the Paris regime of the crack growth curve for $\Delta K > 7 \text{ MPa}\sqrt{\text{m}}$, and $a < 2.5 \text{ mm}$, by finding the gradient of the plot of $\log da/dN$ vs. $\log \Delta K$. The rate of fatigue crack growth for a 0.5 mm starter notch was measured in four specimens, measuring 6.4 with standard deviation of 0.9. The rate fatigue crack growth for the 0.25 mm starter notch was measured on one specimen, 2.5. The rate of fatigue crack growth for the 0.1 mm starter notch was measured on two specimens, giving values of 2.1 and 6.8. The

Notch Size (mm)	ΔK_{th} (MPa $\sqrt{m}$)	$\Delta\sigma$ (MPa)	m
0.1, Specimen A	4.0	331.4	2.1
0.1, Specimen B	4.1	339.7	6.8
0.25	4.0	208.7	2.5
0.5	3.9	142.9	5.5
0.5	4.0	146.6	5.7
0.5	4.0	146.6	6.9
0.5	4.2	153.9	7.6
0.5	4.0	146.6	no data
0.5	4.0	146.6	no data
0.5	4.2	153.9	no data
0.5	3.8	139.3	no data
0.5	4.1	150.3	no data

Table 5.1: The values measured for threshold and the Paris regime of fatigue crack growth.

values measured for both threshold and the rate of fatigue crack growth, m , in all the specimens are shown in Table 5.1. The rate of fatigue crack growth was not measured for five tests due to a change in test conditions affecting the fatigue crack growth curve.

There is no correlation seen between initial notch size and the crack propagation rate. A large amount of variation can be seen in the rate of crack growth in the central Paris regime of the curve, as is exhibited for the 0.5 mm starter notch, ranging from 5.5 to 7.6. The fatigue crack growth curves for two specimens, A and B, with 0.1 mm starter notch can be seen in Figure 5.6. This shows in similarly sized initiating starter notches, the large variability of the crack propagation rate in the Paris regime.

Fatigue crack growth curves for 0.5, 0.25 and 0.1 mm Specimen A and B are shown in Figure 5.7. This shows the similarity in the crack growth threshold values for each of the specimens, but the variability in the crack propagation rate.

It is pertinent to note that the measurement of the crack propagation rate must be in the central region of the crack growth curve. This region is linear on a $\log(da/dN)$ - $\log(\Delta K)$ plot, and therefore approximately $\Delta K > 7$ MPa $\sqrt{m}$. This is because, in this region of the crack growth curve, the crack growth is beyond that of the microstructural effect of colony orientation. However, the da/dN vs. ΔK plot is only valid for $a \leq W/2$ [86], which is approximately 2.5 mm. From Figure 5.8, it can be seen that the fatigue crack growth curve for a starter notch of 0.5 mm extends beyond a crack length of 2.5 mm, marked on Figure 5.8 by a red line, and so the rate

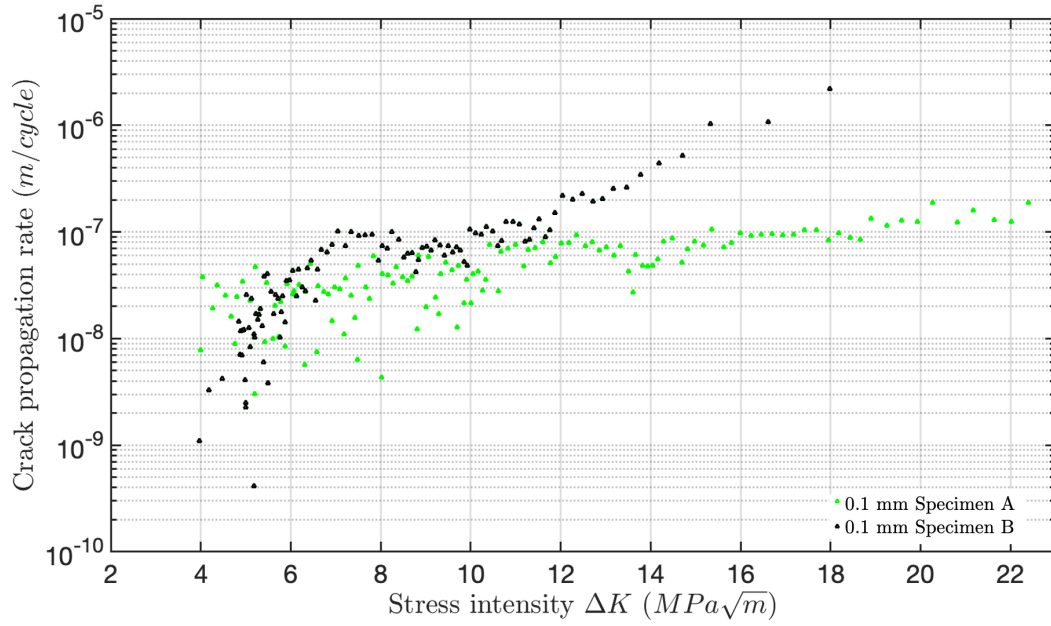


Figure 5.6: Fatigue crack growth curves for Specimens A and B with a 0.1 mm starter notch at 400°C in air, showing large variability in the Paris regime.

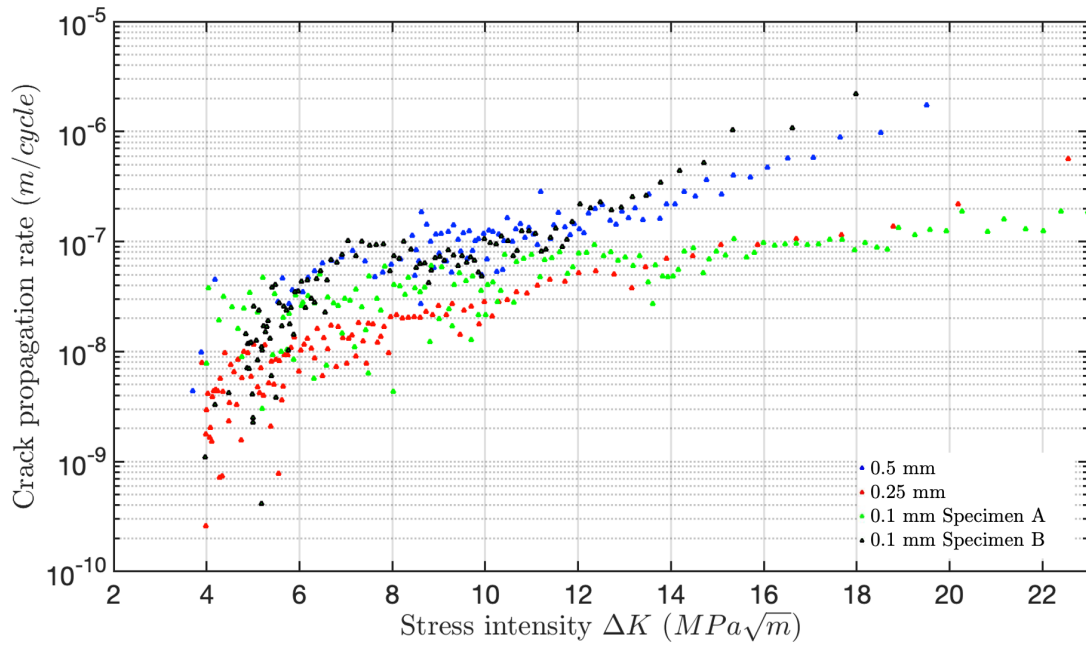


Figure 5.7: Fatigue crack growth curves for 0.5 mm, 0.25 mm and 0.1 mm Specimen A and B starter machined notches at 400°C in air.

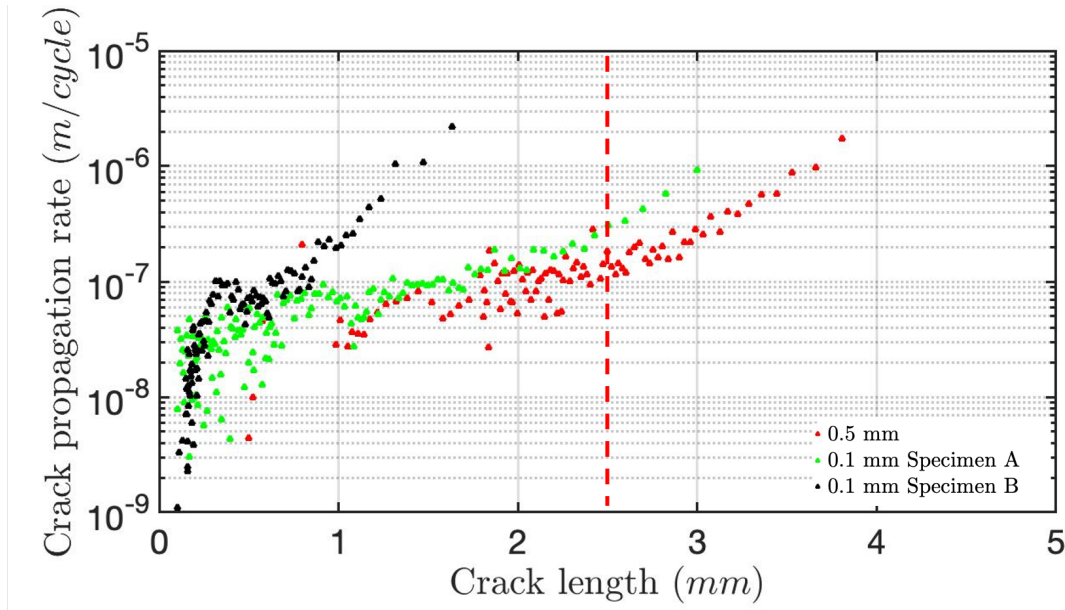


Figure 5.8: Rate of crack growth with the crack length at 400°C in air, for 0.5 mm, 0.1 mm Specimen A and B starter machined notches. A red line at 2.5 mm marks on the maximum crack length for which this curve has validity.

of fatigue crack growth in the Paris regime must be measured before this limit. However, for smaller starter notches of 0.1 mm, the curve and therefore the crack growth rate measurement are valid for a larger portion of the curve.

5.4 Discussion: Methodology for measuring threshold

Methods for measuring threshold are discussed in Chapter 4.3.2, Threshold measurement, and include the K -decreasing technique and compression initiation of a sharp notch. The K -decreasing or load shedding technique could not be successfully applied to γ -(TiAl). This technique was applied to Ti-6Al-4V during this project, but could not be applied to γ -(TiAl) for the reasons previously discussed. An alternative method of finding threshold from a sharp notch, is to initiate a pre-crack from compression [75].

Compressive loads applied to a brittle or ductile material causing far-field compressive stresses can be found to initiate or propagate a crack along a plane of a notch, normal to the direction of the applied stress. This crack is initiated under the compressive load, and grows with a decreasing velocity until its arrest.

This is due to the creation of a cyclic plastic zone ahead of the crack tip due to the far field

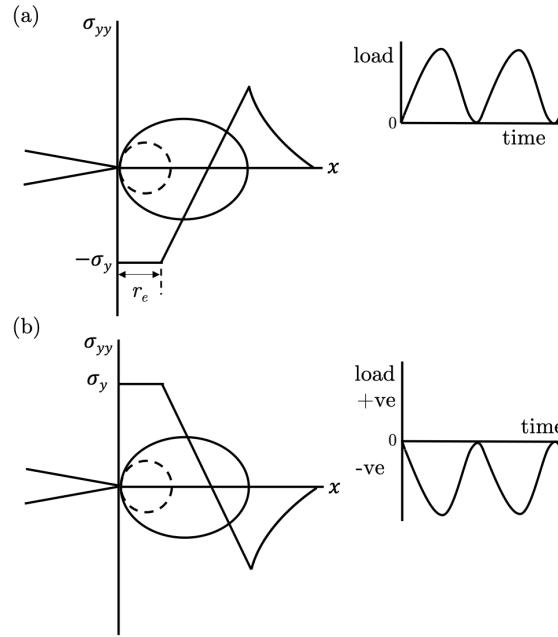


Figure 5.9: (a) Schematic of a zone of residual compression ahead of a sharp notch, with a small angle as a notch tip, far left, subjected to cyclic tension in an elastic-perfectly plastic solid. r_e is the cyclic plastic zone. (b) A zone of residual tension for the non-closing notch subjected to cyclic compression. Schematic adapted from [75].

compressive stress. In zero tension fatigue, ahead of the crack tip there develops a region of compressive plastic strain. This causes the surrounding volume to build tensile residual stresses. As the crack grows, this residual tensile stress offsets the applied compressive stress, causing the crack growth to slow down, and eventually arrest [75]. The total crack length grown in compression is a function of the total tensile residual zone created in the first cycle, stress state, load range and notch tip geometry, amongst others [138]. A comparison of methods to find threshold has been carried out, including cyclic compression pre-cracking in [134]. Pre-cracking with compression is carried out in [139] prior to fatigue testing in γ -based alloy. This comparison of initiation from tension and compression is detailed in the schematic, Figure 5.9.

Initiating a sharp crack through compression from a machined notch, and then running a fatigue test, would validate the assumption that diamond saw machined notches on corner crack specimens could be considered sharp notches. Unfortunately, due to corner crack specimens having a large cross sectional area relative to the notch area, and a large load being applied, when this was attempted the specimen buckled after only a short crack was initiated from the diamond blade machined notch, Figure 5.10.

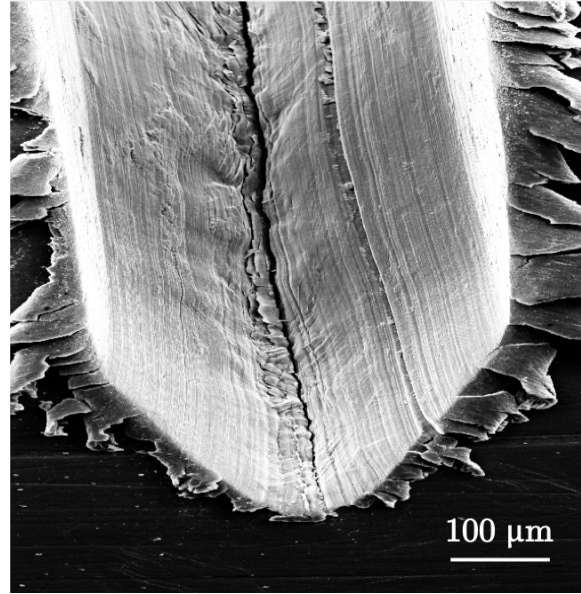


Figure 5.10: Pre-cracking by compression initiated a small notch prior to the specimen buckling.

5.5 Conclusions

Fatigue crack initiation and growth at 400°C in air was studied in a near-net shape cast and HIP γ -(TiAl) alloy notched specimens with notch lengths of 0.03 (FIB), 0.06, 0.1, 0.25 and 0.5 mm (diamond blade). The following observations were made:

- 1) For specimens with a notch smaller than the size of a colony, $\sim 70\ \mu\text{m}$, microstructural initiation was found to dominate.
- 2) Specimens with notches larger than the colony size initiated from the notch. Notch size was not found to have an effect on the fatigue crack growth threshold. The fatigue crack growth threshold for 0.5 mm starter notches was $4.02 \pm 0.12\ \text{MPa}\sqrt{\text{m}}$ for nine specimens, 0.25 mm starter notch was $4.0\ \text{MPa}\sqrt{\text{m}}$ for one specimen, and 0.1 mm starter notches was 4.0 and $4.1\ \text{MPa}\sqrt{\text{m}}$ for two specimens. The fatigue crack growth threshold was therefore found to be $4.03 \pm 0.11\ \text{MPa}\sqrt{\text{m}}$ for the twelve above listed specimens. These specimens initiated in trans-lamellar fracture mode from the randomly oriented colonies local to the notch.
- 3) Crack propagation rate varied within a range of 2.1 to 7.6, and variability was not seen to correlate to initial starter notch size.
- 4) Pre-cracking in compression was attempted but large enough cracks to be detected could not be grown without buckling the sample in this specimen geometry.

Chapter 6

Characterising the Fracture Surface

In this chapter we examine the fracture features that can be observed. Fracture surfaces with known notch site and crack path are first examined and characterised. This in turn is then used to characterise unknown crack paths on fracture surfaces without a known notch site. Notable features are then documented that have been discovered during testing and subsequent microscopy.

6.1 Change in cracking with ΔK

The material condition and preparation are detailed in Chapter 3. Indirect crack growth measurement was carried out using the DCPD method and threshold was determined, as detailed in Chapter 4. The standard test was carried out at 400°C with a 0.5 mm notch. This generated a 5 x 5 mm fracture surface from the cross section of the specimen in Figure 4.1. A fracture surface of this specimen can be seen in Figure 6.1(a). The machined notch is visible in the bottom left corner. The crack propagated in fatigue from this notch, initially with limited roughness, Figure 6.1(b). The crack continued in this mode of growth for approximately 2 mm. The fracture surface then shows an increased roughness with crack growth, Figure 6.1(c), until failure at the top right of the fracture surface in Figure 6.1(a). This change in crack growth mode can be hard to observe optically but can be discerned by careful observation in the SEM, accompanied by optical surface profilometry, as will be shown later in this chapter.

This different roughness can be translated to the type of cracking occurring. The near-notch

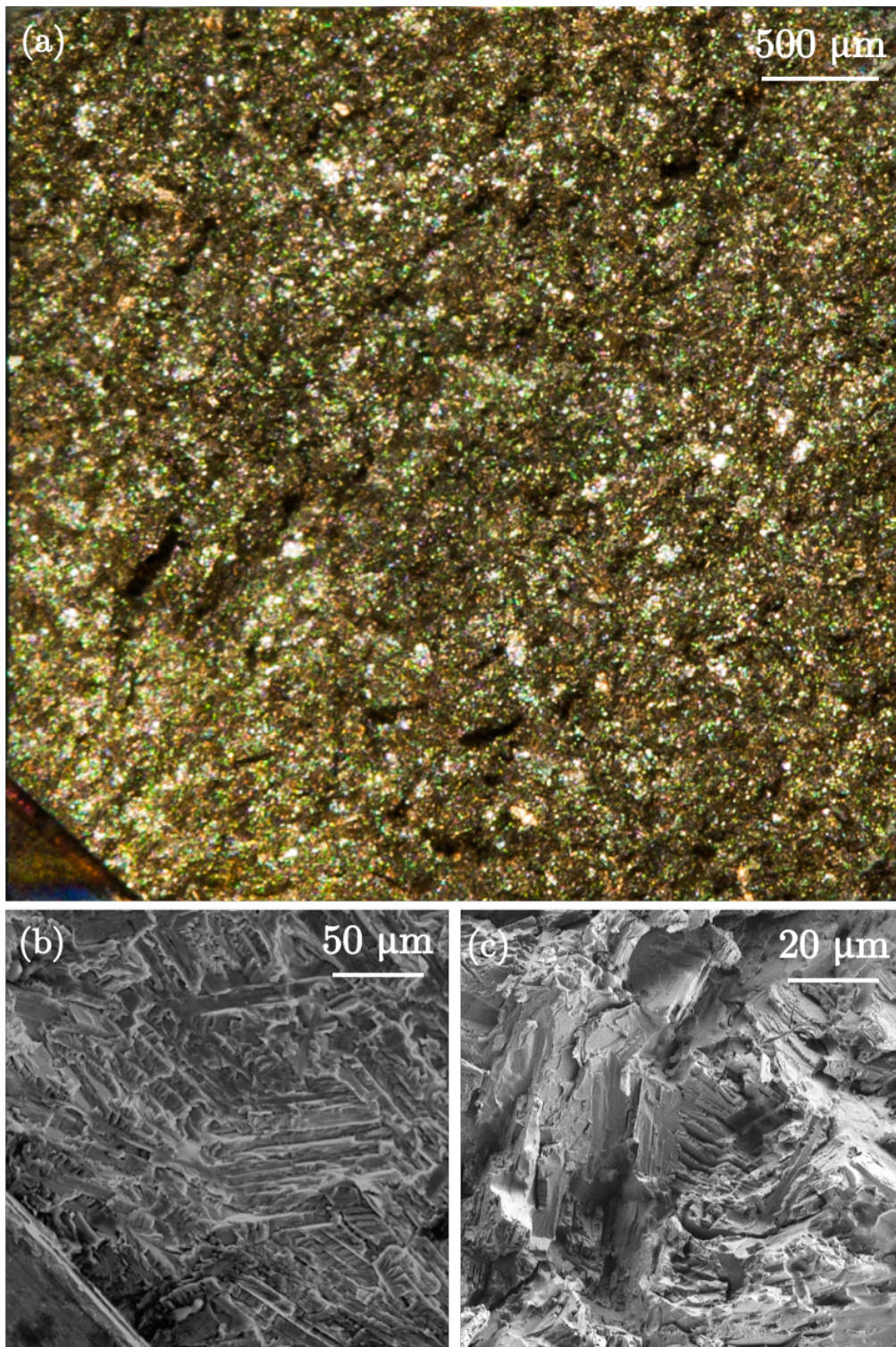


Figure 6.1: (a) Fracture surface of 0.5 mm notch specimen at 400°C, (b) cracking with limited roughness can be seen near-notch, (c) cracking with increased roughness can be seen far from the notch.

environment of limited roughness can be seen to be dominated by trans-lamellar cracking, through the colonies and the lamellae. Due to this cracking mode, the fracture surface tends to

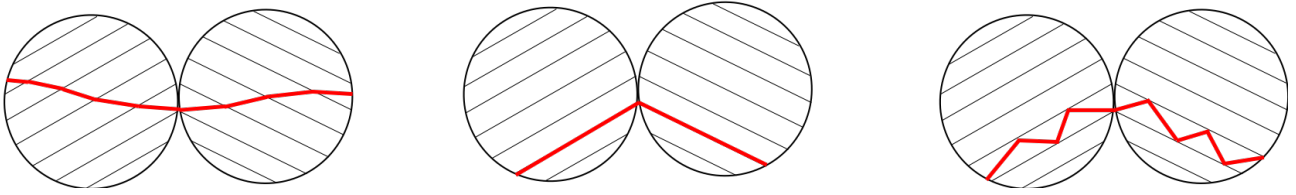


Figure 6.2: Schematic showing (a) trans-lamellar cracking, (b) inter-lamellar cracking, (c) intra-lamellar cracking.

be quite flat when sampling over multiple colonies. At higher ΔK and larger crack lengths, the roughness increases when sampling over multiple colonies and the cracking changes to become predominantly more inter- and intra- lamellar cracking. Trans-lamellar cracking is when the crack travels through the lamellae, giving a flatter crack path across the grain, Figure 6.2(a). Inter-lamellar cracking is the crack path following between lamellae boundaries, Figure 6.2(b). Intra-lamellar cracking is the cracking inside a group of lamellae, Figure 6.2(c).

The fracture surface topography has been measured using the 4D InSpec at Rolls-Royce plc. Derby, courtesy of Andy Curtis. 4D InSpec is an optical interferometry system. This shows the topographical height of the 5 x 5 mm fracture surface for two specimens. Figure 6.3(a) shows the fracture surface height variation for a 0.1 mm notched sample at 400°C. The notch is on the left and the crack grows out to failure on the right. This shows minimal height variation until around 2 mm, then this roughness increases until failure on the right. Figure 6.3(b) shows the fracture surface height variation for a 0.5 mm notched sample at 400°C. This clearly again shows the flatter initial crack growth in near-notch for approximately 2 mm. The height variation and corresponding roughness then increase until failure. These correspond to different load levels, required to grow out from threshold of 330 MPa for 0.1 mm starter notch and 150 MPa for a 0.5 mm starter notch. This height variation around 2 mm in both specimens corresponds to that change in cracking mode from predominantly trans-lamellar to mixed inter-, intra- and trans- lamellar.

Figure 6.4 shows the variation in the RMS roughness across the crack growth diagonal, from the notch diagonally across the fracture surface until failure, for four specimens with varying starter notches and temperatures. The false colour map shows the height surface profile across the fracture surface from initiating notch on the left to failure on the right. Below is the RMS roughness for the corresponding above area. Figure 6.4(a) shows the variation across the 0.1 mm

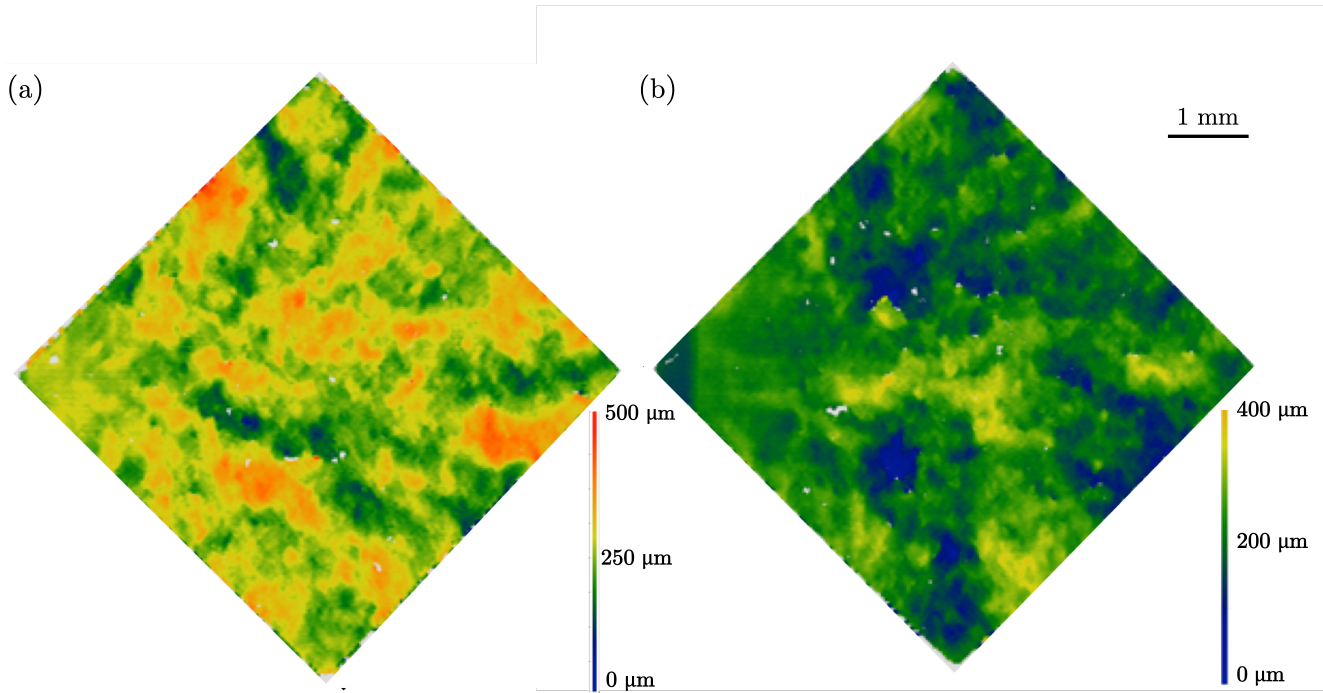


Figure 6.3: The fracture surface topography using 4D InSpec at Rolls-Royce plc. Derby courtesy of Andy Curtis. The colours depict height variation about a mean value for that particular image, with that mean being green, red being high and blue being low. Fracture surface maps at 400°C of (a) 0.1 mm starter notch and (b) 0.5 mm starter notch.

starter notch at 400°C and Figure 6.4(b) shows the variation across 0.5 mm starter notch at 400°C. As can be seen in the previous figure, the variation in roughness for these fracture surfaces increases from a crack length of around 2 mm. This can be seen both by the colour

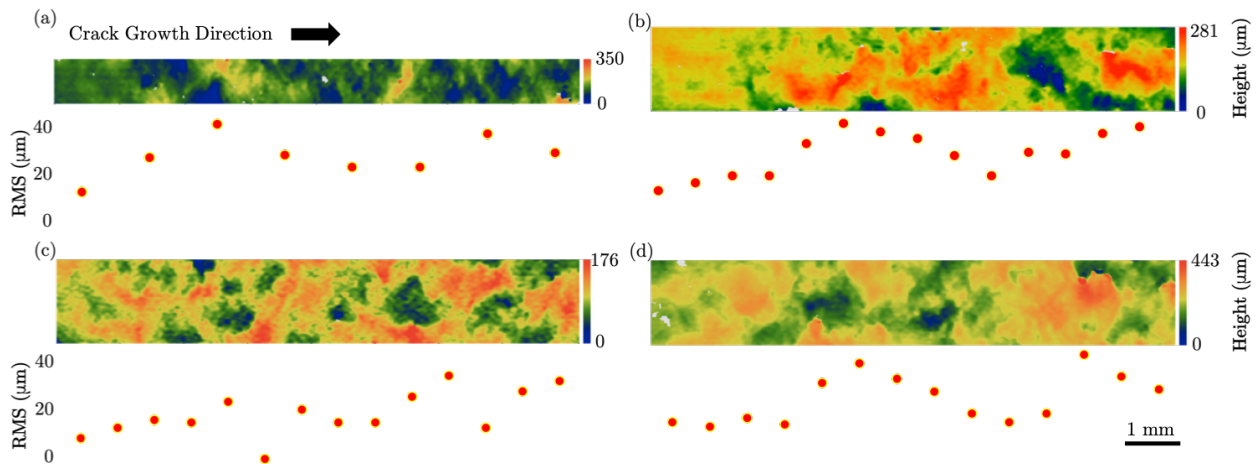


Figure 6.4: Surface profile from notch until failure for four fracture surfaces with the corresponding RMS roughness values below for each using 4D InSpec at Rolls-Royce plc. Derby courtesy of Andy Curtis. (a) 0.1 mm starter notch at 400°C, (b) 0.5 mm starter notch at 400°C, (c) 0.5 mm starter notch at RT and (d) 0.5 mm starter notch at 750°C.

variation in the false colour map, and the corresponding variation in the RMS values below. Figure 6.4(c) shows 0.5 mm starter notch at RT and Figure 6.4(d) shows 0.5 mm starter notch at 750°C. These again show an increase in the variation of height at approximately 2 mm.

It can therefore be difficult to quantitatively discern the change in cracking mode observed in the SEM or in the optical profilometry data, but a small change can, *post hoc*, be found.

Plastic zone size, r_p , can be determined as using Equation 2.13:

$$2r_p = \frac{\Delta K^2}{\pi \sigma_y^2} \quad (6.1)$$

where σ_y is the yield stress, of 430 MPa for γ -(TiAl) at 400°C, and ΔK , is the change in stress intensity applied. From Chapter 5 we can see that fatigue crack growth occurs in the interval between 4 MPa $\sqrt{m}$ at threshold, to final failure at around 20 MPa $\sqrt{m}$. This gives a plastic zone size range from tens of microns, to hundreds of microns. The colony size was found to be 40 - 100 μm . The plastic zone size coincides with this size between stress intensity values of 9 - 11 MPa $\sqrt{m}$, or a crack length of 1.9 - 2.3 mm. This coincides with the change in the crack growth from predominantly trans-lamellar to mixed inter-, intra- and trans- lamellar crack growth in Figure 6.2, at approximately 2 mm. This abrupt transition is shown by Figure 6.5.

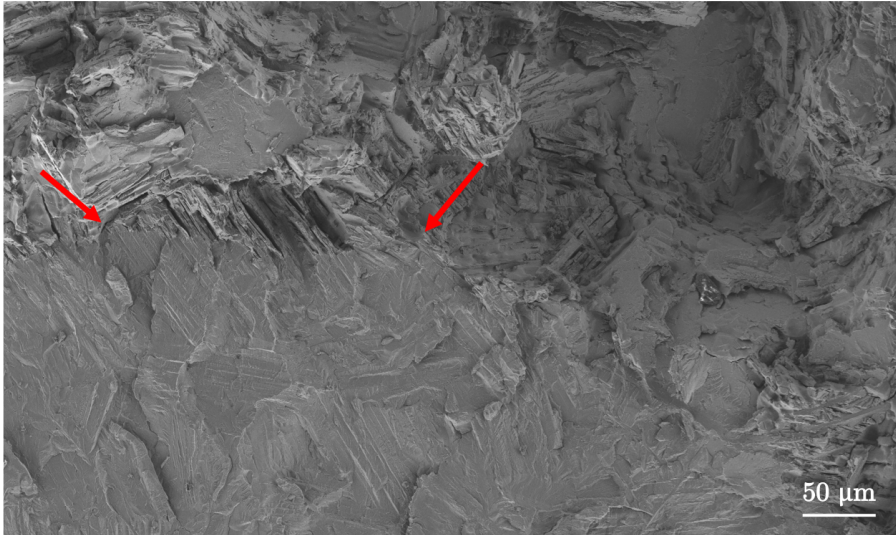


Figure 6.5: Change in plastic zone size to larger than that of a typical colony causes the cracking to transition from wholly trans-lamellar to mixed inter-, intra- and trans- lamellar. This causes the roughness to abruptly increase. This transition point is shown by the red arrows.

6.2 Initiation from microstructure

Notches smaller than the colony size initiated naturally, away from the notch, at microstructural features, Chapter 5. From the area of initiation, crack growth in fatigue was observed to be trans-lamellar, due to the plastic zone size being less than that of a colony, providing a flatter surface. With an increase in crack length, the stress intensity increases. Once this increased the plastic zone size to larger than the colony size, an increase in surface roughness could be observed, coinciding with a change in fracture mode to include inter- and intra-lamellar crack growth.

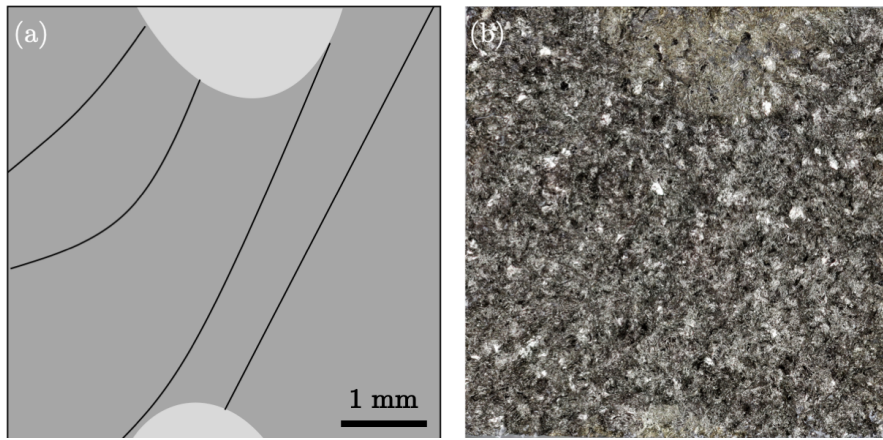


Figure 6.6: Specimen with a 0.06 mm machined starter notch. (a) A schematic showing fatigue in pale grey and river lines in black across the fracture surface. (b) Fracture surface of natural initiation.

The schematic in Figure 6.6(a) shows the features of the fracture surface of the specimen with a 0.06 mm diamond blade machined notch, with the fracture surface shown in Figure 6.6(b). The fracture surface initiated in two locations away from the machined notch. A rougher overload region can be observed centrally, with large prior β colonies being pulled out due to the large plastic zone. Figure 6.7 shows an optical micrograph of the fracture surface from an angle to expose the surface topography. Two flatter regions are observed on the fracture surface, at the top and bottom of the Figure 6.6(a) shown as paler regions. This indicates two initiation sites accompanied by trans-lamellar crack growth. The top fatigue region of initial crack growth can be seen to minimally increase in roughness throughout the fatigue away from an initiation site, and then change abruptly to a rougher cracking appearance, Figure 6.8(a). Within the region of fatigue and trans-lamellar crack growth, the reduction of roughness can



Figure 6.7: Optical micrograph of 0.06 mm notched naturally initiating specimen, 5 x 5 mm fracture surface from an angle showing the surface topography. Image taken using Keyence VHX-5000 at Rolls-Royce plc. Bristol, courtesy of Edward Saunders.

be traced back to find the area of initiation as marked by the red box in Figure 6.8(a) and shown in Figure 6.8(b). River marks down the fracture surface could also be traced, shown as black lines in Figure 6.6(a), showing the direction of crack growth away from the top and bottom regions of fatigue and connecting the crack growth. Initiation occurred from a flat planar feature of similar composition, presumably associated with a colony with its face normal along the loading direction. The neighbouring colony to the left shows striations indicative of intra-lamellar fracture. This would suggest that this colony has a similar orientation to the

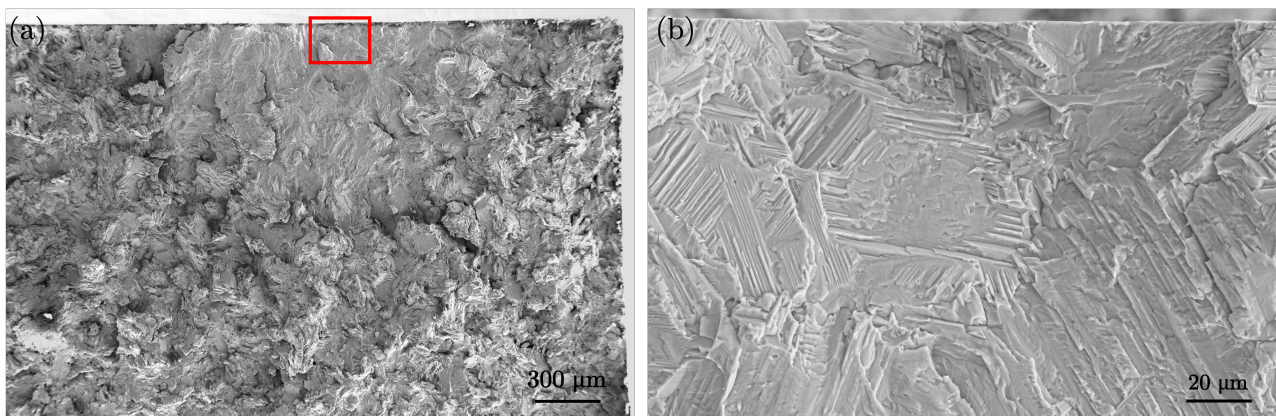


Figure 6.8: SEM Optical micrographs of fracture surface of specimen with 0.06 mm machined starter notch that naturally initiated. (a) The region of fatigue on the fracture surface, with a red box marking the initiation region. (b) Initiation region within the red box with trans-lamellar crack growth.

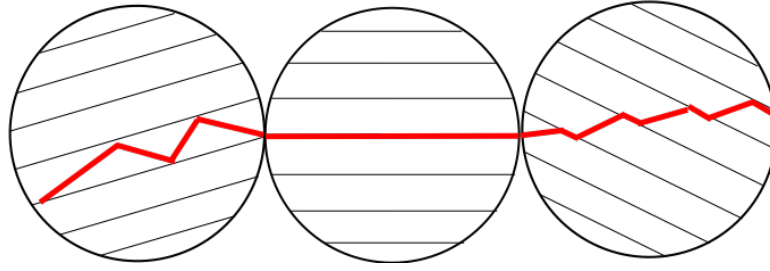


Figure 6.9: Schematic of the crack path in the top initiation site of the 0.06 mm machined notch specimen. The central colony is normal to the loading direction (up-down), and so initiates the fracture with inter-lamellar fracture. The left neighbouring colony has a similar orientation to the central colony and so crack in intra-lamellar fracture mode. The right neighbouring colony has a dissimilar relative orientation and so cracks in trans-lamellar fracture mode.

initiating colony, close to normal to the loading direction. The neighbouring colony to the right shows trans-lamellar crack growth, as is common in regions of fatigue. A schematic of the crack growth through these three colonies can be seen in Figure 6.9. By estimating the area of the planar feature ($5 \times 10^{-9} \text{ m}^2$) and knowledge of the load applied to this specimen during testing (mean load = 5.94 kN), an approximate value of fatigue crack growth threshold can be measured of $4 \text{ MPa}\sqrt{\text{m}}$.

The specimen with the 0.03 mm FIB-machined notch initiated naturally from the central region of the gauge, but away from the notch which is indicated by the red arrow, Figure 6.10(a). The fracture surface, Figure 6.10(b), shows fewer features than that of the sample with the 0.06 mm notch. However, a flatter region indicating fatigue could be observed at the bottom central side of the fracture surface which has been indicated by the red box in Figure 6.10(b). This region of fatigue, which exhibited a near-circular shape, can be seen in more detail in Figure 6.10(c). The roughness in this could be traced back to find an area of initiation. This initiation site is again observed to have a planar feature, with neighbouring intra-lamellar fracture. Initiation was seen to correlate with borides sitting at prior- β grain boundaries, Figure 6.11. Figure 6.12 shows this fracture surface exposing 3D topography, showing the flatter region of initiation on the fracture surface indicated by the red arrow, and limited variation in roughness.

The question therefore arises as to whether the borides at prior β colony boundaries are implicated in natural crack initiation. The borides themselves have a very brittle fracture surface appearance, as might be expected, and there is no secondary cracking or debonding at

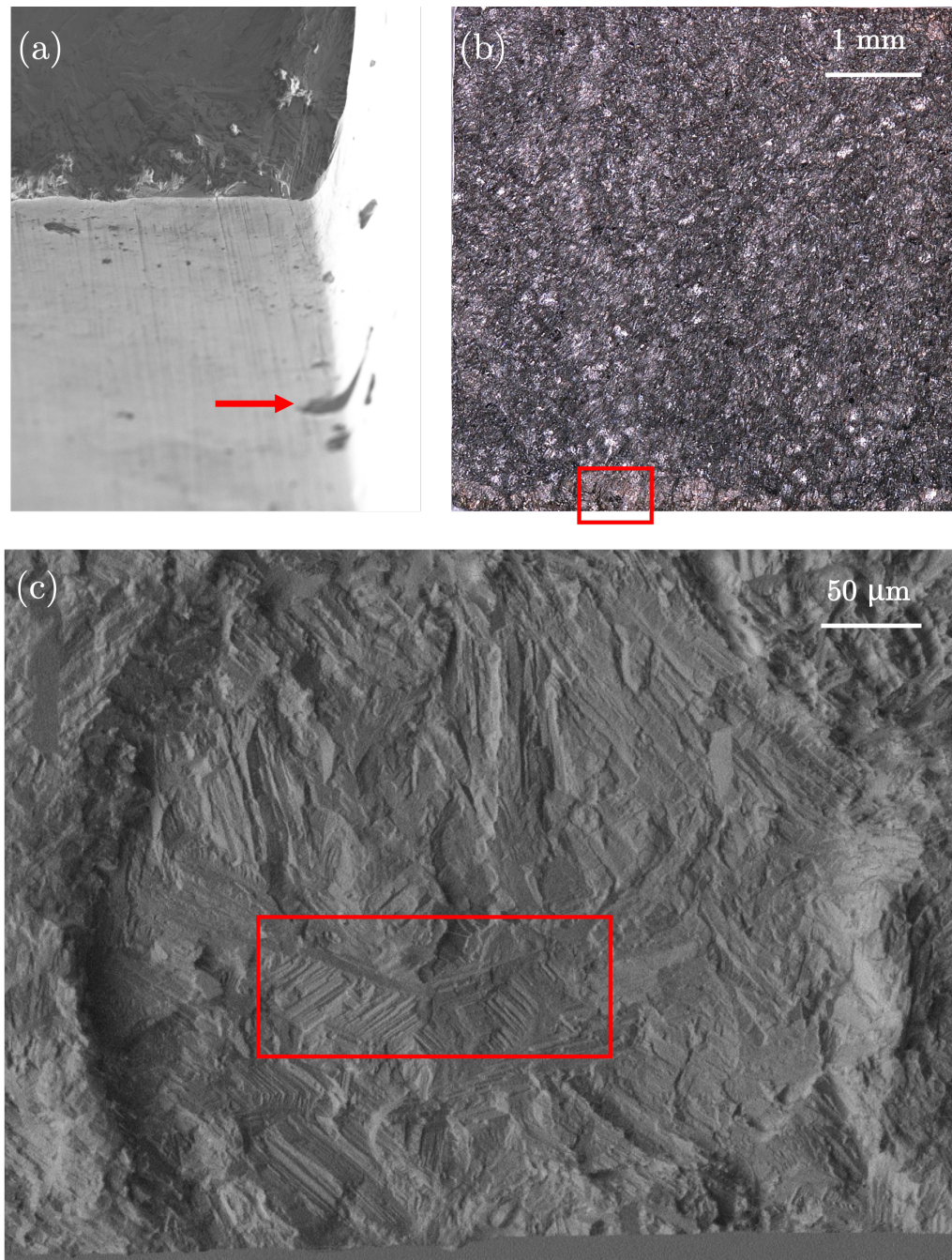


Figure 6.10: The 0.03 mm notched specimen initiated away from the notch. (a) The fracture surface can be seen at the top and the notch is indicated by the red arrow. (b) Fracture surface of the specimen with a small region of fatigue seen at the bottom of the micrograph, area of initiation marked by a red box. (c) Region of fatigue seen at the bottom of the fracture surface showing a near-circular, flatter region with trans-lamellar crack growth.

the boride/colony interface. It may be that failure is associated with the occurrence of multiple colonies of similar orientation, that can deform together, provide a region of locally larger colony size, and therefore the grain boundary borides may be a distraction, not actually a cause of crack initiation. There was nothing different about the borides observed at these initiation sites

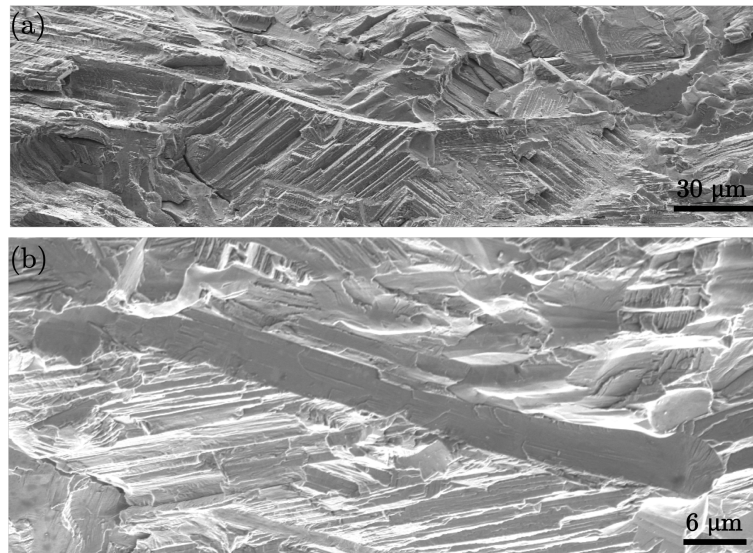


Figure 6.11: The region of initiation for the 0.03 mm notched specimen, marked by the red box in Figure 6.9(c), was seen to correlate with borides, sitting on the prior- β boundaries. Scanning electron micrographs of the initiating region (a) at 45° tilt, (b) at 0° tilt.

to other borides in the microstructure. Therefore it is concluded, tentatively, that borides do not, in themselves, promote cracking in this material. EDX was performed across the fracture surface to decide whether optically lighter regions correlating with the regions of fatigue were more oxidised *i.e.* whether the crack had grown and stopped, or if there were compositional defect, or an anomaly. However, no such difference was found. Therefore it is concluded that the only difference of the lighter colour was that the cracking mode was less rough and trans-lamellar.



Figure 6.12: Optical micrograph of 0.03 mm notched naturally initiating specimen, 5 x 5 mm fracture surface from an angle showing the surface topography, with a red arrow indicating the region of fatigue. Image taken using Keyence VHX-5000 at Rolls-Royce plc. Bristol, courtesy of Edward Saunders.

6.3 Features on fracture surface

The fracture surfaces and other microscopy from all testing throughout the thesis have shown various features which are documented below for posterity and future failure investigation into γ -(TiAl).

6.3.1 Blue-coloured fracture surface features

On some specimens, blue-coloured features could be observed on the fracture surfaces, indicated by the red arrow in Figure 6.13. Fractographic examination suggested that they were not initiating features, with no appearance of river lines or striations leading away from them. They appeared within the overload regions of the crack surface beyond the fatigue development. They are therefore considered to be insignificant with regard to these fractures, and are concluded to be benign. This is shown in Figure 6.13, where such a subsurface coloured feature can be observed. Closer optical and SEM examination of a similar, near-surface feature, is shown in Figure 6.14. This exposes the colour of the shallow surface blue-coloured feature in the optical microscopy, Figure 6.14(a).

Another similar feature is shown in Figure 6.15. This ‘cavity’ (or ‘lump’ on the other side of the fracture surface) showed the presence of a nodular oxide, $<5\text{ }\mu\text{m}$ thick. The ends of dendrite tips can also be observed within the feature, on which the oxide has grown.

EDX analysis of this feature is shown in Figures 6.16 and 6.17, at a working distance of 8.0 mm and at 20 kV. Figure 6.16 shows an increase in both oxygen and manganese in this region, and a marginal depletion in titanium. Aluminium and niobium content remain unchanged in the region of the feature. Figure 6.17 again shows both an increase in manganese and oxygen at the feature site, and unchanged base metal composition. It is concluded that these features are regions associated with cavities occurring at the end of solidification, which oxidised in the presence of entrapped gas during cooling of the cast sample. Again, as this feature was not associated with fatigue crack growth radiating away from it, it is concluded that these features are benign in relation to fatigue crack initiation and development in these cases.

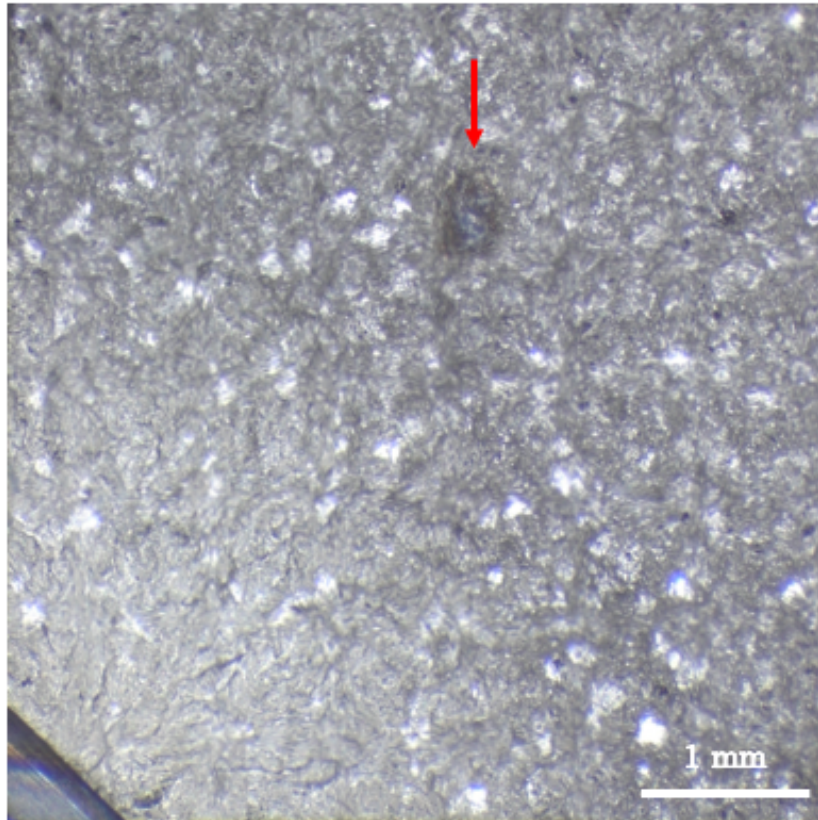


Figure 6.13: A fracture surface showing the blue-coloured fracture surface feature indicated by the red arrow. The crack was not seen to initiate from this sub-surface feature.

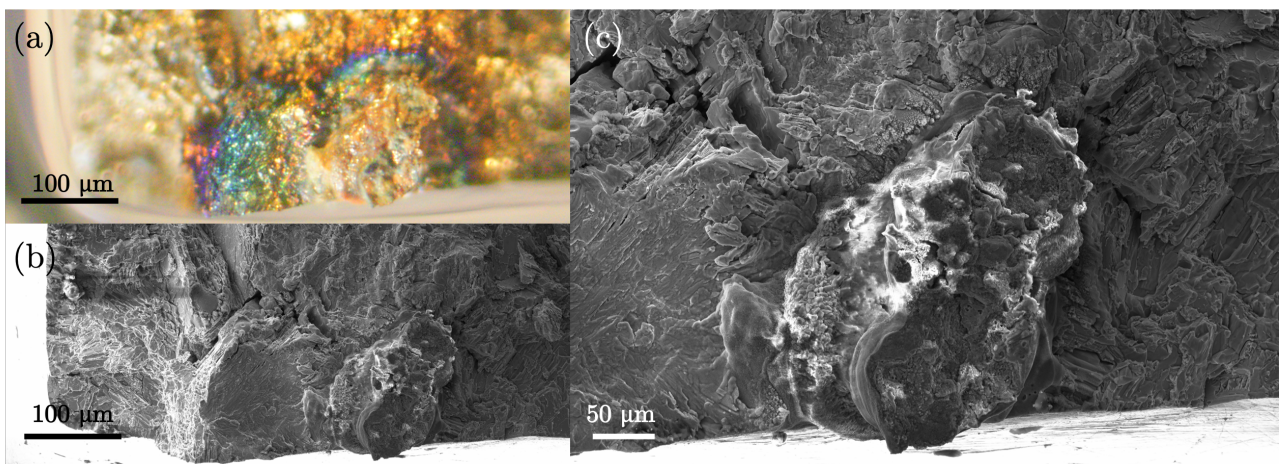


Figure 6.14: Near surface blue-coloured fracture surface feature shown using: (a) optical microscopy exposing its colouration, (b) SEM, (c) increased magnification SEM.

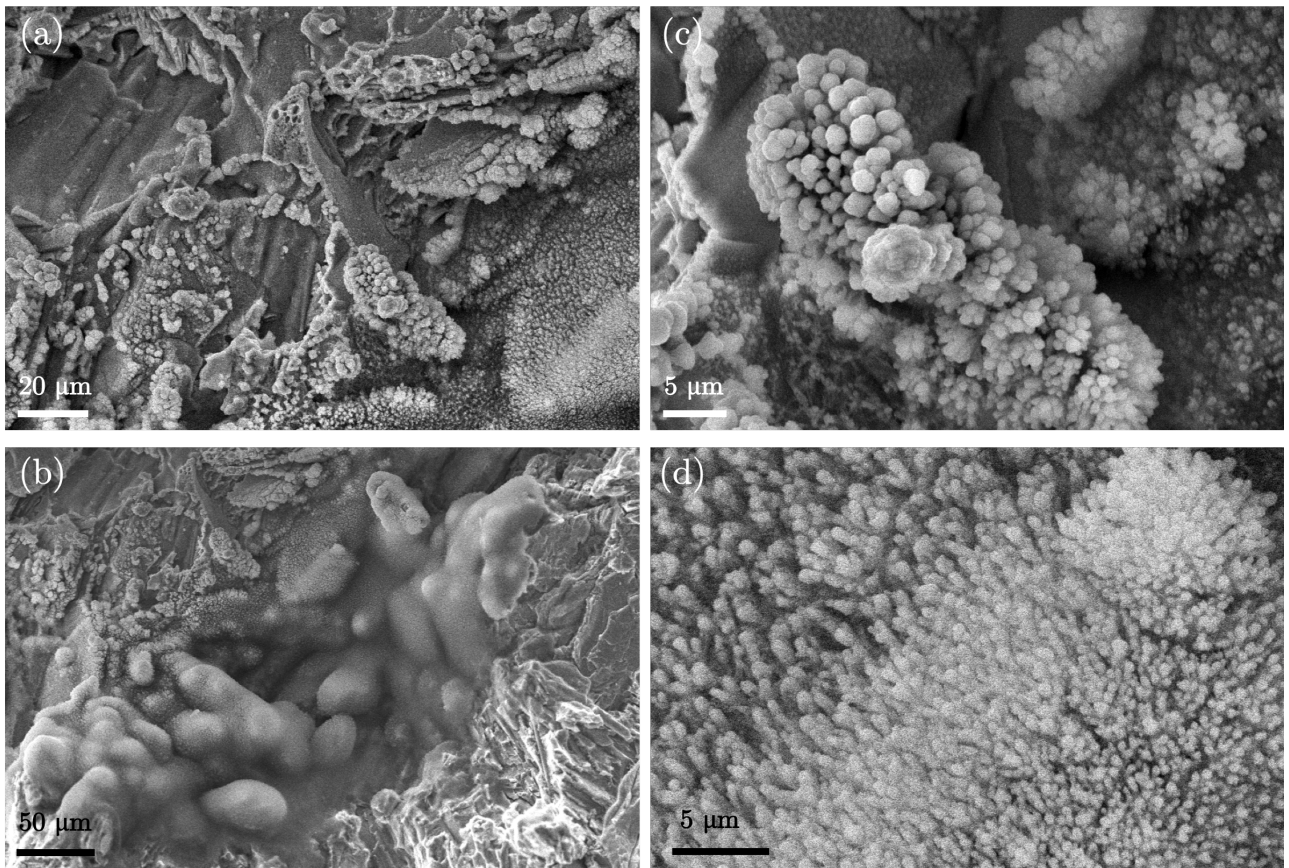


Figure 6.15: (a)(b)(c)(d) Increased magnification images of dendritic features associated with the blue-coloured fracture surface features.

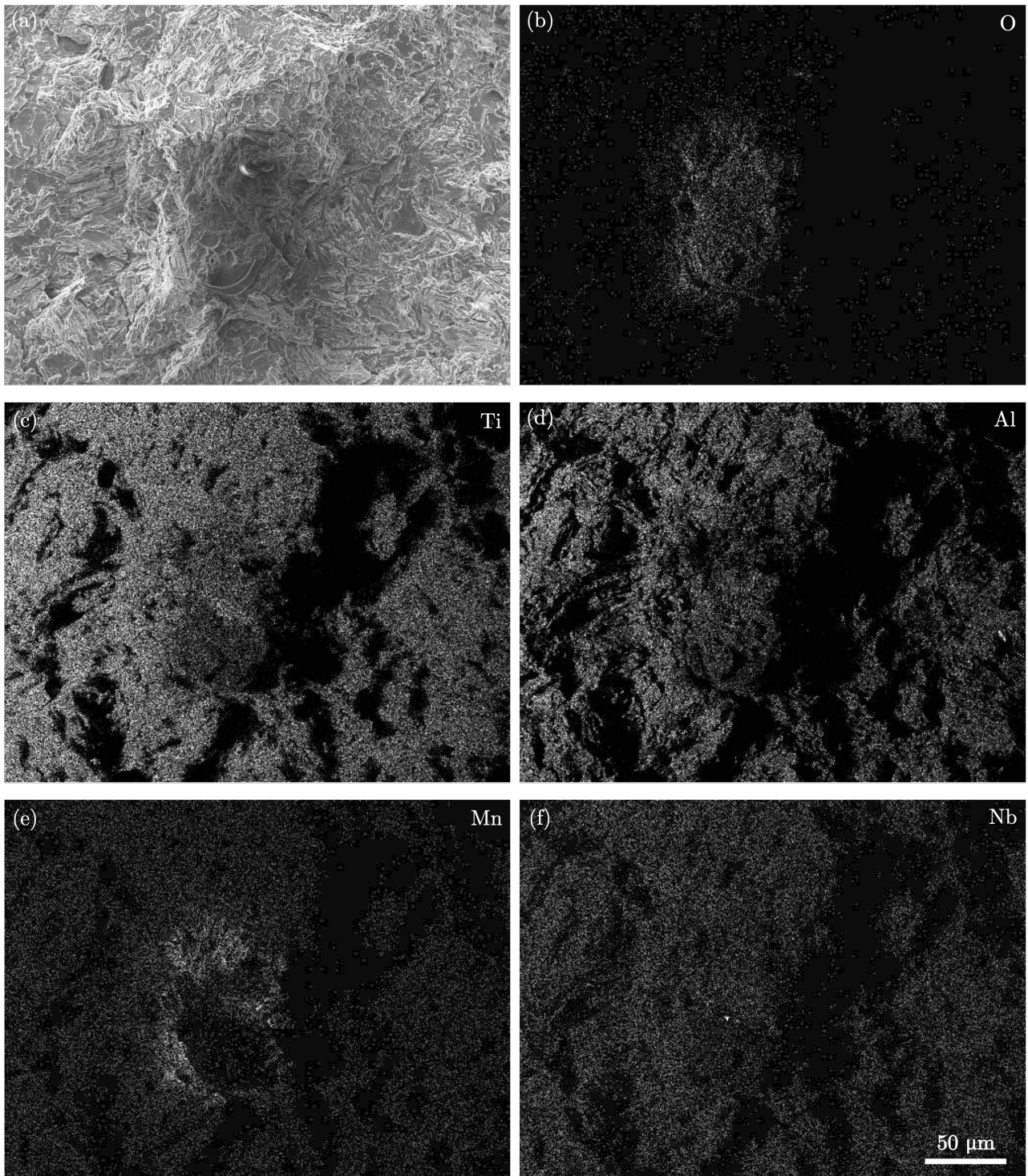


Figure 6.16: (a) SEM image of a blue-coloured fracture surface feature. Qualitative EDX map of this region at a working distance of 8.0 mm and at 20 kV showing elemental: (b) oxygen, (c) titanium, (d) aluminium (e) manganese, (f) niobium.

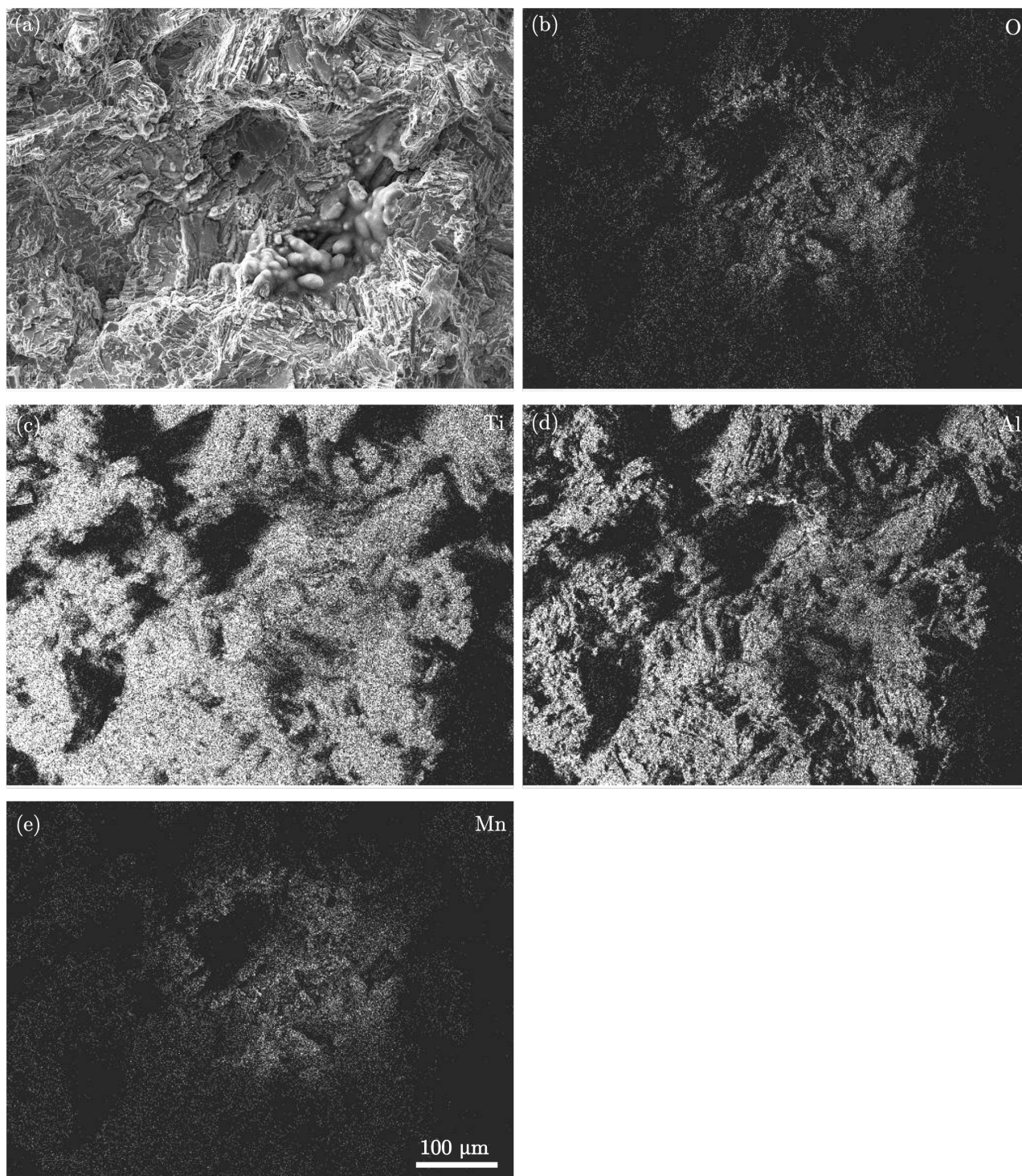


Figure 6.17: (a) SEM image of a blue-coloured fracture surface feature. Qualitative EDX map of this region at a working distance of 8.0 mm and at 20 kV showing elemental: (b) oxygen, (c) titanium, (d) aluminium (e) manganese.

6.3.2 Dislocation activity

A bright field TEM image of an untested part of a sample is shown in Figure 6.18. This shows the presence of a few threading dislocations in the γ phase, which are pinned at the γ/α_2 boundary. This indicates that the initial dislocation density in the material was low, and shows the reluctance of the dislocations to transfer across the $\alpha_2 \parallel \gamma$ interface.

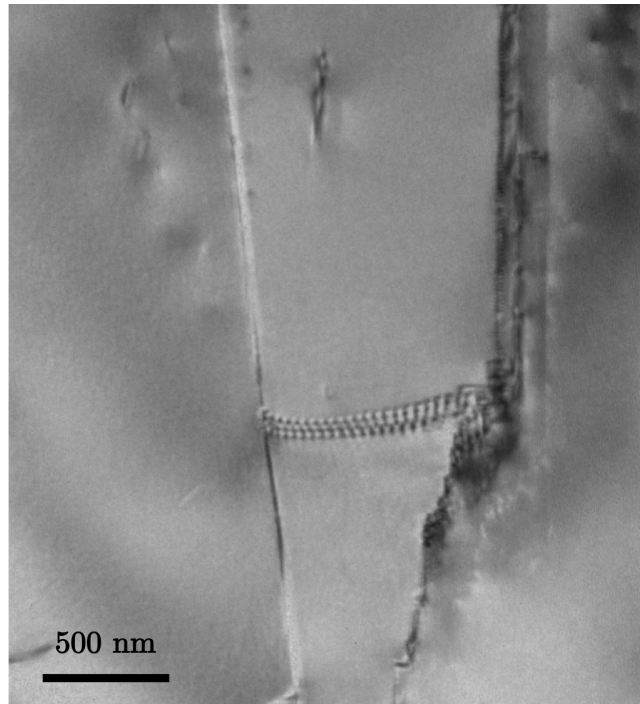


Figure 6.18: A bright field TEM image of an untested part of a sample showing some threading dislocations pinned at the γ/α_2 boundary.

6.3.3 Secondary cracking

Around a crack tip bifurcation of the crack may often take place, but as these link up to provide overall crack advance this may leave behind cracked ligaments that did not ultimately propagate. Furthermore, residual stresses and closure effects may also lead to cracking in the plastic wake. In both cases, these are termed secondary cracking. This can occur during any of the cracking modes shown in the schematic in Figure 6.2, around grain boundaries, or through borides. Borides are wavy as shown in Figure 6.19(a), and account for secondary cracking as can be seen in Figure 6.19(b). Secondary cracking can be seen on the fracture surface in Figure 6.20. The EBSD map is taken perpendicular to this fracture surface. It can be seen that secondary

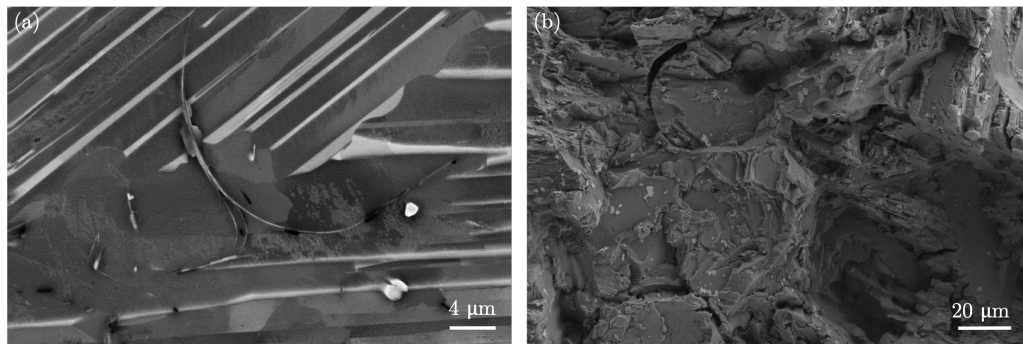


Figure 6.19: (a) Backscattered electron image of a wavy boride. (b) Secondary cracking on the fracture surface around a boride.

cracking occurs in the reverse direction to the crack growth. In this case, the secondary crack follows a grain boundary ahead, which may be related to stress relief in the plastic zone.

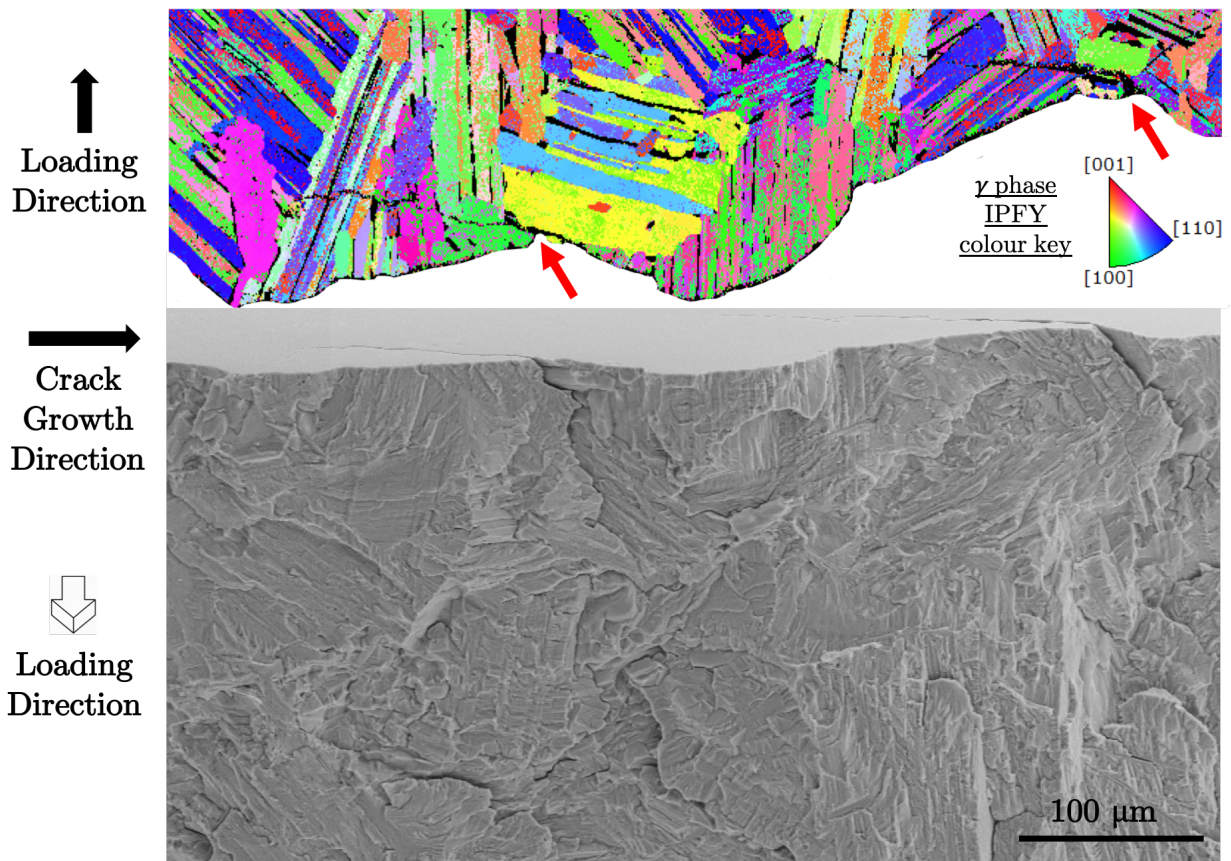


Figure 6.20: Secondary cracking can be seen in the SEM image of a fracture surface, where the loading direction is out of the page. Secondary cracking can be seen in the EBSD map of the cross section perpendicular to the fracture surface, propagating in the reverse crack growth direction. The loading direction and γ direction are aligned out of the page.

6.3.4 Ductility features

Despite this γ -(TiAl) based alloy being characteristically brittle, it shows limited examples of ductility on the fracture surface, so limited that these are noteworthy when found. Figure 6.21 shows some examples of this. Figure 6.21(a) shows signs of flow on the facet in a region of trans-lamellar growth. Figure 6.21(b) shows flow which may be due to cracking along a wavy boride. Figure 6.21(c) shows signs of dimpled rupture fracture, a ductile fracture. Figure 6.21(d) again shows signs of ductility and flow on the fracture surface.

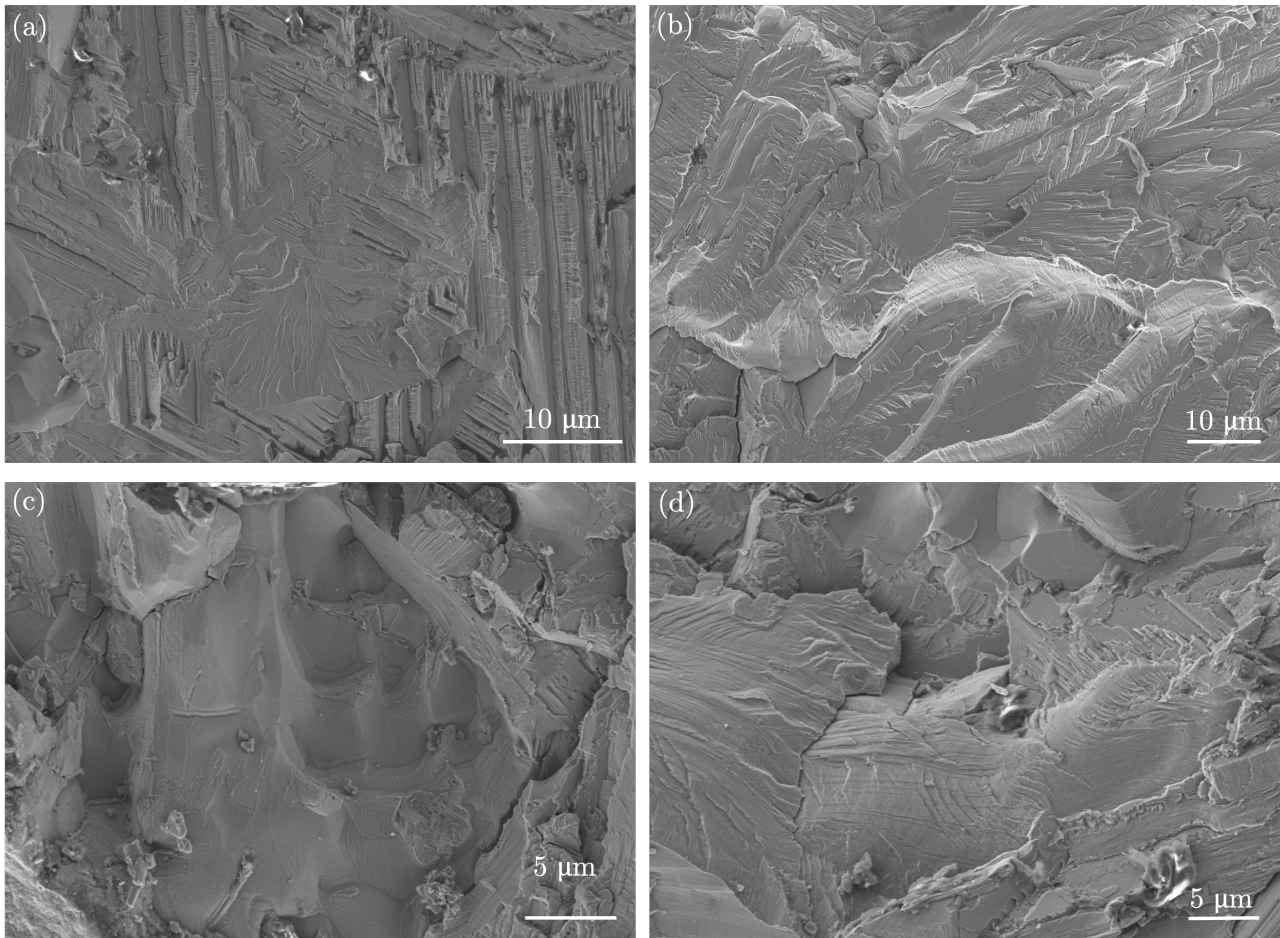


Figure 6.21: SEM images of fracture surfaces showing uncommon examples of ductility, (a) on a facet in a trans-lamellar crack growth region, (b) at a grain boundary that may be coincidental with a wavy boride, (c) dimpling in the overload region, (d) flow in the direction of crack growth.

6.3.5 Facet EBSD on plane

Facets can be seen on the fracture surface where the material cracks along a single crystal plane to create a flat face on the fracture surface. EBSD has been carried out on the facet in Figure 6.22(a). This facet was chosen due to being perpendicular to the loading direction. Figure 6.22(b) shows that this cracking was intra-lamellar through a γ lamella, as is anticipated due to the reduced barrier strength between γ lamellae as opposed to a $\gamma\|\alpha_2$ interface. Figure 6.22(c) shows this cracks along a single crystallographic orientation through this grain. The lower half of this facet shows a different orientation of this γ phase. This shows fracture at a $\gamma\|\gamma$ interface.

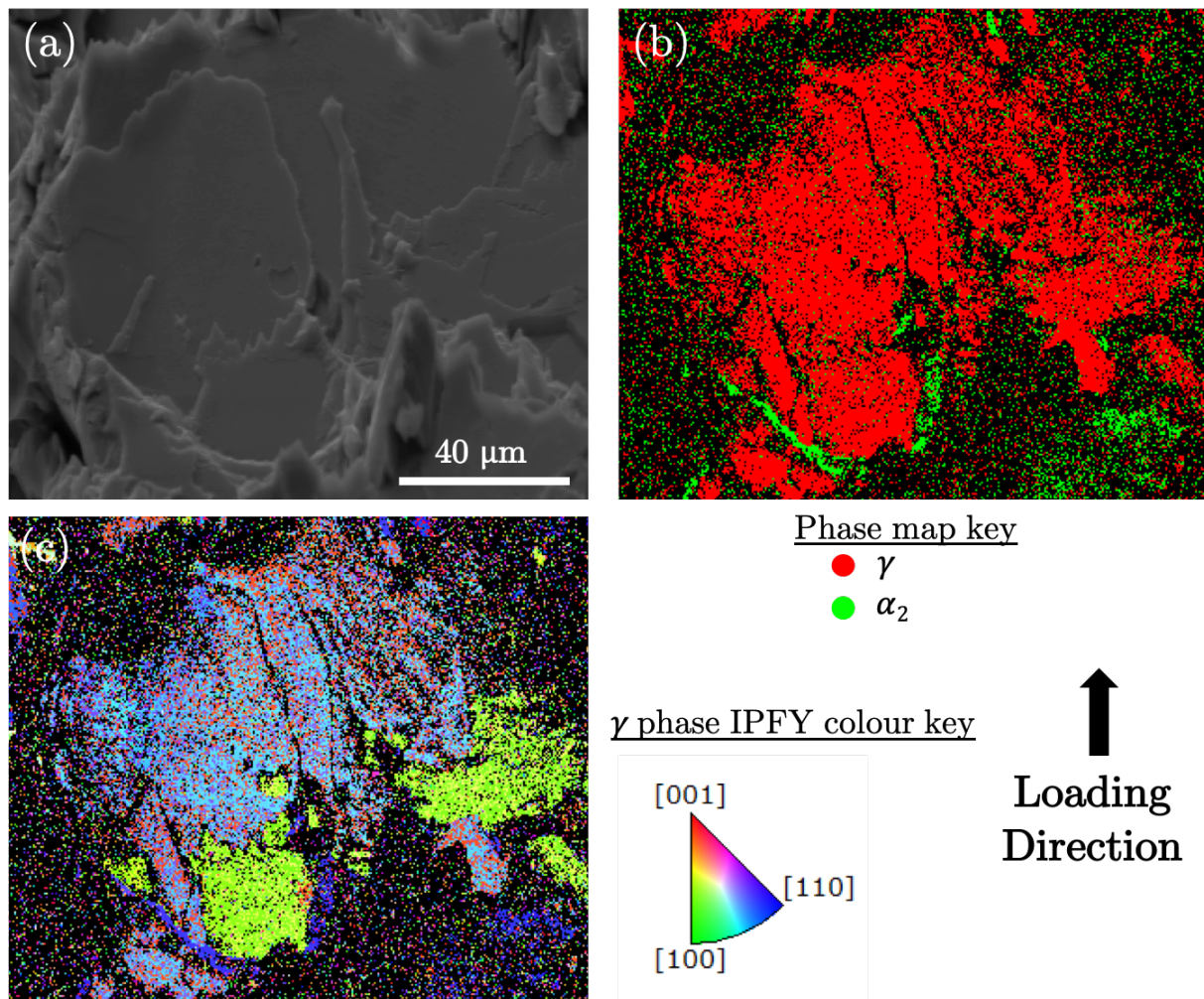


Figure 6.22: Facet on fracture surface where the loading direction and y direction are aligned. (a) SEM image of facet on fracture surface. (b) Phase map of facet. (c) EBSD orientation map of the γ -(TiAl) phase.

6.3.6 Surface delineations

Surface delineations can be seen on the fracture surface at increased magnification, Figure 6.23. These may correspond to γ/α_2 , lamellae, twinning or slip. These can be seen within a single lamella. Due to the symmetric nature of these, mirrored from lamella to lamella these are thought to correspond with the γ -(TiAl) twins or slip bands. Attempts were made to measure the spacing of the delineations across the fracture surface against depth (and ΔK), but no relationship could be found. Figure 6.23(a) shows various delineations within different lamellae. Figure 6.23(b) shows delineations which may be indicative of twinning in neighbouring γ lamellae, indicated by the red arrows.

Figure 6.24 is an EBSD map perpendicular to fracture surface delineations. Figure 6.24(a) shows the phase map, green is the γ phase, blue is the α_2 phase. Within a single colony α_2 has a single orientation due to it originating from the same parent α . It can therefore be deduced that the region depicted is a single colony due to Figure 6.24(b) showing the orientation of the α_2 being the same throughout. Figure 6.24(c) shows just the γ phase. This shows two orientations of the phase. However, the software has had difficulties assigning the correct orientation, as can be seen by the green and pink. This is due to the only marginal tetragonal nature of γ with only 2% difference between the a and c components of the crystal structure.

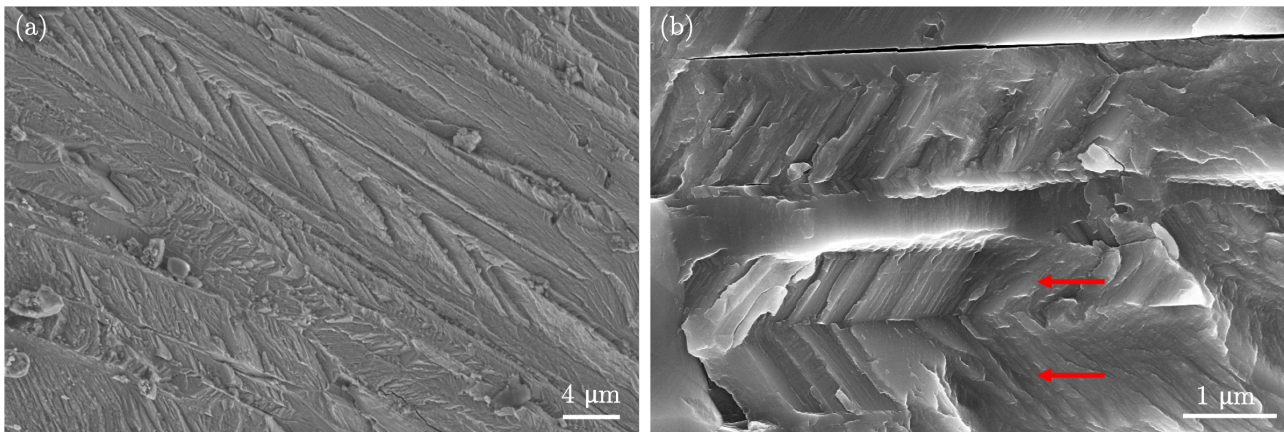


Figure 6.23: Surface delineations. (a) Delineations with varying spacings in different lamellae. (b) Surface delineations indicative of twins marked by red arrows.

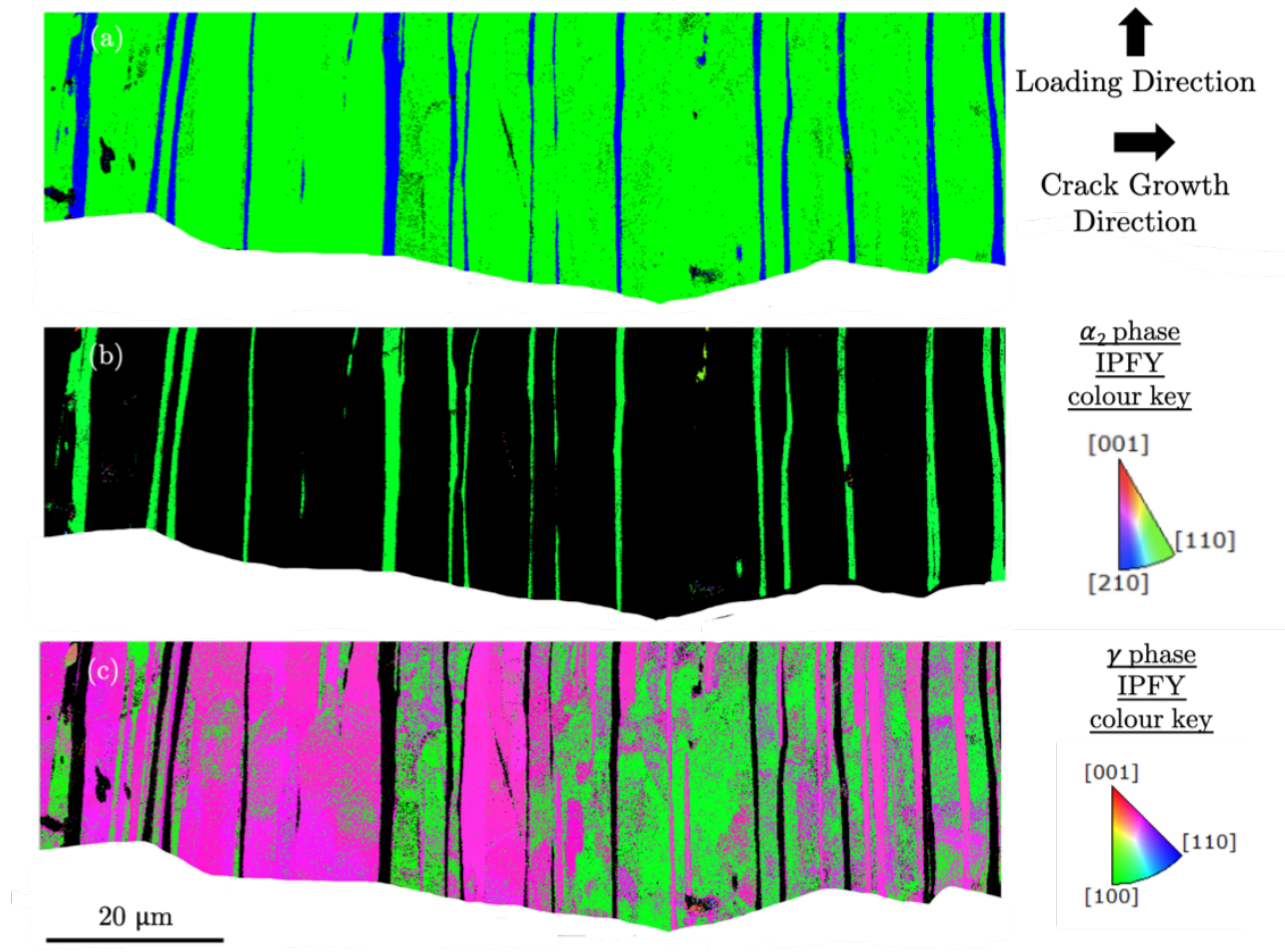


Figure 6.24: EBSD map of delineations where the loading direction and y direction are aligned. (a) Phase map of α_2 and γ . (b) Orientation map of α_2 . (c) Orientation map of γ .

6.4 Findings

1) There is a roughness change at an approximate crack length of 2 mm due to a change in cracking from trans-lamellar to inter- and intra- lamellar. This corresponds to a plastic zone size which is similar to that of the colony size. This is difficult to quantitatively discern. There is a general 3D roughness increase with ΔK , and a significant roughness increase in the overload region versus the fatigue region.

2) Notches smaller than the average colony size initiate from microstructural features. These fracture surfaces that naturally initiated can be characterised into regions of fatigue and overload. The region of initiation can be found due to decreased roughness and trans-lamellar nature, and have a similar fatigue crack growth threshold to specimens that initiated from the machined starter notch of approximately $4 \text{ MPa}\sqrt{\text{m}}$.

3) Borides were seen to correspond with regions of initiation from microstructural features, but it is unclear that they are initiating features themselves.

4) Blue-coloured fracture surface features were discovered but not found to be damaging inasmuch as they did not show any signs of crack initiation or growth from them on the fracture surface.

5) No relationship could be found by investigating the spacing of delineations found on the fracture surface.

Chapter 7

Effect of Colony Orientation on Threshold

7.1 Introduction

The fatigue crack growth threshold has been seen to vary within the same alloy depending on the notch orientation relative to the orientation of lamellae within a colony, where the lamellae all have the same orientation [140]. Yang *et al.* have shown that in a γ -(TiAl) based alloy of cast Ti-46Al-8Nb with a lamellar microstructure at 650°C and R ratio 0.1, lamellar colonies parallel to the stress axis were seen to have the highest threshold. These lamellae can either be parallel or perpendicular to the crack growth direction, Figure 7.1(a)(b)(c). When lamellae are parallel to the stress axis the threshold is higher as the crack has to pass more lamellae per unit distance. Colonies with orientation 40-65° off of the stress axis were seen to have the lowest threshold required to initiate a crack, Figure 7.1(d). Therefore, it is thought that slip or twinning parallel to the lamellar interface of either α_2 — γ or γ — γ_T may interact with cross lamellar slip or twinning to aid the opening of microcracks ahead of the dominant crack. Cracks can be seen to be locally arrested by a change in colony orientation [136]. On the other hand, crack growth is enabled when the orientations of two consecutive colonies are the same and aligned with the direction of crack growth. An incidence of two coincidental consecutive colonies with a ‘bad’ orientation can be seen in Figure 7.2. In this Chapter, the effect of the orientation

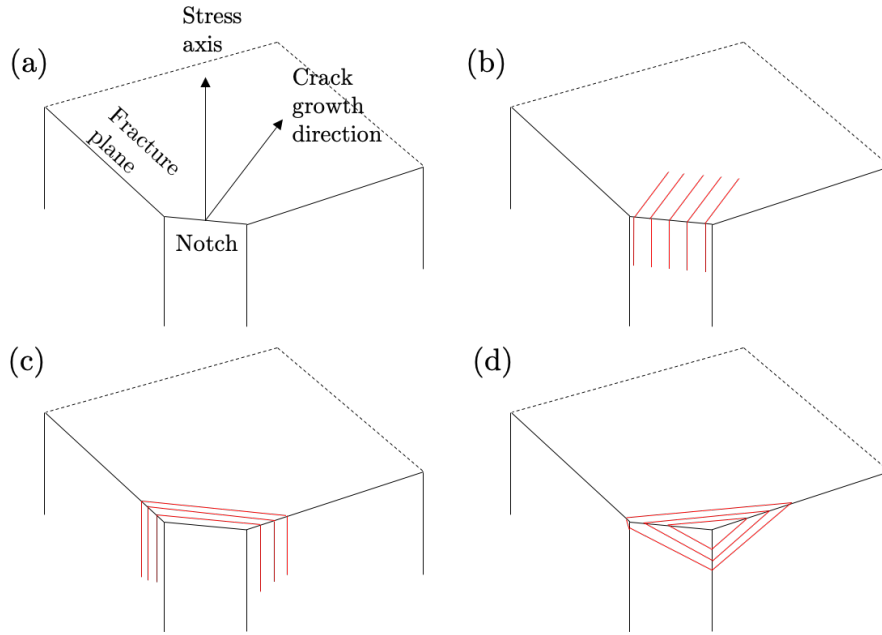


Figure 7.1: Schematic of the central gauge of the corner crack specimen from the notch, with microstructure shown in red: (a) the fracture plane showing the stress axis and crack growth direction from the notch, (b) lamellae from the notch parallel to the stress axis and parallel to the crack growth direction, (c) lamellae from the notch parallel to the stress axis and perpendicular to the crack growth direction, (d) lamellae from the notch at an angle 45-60° from the stress axis.

of a notch relative to an orientation of a colony on the surface of a corner crack specimen will be investigated, and how this affects the fatigue crack growth threshold.

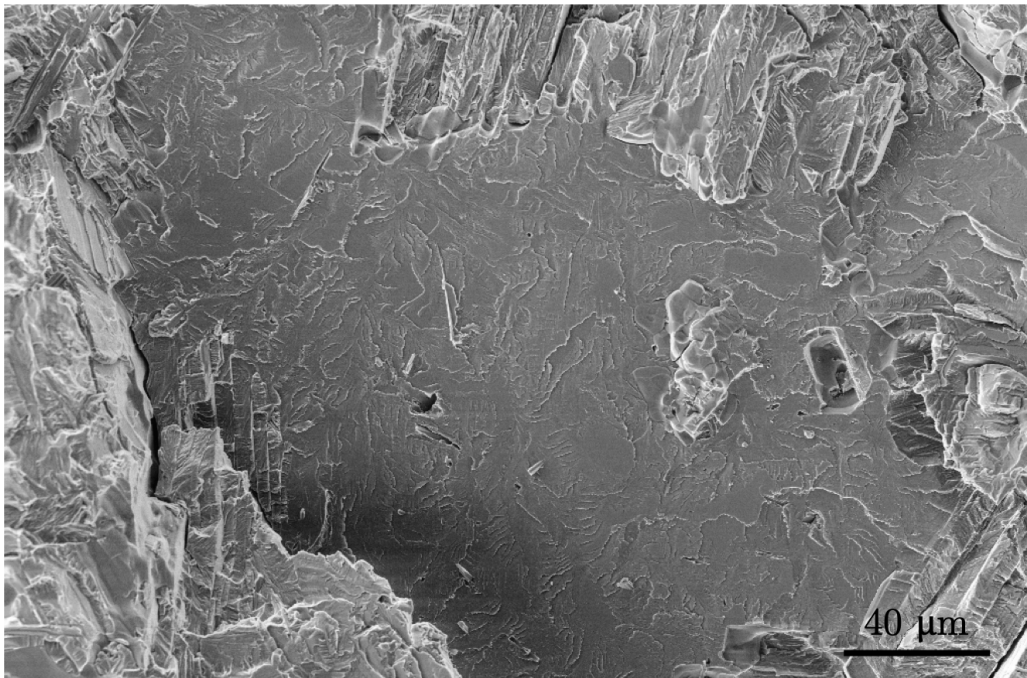


Figure 7.2: Two similarly oriented consecutive colonies giving rise to a large macroscopic facet.

7.2 Experimental methods

The material condition and preparation are detailed in Chapter 3. Indirect crack growth measurement was carried out using the DCPD method and carried out at 400°C in air. Two specimens were machined with twelve notches each along the central gauge, three on each corner of the specimen, spaced equally 3.75 mm apart. Each of these twelve notches were then imaged on both sides of the specimen using backscatter imaging, Figures 7.3 and 7.4, with the darker colour being γ and the lighter colour being α_2 . The measurement of the potential drop across two notches simultaneously is possible. This is achieved by the reallocation of the wires used to measure the reference voltage as a monitor. So the two notches believed to be most favourable for crack initiation were chosen.

It is pertinent to note that the colony shown in backscatter imaging next to the notch is unlikely to cover the entirety of the crack front. The average colony diameter is 0.06 mm, and therefore the average point is the centre of the colony on the surface next to the notch, which means on average each colony penetrates 0.03 mm into the sample along the crack front. This leaves crack front occupied by other colonies, which will mostly likely be oriented differently. On the two specimens with twelve notches, a different notch size was chosen for each specimen. Specimen A had twelve 0.06 mm notches. Specimen B had twelve 0.08 mm notches. Figure 7.5(a) shows a schematic of Specimen A showing one of the twelve 0.06 notches giving a 0.12 mm crack front. Colonies seen in the backscattered image are assumed to occupy 0.06 mm of this crack front, leaving 0.06 mm. This is the average size of one colony. Figure 7.5(b) shows Specimen B showing one of twelve 0.08 mm notches giving a 0.16 mm crack front, so 0.10 mm remaining crack front. There will be a region on each crack front with an uncharacterised orientation of a colony or colonies on both the notches on Specimen A and B.

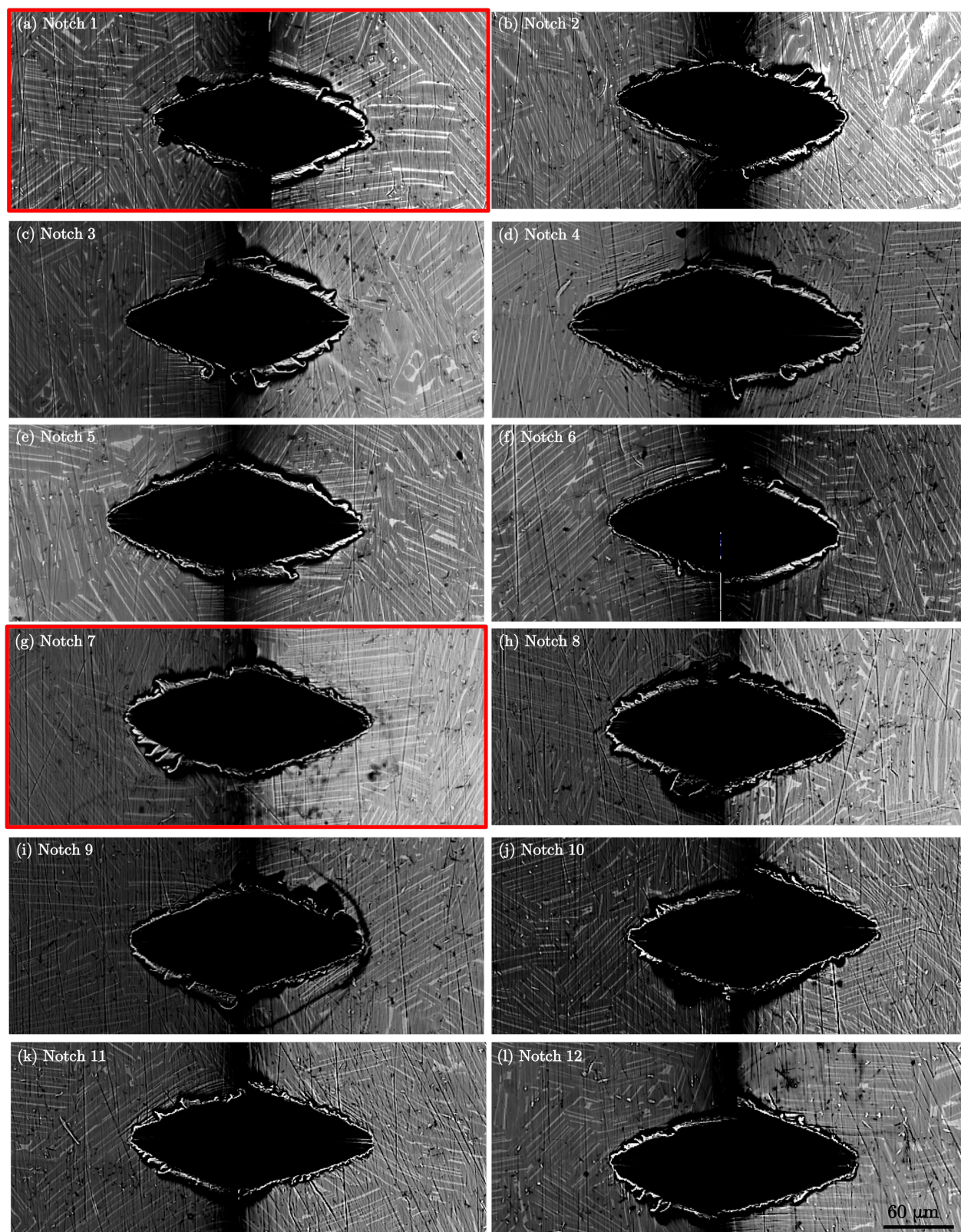


Figure 7.3: Backscattered SEM images of twelve 0.06 mm starter notches on a single specimen, Specimen A. The two selected notches are in red boxes.

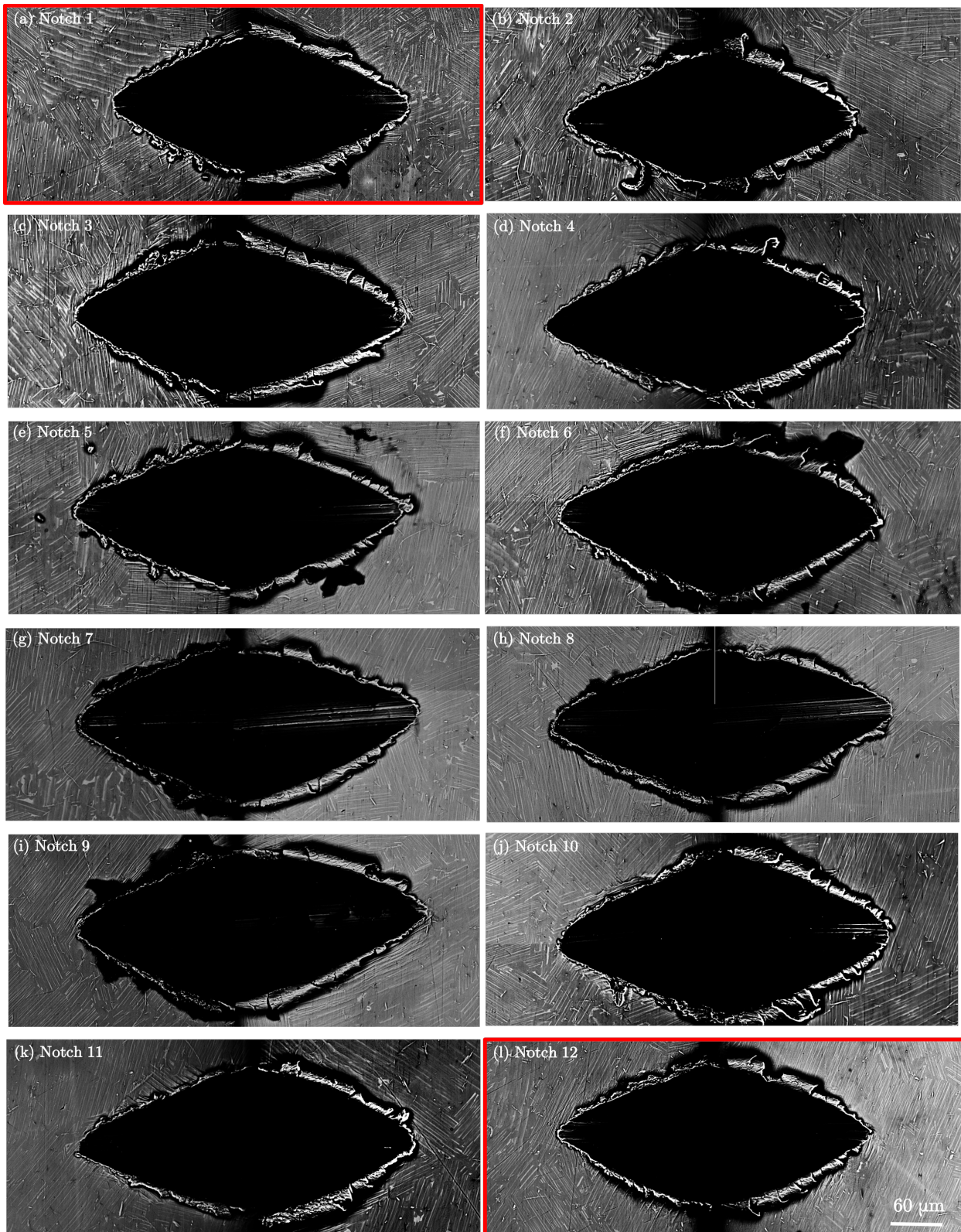


Figure 7.4: Backscattered SEM images of twelve 0.08 mm starter notches on a single specimen, Specimen B. The two selected notches are in red boxes.

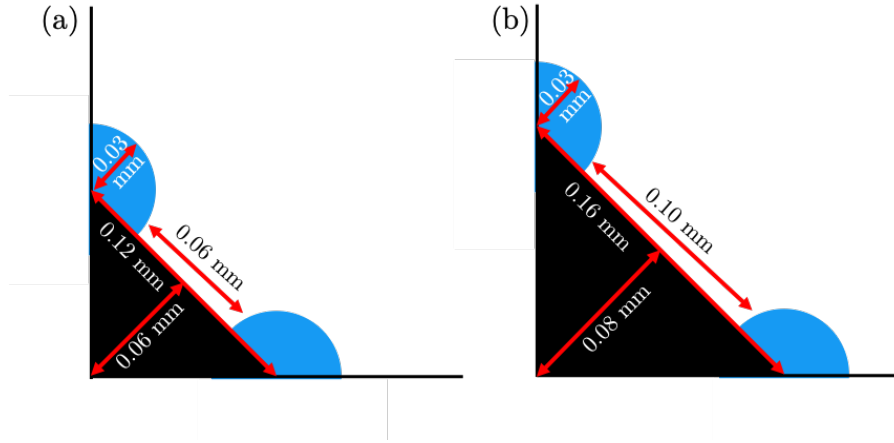


Figure 7.5: Schematic showing notches in black, colonies in blue at the notch edge, and the distance between the known colonies on the crack front for (a) Specimen A with 0.06 mm starter notch, (b) Specimen B with 0.08 mm starter notch.

7.3 Results

The first specimen, Specimen A, was notched with twelve 0.06 mm notches. As discussed in Chapter 5, when a notch is smaller than the microstructural features the crack will initiate from the underlying microstructure. As such it was thought that having multiple small notches would cause one of these to be in a preferential colony and result in earlier initiation. The notches chosen in Figure 7.3 were 1 and 7 are highlighted in red, due to them having favourable colony orientations next to the notch. This sample initiated away from the notch due to machine misalignment. However, initiation was seen from notch 1, but the specimen ultimately failed elsewhere. This occurred at a mean load of 177 MPa with $\Delta\sigma = 144$ MPa, which corresponds to a fatigue crack growth threshold of $2.7 \text{ MPa}\sqrt{\text{m}}$. In Chapter 5 the fatigue crack growth threshold was found at 400°C in air of $4 \text{ MPa}\sqrt{\text{m}}$ through randomly oriented colonies and therefore in trans-lamellar fracture mode. The threshold found here is reduced as the initial crack path is more favourable due to the inter-lamellar fracture mode.

Notch 1 was seen to grow $12.5 \text{ }\mu\text{V}$ in 10 000 cycles. This corresponds to a crack length of 0.13 mm using the calibration curve, Equation 4.2, and therefore to a crack growth rate of $1.3 \times 10^{-8} \text{ m/cycle}$. This cracking from notch 1 can be seen in Figure 7.6, which shows the optical images of the cracks from the notch on both sides overlain in red boxes on the backscattered

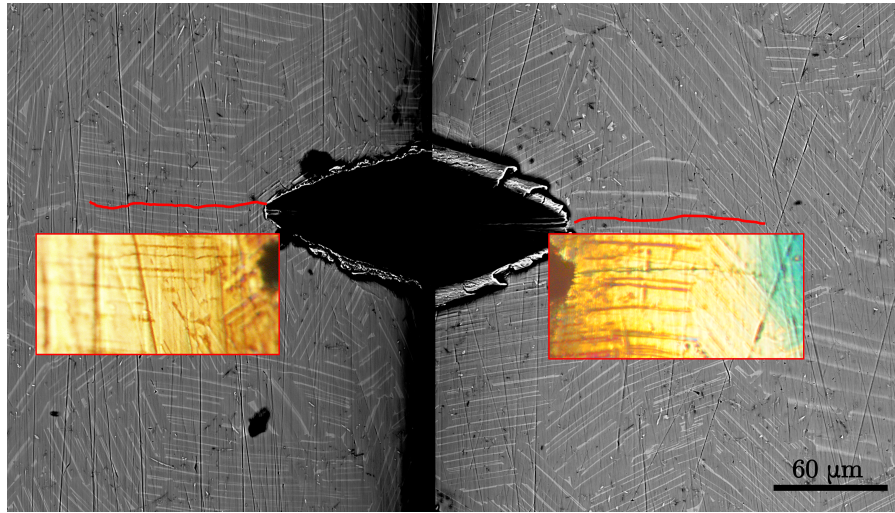


Figure 7.6: A crack initiated from notch 1 on Specimen A along both sides of the notch. Optical images of this crack are inset in red boxes, and the cracks are indicated with the red lines on the backscatter image.

image of the notch. The crack paths have also been marked in red. The right hand side of the notch can be seen to initially crack through a γ lamellar with inter-lamellar cracking, then trans-lamellar cracking straight through the following colony, creating a flat crack path. The left hand side follows a flat crack path mainly along the lamellae boundaries of γ and α_2 . The right hand side was polished to remove the oxidation from testing and an EBSD map made, Figure 7.7. The colony orientations can be seen to differ due to polishing back through the material to expose the microstructure. However, the underlying colony can be seen to have a favourable orientation. It can be seen that once a crack is growing, that a change in the colony

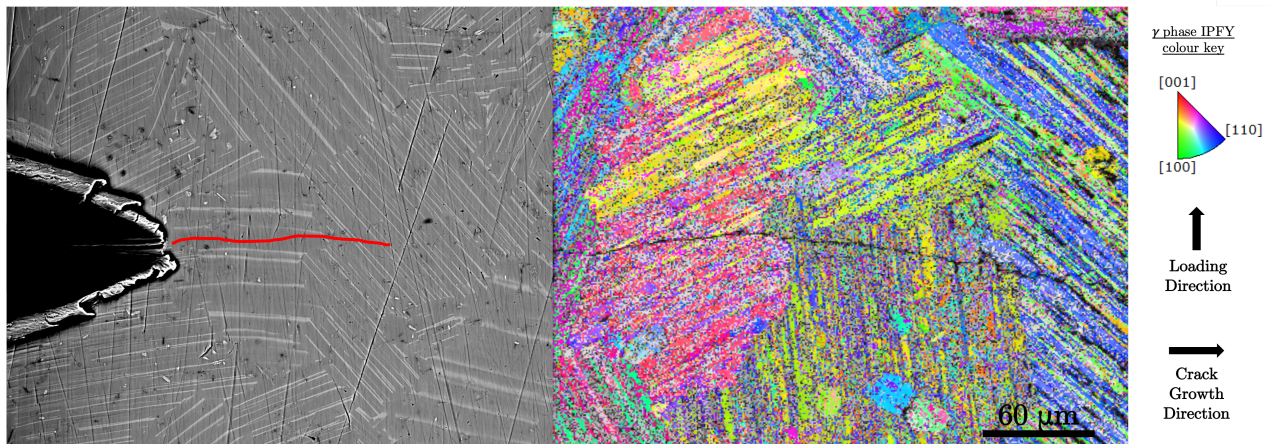


Figure 7.7: Backscatter image of Specimen A notch 1 with the crack path indicated in red with the corresponding EBSD map showing the crack path and colony orientation.

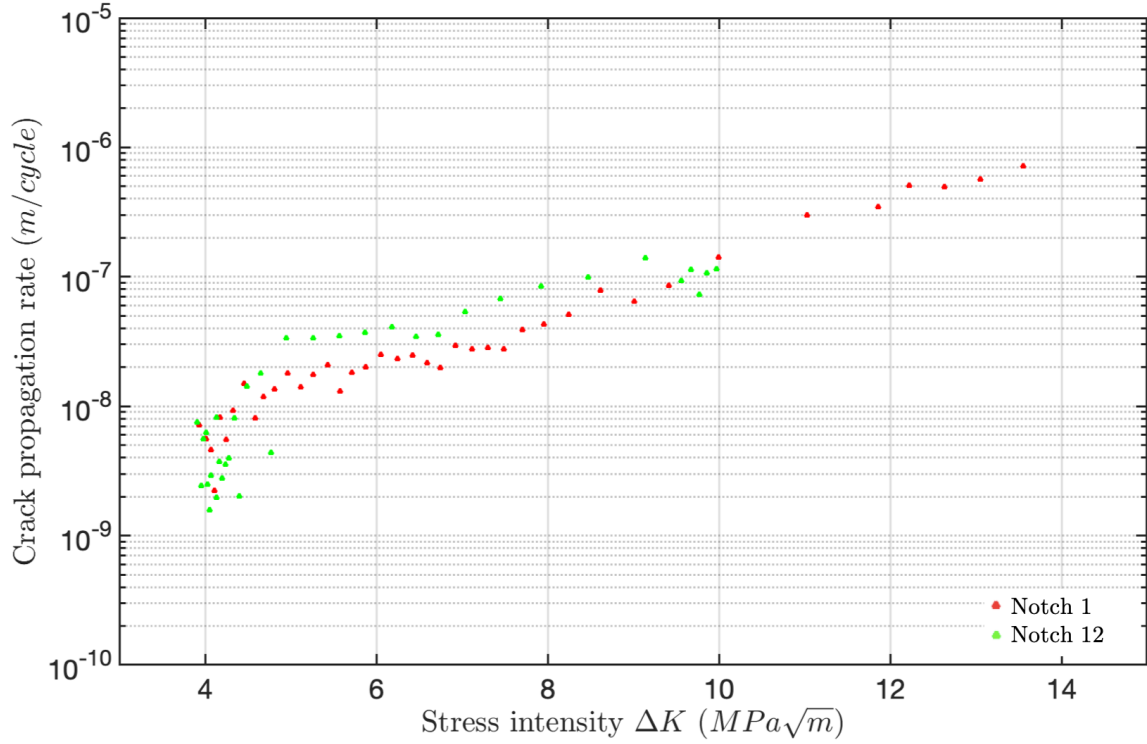


Figure 7.8: The fatigue crack growth curves for 0.08 mm starter notches 1 and 12 on Specimen B, showing similar values for threshold of 4.1 and 4.0 MPa $\sqrt{m}$ respectively, and similar crack growth rates.

orientation does not arrest the crack growth, nor does the crack path migrate to the local grain boundary. Notch 7 was not seen to grow at all from the notch.

The second specimen, Specimen B, was machined with twelve 0.08 mm notches so as to promote initiation from a notch rather than from elsewhere in the microstructure. The notches chosen in Figure 7.4 were 1 and 12 and were on the same plane. This specimen was seen to initiate at the notch at a mean load of 226 MPa with $\Delta\sigma$ of 370 MPa. The threshold for notch 1 was 4.1 MPa $\sqrt{m}$ and the threshold for notch 12 was 4.0 MPa $\sqrt{m}$. None of the other notches were seen to initiate a crack. Due to the large number of colonies on the crack front, the fatigue crack growth threshold is the same as that found in Chapter 5, for trans-lamellar fracture mode initiation. The fatigue crack growth curve for both of these notches is reported in Figure 7.8. This shows that the thresholds were similar, as were the crack growth rates of the two notches. Notch 1 can be seen to grow to a larger ΔK of approximately 14 MPa $\sqrt{m}$, corresponding to a crack length of approximately 1 mm before final fracture. Whereas notch 12

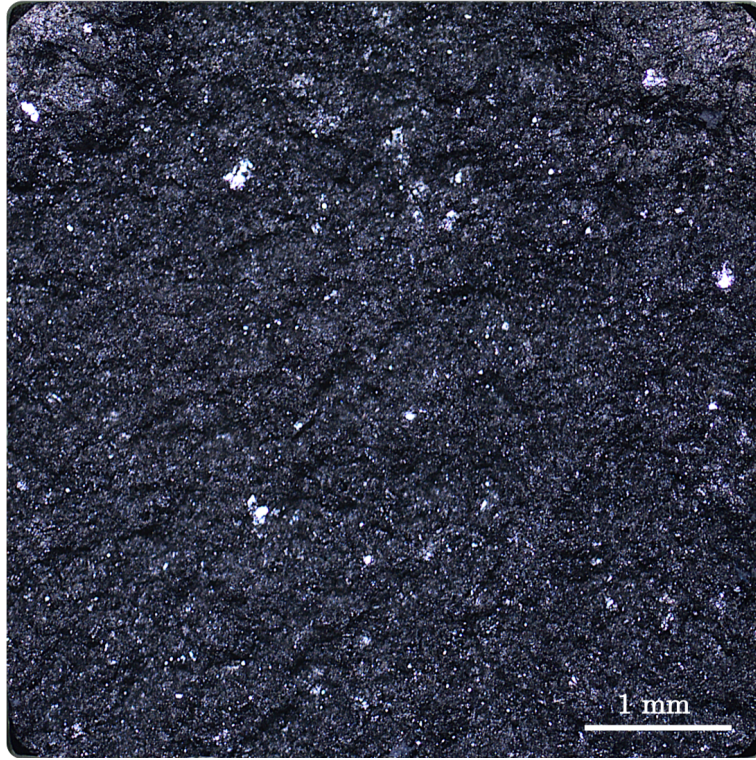


Figure 7.9: Fracture surface of Specimen B. Notch 1 is in the top right and shows a larger region of fatigue. Notch 12 is top left and shows a smaller region of fatigue.

grows to approximately $10 \text{ MPa}\sqrt{\text{m}}$, corresponding to a crack length of approximately 0.5 mm before final fracture. Notch 12 shows deceleration of its crack growth rate at ΔK greater than $9 \text{ MPa}\sqrt{\text{m}}$. Although it is far from the dominant crack at notch 1, 5 mm at crack initiation and 3.5 mm at last measurement before final fracture. Notch 12 may have been protected due to strain relaxation-unloading of this crack due to opening at notch 1.

Figure 7.9 shows the fracture surface of Specimen B. Crack growth can be seen from two

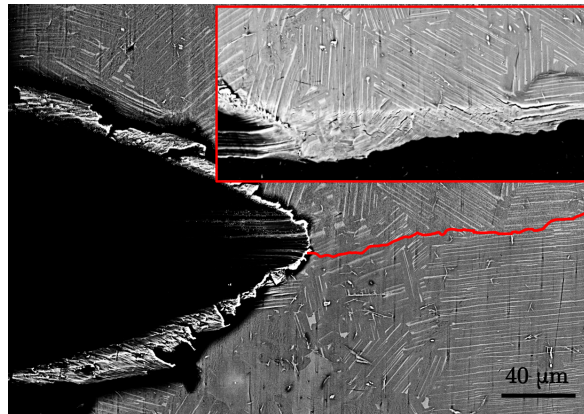


Figure 7.10: Specimen B notch 1 with the crack path indicated with the red line. Inset in the red box is the backscattered image of the fractured specimen gauge.

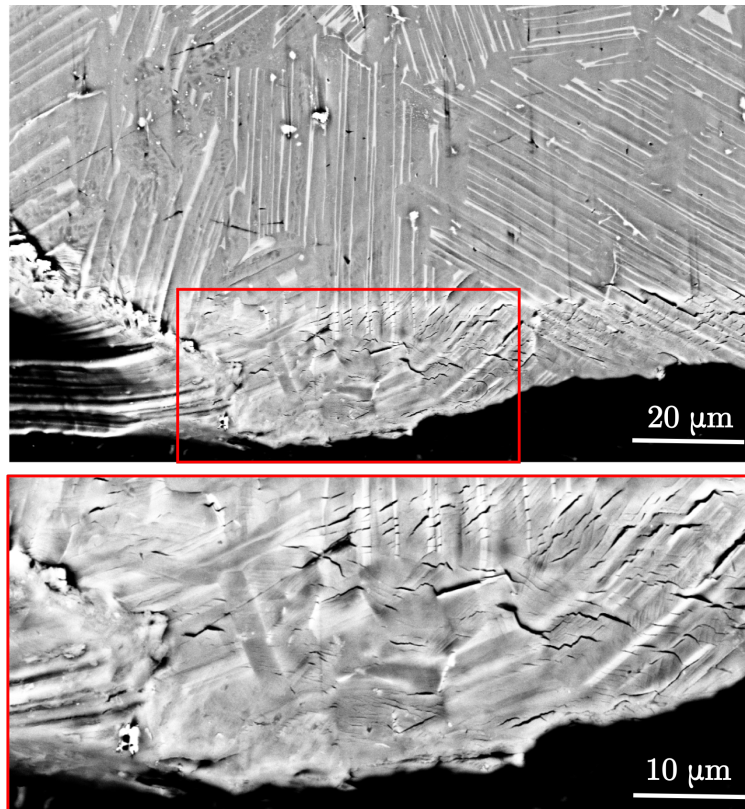


Figure 7.11: Specimen B notch 1 near-notch subsurface deformation.

notches. The top right notch shows a larger region of fatigue crack growth of approximately 1 mm, relating to notch 1. The top left region of crack growth relates to notch 12 of approximately 0.5 mm, showing a smaller region of fatigue. Upon careful inspection, it can be seen that there are river lines connecting these two regions of fatigue.

Figure 7.10 shows notch 1 in Specimen B with the crack path in red. Inset above the crack

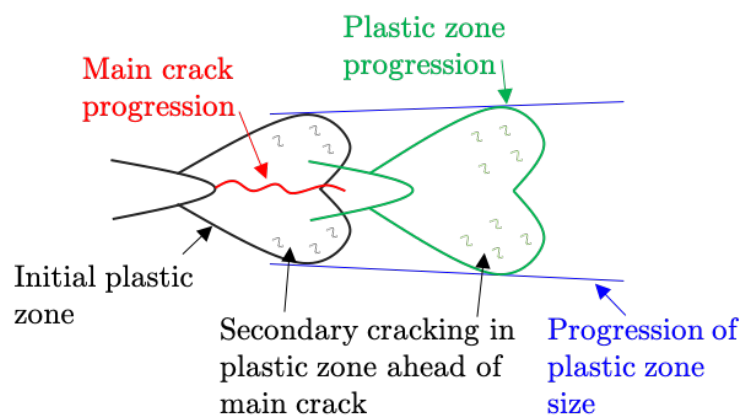


Figure 7.12: Schematic showing crack progression being preceded by the deformation by the plastic zone.

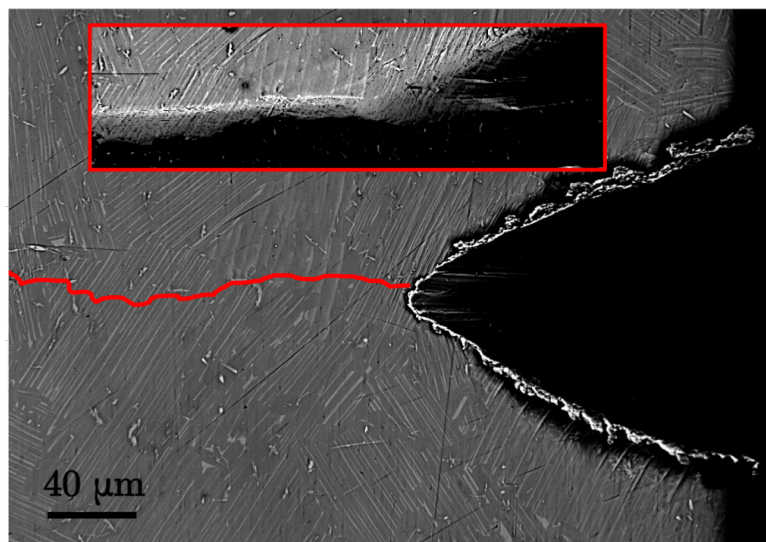


Figure 7.13: Specimen B notch 12 with the crack path indicated with the red line. Inset in the red box is the backscattered image of the fractured specimen gauge.

path is the backscatter image of the fractured specimen gauge. This crack path is found to travel along the lamellae and around grain boundaries to give a flat crack path. The deformation seen in the near-notch environment and is shown in Figure 7.11. This shows the plastic deformation that has been undertaken a distance into the material including microcracking. Confusingly, the extent of the microcracking does not seem to be at a constant depth relative to the fracture surface. However, it is at a constant depth relative to the average fracture plane. It is inferred that the microcracking occurs in the plastic zone prior to progression of the main crack front, schematically depicted in Figure 7.12. However, such extensive microcracking was not observed in sectioned samples, only at the surface of the test specimen, although some secondary microcracking is frequently observed in sections. The occurrence of microcracking in the plastic zone does give some hope that, at least in some stress states, there may be toughening mechanisms that can operate.

Figure 7.13 shows notch 12 in Specimen B with the crack path in red and backscatter image of the specimen gauge inset. This crack path is trans-lamellar through the colonies. In both the specimens it can be seen that trans-lamellar cracking is preferred over lamellar decohesion.

7.4 Conclusions

1) Fatigue crack growth threshold at 400°C in air in inter-lamellar fracture mode is $2.7 \text{ MPa}\sqrt{\text{m}}$. This is reduced from the fatigue crack growth threshold of $4 \text{ MPa}\sqrt{\text{m}}$ through randomly oriented grains near-notch in trans-lamellar fracture mode as found in Chapter 5.

2) ΔK_{th} and crack growth rate similar to bulk material for the larger 0.8 mm notches due to the fracture mode again being trans-lamellar as in Chapter 5.

3) Microstructural initiation can occur even if the worst single colonies are selected *i.e.* of multiple similar colonies.

4) Once a crack is growing, carries on into adjacent colonies rather than locally arresting, or migrating to a crack path on a grain boundary.

Chapter 8

Effect of Temperature Excursions on Crack Growth

8.1 Introduction

The two phase alloy FCT γ -(TiAl) and HCP α_2 -(Ti₃Al) only has a restricted number of available slip systems below around 700°C [2, p. 172]. Above this point, there is a change in deformation mechanisms, so more slip systems are available, dropping the yield strength but increasing the ductility. It is interesting to ask how this affects fatigue crack growth behaviour given the potential for over-temperature events in operation, although probably not of the magnitude examined here. This chapter aims to explore these effects.

The overall ductility in both the α_2 and the γ phase independently increase as temperature rises from room temperature to 800°C, increasing the ductility of the overall alloy Ti-4522XD. Deformation at room temperature is mainly confined to the γ phase of the two phase alloy [2, p. 167]. α_2 is seen to deform less, attributed to its increased solubility for alloying elements compared to the γ phase. This is understood to happen because dislocations are more easily pinned, *e.g.* at solute atoms, and so its plasticity is more limited [2, p. 19]. The γ phase deforms by octetedral glide of ordinary dislocations with the Burgers vector $\mathbf{b}=\frac{1}{2}\langle 110 \rangle$ and superpartial dislocations with the Burgers vectors $\mathbf{b}=\langle 101 \rangle$ and $\mathbf{b}=\frac{1}{2} \langle 11\bar{2} \rangle$. Mechanical twinning in the γ is a secondary deformation mode [141].

At 800°C the γ phase deforms under glide modes similar to those occurring at room temperature, with strain accommodation occurring by climb of ordinary dislocations [2, p. 171], relaxing the need for additional independent slip systems. γ also undergoes increased mechanical twinning at higher temperatures. The increased ductility of α_2 at the increased temperature of 800°C can be attributed to activation of prismatic slide and a relatively dense population of dislocations on the prismatic planes. Climb of these dislocations allows increased deformation. Temperatures above 700°C also increase the activation of pyramidal slip. These additional deformation mechanisms of both phases accelerate the interfacial accommodation problem between the two phases and contributes to the increased ductility of the alloys at increased temperatures, *e.g.* due to improved slip transfer.

In this chapter, the effect temperature excursions on the fatigue crack growth behaviour and associated sub-surface deformation mechanisms are examined. Specifically, this analysis will investigate whether the plasticity introduced at 750°C local to the crack tip will protect against further crack growth at a lower temperature of 400°C due to the introduction of additional slip systems.

8.2 Experimental methods

The material condition and preparation are detailed in Chapter 3. Indirect crack growth measurement was carried out using direct current potential drop (DCPD) and threshold was determined as detailed in Chapter 4. The starter notch size was 0.5 mm throughout. During fatigue testing, after crack growth at 400°C to a pre-determined ΔK , the loading waveform was paused, load decreased to a nominal value and the temperature increased to 750°C, stabilised within $\pm 0.5\%$ of the desired temperature, using a three-zone radiant furnace. The load was then returned to the mean value, and the loading waveform re-applied. The crack was then left to grow until a pre-determined ΔK . The waveform was again paused, load decreased, and temperature returned to 400°C, and left to stabilise. The same waveform was again re-applied and the crack allowed to grow to failure. This can be seen in detail in Figure 8.1.

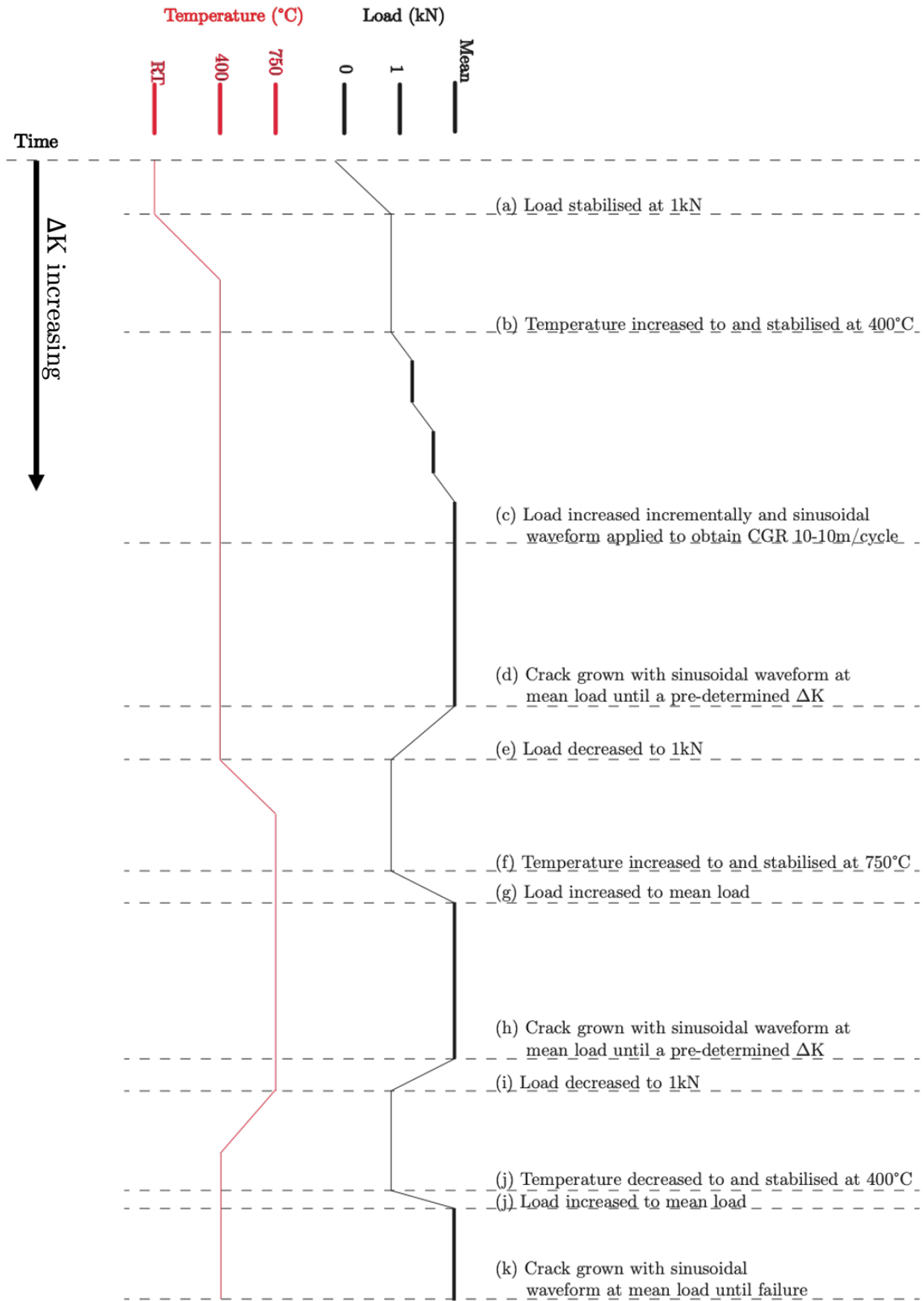


Figure 8.1: Evolution of temperature and maximum load during the fatigue testing through the test sequence, showing how the changes in temperature were applied.

8.3 Results

Isothermal response fatigue crack growth curves at 400 and 750°C are shown in Figure 8.2. At 400°C ΔK_{th} in trans-lamellar fracture mode was 4.0 MPa $\sqrt{m}$, Chapter 5. At an increased temperature of 750°C, ΔK_{th} increases to 7.1 MPa $\sqrt{m}$. This was tested using a 0.5 mm notched specimen, and therefore initiating through randomly oriented colonies local to the notch and therefore in trans-lamellar fracture mode. However, the fatigue crack growth rates in the steady-state (Paris) fatigue crack growth regime are observed to be around 3 - 10 $\times$ greater, with a concomitant reduction in life.

Four tests were carried out in which the temperature was increased from 400 to 750 °C for differing ranges of ΔK , which has been summarised in Table 8.1. Each of the fatigue crack growth curves show points with the data, blue data points at 400°C and red data points at 750°C. This data is overlain with a blue line for the isothermal response curve at 400°C and a red line for the isothermal response curve at 750°C.

Test 1, Figure 8.3, had an increase in temperature in the range of 7.8 MPa $\sqrt{m}$ to 9.4 MPa $\sqrt{m}$.

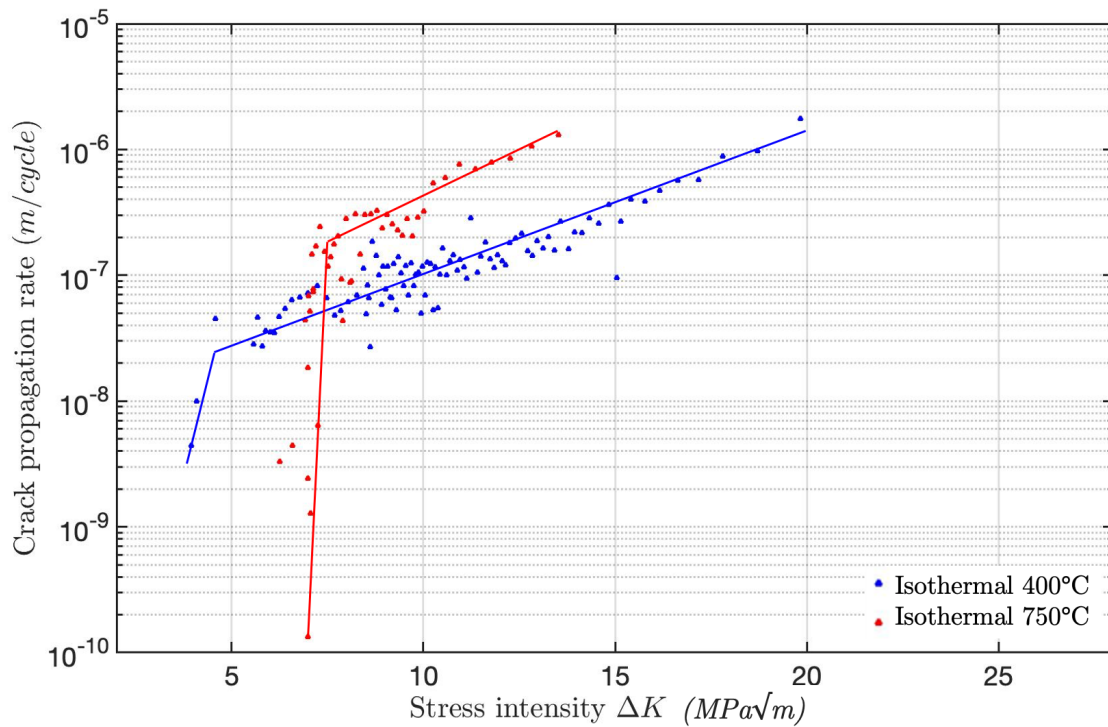


Figure 8.2: Isothermal fatigue crack growth curves obtained at 400 and 750°C in γ -(TiAl) alloy 4522XD in the as-cast and HIP condition. Guides to the eye are added to aid the reader.

Test	ΔK temperature increase MPa $\sqrt{m}$	ΔK temperature decrease MPa $\sqrt{m}$
1	7.8	9.4
2	5.6	8.5
3	7.0	11.1
4	7.4	n/a

Table 8.1: Table of tests and their values of ΔK for temperature increase and decrease.

In the increased temperature region there is seen to be an initial burst in growth, showing a high crack growth rate. This is thought to be due to the crack growing in inter-lamellar fracture mode through a colony. This high crack growth rate is short lived. The fracture path is impeded by the following colony. The ΔK is then close to the trans-lamellar fatigue crack growth threshold at 750°C in air, and therefore the crack growth rate slowly stabilises. It is uncertain whether it grows out to equilibrium for 750°C. When the temperature is then decreased again, the crack growth rate retards due to a larger plastic zone size and larger yield stress, 430 MPa at 400°C and 350 MPa at 750°C [142]. This slowly grows out to equilibrium to the isothermal response curve for 400°C. Test 3, Figure 8.4, can be seen to give similar results, with the elevated temperature portion in the ΔK range of 7.0 MPa $\sqrt{m}$ to 11.1 MPa $\sqrt{m}$. In the increased temperature region, this test can be seen to show an initial burst of growth due to growing in inter-lamellar fracture mode, and then being impeded by the following colony. The crack growth rate grows out to equilibrium more slowly than test 1 due to stress intensity factor range being close to the threshold at 750°C in air. Test 3 again gives no indication that the crack growth rate would have grown out to the isothermal response curve.

In test 2, Figure 8.5, the increase to 750°C was performed below the isothermal fatigue crack growth threshold in trans-lamellar fracture mode at 750°C. The temperature was increased from 400°C to 750°C at ΔK of 5.6 MPa $\sqrt{m}$, but the ΔK_{th} is 7.1 MPa $\sqrt{m}$. This causes the crack growth to retard by 1000 x. However, due to the sharp crack already initiated, the crack slowly grows out. This gives the fatigue crack growth threshold in inter-lamellar fracture mode at 750°C in air to be 5.8 MPa $\sqrt{m}$. It is again uncertain whether this increase in temperature region grows out to the 750°C fatigue crack growth curve. Once the temperature was decreased again at ΔK of 12 MPa $\sqrt{m}$ the bigger plastic zone size again retards crack growth.

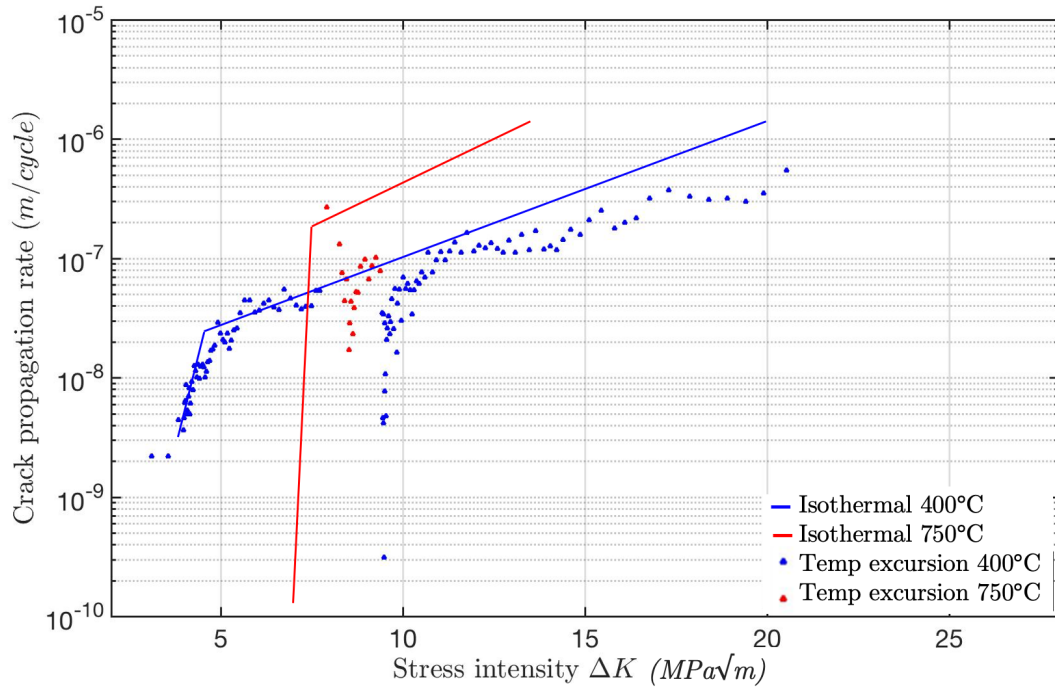


Figure 8.3: Fatigue crack growth test 1, with a temperature increase from 400 to 750 °C for the interval from 7.8 - 9.4 $\text{MPa}\sqrt{\text{m}}$. The isothermal behaviour guides-to-the-eye are overlain.

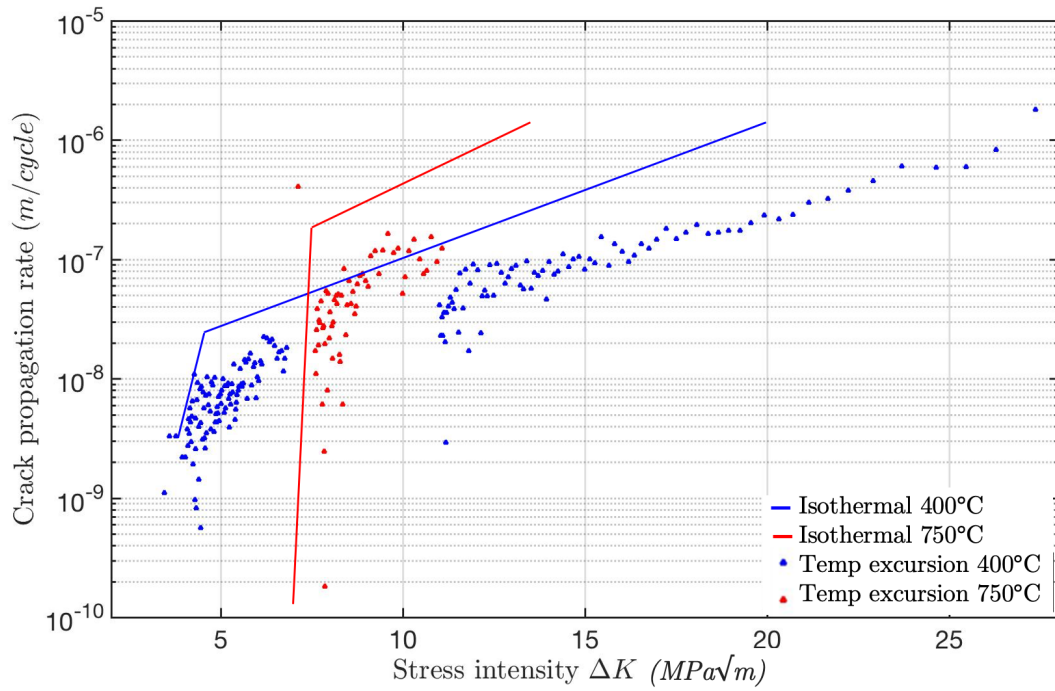


Figure 8.4: Fatigue crack growth test 3, with a temperature increase from 400 to 750 °C for the interval from 7.0 - 11.1 $\text{MPa}\sqrt{\text{m}}$. The isothermal behaviour guides-to-the-eye are overlain.

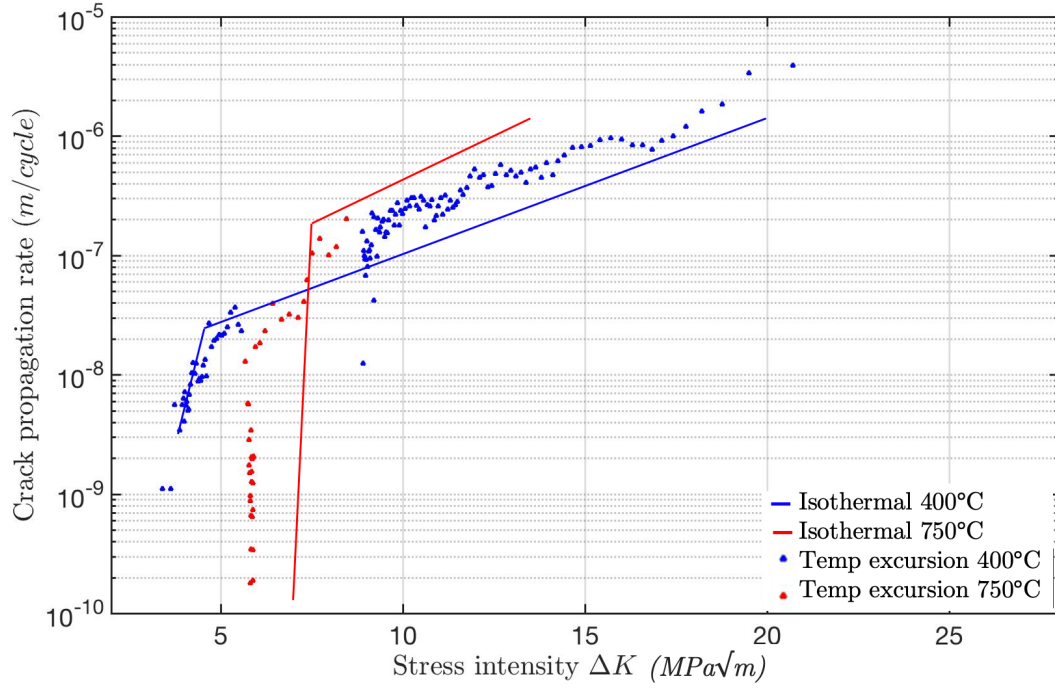


Figure 8.5: Fatigue crack growth test 2, with a temperature increase from 400 to 750 °C for the interval from 5.6 - 8.5 $\text{MPa}\sqrt{\text{m}}$. The isothermal behaviour guides-to-the-eye are overlain.

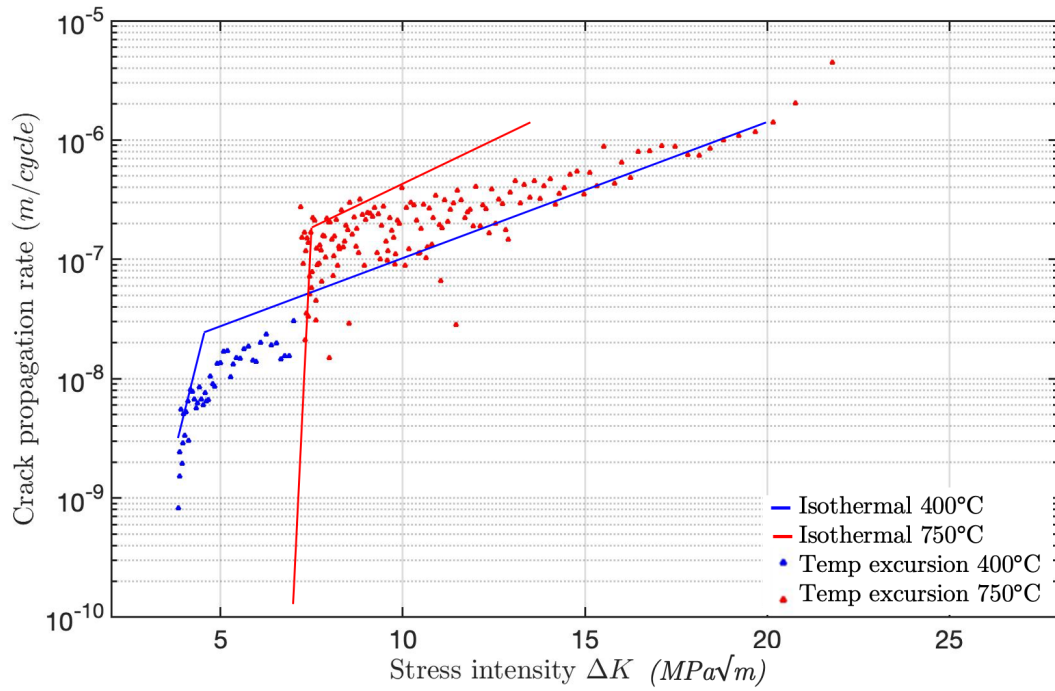


Figure 8.6: Fatigue crack growth test 4, with a temperature increase from 400 to 750 °C after 7.4 $\text{MPa}\sqrt{\text{m}}$. The isothermal behaviour guides-to-the-eye are overlain.

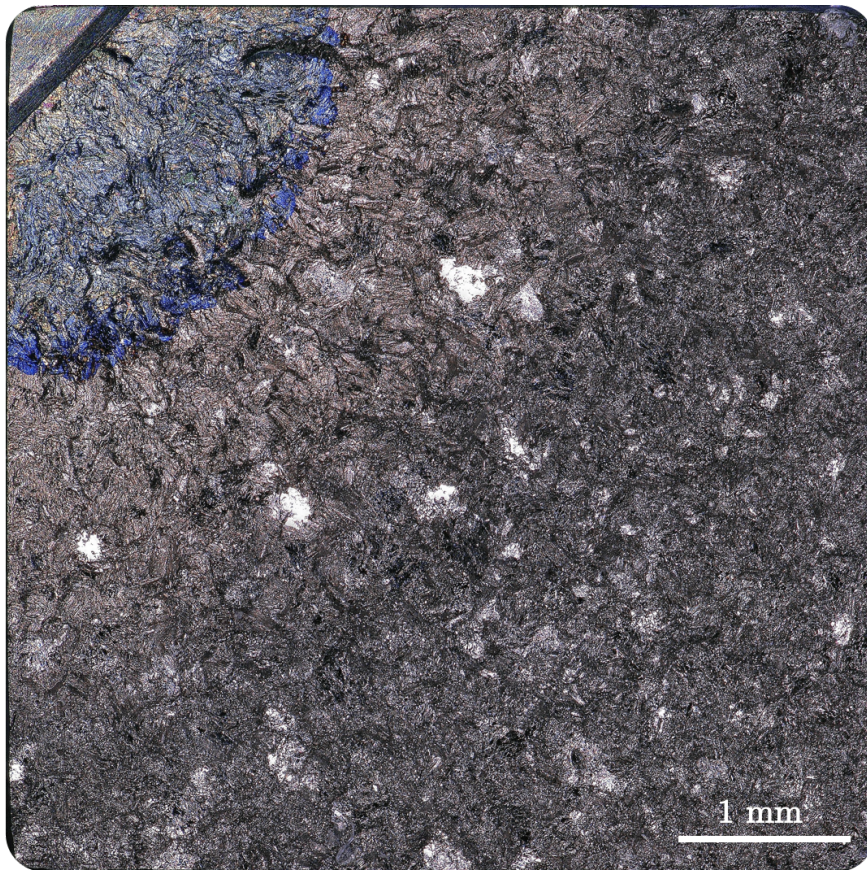


Figure 8.7: Fracture surface from the sample in Figure 8.3, with temperature excursion in the ΔK range 7.8 - 9.4 MPa $\sqrt{\text{m}}$. A thick blue oxide scale is present in the region up to 7.8 MPa $\sqrt{\text{m}}$, and a thinner blue band in the over temperature region of fatigue crack growth.



Figure 8.8: Tilted view of the fracture surface in Figures 8.3 and 8.7. It can be observed that at larger crack length shiny flat regions are observed and pull out of whole colonies, whereas cleavage of the colonies is observed as shorter crack lengths.

In test 4, Figure 8.6, the temperature was increased to 750°C at a ΔK of 7.4 MPa $\sqrt{\text{m}}$ until failure, without returning to 400°C. It was shown in Chapter 5 that there is large variability in the crack propagation rate for the same material in the same test environment. In Figure 8.6 it can be seen that there is an increase in the crack growth rate from 400 to 750 °C. This is therefore indicative of the crack growth rate growing out to the isothermal response curve.

Optically, the fracture surface can be seen to show discolouration from the oxide scale where the fracture surface was exposed to elevated temperatures. Figure 8.7 from test 1 shows the crack growing out from the starter notch (top left) until ΔK of 7.8 MPa $\sqrt{\text{m}}$. This initially grows out at 400°C. There is then the temperature increase to 750°C for a short period until ΔK of 9.4 MPa $\sqrt{\text{m}}$, shown by the darker blue band. The temperature is then dropped to 400°C until failure. The last region has only experienced 400°C and therefore can be seen to have little-to-no colouration. The thin region of darker blue has experienced both 400°C and 750°C. The initial, most oxidised region of lighter blue has experienced 400°C then 750°C then finally 400°C. Figure 8.8 shows this same fracture surface, from a tilt, enabling morphology to be exposed. Typically in the near-notch environment of the fracture surface the crack growth is more trans-lamellar. As the value of ΔK increases with the increase in crack size, there is an increase in the plastic zone size. This causes the plastic zone size to increase to larger than that of the colonies, thus causes the surface morphology to change to pull out entire prior β grains, leading to inter- and intra-lamellar crack growth and increased roughness. The fracture surface shows no considerable change in surface morphology in the transition across temperatures despite the change in the plastic zone size and deformation mechanisms.

This same fracture surface of test 1 was polished back perpendicular to the surface and an EBSD map was generated of the region before, during and after the temperature excursion. This map only contains the orientations of the γ phase. This is shown in Figure 8.9 with an SEM image below of the corresponding perpendicular fracture surface. The SEM image shows no visible change in either morphology due to the increase in plastic zone size during the temperature excursion, or in colour. The EBSD map shows no noticeable change through the temperature excursion with increased mechanical twinning or increased deformation. The optical micrograph shows the only indication of the temperature excursion with the oxide scale.

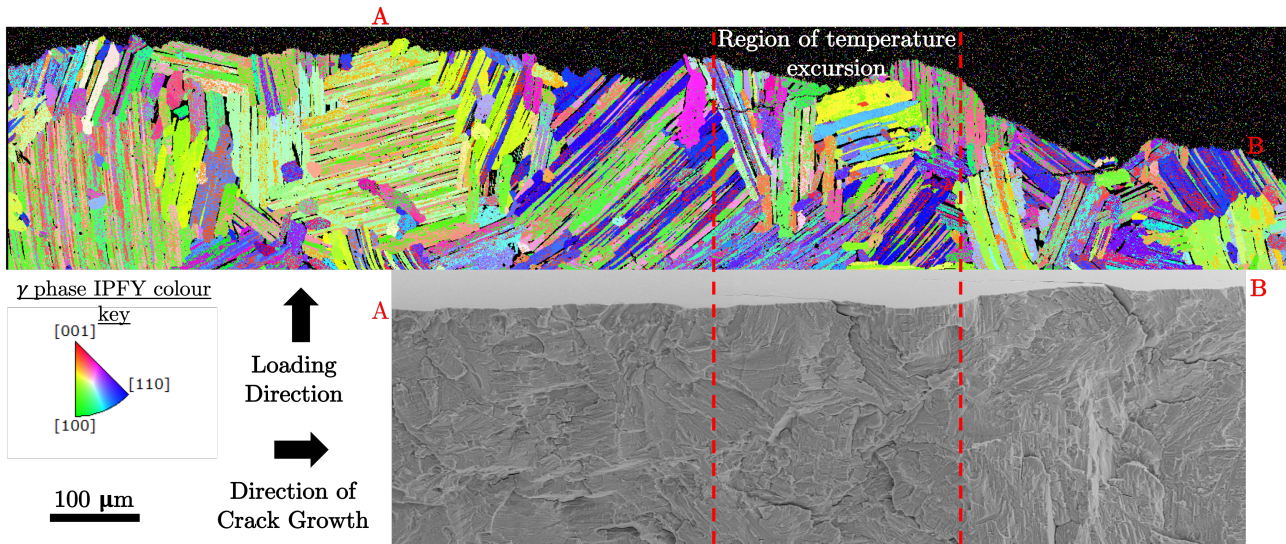


Figure 8.9: Temperature excursion test 1 EBSD map of the cross section perpendicular to the fracture surface through the region of temperature excursion, SEM image below directly relating to the EBSD. The loading direction and y direction are aligned up the page for the EBSD.

8.3.1 Compressive event

Application of a compressive load during crack growth causes crack retardation. Figure 8.10 shows the initial crack growth following that of the isothermal response curve at 400°C. At a ΔK of approximately $5 \text{ MPa}\sqrt{\text{m}}$ a compressive load of approximately 6 kN is applied. The

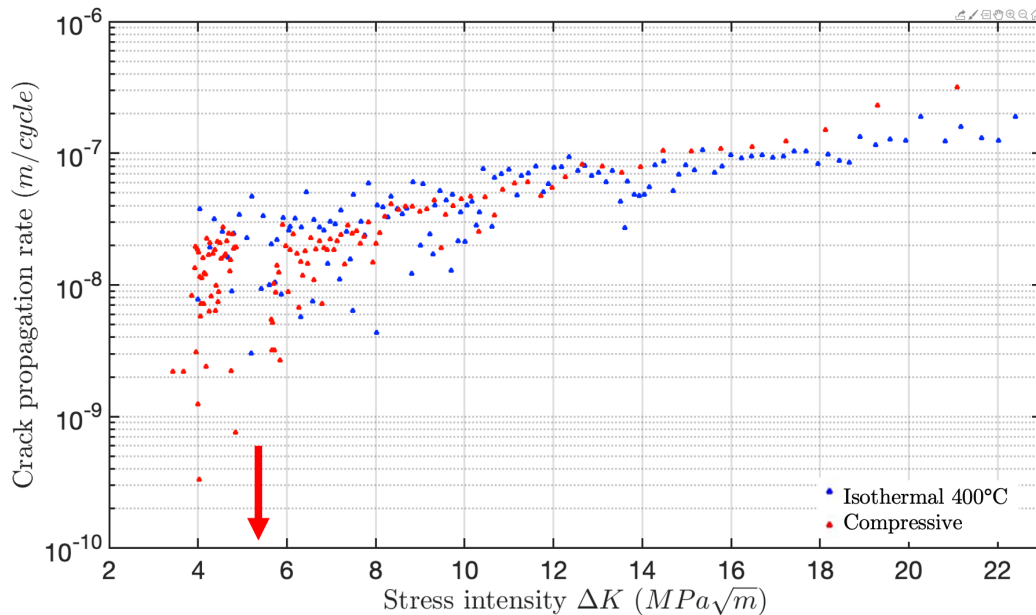


Figure 8.10: Fatigue crack growth curve of the application of the compressive load of 6 kN at a ΔK of approximately $5 \text{ MPa}\sqrt{\text{m}}$ indicated by the red arrow, compared to the isothermal response curve at 400°C.

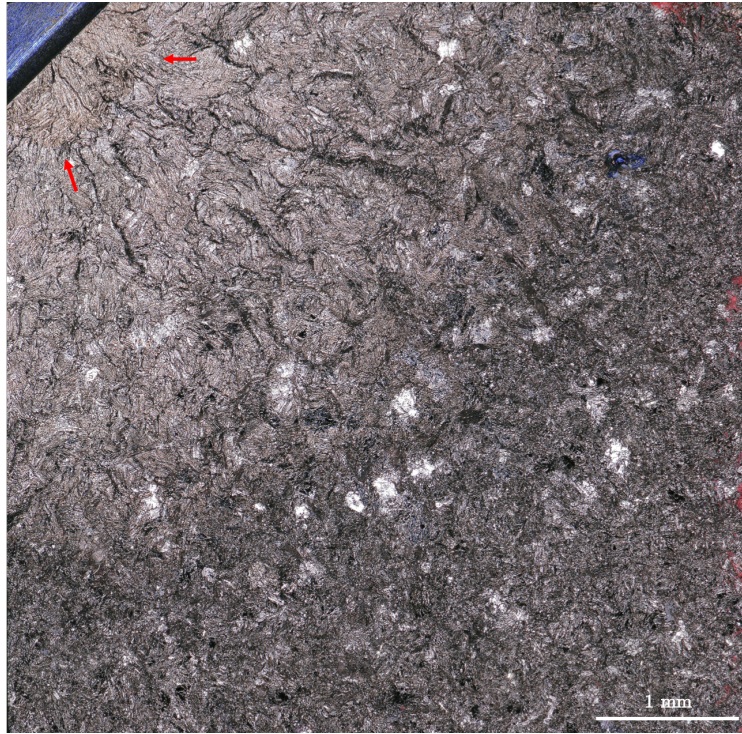


Figure 8.11: Fracture surface of the application of the compressive load during crack growth. There is a brown ring in the early crack growth marked by red arrows. This is due to oxidation because of being held at temperature for longer than rest of sample when trying to grow crack out after the application of the compressive load.



Figure 8.12: Fracture surface at a tilt of the application of a compressive load during crack growth.

crack growth rate can be seen to decrease, and slowly grow out and return to the isothermal response curve. Figure 8.11 shows the fracture surface of the application of the compressive load during the crack growth. There is a brown ring that can be seen in early crack growth marked by the red arrows. This is due to the region being held at temperature for longer as the crack retarded and took longer to grow out after the compressive event. Figure 8.12 shows this same fracture surface tilted to expose the surface morphology. This shows that there is no indication on the fracture surface of this compressive load being applied other than the region of increased oxidation. This application of a compressive load was due to a mechanical failure, and was therefore was not an intentional test.

8.4 Conclusions

- 1) The fatigue crack growth threshold at 750°C in air for trans-lamellar fracture mode was found to be $7.1 \text{ MPa}\sqrt{\text{m}}$.
- 2) The fatigue crack growth threshold at 750°C in air for inter-lamellar fracture mode was found to be $5.8 \text{ MPa}\sqrt{\text{m}}$.
- 3) A step increase in temperature between the ΔK_{th} values for the inter-lamellar and trans-lamellar threshold values for the increased temperature causes an initial burst in the crack growth rate, quickly followed by a retardation in the crack growth rate. The crack growth rate is found to have greater retardation the closer the value of ΔK at which the temperature is increased at is to the inter-lamellar fracture mode ΔK_{th} .
- 4) A step decrease in temperature above the ΔK_{th} values shows crack growth rate retardation due to the larger plastic zone formed at the previously increased temperature.
- 5) The only method found in this work of detecting change in temperature is by optical microscopy, looking at the oxide scale colour. This oxide scale is not easily measurable, being less than a micron in thickness, in cross section. It not detectable by increased mechanical twinning in the region by looking at the EBSD maps obtained.
- 6) Application of a compressive load during crack growth causes a retardation in the crack growth.

Chapter 9

Conclusions and Further Work

9.1 Conclusions

Although γ -(TiAl) based alloys have limited ductility, they can be relatively tough. However, their fatigue strengths are relatively poor and their fatigue crack growth rates are high. Therefore it is imperative that cracks not be allowed to initiate and propagate, making the fatigue crack growth threshold critically important. This threshold is strongly microstructure and temperature dependent. The γ -(TiAl) based alloy under investigation was Ti-45Al-2Mn-2Nb-0.8 vol.% TiB₂-XD. Crack growth measurements were carried out using the DCPD method. In order to relate the voltage to the crack length, calibration was necessary. Usual methods for this include ‘beach marking’, the formation of marks on the fracture surface by applying an overload. This did not form any such marks on the γ -(TiAl) fracture surface, so calibration was instead carried out using heat tinting to form concentric bands of oxide scale in order to directly relate the colour banded crack lengths to the known voltage at each crack length. Due to having a small, 5 x 5 mm gauge cross-section, the usual method for fatigue crack growth threshold determination, pre-cracking, was difficult. Therefore, initially the effect of notching and this notch size was investigated on the threshold.

Corner crack specimens were fractured from machined notches of varying sizes: 0.5, 0.25, 0.1, 0.06 (diamond blade) and 0.03 (FIB) mm at 400°C in air. Specimens with notches larger than that of the average colony size (70 μ m) initiated from the notch. The fatigue crack growth

threshold for 0.5 mm starter notches was $4.02 \pm 0.12 \text{ MPa}\sqrt{\text{m}}$ for nine specimens, 0.25 mm starter notch was $4.0 \text{ MPa}\sqrt{\text{m}}$ for one specimen, and 0.1 mm starter notches was 4.0 and $4.1 \text{ MPa}\sqrt{\text{m}}$ for two specimens. The fatigue crack growth threshold was therefore found to be $4.03 \pm 0.11 \text{ MPa}\sqrt{\text{m}}$ for the twelve above listed specimens. These specimens fractured from the machined notch and therefore initiated through unknown and random orientations of lamellae within the colonies. Due to the large number of colonies being on the crack front, and the small crack growth per cycle, the nature of the crack growth would predominantly be trans-lamellar. The fatigue crack growth threshold determined for these starter notches is therefore the threshold for trans-lamellar growth.

The fracture surface for a specimen initiating from the machined starter notch can be seen to have an initial region of flatter crack growth in fatigue, then an increased roughness into tensile overload when sampling an area of multiple colonies. This is due to a change in fracture mode from predominantly trans-lamellar to mixed trans-, inter- and intra-lamellar. This transition corresponds to the plastic zone size becoming similar to that of the colony size. This is challenging to quantitatively discern.

Corner crack specimens with 0.06 and 0.03 mm starter notches fractured away from the notch. Fractures surface analysis was carried out on these using the knowledge gained from analysing fracture surfaces of specimens that failed from a known notch site. This allowed the fatigue region to be determined from the tensile overload region, and the initiation site to be determined within the fatigue region. The initiation site was seen to be a colony or colonies with lamellae orientation perpendicular to the stress axis. This is seen by a facet of inter-lamellar crack growth, with a neighbouring colony with intra-lamellar crack growth. These two unfortunately oriented grains on the 0.06 mm notched specimen were found to initiate, giving a fatigue crack growth threshold of approximately $4 \text{ MPa}\sqrt{\text{m}}$ at 400°C in air.

A single corner crack specimen had 12 x 0.06 mm notches machined and the local colony orientations imaged. Small 0.06 mm notches were previously found to not initiate a crack from the notch, but for the specimen to initiate elsewhere in the microstructure. However, it was thought multiple notches would increase the probability of a notch being next to a colony orientation that is preferential for initiating a crack. 0.06 mm notches allow most of the

notch-front colony orientations to be characterised by imaging, with only 60 μm of the crack front remaining unknown. It was possible to measure the crack growth data from two notches. The notches were chosen by the orientation of the local colony to the notch in respect to the stress axis. Notches chosen were those which would most likely grow by inter-lamellar fracture. Initiation was measured from one of the selected notches. Post-test imaging showed the initial inter-lamellar nature of the crack growth. The fatigue crack growth threshold was found to be $2.7 \text{ MPa}\sqrt{\text{m}}$ for inter-lamellar crack growth at 400°C in air. Once a crack was growing, it carried on into adjacent colonies rather than locally arresting or migrating to a crack path on a grain boundary. Another single corner crack specimen was notched with $12 \times 0.08 \text{ mm}$ notches. Due to the larger nature of these notches, there was a larger crack front. This therefore led to the initial crack growth being through multiple colonies and therefore trans-lamellar in nature. The fatigue crack growth threshold was found to be $4 \text{ MPa}\sqrt{\text{m}}$ at 400°C in air. This is in agreement with earlier results.

At 750°C in air a 0.5 mm notched corner crack specimen was found to have a fatigue crack growth threshold of $7.1 \text{ MPa}\sqrt{\text{m}}$. Again, due to the large number of colonies on the crack front this would be a trans-lamellar fatigue crack growth threshold. Fatigue crack growth was carried out at 400°C with a step change in temperature up to 750°C for a period of the crack growth. The temperature was then reduced back to 400°C and grown until failure. The ΔK range for this temperature increase varied. A step increase in temperature from 400 to 750°C at $5.6 \text{ MPa}\sqrt{\text{m}}$ was applied, below the fatigue crack growth threshold in air at 750°C . This was found to retard the crack growth rate, but to slowly grow out to the isothermal response curve at the current temperature. This is thought to be due to the crack having already initiated and been growing at the lower temperature, and there being a sharp pre-crack, and the crack growth being inter-lamellar and therefore again showing a lower threshold. The inter-lamellar fatigue crack growth threshold at 750°C in air is therefore $5.8 \text{ MPa}\sqrt{\text{m}}$.

When the step increase in temperature is carried out at a larger value of ΔK , $7 \text{ MPa}\sqrt{\text{m}}$, it can be seen that there is a brief burst of crack growth, the crack growth then retards substantially. This is due to the crack growth initially growing inter-lamellar through a colony, then reaching a colony boundary, impeding crack growth. The ΔK is then close to the trans-lamellar fatigue

crack growth threshold at 750°C in air, and therefore the crack growth rate slowly grows out to the isothermal response curve. When the step increase of temperature is again higher, *i.e.* 7.8 and 9 MPa $\sqrt{\text{m}}$, the retardation of the curve is seen to be less. This is due to the step temperature increase being larger than the trans-lamellar fatigue crack growth threshold at 750°C in air. When the temperature is step decreased from 750 to 400 °C it can be seen to cause a retardation in the crack growth rate. This is due to there being a large plastic zone size that was formed previously when the crack was growing at a higher temperature and therefore this retards the crack growth rate.

The temperature excursions are clearly visible using optical microscopy. This oxide scale is not easily measurable, being less than a micron in thickness in cross section. It not detectable by increased mechanical twinning in the region by looking at the EBSD maps obtained. Application of a compressive load during crack growth causes a retardation in the crack growth.

These fracture surfaces were seen to be a host for other features. Borides were seen to correspond with regions of initiation from microstructural features, but were not thought to be initiating features themselves. Blue-coloured fracture surface features were discovered but not found to be fatal, or show any signs of crack growth from them on the fracture surface. Limited ductility can be seen across the fracture surface. Surface delineations occur across the fracture surface and were not seen to have any relationship to the crack growth rate. Plastic deformation, including microcracking, was observed near the crack on the gauge length in the direction of the stress axis, a distance into the material. The depth of the microcracking was relative to the average fracture plane due to the microcracking occurring in the plastic zone prior to the progression of the main crack front. Secondary microcracking is frequently observed. This microcracking in the plastic zone gives indication that in some stress states, there may be toughening mechanisms that can operate.

9.2 Future work

After concluding the thesis, there are a few suggestions of future research that may be carried out continuing on from some of the themes.

Due to the γ phase being marginally tetragonal, less than 2%, EBSD techniques struggle to differentiate when indexing between the a and c components. Further work could be done in processing this data to distinguish between these components. Therefore, this would give the possibility of distinguishing twins. Being able to correctly index the γ phase would also open up possibilities for GROD, grain rotation orientation deviation. This would allow the grain misorientations to be calculated and therefore plasticity characterisation. This could be used to verify results in Chapter 8; the depth of the plastic zone into the material perpendicular to the fracture surface could be determined. This would verify the findings on the changes in plastic zone size causing the crack growth rate to vary. At a constant temperature the plastic zone size would slowly increase throughout the fracture surface. The plastic zone size would then rapidly increase in the region of increased temperature. Upon a step decrease in temperature, therefore would be too large a plastic zone size for the new test conditions and therefore the crack takes longer to grow out of this plastic zone, retarding the crack growth, until slowly growing out to the isothermal response curve crack growth rate.

Methods could be developed to discern fatigue from overload regions by interpreting the roughness, potentially using image analysis techniques associated with machine learning methods. Further work could also be carried out to explore natural initiation of the specimens, and characterise those regions further. This would include further investigation into the role of the borides into the initiation region, their role in toughening and their debonding stress. Investigation could also be made to further look at the role of α_2 in the crack front, to discover whether it, for example, can adsorb oxygen at the crack tip, analogous to its claimed role in chemically purifying the γ during microstructure formation.

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