

THE DEFORMATION OF ICE

by

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ABSTRACT

The aim of this investigation was to study the way in which single crystals of ice deform. However, as a preliminary to this, a modus operandi was developed for growing single crystals of known purity and preparing them for the experiments. This not only minimises the mechanical damage sustained by the crystals but also provides facilities for their controlled doping.

The surface changes that occurred when rectangular specimens of single crystalline ice were compressed under a constant load have been studied, and apart from the usual basal slip lines, several instances of non-basal ones were recorded. In addition, the distortion of the basal slip lines was studied, especially near grain boundaries and some instances of polygonisation boundaries were observed.

By using a modified formvar technique, silvered replicas of deformed ice surfaces were obtained, and the spacings of the basal slip bands were measured as a function of the resolved shear stress. For any specimen these showed considerable spread and Wakahama's relation gives only approximate values - especially at low stresses where very finely spaced lines were often observed.

The changes that occurred in the appearance of a deforming crystal when it was viewed between crossed polaroids were also studied and several interesting effects observed. Thus apart from the mass transport of material and interference effects at grain boundaries, some interference lines were seen, which were parallel to the basal plane. If the explanation given for them is correct, their presence signifies the active participation of non-basal dislocations in the deformation of ice.

Finally, by studying the stress-optical effect reported by Nakaya (1956), some information on the way the stress distribution changes during deformation and the associated relaxation was obtained.

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CHAPTER 1.

SURVEY OF PREVIOUS WORK.

1.1 INTRODUCTION.

Though it is probably quite correct to say that the plastic deformation of ice is not fully understood, the situation has improved considerably since 1950, when a renewed attack on the problem was made. At first glance, the lack of knowledge before this is rather surprising, as a large number of papers on the subject were already available. However most of these were concerned with the behaviour of a particular type of polycrystalline ice and so are of only limited relevance to the general problem. Recent investigators have started to remedy this by concentrating more on the fundamental parameters - thus intergranular effects have been eliminated by working with single crystals. Many experimental techniques have been used in these studies and will be considered below, but first, the few basic studies before 1950 must be surveyed.

1.2 EARLY WORKERS.

Although his main interest lay in the absorption and conduction of heat, Tyndall (1857) also studied the optical changes that occurred when a block of ice was

deformed. He found that axial compression produced cloudy lines in his specimens, which were probably parallel to the basal plane. This is not explicitly stated, but seems to be a reasonable conclusion as they were usually parallel to his flowers, which do form in this plane. (These "flowers" are actually melt figures and have been thoroughly studied by Nakaya).

Twenty years later, McConnell and Kidd (1888, 1890, 1891) performed the first experiments that were solely concerned with the deformation of ice. Though the apparatus was crude by modern standards, some of their conclusions are still valid today. Thus they found that:-

1. When deformed, single crystals of ice behaved like packs of cards.
2. The rate of deformation showed a tendency to increase with time and temperature.
3. This rate was related to the applied stress by a three-halves power law:-

$$\Delta \dot{\epsilon} \propto \left(\frac{\sigma_p}{\sigma_m} \right)^{3/2}$$

(where $\Delta \dot{\epsilon}$ is the change in the strain rate produced by changing the applied stress from σ_m to σ_p).

About the same time a rather similar investigation was carried out by Mücke (1895, 1900). However, he was

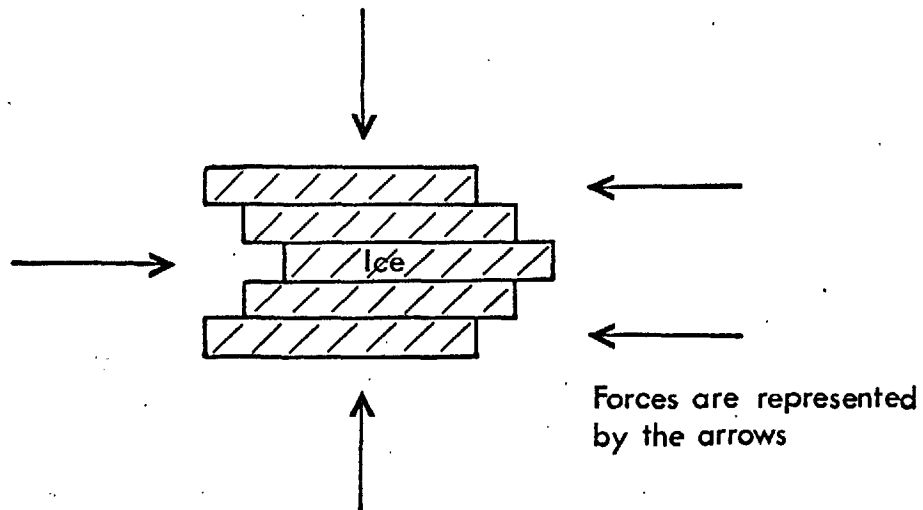
more concerned with the optical changes that occurred and in one of his papers there are some photographs which show a layer structure that formed when a block of ice was deformed. His stress system was such that the specimens were badly bent at the end of an experiment and the way the layers followed the contours of the bends is clearly visible in some of the photographs. In his later paper he argues that there is no reason to believe that internal melting has to occur for ice to deform and this conclusion is, of course, of fundamental importance.

The results of Tamman and Salge's (1928) were similar to those of previous workers, with one important exception. They measured the rate at which their specimens crept under a shear stress when a compressive stress was also present. (See Fig. 1). The creep rate seems to have been affected by the latter, though not unidirectionally. This raises the question of the compatibility of shear and uniaxial tests and may signify the presence of active non-basal dislocations.

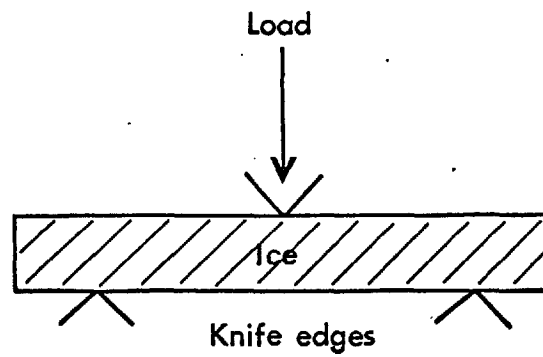
It is interesting to note that both Miggé and Tamman and Salge attempted to find a preferred glide direction in the basal plane by measuring the stress required to shear ice in various directions in this plane. None of these investigations gave a positive result, which may mean that such a direction does not exist.

Fig.1 Stress systems used by certain workers

(a) That of Tamman and Salge (1928)



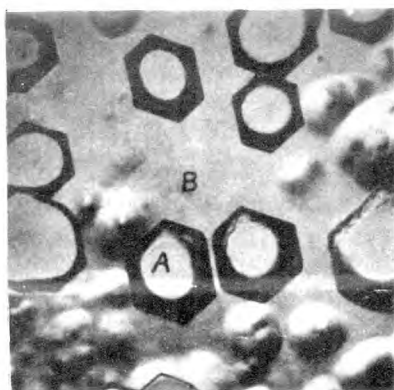
(b) That of Nakaya (1958)



1.3 ETCHING.

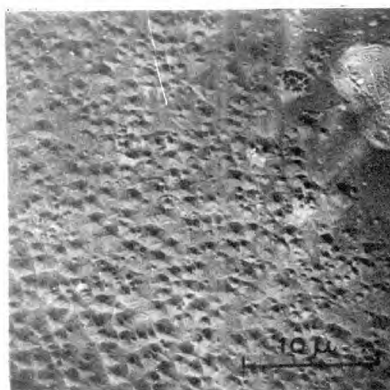
(In Fig. 2 a selection of photographs of the various types of etch pits is given as an aid in following the discussion).

It is usual to list etching techniques according to their mode of action - hence the terms chemical and thermal etchants (the former acting by dissolution and the latter by evaporation of the surface being studied). In the case of ice a great deal of confusion arose because the distinction between the two was not realised at the outset. This occurred because the action of the formvar films was not fully understood. Higuchi (1958), who discovered that a solution of formvar in ethylene dichloride could be used to etch ice, thought that its action was thermal. However, as Kuroiwa and Hamilton (1962) showed so clearly, though this is true in the case of the first coat, the bottoms of the pits formed by this are chemically etched by subsequent coats. This conclusion is really quite logical and could have been anticipated - for a thermal pit is formed where there is a hole in the formvar film and holes are least likely where it is thickest (i.e. over the bottoms of pits produced by the first application). Thus it seems that by retarding the chemical action of the ethylene dichloride on the ice surface with formvar, imperfections may be detected.

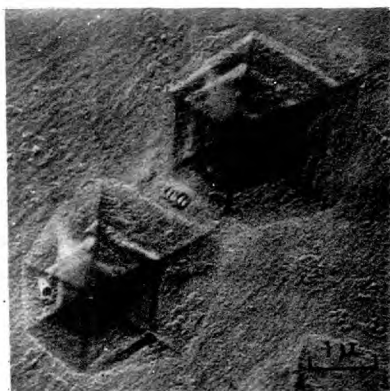


1/4 mm.

Thermal etch pits (Muguruma)
on basal plane

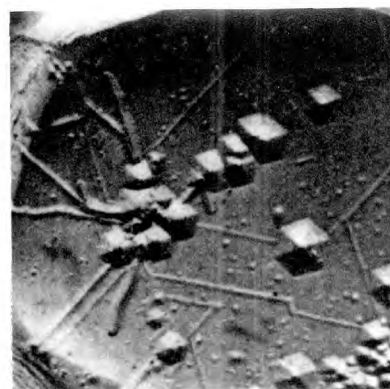


Chemical etch pits (Muguruma)
at B

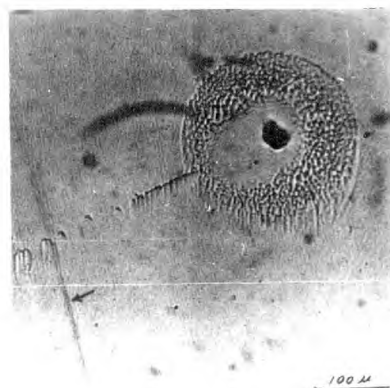


.05mm.

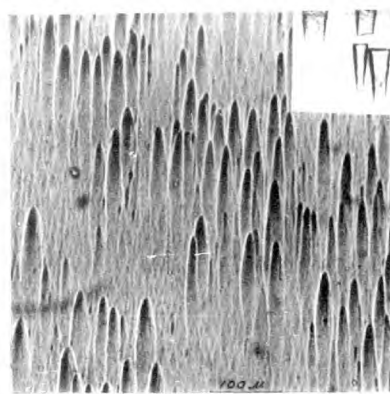
Chemical etch pits (Muguruma)
at A



$\langle 10\bar{1}0 \rangle$ Etch channels on (0001)
plane (Muguruma and Higashi)



Interaction between dislocations
and an inclusion (Kuroiwa and
Hamilton)



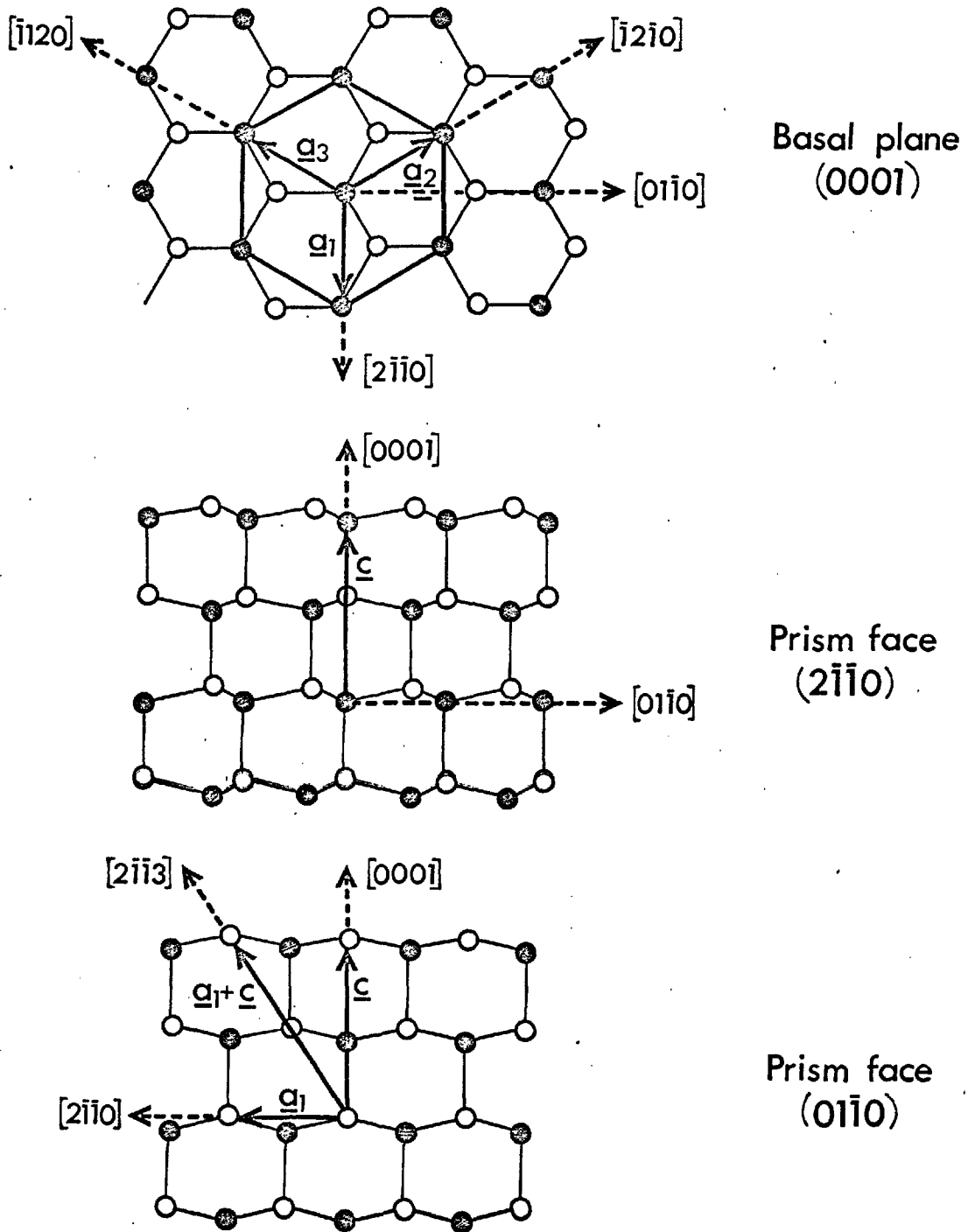
Elongated etch pits on prism face
(Kuroiwa and Hamilton)

By using simple thermal evaporation, Truby (1955) was able to obtain hexagonal pits on the basal surfaces of his specimens. These pits tended to overlap each other and some had steps in them. When the ice had been doped with caesium fluoride, both the depth and density of these pits was reduced. Again with the aid of thermal evaporation, Kuroiwa and Hamilton (1962) produced channels on the surfaces of their specimens. On prism faces these were parallel to the basal plane, but on the latter they trisected or bisected the $\langle 10\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ directions. (See Fig. 3). This result was quite unexpected and still requires an explanation.

There is no obvious correlation between the thermal pits produced with the aid of formvar replicas and any imperfections in the crystal's surface. However the crystallographic orientation of the surface is discernible from their shape - this being given by cutting a hexagonal column, whose axis is parallel to the c-axis, with a plane that is parallel to the surface of the crystal. The bottoms of these large thermal pits were found to be extremely smooth and have been used extensively in chemical etching.

Chemical etching with formvar solution also produces pits on the basal and prism faces which may be used to determine the orientation of the specimen's surface.

Fig. 3 The principle directions in ice



[The black and white atoms lie in different planes]

However thermal ones are better than these for this, as they have sharper boundaries and are bigger. It is now reasonably certain that the chemical pits occur where dislocations cut the surface, as the following observations show:-

1. Some of the pits have steps in them which can be explained by the presence of screw dislocations (Muguruma (1961); Bryant and Mason (1960)).

2. The density of the pits is lower at the bottom of a thermal etch pit than on the surface. This is to be expected as the surface is stressed during its preparation (Muguruma (1961, 1963); Muguruma and Higashi (1963 b.)).

3. If the surface is scratched, the density of these etch pits goes up by a factor of a hundred (Kuroiwa and Hamilton (1962)).

4. The pits interact with impurities and pile-up at grain boundaries (Kuroiwa and Hamilton (1962); Muguruma (1963)).

5. Slip bands are marked by rows of these pits and it is interesting to note that the rows are a trifle wavy, which may indicate cross-slip (Muguruma (1963)).

The pits produced by Truby (1955) were very similar to those discussed above, both in size and density. Thus it is quite possible that he did succeed in etching ice thermally.

There have been two attempts to correlate chemical etch pits on the two faces of an ice crystal and both failed. Bryant and Mason (1960) assumed this was due to the devious paths followed by dislocations in traversing the crystal; while Kuroiwa and Hamilton (1962) argued that it signified that the dislocations did not penetrate the crystal. At present it is impossible to decide between these two alternatives. An attempt to correlate the etch pits on two fracture faces (Kuroiwa and Hamilton (1962)) also failed, probably due to relaxation phenomena.

Rows of pits may be seen at grain boundaries and several workers (Kuroiwa and Hamilton (1962); Muguruma (1963); Bryant and Mason (1960)) have used their spacing to measure the angle of misfit. Unfortunately, none of these measurements was checked by using another technique such as the extinction of two grains between crossed polaroids. Such a check might provide additional information about the distribution of dislocations at these boundaries, especially if the two methods disagreed.

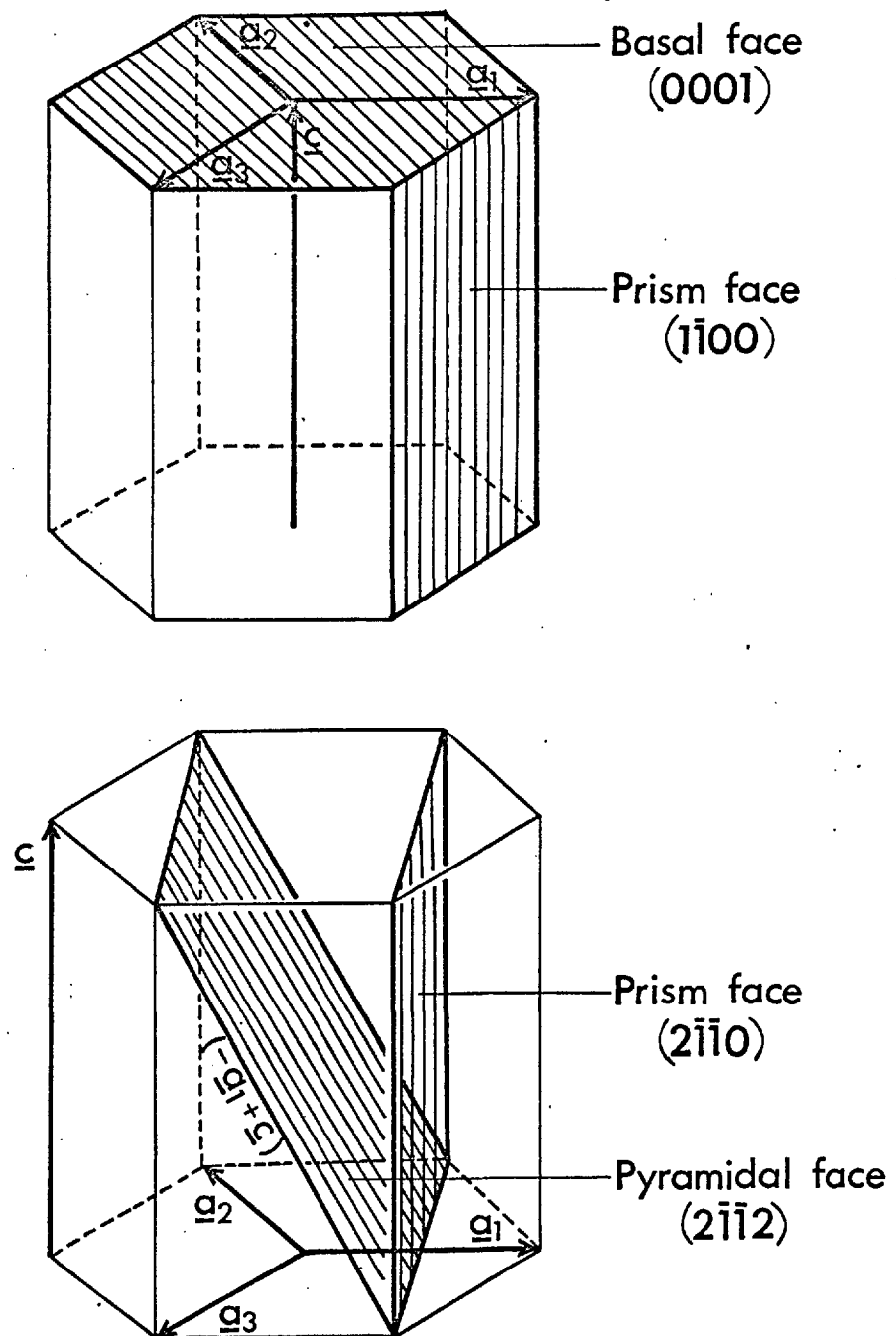
Several workers have reported the presence of etch channels, some of which have actually been produced by the application of a stress (Kuroiwa and Hamilton (1962); Bryant and Mason (1960); Muguruma and Higashi (1963 b.)). They occur on both basal and prism faces. In his study

of the latter, Muguruma (1963) found that they were tapered and tended to elongate parallel to the basal plane when the specimen was stressed. Assuming that they consisted of piled-up dislocations, which were too close to develop separate etch pits, he showed that the spacing of the channels was consistent with the magnitude of the applied stress.

The channels on the basal plane run parallel to the $\langle 11\bar{2}0 \rangle$ and $\langle 1\bar{1}00 \rangle$ directions and in some cases actually switch from one to the other. Their width often changes, both abruptly and gradually (i.e. they assume a taper) and etch pits have been found at their ends. Most workers agree that the channels trace the motions of dislocations and Muguruma and Higashi (1963 a. 1963 b.) have proposed the combinations of glide planes and Burger's vectors shown in Table 1.1. (N.B. Fig. 4 shows the relevant planes).

Where channels change their directions between $\langle 10\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ Muguruma and Higashi (1963 b.) believe the dislocation has an $(a + c)$ Burger's vector and is slipping on the $(\bar{1}122)$ and $(10\bar{1}0)$ planes. They point out that the taper of some of the channels could be due to impurities or vacancies that are deposited on the surface by a moving dislocation. However, Kuroiwa

Fig. 4 The principle planes in ice



and Hamilton (1962) are of the opinion that the width of a channel at any point is proportional to the speed with which the dislocation passes it. Neither of these explanations seems satisfactory and the correct one may well be a compromise between the two.

TABLE 1.1 - Possible combinations of glide planes and Burger's vectors to give the etch channels.

Burger's Vector	Glide Plane	Direction of Trace on Basal Plane
$\underline{a} + \underline{a}$	$(11\bar{2}2)$	$\langle 10\bar{1}0 \rangle$
$\underline{a} + \underline{a}$	$(10\bar{1}0)$	$\langle 11\bar{2}0 \rangle$
$\underline{a}$	$(10\bar{1}0)$	$\langle 10\bar{1}0 \rangle$

1.4 OPTICAL CHANGES.

Up to the present, Nakaya (1958) has carried out the most thorough study of the optical changes that occur in a single crystal of ice when it is deformed. The crystals, which had been cut into beams, were stressed by three knife edges (Fig. 1) and any changes that occurred were recorded by means of shadow photography. As deformation proceeded, he found that a series of fine lines appeared, which were parallel to the basal plane. They were about 0.06 mm. apart and divided the specimen into a series of

layers. After studying the behaviour of beams of various orientations, Nakaya concluded that the plastic deformation of ice is due to gliding between these layers, which, though flexible, cannot be stretched. He also observed the cloudy lines, referred to by Tyndall (1857), and succeeded in showing that they were due to the stress concentrations that arise when Tyndall Flowers refreeze.

In one of his experiments with a specimen oriented so that basal glide could not occur, Nakaya (1956) saw a rather complex pattern between crossed polaroids. The load had already been removed when it was observed and it changed as the polaroids were rotated. Furthermore, there was a dark shadow at the central knife edge which followed the maximum stress contour predicted by elastic theory. However, this could not have been a simple photo-elastic effect as the pattern was visible when the load was removed.

By using the difference in the extinction positions of two grains on either side of a grain boundary, Higashi and Sakai (1961) were able to study the motion of such boundaries under an applied stress. Provided the angle of misfit was greater than one degree, the boundary was quite visible; though if it was greater than ten degrees several sub-boundaries were formed and, instead of the usual single row of etch pits, several were visible. They were able to

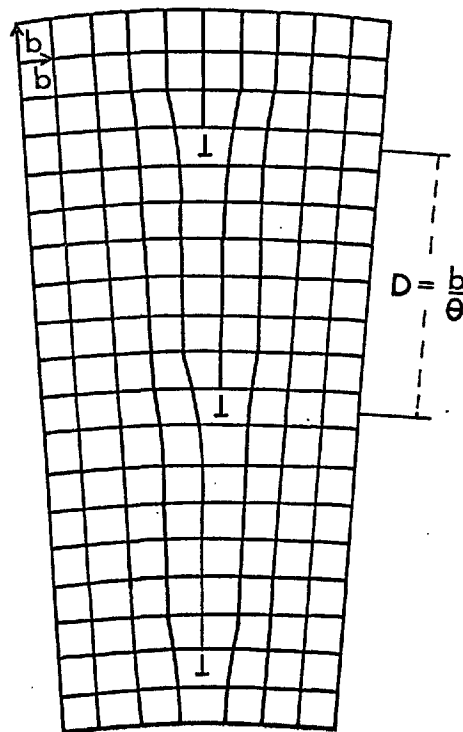
explain most of their observations by assuming that the arrangement of dislocations at the boundary was given by Burger's model (Fig. 5). As they did not calculate the angles of misfit from the spacing of the etch pits at the boundary and compare this with the value given optically, it is not certain that this assumption was justified, and it may be the source of the discrepancies that occurred between theory and experiment.

When the stress was increased, the activation energy for the motion of the boundary did not decrease, which may mean that other slip mechanisms, besides the basal ones, were operative. Another interesting result was the variation of this energy between 11 Kcal/mole and 17 Kcal/mole as the orientation of the a - axes with respect to the boundary was varied. Higashi and Sakai showed that this might be due to the changes that occur in the distance between potential energy valleys as the crystal is rotated. Finally, when the stress was reversed several times, the rate of motion of the boundary tended to decrease. This may have been due to an increase in the number of blocking dislocations in the crystal.

1.5 SURFACE CHANGES.

Many workers have reported that ridges appear on the surfaces of deformed ice crystals which are usually parallel

Fig. 5 Burger's model for a grain boundary



to the basal plane. These topological features probably trace the intersection of an active glide plane with the specimen's surface, and, as they have also been detected by shadow photography, some form of disorder must ensue in these planes - though Rigsby (1957) believes that no cracking or breaking occurs. By immersing his specimens in paraffin, Gold (1962) was able to follow the development of such features during deformation. However, some care must be taken in interpreting his results as the ice was polycrystalline and not very pure. In common with other workers, he often observed basal slip lines, but in addition, on two occasions he saw non-basal ones also. As both of these were associated with an unusual feature, he concludes that prismatic slip is an extremely uncommon occurrence. When the deformation was greater than $1\frac{1}{2}$, the basal slip lines either curved or some short lines were formed that were perpendicular to them. As these were usually associated with the formation of kink bands, Gold suggests that they were small angle boundaries.

In his study of surface features, Wakahama (1962) failed to detect any non-basal slip planes even when his specimens were clamped for non-basal glide. However, he measured the spacings of the non-basal slip bands on one occasion and using an empirical relation, he derives a

formula for predicting it for other stresses. He justifies this rather dubious practice by showing that it gives a value which agrees with Nakaya's measurements. However, not only was the exact value of the stress unknown in these experiments, but it is probable that other factors, such as impurity content, also affect the spacing. Furthermore, the dislocation model he uses - though able to explain his observations - seems to be an oversimplification and further comments on this will be made later.

By using the replica technique and an electron microscope, Muguruma (1963) was able to study the form of the slip bands in more detail than previous workers. He found that they consisted of wavy rows of pits and believes that this may indicate that cross-slip has occurred. The slip lines could be divided into two classes - fine ones about 0.1μ apart and well-developed ones about 10μ apart. Wakahama and Nakaya seem to have measured the latter, which could consist of bundles of the former, and it would be interesting to ascertain the relevance of these two types in creep. Once again it is evident that Wakahama's model is a simplification of the actual situation, though it must be emphasised that his model is very useful as a rough guide as to what happens when ice creeps.

1.6 DEFORMATION CURVES.

Though many methods have been used to study the deformation of ice, those described in this section really form the "backbone" to the investigation - for by measuring the creep curves of single crystals, some insight into the basic mechanisms may be obtained. This can then be extended by using more specialised techniques.

In 1954 Steinemann (1954) published the results of his investigation into the flow law and glide elements of single crystalline ice. Having clamped his specimens so that only non-basal flow could occur, he attempted to bend them; but found that there was either no measurable deformation or fracture occurred. The only marks that ever appeared on these beams were parallel to the basal plane and so he concludes that this was the glide plane. In the second part of the investigation he sheared his specimens parallel to this plane, partly to discover a preferred glide direction and partly to find the flow law. Unfortunately he failed to prove the existence of such a direction, for though the creep rate for a constant stress exhibited a slight anisotropy at higher stresses, this lay within his experimental errors. However, the results of his investigation into the flow law were rather interesting - for, though he found that the creep rate was related to the

stress by a power law of the form:-

$$\dot{\epsilon} = k\sigma^n$$

(where $\dot{\epsilon}$ = strain rate; σ = applied stress:

"k" and "n" are constants)

the exponent changed from 1.6 to 3.2 at a strain of about 10% - irrespective of the stress. This change was quite irreversible unless recrystallisation occurred and the results showed less scatter after it. However, some years later, (Gold (1962)) observed a similar transition when he compressed cylinders of single crystals - but this time the specimens changed from soft to hard. These two observations have yet to be explained.

Steinemann's results on the glide plane have been confirmed by Glen and Perutz (1954) who axially extended cylinders of ice and measured the orientations of the tapes that formed - these always tended to be basal. Though most of their creep curves were not very useful, it is interesting to note that one of them could be explained if a power law was assumed. A similar law also fitted the curves obtained by Griggs and Coles (1954) except that they added a constant to allow for the temperature at which the run was made:-

$$\dot{\epsilon} = a (\sigma - bT^2)^2 t.$$

(where T = absolute temperature, t = time and "a" and "b" are constants).

Their results showed a lot of scatter which they attributed to many factors including uncertainty in the orientation, eccentric loading and inhomogeneous crystals. Once again non-basal deformation only produced erratic behaviour.

Assuming that shear and compressive tests are compatible (despite the results of Tamman and Salge (1928)) Butkovich and Landauer (1959) tried to fit three different creep laws to their experimental curves. They found that there was least scatter about a power law and so they opted for it in the form:-

$$\dot{\epsilon} = k\sigma^n$$

(where $n \approx 3$)

Though this expression fitted the experimental points obtained from both basal and non-basal deformation, the multiplying constant k was about a hundred times smaller in the latter case. Polycrystalline samples were also tested and again the power law was most appropriate, with the constant taking the same value as for non-basal deformation. On comparing their results with Weertmann's (1951 a. 1957 b.) theoretical expressions, they were unable to distinguish between dislocation climb and motion controlled by a Peierls force, as the rate controlling process.

When Rigsby (1957) tried deforming single crystals under a hydrostatic pressure, he found that the only effect

was to change the effective melting point. This result is very interesting if that of Tamman and Salge (1928) is also considered.

Although the above experiments have all used a constant load to deform the ice specimens, constant strain rate studies have also been made. Using this method Wakahama (1962) found that:-

$$\dot{\epsilon} = a(\sigma - \sigma_c)^3$$

(where a is a constant and σ_c is the critical stress) could be used to predict the steady-state stress for a given strain rate, and estimated that the critical stress could not be greater than 200 gm wt/cm² in agreement with Steinemann. When he tested specimens that were oriented so that basal flow could not occur, he found that he did not observe any surface features until fracture started. Thus assuming that there is a three-dimensional network of dislocations present in ice and that the non-basal ones are immobile, he was able to explain all his observations using the resultant Frank-Read sources.

Using:-

$$\dot{\epsilon} = k\epsilon^m \sigma^n$$

(where m and n are indices and k is a function of temperature only)

Readey and Kingery (1964) succeeded in predicting both their transient and steady-state curves. The value of

the index "m" was found to be unity, but that of index "n" varied with increasing strain from 2.5 to 1.5 - probably as a result of changes in the dislocation networks. The existence of such changes was confirmed by the increase in the density of non-basal dislocations during deformation, which they detected by chemical etching. This of course confirms the oversimplification of Wakahama's model.

From their results Readey and Kingery calculated that the activation energy of the deformation process was about 14 Kcal/mole which could mean that the creation of imperfections was the rate controlling process. Though this would be necessary if basal dislocations were cutting a forest of non-basal ones, they do not rule out the possibility of dislocation climb in view of the low value of n. Their results show far less scatter than previous workers and they consider that this was mainly due to the specimen's being well-annealed (though in another part of the paper they say it has no effect!) and certain geometric considerations. Though sublimation did not affect their results, they did not check whether size had any or not - and thus the results of Jellinek and Brill (1956), which showed that size affected the strength of an ice crystal, are still unconfirmed.

(N.B. For future reference the various power laws are listed in Table 1.2).

TABLE 1.2 - The flow laws for Ice.

Workers	Flow Law	Remarks
Steinemann (1954)	$\dot{\epsilon} = k \dot{\sigma}^n$	"n" changed its value from 1.6 to 3.2 at a strain of about 10%.
Griggs and Coles (1954)	$\dot{\epsilon} = a(\dot{\sigma} - bT^2)^2$	
Butkovich and Landauer (1959)	$\dot{\epsilon} = k \dot{\sigma}^3$	
Wakahama (1962)	$\dot{\epsilon} = a(\dot{\sigma} - \dot{\sigma}_c)^3$	
Readey and Kingery (1964)	$\dot{\epsilon} = k \dot{\epsilon} \dot{\sigma}^n$	"n" changed from 2.5 to 1.5 as the strain increased.

1.7 X-RAY STUDIES.

X-rays may be used to investigate many crystalline features such as symmetry and other lattice characteristics. However in this section such studies are only of interest inasmuch as they provide some insight into the way ice deforms.

As already stated, Glen and Perutz (1954) extended cylinders of ice axially and studied the tapes that formed. Though they were able to determine the orientations of the planes of these tapes conoscopically, X-rays were required for the a - axes. On using them to compare the orientations

of the crystals before and after straining, they found that the results were inconclusive and were unable to decide whether a preferred glide direction existed or not.

Although an X-ray plate showed a series of "smudged" points with undeformed crystals, this "smudging" increased considerably after deformation and in some cases a series of discrete spots were superimposed on them. This "smudging" of the spots is called asterism and Glen and Perutz consider that it shows how the lattice was distorted during deformation. However the series of discrete spots is what would be expected if a pile of ice plates of slightly different orientations were X-rayed. Thus this may mean that the ice had polygonised (i.e. the distorted lattice had broken up into a series of perfect crystals, each of slightly different orientations).

1.8 GENERAL DISCUSSION.

So far, any discussion of the work has been of a rather limited scope, the remarks being kept within the sphere of the particular section in which the paper is referred to. Now in this section it will be extended and the results as a whole will be considered in order to determine our present state of knowledge. Thus it should be possible to decide what the next steps in the investigation should be.

It is now quite certain that basal dislocation lines exist in ice, as both etching and the study of deformed specimens have shown. However there is a distinct possibility that non-basal ones also exist and may play an important part in the deformation of ice. Their presence has been revealed by etching techniques which have also shown that their density and distribution changes during creep. Thus the question of what role they take during the deformation of ice, must be answered as soon as possible. Is it a purely passive one? Wakeham is evidently of this opinion for he argues that they just act as pivot points for the Frank-Read sources. Similarly Readey and Kingery believe that a forest network is formed by them which has to be cut by moving basal dislocation lines.

However, suppose they take an active role by actually moving on non-basal planes. This idea is not so implausible as it at first appears for not only has prismatic glide actually been observed by Gold, but such a mode of deformation will probably be required to explain the results of certain workers such as Landauer and Butkovich. Indeed this could account for the variety of power laws that have been proposed to predict the creep of ice and also resolve the discrepancy between the results of Gold and Steinemann. Further evidence for them taking such an

active part may be provided by the wavy nature of the basal slip lines and the hardening that Higashi and Sakai detected, when they tried reversing the direction of motion of a grain boundary several times.

Of course, if they do take this active role, the question of glide planes other than basal ones arises - as also does the reason for the absence of hardening that might be expected when dislocations on these planes interact with others on the basal ones. It would be a great help in deciding what these planes actually are if a proper explanation of the etch channels was forthcoming. Furthermore the existence of two types of basal slip bands reveals that even pure basal slip may not be as simple as at first appears, and their relative importance in deformation must be resolved as soon as possible.

So far no-one has succeeded in proving that there is a preferred glide direction in the basal plane and Barclay Kamb (1961) has shown theoretically that, if a power law is applicable, then it should be impossible to detect this directly by stress-strain measurements. This leaves X-rays and etching as the possible techniques and, as Glen and Perutz have shown that the former is very difficult, one hopes that refined etching techniques will be successful.

If a definite value for the activation energy could be determined, this would be a great help in deciding what

the rate controlling process is. However, though the value of 14 Kcal/mole appears quite frequently in the literature, until the question of the creep law is resolved, this will not be possible. In fact, if non-basal glide is relevant, this value may vary considerably - depending very much on how the specimen is deformed. It is interesting to note that such a variation has actually been observed by Higashi and Sakai. Though this was the special case of grain boundary motion it makes the possibility well worth considering.

The nature of the unusual form of stress-optical effect reported by Nakaya remains obscure. If this could be studied more thoroughly, a new technique for use in the investigation might become available, for it could give the stress distribution in a crystal at any instant. Thus the stresses required to make certain processes occur could be determined more accurately and this would be a great help in deciding what the rate controlling mechanisms are.

So far the question of purity has not been taken very seriously, which is rather surprising when the scatter of results and Truby's observation are considered. This must be thoroughly studied as it is quite possible that the presence of impurities affects the mode of deformation quite radically.

No reference to either transient or polycrystalline

creep has been made in this chapter. This is quite intentional as the situation is complex enough without introducing unnecessary complications at this stage. It might be argued that only a distorted picture will emerge from the relatively specialised field considered. However it is felt that until the parameters for this are known, the introduction of further unknowns will only succeed in obscuring the former still more.

1.9 AIMS OF THE PRESENT WORK.

It must be quite evident from the preceding discussion that, although much is already known about the deformation of single crystals of ice, a great deal of quantitative work has still to be done. As a preliminary to this some procedure must be set up for preparing single crystals of ice of any required shape. Furthermore it should be possible to control the purity of these crystals quite precisely (i.e. of the order of ppm) so that the effects of impurities can be investigated. Another point that must be considered is the internal damage sustained by the specimens during their preparation, as this could change the form of the dislocation networks and so alter the mode of deformation. Thus it seemed quite logical to devote the first part of this investigation to setting up such a system.

Having done this, the next step was largely a matter of preference. However, as the optical changes that occur in a deforming crystal had not received much attention, it was decided to investigate these in both pure and doped crystals. In doing this, surface changes were also studied as the same piece of apparatus was suitable for both investigations.

CHAPTER 2.

PRODUCTION AND PREPARATION OF SINGLE CRYSTALS OF ICE.

2.1 INTRODUCTION.

At present, mainly due to the scatter in experimental results, it is impossible to say which, if any, of the many creep laws is the correct one, and as this relation might enable the activation energies associated with the various modes of deformation to be determined, the situation must be remedied as soon as possible.

Though there are many possible sources of this scatter, the most probable intrinsic ones seem to be - the age and impurity content of the ice; the method used to shape the ice. These will now be considered in turn.

IMPURITY CONTENT.

As both the arrangement and movement of dislocations are altered by the presence of impurities, variable purity content must lead to scatter. This view is supported by the results of Truby (1955) who showed that the density of dislocations could be changed by doping the ice. Furthermore, the results of Readey and Kingery (1964), who have published the latest paper on the deformation of ice, show less scatter than those of previous workers, and the main precaution they took was to use pure water in growing their specimens. So it seems reasonable to conclude that to reduce the scatter in the results of an individual worker,

the impurity content must be kept fixed; while to explain some of the scatter between different workers, the effects of impurities must be studied.

METHOD OF SHAPING.

Stresses are present when an ice crystal is machined and thus dislocations and other imperfections must be introduced. As their number and form must vary, this leads to scatter - though for a particular worker this may be negligible. Thus to make the results of various workers more compatible, its effect must be studied and perhaps the easiest way would be to test specimens, which had been thermally cut, at the onset and then see what difference machining makes.

AGE.

Due to relaxation phenomena, the way a block of ice deforms may depend on its age - though this might be hard to detect if the time scale were too long. Thus, though age may not be responsible for the scatter in the results of an individual worker, it is probably very relevant when the results of various workers are compared as their specimens will have come from many different sources.

In addition to these intrinsic sources of scatter, there may be extrinsic ones due to such things as eccentric mounting of the specimens in the apparatus. Thus it is evident that the problem of scatter is quite a complex one

which will require further work.

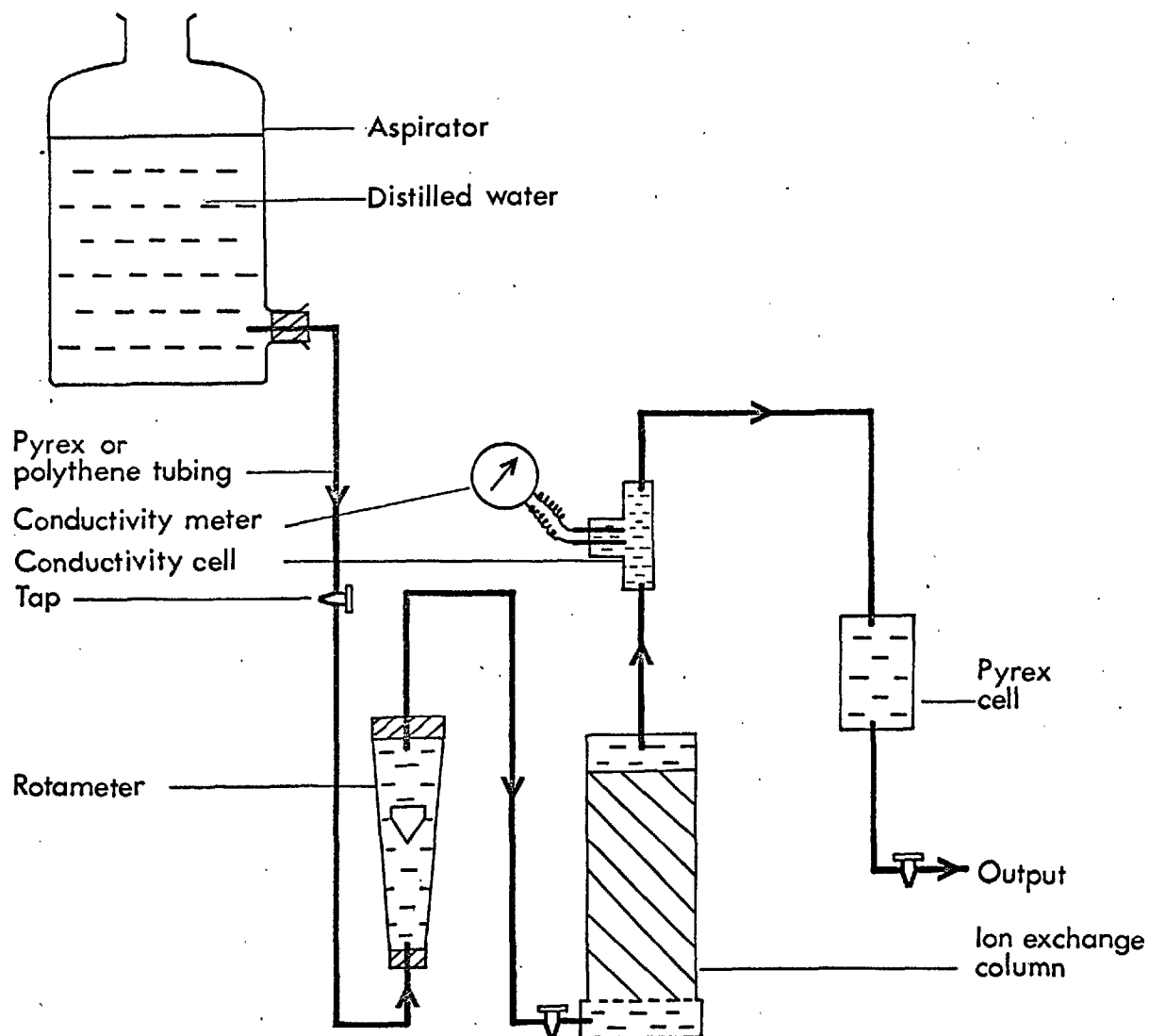
In this Chapter a system of preparing single crystals will be described which is believed to form a good basis for this study, and in later chapters some consideration will be given to it, though it was not the principal consideration of this work.

2.2 WATER SOURCE.

The system for producing the water used in the crystal growth apparatus is described in this section. No mention of impurity content is made here as it is felt that such an important topic requires a separate section. (See Section 2.6).

The distilled water used for growing the ice crystals came initially from an aluminium still and was purified by passing it through the system shown in Fig. 6. The ion exchange column illustrated in this figure was an Elgastet Major Type B10⁴/CON and consisted of a mixed bed resin, which was contained in a "Plexiglass" cylinder. On emerging from this column, the water passed through a "Vulcathene" conductivity cell so that a rough check on its purity could be made and then its particulate content was measured by a light scattering device with a pyrex cell, before it finally arrived at the outlet. These various parts were connected by polythene and pyrex tubing, and the

Fig. 6 Schematic diagram of water source



maximum flow rate obtained was about 36 litres/hour.

2.3 GROWTH FRAME.

Though the simplest method of obtaining single crystals is to cut them from a polycrystalline block, this is not very satisfactory due to the variable stresses that exist between grains, and the associated relaxation phenomena that occur when the crystal is separated from the block. Furthermore, owing to the great affinity of grain boundaries for impurities, controlled doping is well nigh impossible. Thus it is better to use a method which produces one crystal at a time, and satisfies certain other requirements:-

- (1) The water put into the frame must be kept pure.
- (2) Controlled doping of the crystals must be possible.
- (3) The stresses acting on the crystals must be kept to a minimum.
- (4) The crystals must be uniform and large enough for the experiments.

Of the many methods described in the literature, that used by Jaccard (1959) seemed the most suitable and so a modified version of his cryostat was built. This is shown in Fig. 7 and it can be seen that it consisted of four parts - a cold reservoir, a heater, a mould and a system for lowering the mould. These will now be considered in turn.

Fig. 7a Schematic diagram of growth frame

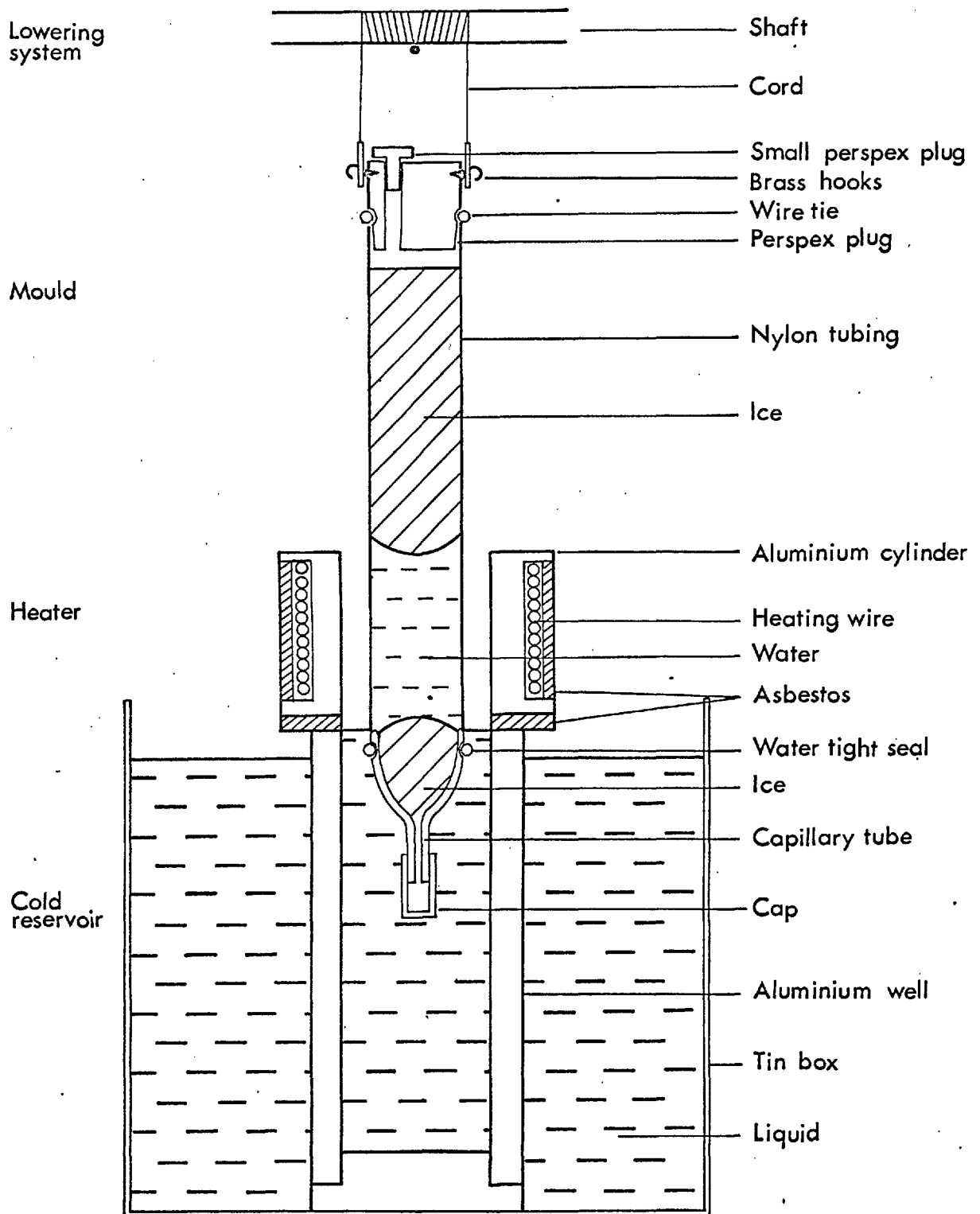
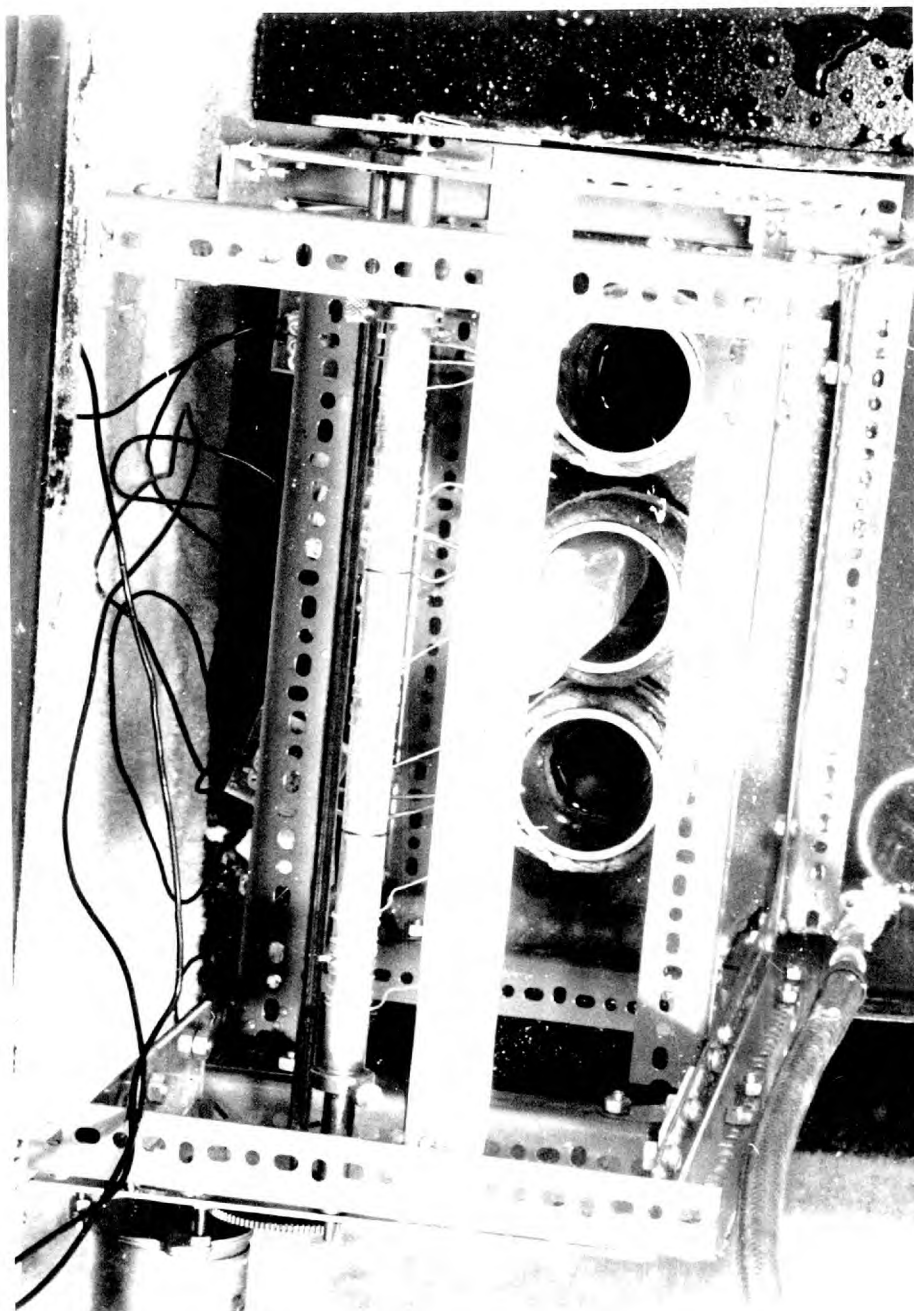


Fig. 7(b) Photograph



THE COLD RESERVOIR.

The cold reservoir used in this frame had to satisfy the following requirements:-

(1) It had to have a thermal capacity large enough for its temperature to remain stable despite the heat evolved by the growing crystal and variations in the ambient temperature.

(2) In order that a high temperature gradient could be maintained between the heater and the cold reservoir, the cold region had to start suddenly and not fluctuate during growth.

(3) The actual temperature of the main body of the reservoir had to be low enough for ice to form easily.

From the diagram it can be seen that the reservoir consisted of a large tin can full of a liquid, whose level was kept below that of the liquid contained in the aluminium cylinder into which the mould was lowered. In this way both a stable interface and a large thermal capacity were obtained. Furthermore, by using a liquid good thermal contact was always maintained between the mould and the cold reservoir as it was lowered into the aluminium well.

The cryostat was kept in a refrigerator to ensure that the ambient temperature did not vary greatly. However, the cold reservoir had to be further cooled to

about -25°C by a Townson and Mercer Laboratory Refrigeration Unit to satisfy the third requirement.

THE HEATER.

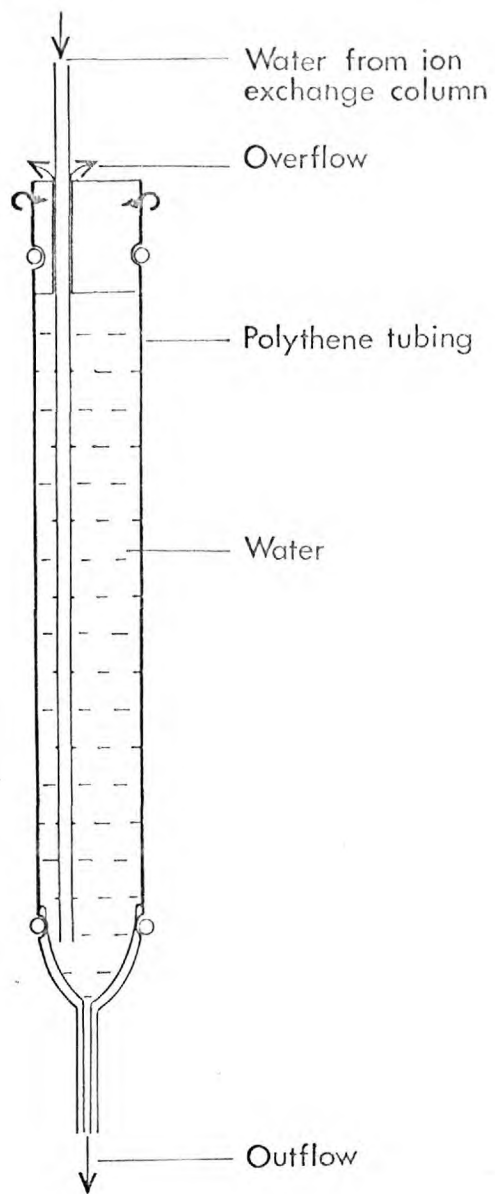
From the diagram it can be seen that the heater consisted of an aluminium cylinder, with "Kumunal" heating wire wound on it and lagged with asbestos cord to reduce heat losses. By placing a thin asbestos sheet between the heater and the cold reservoir, any direct heat exchange between the two was prevented. Thus a sharp and stable temperature gradient was obtained between them - especially as air circulation in the heater was retarded by keeping the clearance between it and the mould to a minimum.

THE MOULD.

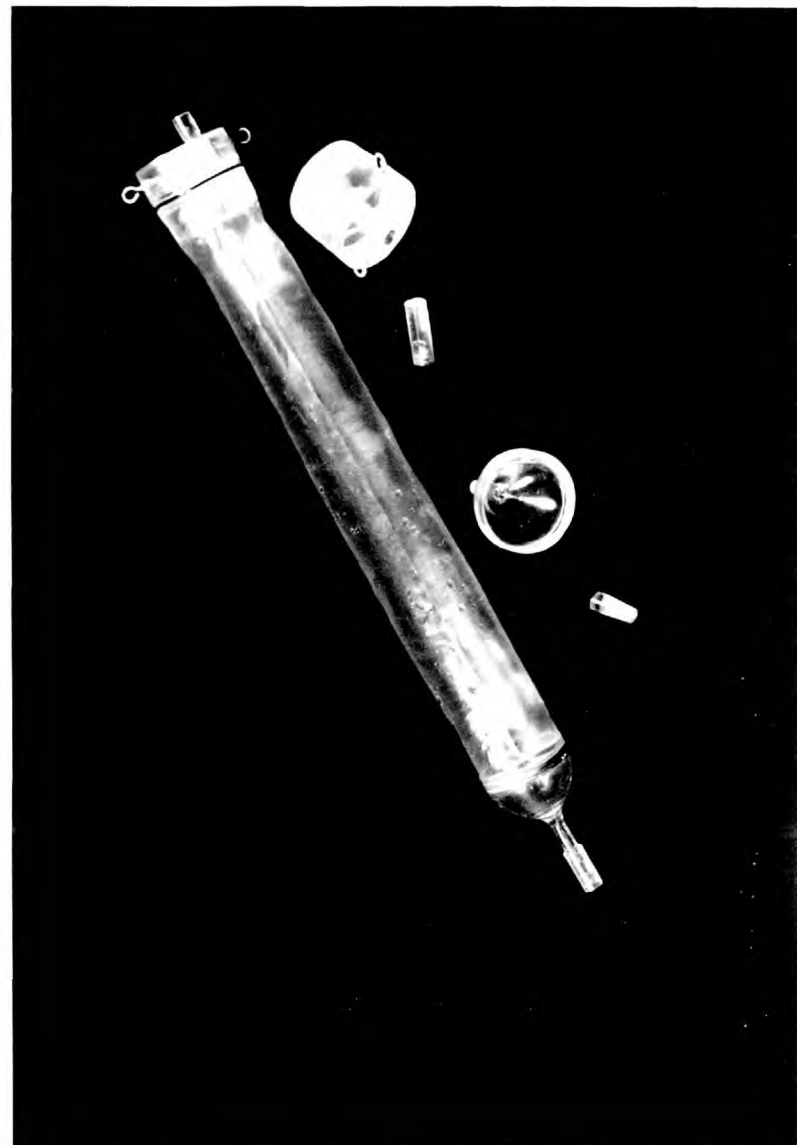
The mould, in which the crystals were grown, consisted of a length of nylon tubing which was closed at the top by a perspex plug and at the bottom by a piece of machined perspex. This had been turned to terminate in a narrow capillary and a small cavity was obtained at the end of this by fixing a perspex cap to it.

The method of filling the mould with water is shown in Fig. 8. When the water had been run through this system for about ten minutes, it was switched off and the mould sealed with a small perspex plug and the cap. In doing this, care was taken to ensure that no air was

Fig. 8(a) Schematic diagram of washing system



(b) Photograph of a complete mould



trapped in the capillary or the small chamber, as otherwise the frame would not have worked properly. The mould was then suspended (See Fig. 7) and lowered slowly through the temperature gradient into the cold reservoir. As this occurred, polycrystalline ice was formed in the small chamber but the small diameter of the capillary allowed only one of the many dendrites to pass through it to the main chamber. It was essential that this occurred before the 0°C isotherm reached the main chamber as otherwise the water in this chamber might have nucleated. To ensure this the length of the capillary tube, the temperature of the reservoir and the rate at which the mould was lowered, were adjusted to appropriate values.

When the single crystal had started to grow in the main chamber, stray crystals had to be prevented from forming and so in setting up this system the following points were taken into account:-

(1) The probability that an ice crystal will form in supercooled water depends among other things on its volume and temperature. Thus the larger this volume and the lower its average temperature the more likely it is to nucleate. These two points had to be borne in mind when the growth frame was set up, as otherwise stray crystals formed. One consequence of this was that the rate at which the mould was lowered had to be kept less than the rate at which the

ice could grow, so that the ice-water interface did not lag increasingly behind the 0°C isotherm. This would have led to an increasing volume of supercooled water and hence, ultimately, to the formation of stray crystals.

(2) If the thermal capacity of the cold reservoir had not been large enough to absorb the latent heat developed by the growing crystal, with negligible temperature changes, its average temperature would have risen. This would have lowered the rate of growth of the crystal and hence increased the volume of supercooled water. Furthermore, the temperature gradient would also have been reduced and this also would have led to an increased possibility of stray nucleation. (See below).

(3) If the temperature gradient at the interface was too shallow, the latent heat evolved could not be removed quickly enough and so the thickness of the layer of supercooled water increased. Furthermore, with a steep temperature gradient even a short distance represents a large temperature change and as the growth rate of ice increases with decreasing temperature, it becomes increasingly more difficult for the ice-water interface to lag behind the 0°C isotherm. Thus by using such a gradient, the probability of stray nucleation was reduced.

(4) It was essential to keep the ice-water interface convex towards the water, as with a concave one not only was

the supercooling increased by the curvature, but any stray crystals tended to grow into the main crystal. In actual practice, the desired shape was attained by balancing the temperatures of the hot and cold regions.

(5) The inside of the main chamber had to be smooth so that the possibility of it nucleating stray crystals was low.

Because of the low value of the partition coefficient, the concentration of gas in the water increased as the crystal grew and if it became too high gas bubbles formed. In order to avoid this, care was taken when loading the moulds to ensure that the gas and dust content were kept to a minimum - the latter because dust particles can nucleate gas bubbles. By taking these precautions, the probability of gas bubble formation was reduced to almost zero except at the top of the crystal and so the liquid did not have to be stirred. This was fortunate as the water above the heater was frozen (see Fig. 7) and so some difficulty would have been experienced!

PURITY.

In his review of the principles of solidification, Tiller (1963) discusses the solute distributions that arise from various freezing conditions. One of these was Partial Mixing, which he defines as the case when the solute distribution in the liquid is affected by

both diffusion and convection, and it is this one which seems to fit the experimental set-up most closely. Furthermore the growth frame operated as a Zone Refiner (owing to the frozen ice above the heater) and so it follows from his analysis that the solute distribution along the crystal's length should have been fairly uniform except at its ends. Thus these were always discarded.

The crystals were kept pure by completely enclosing the liquid in the mould and yet at the same time there were facilities for doping. Unfortunately some of the liquids used in the cold reservoir diffused through the nylon and this led to contamination. This caused trouble and is the reason why the liquid used in this reservoir was not mentioned in the appropriate section. This and other aspects associated with the purity of the crystals will be discussed in Section 2.6.

THE MOULD LOWERING SYSTEM.

As shown in Fig. 7, the mould was suspended from two lengths of linen thread and was lowered at about 1 micron/sec. by a geared down synchronous motor.

CONCLUSION.

It is evident from the foregoing that there are many factors to be considered in operating a growth frame and for success a certain amount of luck is required. The actual frame used in this investigation gave about two

good crystals out of a possible three per run.

2.4 THE SHAPING OF THE CRYSTALS.

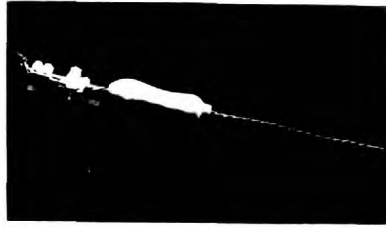
One of the aims of this investigation was to reduce the amount of scatter between the crystals and, as machining introduces variable numbers of dislocations, this method of shaping was not considered suitable. The only way of doing this without introducing dislocations is by melting and so it was decided to cut the specimens thermally. With this technique it was more difficult to avoid mounting them eccentrically in the apparatus but the maximum errors were calculable. The other method would be to actually grow the crystals to the required shape. However this was rejected; as quite apart from the problems of stray nucleation during growth, considerable thermal stresses must arise.

EXPERIMENTAL TECHNIQUE.

Single crystals, in the form of either rectangular blocks or cylinders, were required in this study and the procedure used to obtain the former was as follows:-

(1) A cylindrical single crystal of ice was diametrically cut into two parts, with a thermal cutter made of Kumunal heating wire (Fig. 9(a)), after a check had been made to ensure that the plane of this cut was of the required orientation. (The Universal Stage was used for this).

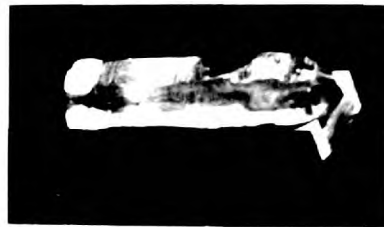
Fig. 9



(a) Kumunal cutter



(b) Rectangular cutter



(c) Rectangular specimen — half cut

(d) Finished rectangular specimen
mould

(2) A rough rectangular shape was made on one of the exposed faces with the brass block shown in Fig. 9(b) - which was initially at room temperature.

(3) This block was then cooled until its temperature was just above 0°C and the crystal again placed in contact with it, so that further melting occurred. However this time it was very much less, and after any trapped air bubbles had been removed, the ice was left in contact with the brass so that this film of water could refreeze. (See Fig. 9(c)).

(4) When this freezing was complete, the excess ice (shown shaded in Fig. 9(c)) was melted away and a rectangular specimen obtained by placing a glass slide on the block as shown in Fig. 9(d) - whilst taking care to ensure that no air bubbles were trapped in the water between the slide and the ice.

(5) When it was completely frozen, the ice was removed by slightly warming the brass block and a check was made to see that it was still a single crystal.

At any temperature the best surface for nucleating the growth of an ice phase in supercooled water is ice itself - though as the temperature falls an increasing number of foreign bodies may serve as nuclei. Now the water present during thermal cutting was not only close to the freezing point but was also in contact with ice, and

so was nucleated by it. The new ice formed from this water was a continuation of the nucleating single crystal and so the crystal of the desired shape was obtained. In this connection it should be noted that if the thickness of the water between the brass and the ice was too large, stray crystals and air bubbles were liable to occur. This follows directly from the discussion in Section 2.3.

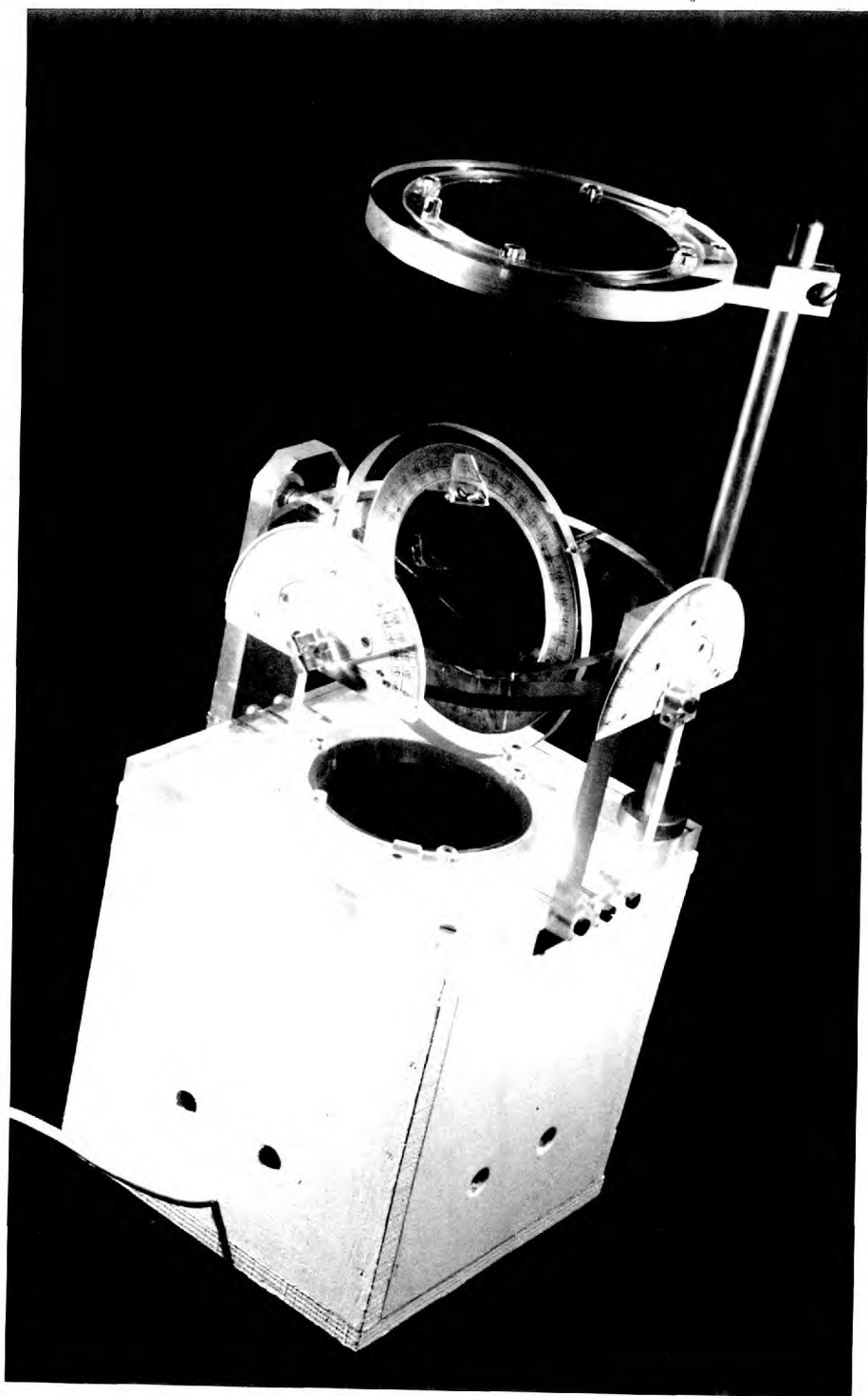
2.5 DETERMINATION OF THE ORIENTATION OF CRYSTALS.

Ice is a uniaxial substance and so the orientation of its c - axis may be determined optically, but not the orientation of the a - axes. X-rays or etching could give their orientation, but it is extremely difficult to obtain accurate results with either technique and so it was decided only to fix the c - axis, unless experimental results showed that this was not sufficient.

A Universal Stage without hemispheres (See Fig. 10) was used to determine the orientation of the c - axis. Full details on the use of such a stage are given by Langway (1958). He also includes a correction table in this paper which allows for the refraction of the light as it passes through the specimen.

With the aid of this stage the angles that the optic axis makes with the face of the crystal (g_2) and ^{that the projection of the optic axis} with the ~~on the surface~~ ^{makes} long side of the rectangular specimens (g_1) were determined.

Fig.10 Universal stage



However in the case of these specimens the angle that the optic axis made with the compression axis (g) was not equal to g_2 but was given by:-

$$\cos g = \cos g_1 \cos g_2 \quad (\text{See Fig. 11})$$

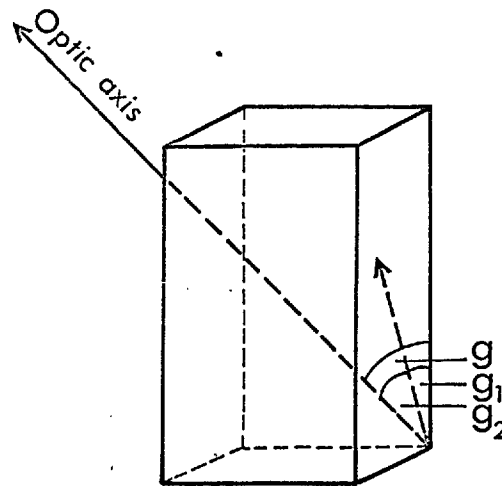
(This equation is derived in Appendix I).

2.6 PURITY.

The observation that alcohol could penetrate nylon tubing led to the serious consideration of the possibility of the ice crystals being contaminated, as the liquid used in the cold reservoir was alcohol. An idea of the extent of this contamination was obtained by measuring the organic carbon content of the specimens with the oxidation technique of Montgomery and Thom (1962) and the results are given in Table 2.1.

Fig.11 Angles associated with rectangular specimens

(a) Typical specimen



(b) Figure used for analysis

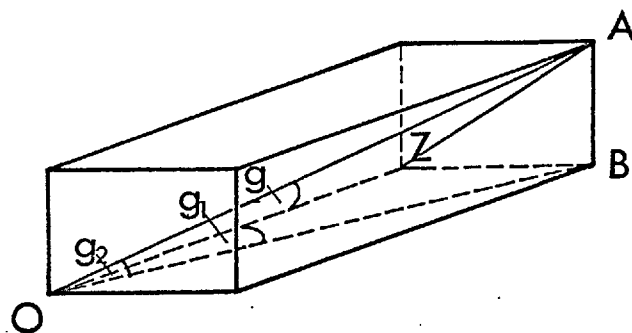


TABLE 2.1 ORGANIC CARBON CONTENTS

Nature of Sample	Organic Carbon Content
Ice grown with alcohol in the cold reservoir	12.3 ppm
Ice grown with paraffin in the cold reservoir	> 10 ppm
Ice grown with calcium chloride solution in the cold reservoir	2.3 ppm
Water from the ion-exchange column	0.1 ppm
Water held in the mould at 20°C	1.1 ppm/day
Water held in a mould which was half immersed in calcium chloride solution at 20°C	2.0 ppm/day

It can be seen that the ice specimens contained about a hundred times more carbon than did the pure water. In order to reduce this the alcohol was replaced by paraffin as it is less soluble in water and not so volatile as the former. However it can be seen from the table that this did not improve matters - probably for three reasons:-

- (1) Nylon is not impervious to paraffin.
- (2) Paraffin tends to creep over any body that is partially immersed in it.

(3) The solubility of paraffin in water lies between 0 ppm and 50 ppm and so is not negligible.

As most organic substances suffer from similar defects, it was decided to try a salt solution instead. Though both ferric chloride and calcium chloride are suitable, the former attacks tin and so a solution of 2 kg calcium chloride/gallon of water was used. With this solution in the cold reservoir, the organic carbon content was reduced by a factor of five and furthermore, a flame photometer test showed that any calcium contamination was less than 1 ppm.

The flame test seems to show that the organic contamination did not arise from the calcium chloride solution. This is not unreasonable, as though such a solution could give the required contamination, there must be water on the other side of the nylon and, as calcium chloride solution (unlike the organics) did not creep up the outside of the moulds, there was only ice. In fact all the organic carbon observed could be derived from the nylon tubing and so it seems reasonable to conclude that this was the main source.

The water obtained from the still had a particulate content of between 30 and 200 particles per cc, depending on how much water had passed. It was to conserve this that the enclosed mould loading system shown in Fig. 8

was set up. This seems to have been quite successful as the particulate content of the ice was undetectable with the apparatus available. Whether this result means that the growing ice surface rejected particulate matter or results from the limitations in the experimental technique, is impossible to decide at present.

According to the manufacturers, the output from the ion exchange column should not contain more than 1 ppm of non-volatile matter and most of this should be pyrogenic or colloidal. Both the scattering the organic carbon content measurements seem to agree with this.

Neutral gases, such as nitrogen and oxygen, pass straight through the column and their presence was confirmed by the occurrence of air bubbles in some of the ice specimens. Not being a neutral gas when in solution, carbon dioxide is removed by the ion exchange column. However it seems likely that more diffused in from the atmosphere after the water had left the column as:-

- (1) The pH of the ice specimens lay between 5 and 6 (i.e. it was slightly acidic).

- (2) A graph showing the decay in the pH value with time had a time constant which was about right for the diffusion of carbon dioxide in water.

- (3) When flow was stopped the resistivity of the

water fell from about 2×10^7 to 10^6 ohm cms.

So it seems reasonable to conclude that the ice specimens contained negligible impurities apart from:-

(2) Nitrogen, oxygen and carbon dioxide.

(b) $\leq$ 2.3 ppm organic carbon from the nylon tubing.

CHAPTER 3.

SURFACE FEATURES DEVELOPED DURING THE DEFORMATION OF ICE.

3.1 INTRODUCTION.

When a body deforms topological changes often occur, and by studying them information on the way the deformation occurred may be obtained. Thus, for example, it may be possible to identify the active glide elements by noting the orientations and forms of the slip lines.

This extremely useful technique has been used quite successfully with ice and the main results have been discussed in Chapter 1. However, though many interesting effects have been reported, some of their explanations are rather tentative and it was in the hope of improving this situation that the experiments described in this chapter were carried out.

This work has been divided into two sections. The first part deals with direct observation of the surface during deformation; while the second reports the quantitative measurements of slip band spacings that were made with the aid of a replica technique.

3.2 THE SURFACE CHANGES DURING CREEP.

In this section the results of the study of the surfaces of deforming single crystals of ice are described and it should be noted that many of these have already been

reported by other workers. However, they are considered to be worth describing as (a) the ice specimens were single crystals of known purity, (b) the stress was relatively uniform, and (c) the resolution obtained was better than that of previous workers - so enabling the phenomena to be studied in more detail than had been possible before. Furthermore, some instances of "non-basal" slip were observed and as this is a rather important topic at present, any information on it is valuable.

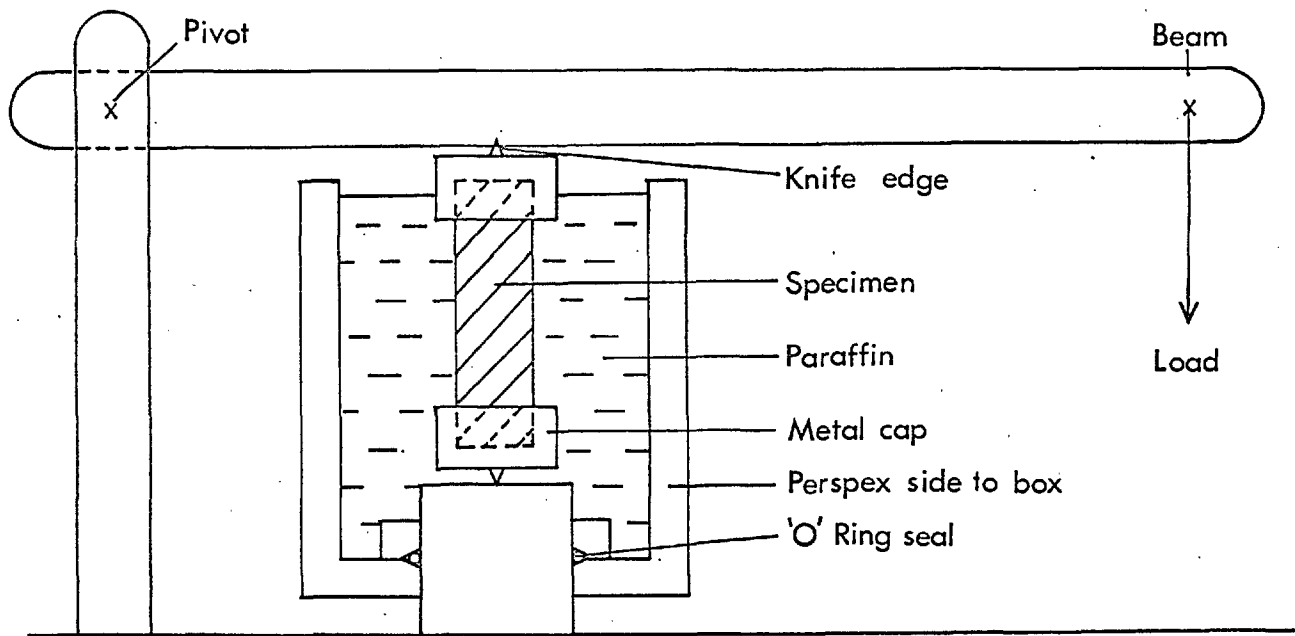
3.2.1 APPARATUS AND TECHNIQUE.

The experiments were carried out in a cold room in which, due to the air circulation caused by the fan, unprotected ice surfaces evaporated quite quickly. Furthermore, owing to the small radii of curvature of any topological features developed during deformation, these evaporated faster than the surrounding plane surface and so were erased. To prevent this the specimens were immersed in paraffin during the experiments - ice being only slightly soluble in it.

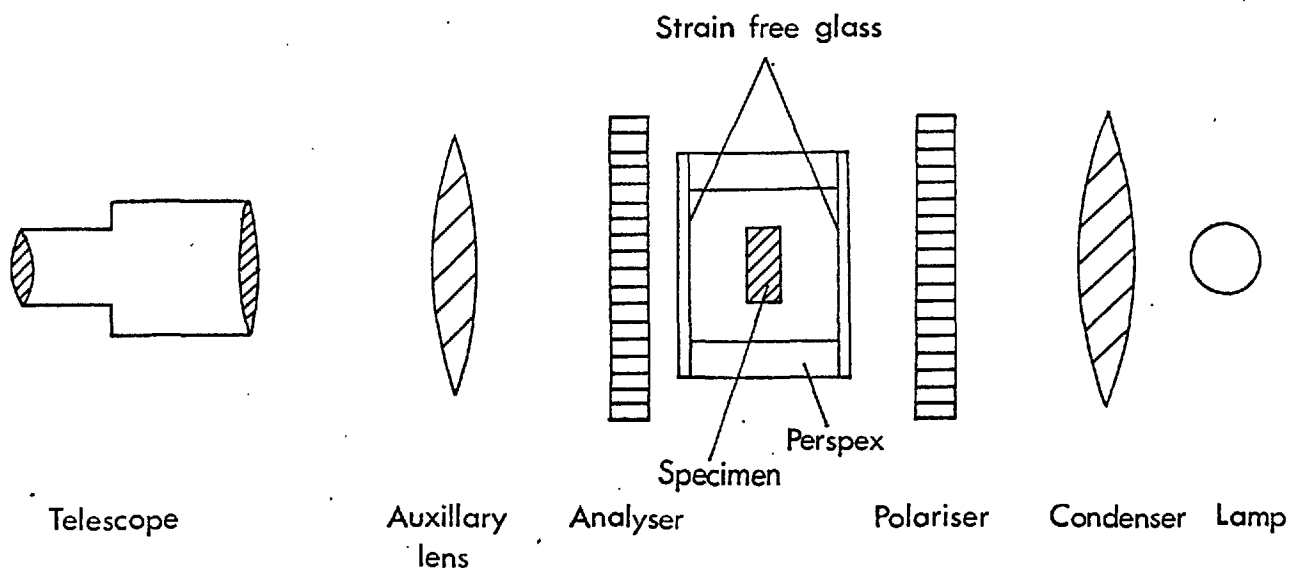
As may be seen from Fig. 12(a), the rectangular specimens, which had been cut from single crystals of ice and frozen into brass caps, were stressed between two knife edges by loading a beam. Any deformation was measured with a screw micrometer, using a battery-lamp circuit to show when contact had been made between it and the beam. (This

Fig.12 Apparatus for optical studies

(a) Stress system (front view)



(b) Optical system (top view)

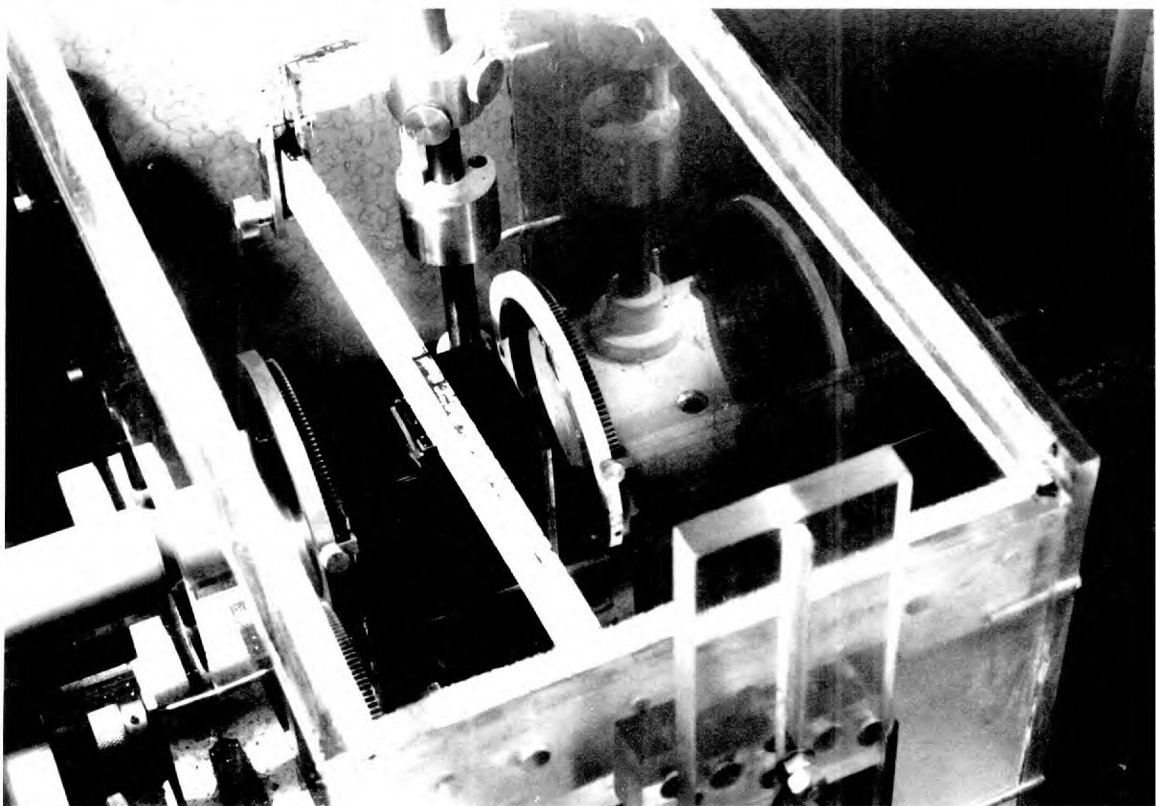
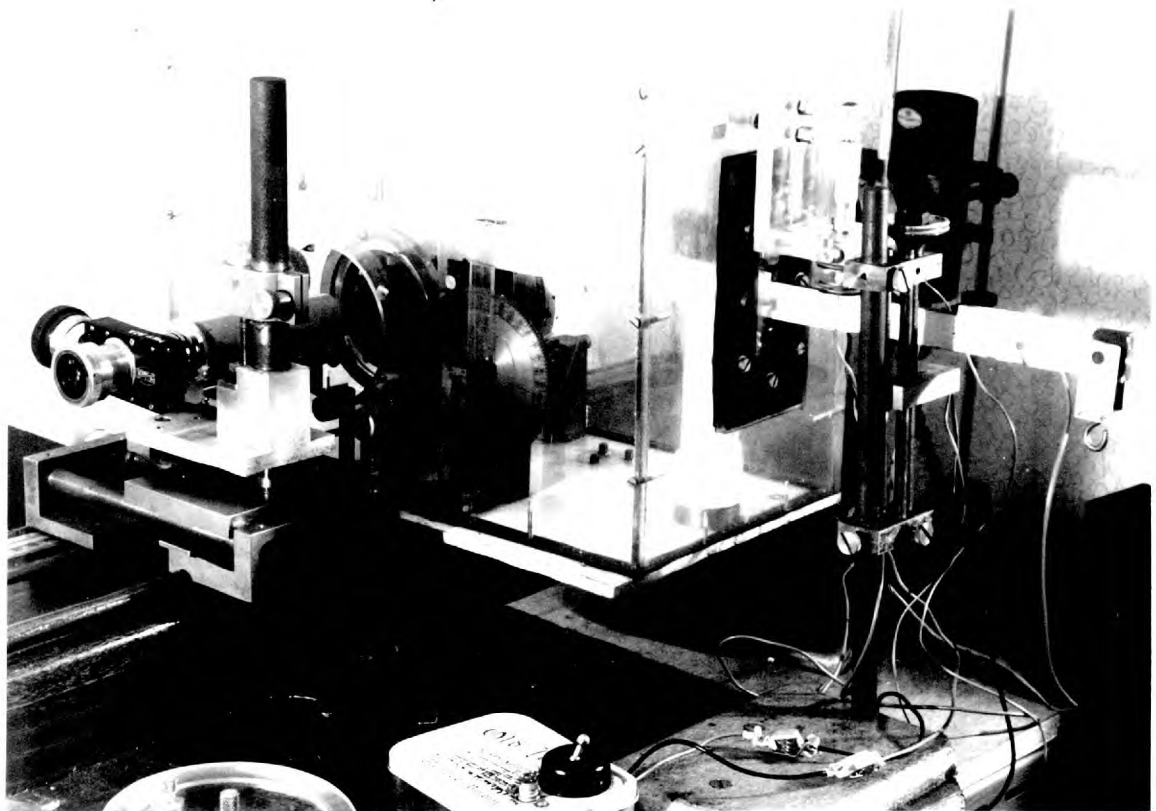


is shown quite clearly in Fig. 13).

By surrounding the specimen with a rectangular box, it was possible to keep it immersed in paraffin during the run and so prevent evaporation. As may be seen from Figs. 12 and 13, this box consisted mainly of perspex except for two faces of strain-free glass through which the specimens were viewed. These were necessary because the crystals were examined between crossed polaroids, and perspex, being a uniaxial substance, exhibits an extinction position similar to that of ice.

With the aid of a condenser lens, the specimens were illuminated with either sodium or mercury light and studied through the telescope barrel. This had a X10 vernier micrometer eyepiece and a 50 mm objective attached, and gave quite a high magnification. However lower magnifications could be obtained by placing an auxiliary lens between it and the specimen. This is the arrangement shown in Fig. 12(b).

As the same piece of apparatus was also used for studying the optical changes that occur during creep, a polariser and an analyser were included. Each of these consisted of a piece of polaroid which had been sandwiched between two pieces of strain-free glass, mounted on a gear wheel and fixed so that they could be rotated together without being uncrossed. Though facilities for uncrossing



or removing the polaroids were provided, these were not used very often as any surface features could be seen more clearly by reducing the light intensity. Another technique that was used to throw them into relief was to alter the relative positions of the lamp and the condenser lens.

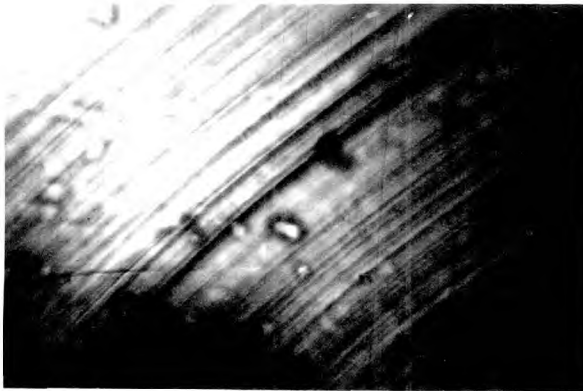
3.2.2 SURFACE CHANGES ON A SINGLE CRYSTAL.

This section discusses the features that develop on the surface of a single crystal and the next (Section 3.2-3) refers to the changes that occur in the region of a grain boundary.

When a load is applied to a single crystal of ice, it suffers the usual elastic deformation and then proceeds to creep at an increasing rate. This "creeping" is achieved by extensive slip occurring on a few preferred planes which may be detected by the ridges that form where they intersect the surface of the crystal, provided the strain is great enough.

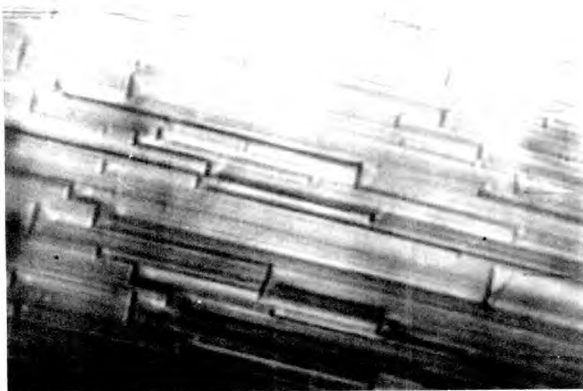
Using the apparatus just described, single crystals of ice were compressed by stresses of various magnitudes and their surfaces studied during the actual deformation. It was found that, provided the optic axis of the specimen did not make too great an angle with the surface, lines parallel to the basal plane were eventually formed. Typical examples of these basal slip lines are shown in Fig. 14 and it can be

Fig. 14



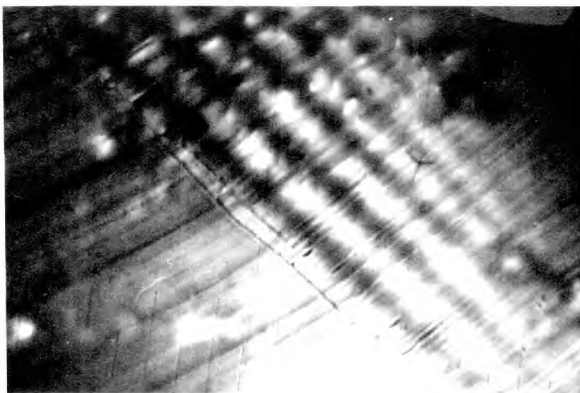
(a) Curvature of the basal slip bands

— $\frac{1}{2}$ mm.



(b) Basal and non-basal slip bands

— $\frac{1}{2}$ mm.



(c) Grain boundary

— $\frac{1}{2}$ mm.

seen that they have the following properties:-

- (1) Some are more prominent than others.
- (2) Their spacings are not equal.
- (3) They are able to curve when necessary. (See Fig. 14(a)).

The first property is rather interesting, as though it may not mean that Rigsby (1957) was wrong in assuming that all the active planes start slipping at the same instant, it does show that some slip more than others. This was quite unexpected as the stress was quite uniform over the area studied, but probably resulted from variations in the distribution of forest dislocations. These could retard slip on some of the planes besides preventing it on others. However this and the non-uniform spacing will be considered more fully in the latter part of this chapter.

Often, in regions where the lattice was curved, short lines perpendicular to the basal plane were also present. Some of these are shown in Fig. 14(a) and it can be seen that they have a symmetrical appearance. Furthermore, though not parallel to each other, they are all perpendicular to the basal plane and so must follow the curvature of the lattice. Thus it seems likely that they are small angled grain boundaries - similar to that shown in Fig. 5.

In Fig. 14(b) some more non-basal lines are shown. These are also perpendicular to the basal plane but differ

from those just discussed in certain important aspects:-

(1) They do not have a symmetrical appearance (i.e. the surface on one side of a line is more blurred than that on the other).

(2) By following alternate basal and non-basal lines it is possible to trace a number of prominent zig-zag lines across the specimen.

These facts seem to favour them being some form of non-basal slip line, which of course means that non-basal glide elements were present during creep. Although it is impossible to identify these for certain, the angles between the two types of lines are what would be expected if basal and prism planes were involved and so the dislocations probably had $\frac{a}{2}$ or $\frac{c}{2}$ Burgers vectors. (See Figs. 3 and 4). However as the non-basal slip lines always terminated on active basal ones, the $\frac{a}{2}$ Burgers vector seems most likely as this would give simple cross-slip. This phenomenon was only observed on a few occasions and in all cases the resolved shear stress was higher than usual - being about 1 kg/cm^2 instead of 0.5 kg/cm^2 . However there was no visible stress inhomogeneity which means it may occur more often than Gold (1962) has suggested. In fact these observations would be more in accord with Tegart's (1964) theory of a higher critical stress being associated with the formation of non-basal slip bands. It is possible that

the reason these experiments did not reveal their presence on more occasions was because grain boundaries formed before the slip bands were detectable. (See the next section).

On two occasions non-crystallographic ridges formed on the ice surface shortly after increases in the applied stress. These were quite irregular in both form and spacing and were probably cracks that arose because of the sudden stress change.

Finally, it is interesting to note that no difference between the behaviour of pure and impure crystals could be detected, even with regard to cross-slip.

3.2.3 SURFACE CHANGES AT A GRAIN BOUNDARY.

As deformation proceeds the total length of the dislocation lines in the specimen increases enormously. This leads to a significant increment in the value of its internal energy, due to the interactions between these dislocations and their stress fields. In localised regions the energy may attain such a high value that the lattice is able to reduce it by allowing the dislocations to arrange themselves in ordered patterns. This "ordering" of the dislocations often involves climb and so the internal energy has to be quite large before this can occur. (i.e. The single crystal must suffer a certain amount of deformation).

If this "ordering" is sufficiently extensive, a grain boundary will form (see Fig. 5 for example) and, as the lattices on either side of such a boundary will have slightly different orientations, this means that the original crystal has been broken up into two. Once present, such a boundary continues to attract dislocations and so, as deformation proceeds, the difference in the orientation of the two grains will increase.

Ice is a uniaxial substance and so if a single crystal is viewed between crossed polaroids, no light will be transmitted if its principal axes are parallel to the transmission axes. This fact may be used to detect boundaries between grains of different orientations as their extinction positions will not coincide. Thus one grain may be "blackened-out" while the other still transmits some light - hence enabling the exact position of the grain boundary to be found. With ice this was by far the most satisfactory way of doing this, as though grooves can form which trace their locus, these are not only hard to detect but also infrequent.

When single crystals of ice were allowed to creep under a constant load, grain boundaries were often detected after a certain amount of deformation. Their formation was usually followed by an increase in the strain rate, with the result that the specimens collapsed - often with the formation of further grain boundaries. Such specimens were invariably

kinked and not necessarily in the plane of observation.

These grain boundaries did not follow any definite crystallographic orientations and though some were straight, just as many were curved. Thus it seems likely that non-basal dislocations were necessary for their formation.

A picture of a grain boundary is given in Fig. 14 and it can be seen that on this occasion it is marked by a groove which could arise either from displacements of the crystal lattice or else from etching by the paraffin. Slip bands are also visible and these clearly show the difference in the orientations of the two grains. The extensive distortion of the lattice, shown in this photograph, was not observed on every occasion but confirms the high state of internal stress that must have existed prior to the formation of the boundary. Dark bands parallel to the boundary are also visible but these are an interference effect which will be discussed later. (See section 4.4).

Thus it seems that as the single crystal deforms a high state of internal stress builds up, which is often relieved by the formation of a grain boundary. As a result of this the crystal is able to deform more easily.

It is convenient to point out here that there was no detectable difference in the way these boundaries formed in impure crystals from that in pure ones. However, as only organic impurities were present, this may not be generally

true as the internal energy of a crystal must depend on the impurities present.

3.2.4 CHANGES THAT OCCURRED AFTER THE LOAD WAS REMOVED.

Although no changes in the specimen's appearance were normally seen if it was left in position with the load removed, on two occasions recrystallisation did occur. This was detected with crossed polaroids and was to be found near the base of the specimens in the region where ice had accumulated during deformation. (See section 4.3). The individual crystals were quite irregular in shape and did not extend across the full width of the specimen - unlike the grain boundaries described in the previous section. As this phenomenon has already been extensively studied by Steinemann (1954(b)), no further comments will be made here, except to point out how this confirms the increase in internal energy that must result from the deformation.

3.3 SLIP BAND SPACINGS.

A study of the literature revealed that the only measurements that had been made of the spacing of the basal slip lines were by Wakahama (1962) and that these were not very satisfactory for the following reasons:-

(a) His measurements were only made at one value of stress.

(b) He quotes no spread in values which disagrees with results already reported in this thesis. (See Section 3.2.2).

(c) His extrapolation formula is purely empirical, takes no account of possible impurity effects and was not tested experimentally.

Thus there was no alternative but to determine these values experimentally as not only were they required to test a theoretical explanation for the Interference Bands (see Section 4.5) but they might show how impurities affect the deformation of ice.

3.3.1 APPARATUS AND TECHNIQUES.

As the slip bands could not be resolved properly without the use of a microscope, the apparatus described in Section 3.2 was not suitable. This meant that the specimens could not be studied directly, because the small working distance left no room for a paraffin container. An attempt to overcome this difficulty by mounting the specimen on the horizontal stage of a microscope failed, because any surface features were erased by the inevitable movement of the paraffin.

When ice is immersed in a solution of formvar in ethylene dichloride for some time, any surface irregularities are removed by its etching action. However, if the specimens are coated with paraffin before being put in the formvar, dissolution can be prevented because paraffin reacts with the formvar to form a protective film. Thus, by ensuring that the specimens were covered by only a thin layer of

paraffin, it was possible to obtain a formvar replica of the surface without etching having occurred.

Rectangular specimens, which had been cut from single crystals of ice, were used in the experiments. These were frozen into brass caps, which had knife edges attached to them, and stored in paraffin until required. As not all the cut ice was required for this, it was possible to use a piece of the excess for determining the orientation of the c - axis in the specimens.

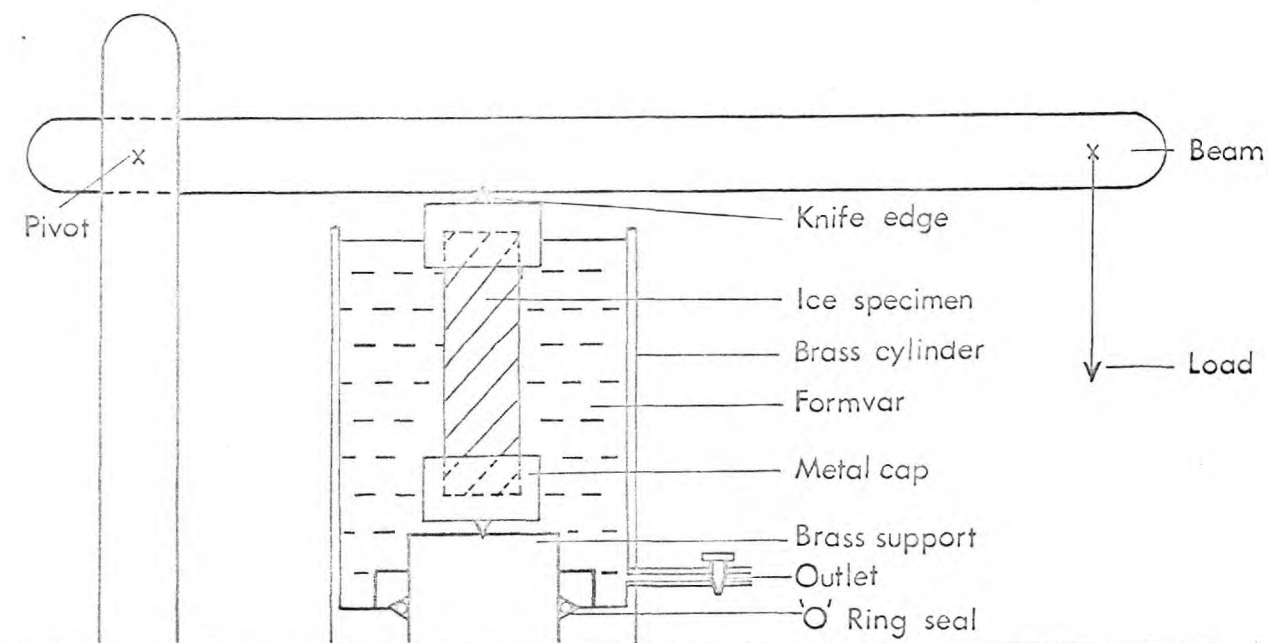
The specimens were stressed in the apparatus shown in Fig. 15 and it can be seen that this is essentially the same as that used in the Optical Studies except that the container was made of brass and there are no optical attachments. When necessary, the deformation could be measured with the vernier scale shown on the left of the photograph.

The brass cylinder held no liquid when the specimen was first mounted in the apparatus, as about fifteen minutes had to be allowed for the excess paraffin to drain off. If this had not been done, the film would have been too thick and so the deformation of the specimen might have been impeded. After this the 6% formvar solution was added and the run started.

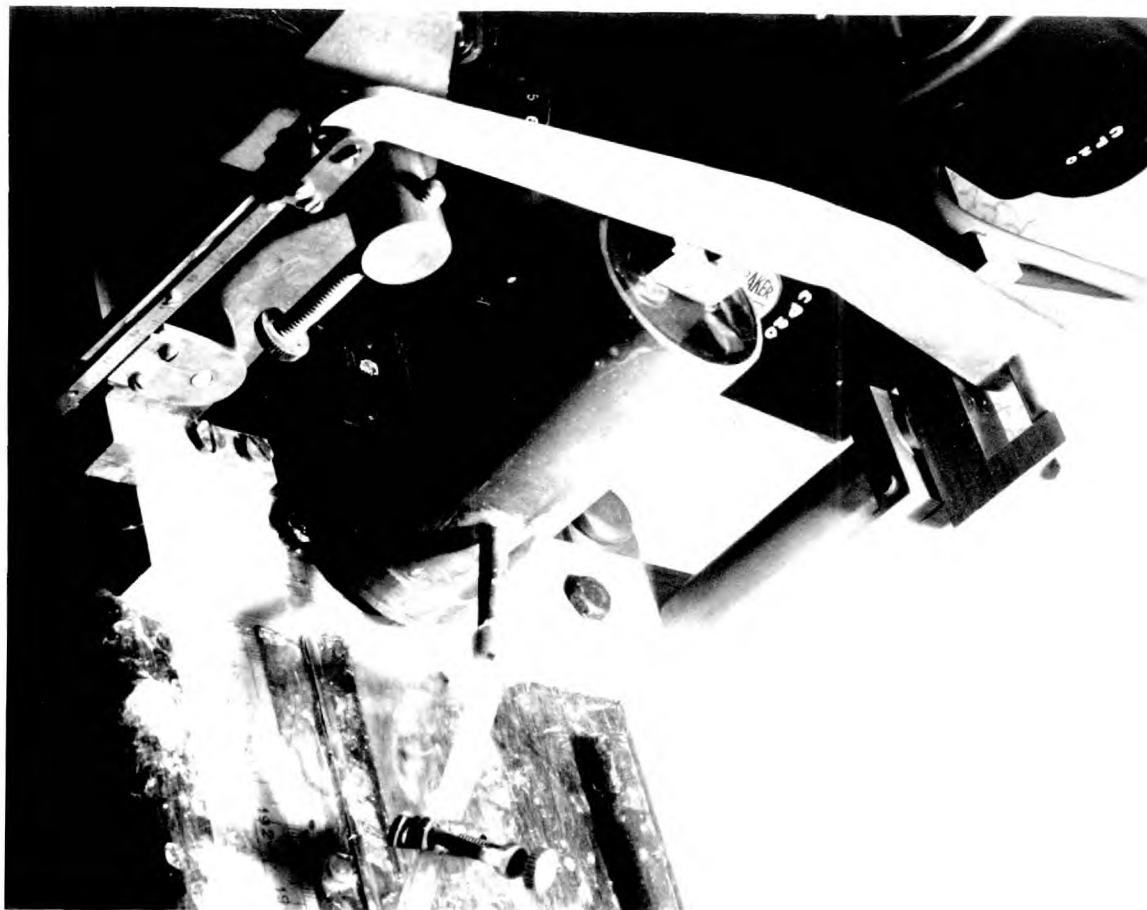
At the end of the allotted time (usually about a week) or sooner if it had collapsed, the specimen was removed and, after the excess solution had been drained off, it was left

Fig.15 Apparatus used to obtain replicas of slip band spacings

(a) Schematic diagram



(b) Photograph



so that the remaining film could harden. This took about 24 hours. The replica films were then separated from the ice, with the help of a razor blade and some water, and mounted on glass slides.

These slides were silvered at a small angle to the normal with Alumina^{um}, so enabling the spacings to be measured with a microscope using vertical illumination. The actual measurements were made with a vernier micrometer eye-piece and a typical replica is shown in Fig. 16.

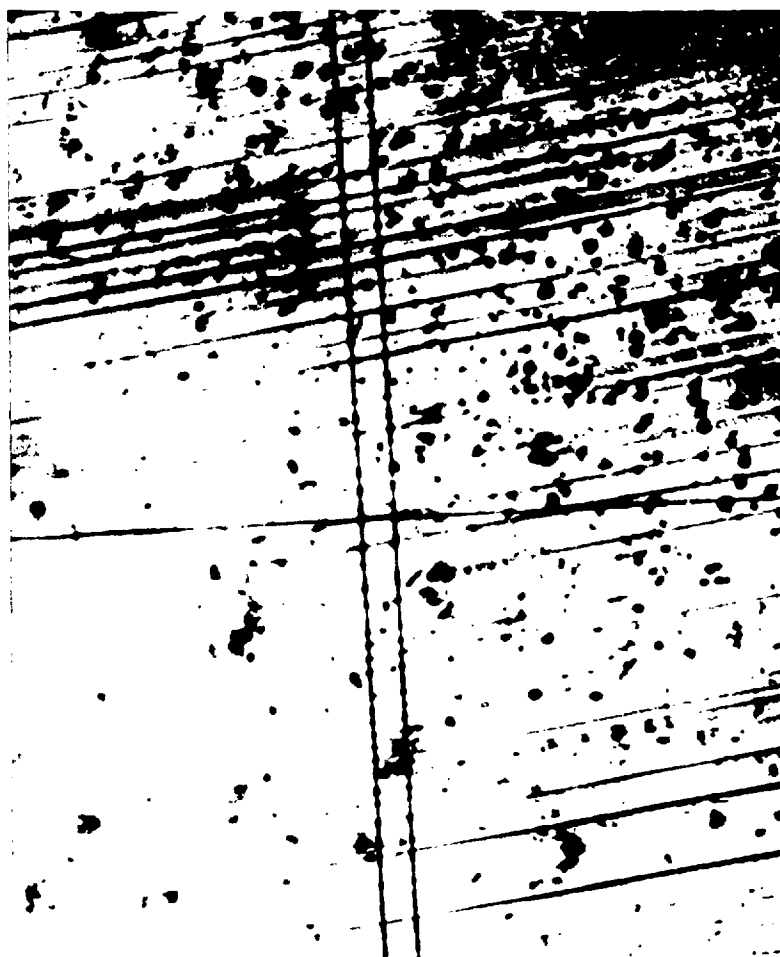
3.3.2 SOME PRELIMINARY REMARKS ON THE TREATMENT OF THE RESULTS.

At this juncture it is convenient to make a few introductory remarks on the treatment of the results.

Fig. 17 shows a typical set of results, which were obtained by measuring the spacings of the slip bands on a silvered replica (See Fig. 16). It can be seen that the values exhibited a considerable spread and so it seemed logical to display this on a histogram - the readings being divided into the largest number of classes that would give a sensible graph.

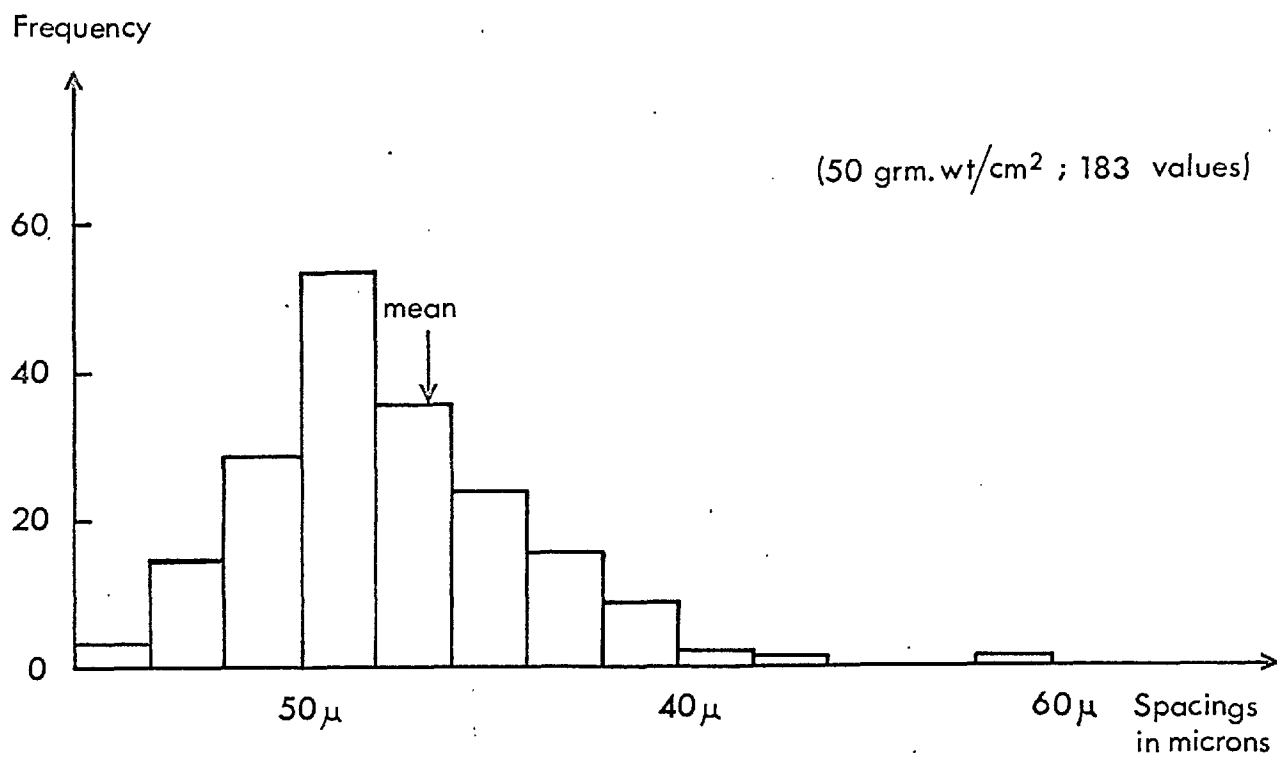
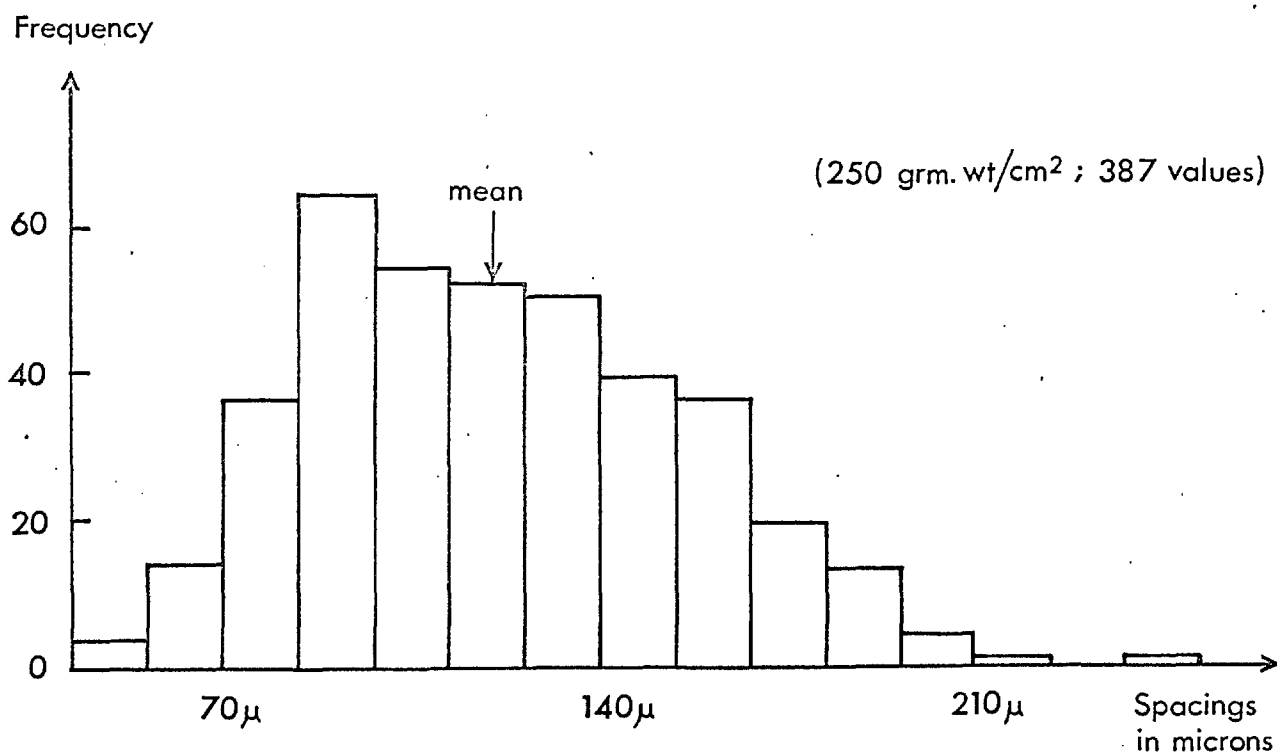
Although the mean spacings were calculated from the readings in the usual way, only a rough measure of the spread seemed necessary and this was obtained by calculating the root mean square deviation using the classes. With reference to this last step, it should be noted that the

Fig. 16



Replica of slip bands
Distance between the ^{cross-wires} graticules = 50μ

Fig. 17 Typical set of results



mean was represented by the class in which it fell.

The resolved shear stresses on the basal plane were calculated from the total applied compressive stress in the usual manner by multiplying it by the function " $\sin g \cos g$ " (See section 2.5). This angle was subject to an error of about $2\frac{1}{2}^\circ$ and it is this error which was used in plotting the results.

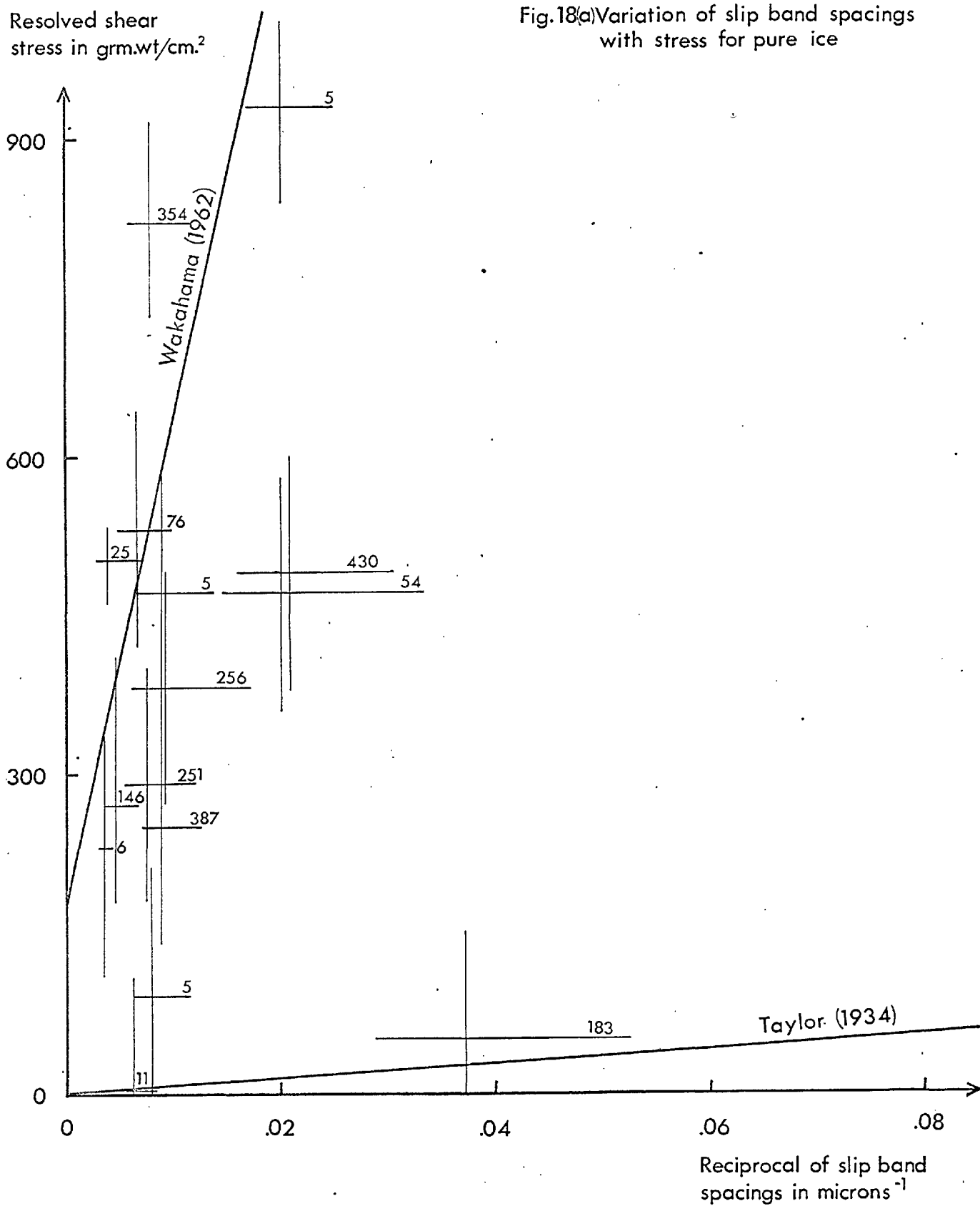
3.3.3 THE RESULTS.

The variations in the spacings of the slip bands for any specimen are illustrated by the two histograms which are plotted in Fig. 17. These are quite typical and show the asymmetric nature of the distribution with the mean value invariably larger than the mode. As these were the only features common to all the distributions, the failure of attempts to fit Poisson and Gaussian Distribution Curves to the results is hardly surprising.

All the results obtained are plotted in Fig. 18 and the number of readings on which each point is based is inscribed on its right hand side. It can be seen that the "poorer" points do not noticeably disagree with those based on a large number of readings.

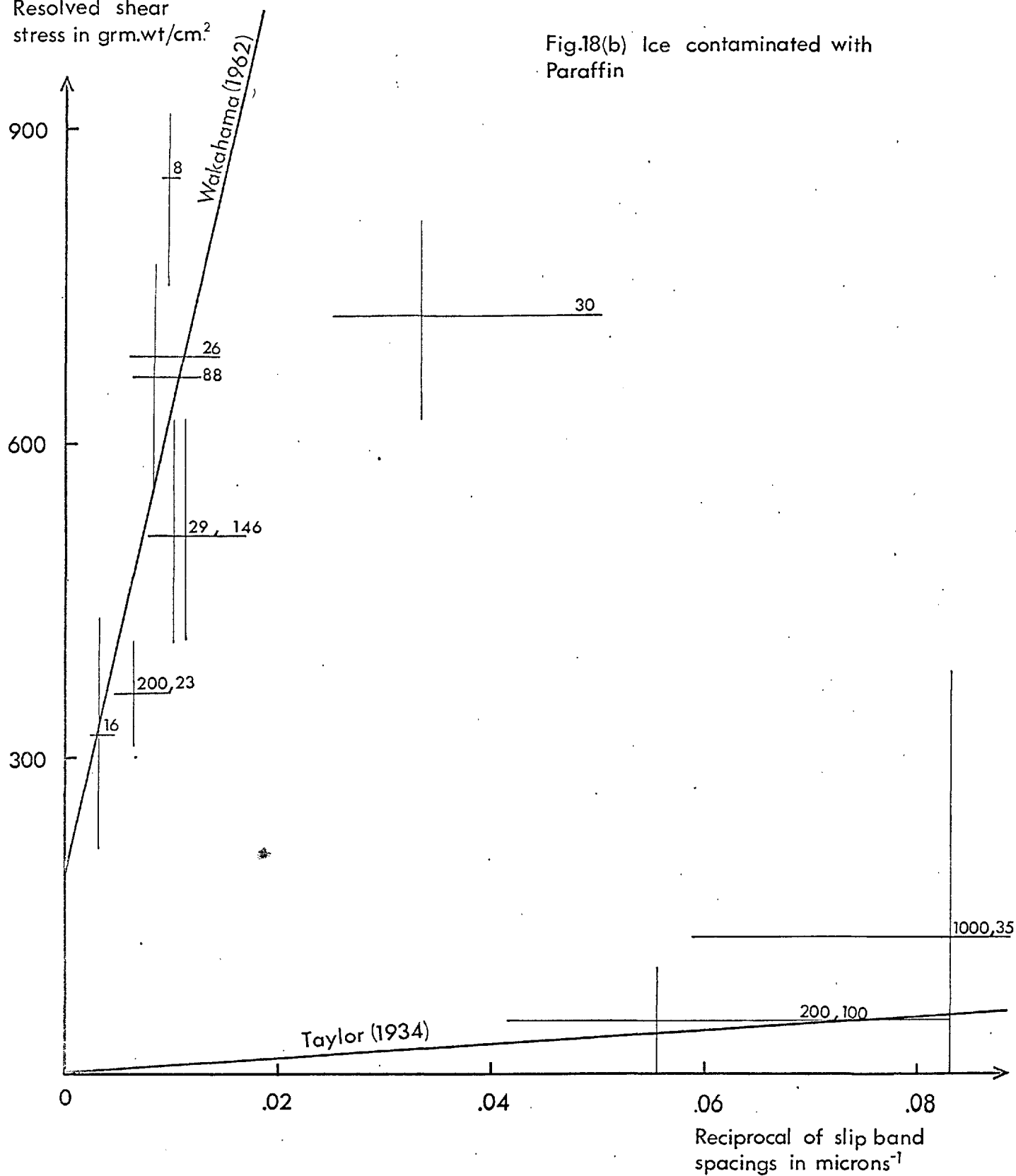
Figs. 18(a) and 18(b) show the way the slip band spacings vary with resolved shear stress both for pure ice and for ice contaminated with paraffin (the results having been corrected for the inclination of the basal plane to

Fig.18(a) Variation of slip band spacings with stress for pure ice



Resolved shear
stress in grm.wt/cm^2

Fig.18(b) Ice contaminated with
Paraffin



the surface). Although the spacings seem to decrease with increasing stress, they show a considerable spread and, at low stresses, unusually fine spacings are sometimes recorded. These readings show that the apparatus would have been quite capable of resolving such spacings had they been in any prominence on the other specimens.

A film of solid formvar invariably formed at the air-formvar interface and, though it was kept flexible by coating it with ethylene dichloride, the actual resolved shear stresses must have been slightly smaller than those quoted. However the error introduced cannot have been very large as the film could not even support the unloaded beam, and even hanging weights smaller than the usual 500 gm wts. from the beam, did not produce unusual results. Furthermore this error cannot account for the anomalous plots at low stresses, as it would have tended to increase not decrease the spacings of the slip bands.

Except for the number of anomalous plots at low stresses, no difference could be detected between the two sets of results for pure and impure specimens (compare Figs. 18(a) and 18(b)). This may not be true for all contaminants and it would be interesting to repeat the experiments with ice which had been contaminated with caesium fluoride, as this was the compound that affected Truby's (1955) results.

3.3.4 DISCUSSION.

In addition to the actual results, two straight lines have been plotted in Fig. 18. The one labelled Wakahama (1962) is a plot of the relation he proposed in his paper:-

$$(\sigma - 200) \cdot d = 4.5 \quad \dots \dots \dots (1)$$

(where σ = applied shear stress in gms wt/cm²; d = slip band spacing in cms).

It can be seen that for stresses in excess of 200 gm wt/cm², the values predicted by this formula are quite a good guide to the experimental ones - though sometimes the slip bands were closer together than predicted. Furthermore, as some of these points were obtained with stresses that were smaller than the minimum value Wakahama says is required for slip, the question arises of just what equation (1) means.

A possible answer to this problem may be found in the literature on the deformation of metals. Thus, Andrade and Roscoe (1937) found that if the surface of a deformed metal was examined under an optical microscope, irregularly spaced slip lines were seen. However, at higher magnifications, more finely spaced ones became visible and these were quite regularly spaced. In the case of lead, they succeeded in measuring these spacings and their frequency plot is a normal Gaussian one. Though this is quite different from the skew plots obtained with ice (Fig. 17),

there was a tendency for the mode and mean to be closer together than usual with the anomalous points. (i.e. Those with smaller spacings than usual).

With metals, the spacings of the regularly spaced slip bands can usually be quite well represented by the following formula derived by Taylor (1934):-

$$G \cdot d = 0.16Ga$$

(where G = shear modulus; a = lattice parameter).

If one assumes that $a = 4.5A^\circ$ (Barnes (1929)) and that $G = 10^{10}$ dynes/cm² rad. (Dorsey (1940)), then in the case of ice this becomes:-

$$G \cdot d = 0.72 \dots\dots\dots (2)$$

This equation is plotted in Fig. 18 (see line marked Taylor (1934)) and it can be seen that, though the anomalous points are in quite good agreement with it, it predicts values that are much smaller than those obtained from equation (1).

Thus it seems that one normally measures the coarse slip band spacings and that these are quite well represented by Wakahama's formula. However, at low stresses the more finely spaced slip bands were sometimes detected and their spacings lay closer to the values predicted by Taylor. These were not detected at higher stresses probably because the minor topological features due to them were completely masked by the more prominent coarse slip bands. It is

interesting to note that Muguruma (1963) may have detected both types of band at the same time during his etching experiments.

CHAPTER 4.

THE OPTICAL CHANGES THAT OCCUR IN ICE DURING ITS DEFORMATION.

4.1 INTRODUCTION.

When Nakaya (1956) studied some of his specimens between crossed polaroids, he observed some rather complex interference patterns. These were very similar to what would have been predicted by photo-elastic theory, but as no stress was being applied when the observation was made, this cannot be the full story (see Chapter 1.)

As no other workers had reported this effect, it was decided to try and detect it again in order to elicit its true nature, and it was hoped that it might provide a new tool for studying the deformation mechanisms in ice.

Several other effects have been observed during the course of this investigation, which have provided some very interesting information on the way ice deforms. Though these are described in this Chapter, the mathematics necessary for their explanations is rather complicated and so most of it has been put into Appendices.

4.2 APPARATUS AND TECHNIQUE.

As the same specimens and apparatus were used for both this study and that on the surface changes that occur during creep, very few comments are necessary because full details have already been given in Section 3.2.1. However, it must be stressed that in this case the specimens were illuminated by a parallel beam of light.

Both the optical arrangements described in Section 3.2.1 were used as some effects required a close-up of certain regions and others required a general view of the entire specimen.

4.3 THICKNESS CONTOURS.

Ice is a uniaxial substance and therefore exhibits the phenomenon known as Double Refraction. As the theory is given in many standard texts (see for example Jenkins and White (1953)) it will not be reproduced here. Any unusual facets of the optical behaviour of ice will be considered as they arise.

Before discussing the effect known as "Thickness Contours" in this Thesis, one point must be settled. In general, every beam of light that is incident on a block of uniaxial material will divide into two beams during transmission and often the angle between these two is appreciable. However, it is shown in Appendix II that, because the two principal refractive indices in ice are so nearly equal, the ordinary and extra-ordinary waves may be considered to travel along the same paths.

In Appendix IIIB the intensity of the light emerging from an optical system, which consists of a piece of ice situated between two crossed polaroids, is considered. It is shown that this intensity is zero not only when the

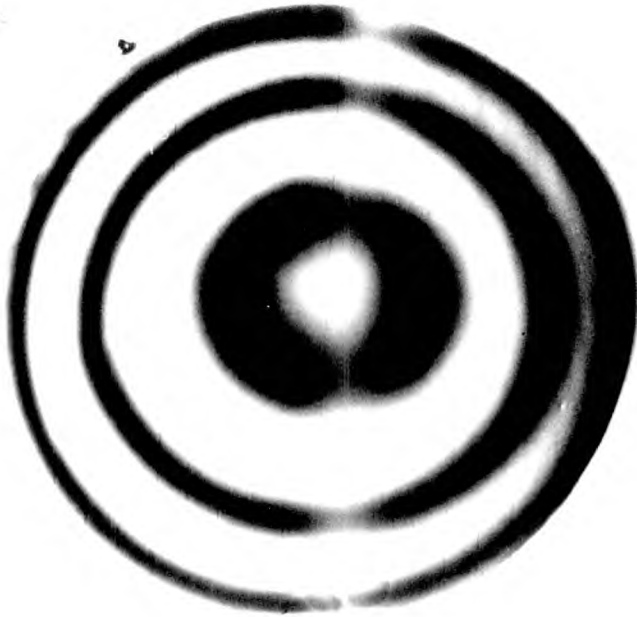
transmission axes of the polaroids are parallel to the principal axes in the ice, but also when the optical thickness of the specimen has certain values. If any part of the specimen is of this thickness, it will remain dark no matter how the crossed polaroids are rotated.

Such regions were often encountered with specimens in which the optic axis did not make too great an angle with the surface. When the optic axis is almost directed towards the observer the difference in the two refractive indices is so small that the minimum thickness required for the effect to occur (i.e. the distance, t , when $m = 1$ in equation (3) of Appendix IIIB) is greater than any likely thickness variations. If these are assumed to be about 1 m.m. then they become smaller than the theoretical minimum thickness when the optic axis makes an angle which is greater than 42° with surface of the specimen.

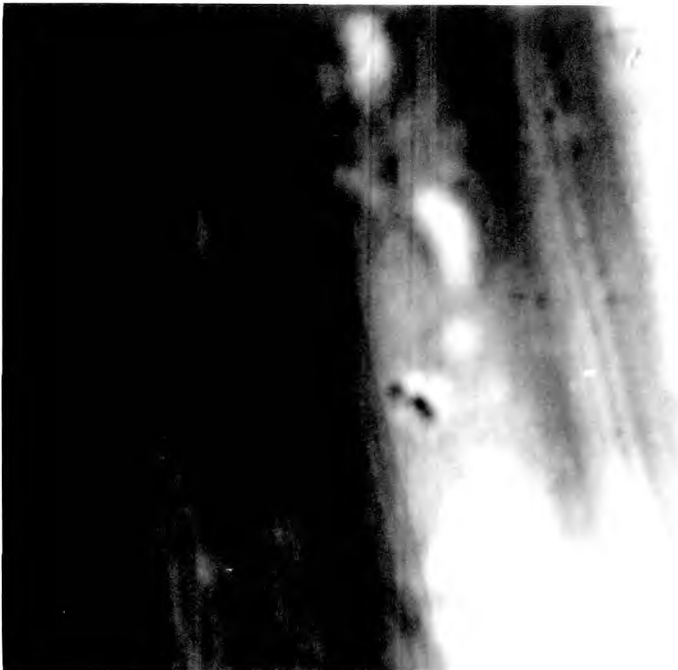
The existence of the effect was confirmed by deliberately contouring a specimen by pressing a warm piece of metal such as a ball bearing into the crystal. Fig. 19(a) establishes this phenomenon quite well as not only do the dark lines in the photograph follow the expected contours and do not move as the polaroids are rotated, but the diameters of the circles agree quantitatively with the calculated values.

This effect has been treated in some detail because it revealed an important change that occurred as the specimens

Fig. 19



(a) Artificial thickness contours

 $\frac{1}{2}$ cm.

(b) Flow contours

 $\frac{1}{2}$ cm.

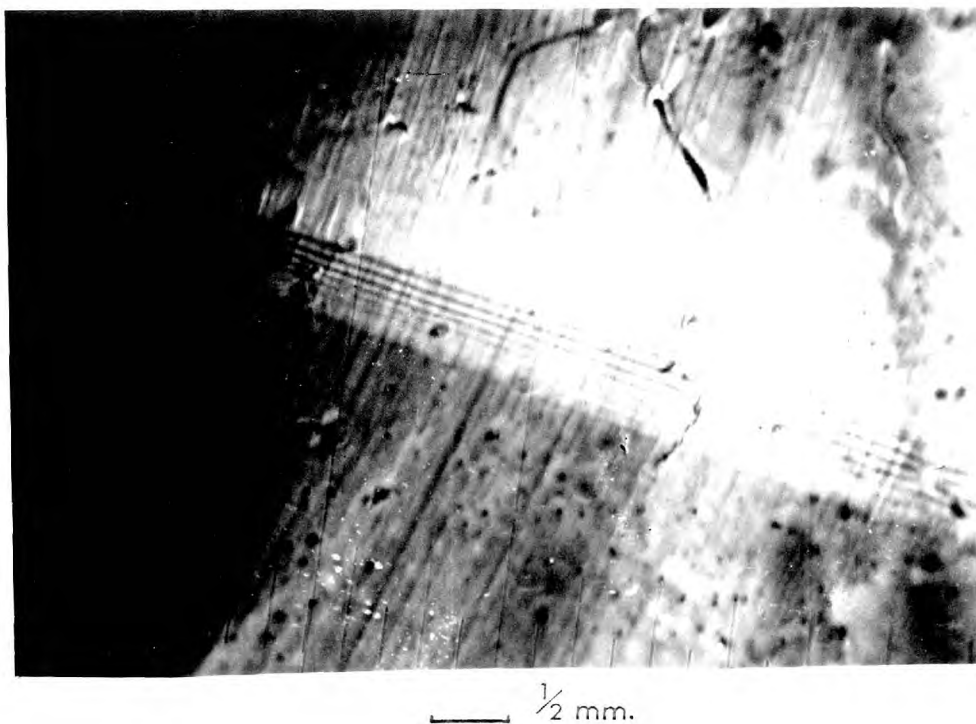
deformed. It was found that in regions where prominent slip bands were visible, the intensity of the light diminished often practically to zero (see for example Fig. 19(b)). As the areas remained dark even when the crossed polaroids were rotated, a change in the thickness of the specimen must have occurred there (see Chapter 5).

4.4 INTERFERENCE BANDS AT GRAIN BOUNDARIES.

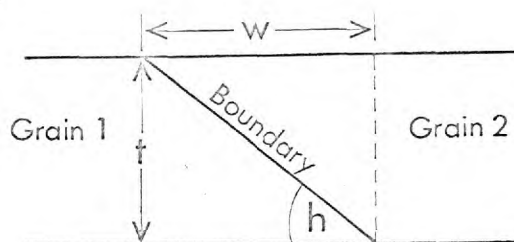
Some dark lines may be seen in Fig. 14(b) which follow the contour of the grain boundary. It was pointed out in Section 3.2.3 that these were an interference phenomenon and disappeared if the polaroids were removed. As the crossed polaroids were rotated they moved and there was an optimum position of the microscope barrel for them to be seen most clearly. However these bands were not observed at every grain boundary, nor were they always so regularly spaced.

Another example of these bands is given in Fig. 20(a), and Fig. 20(b) is a schematic cross-section of the specimen in the vicinity of this boundary. It can be seen that the optical model used in Section 4.3 must be modified in such regions by replacing the single crystal of ice by two similar crystals of different orientations and thicknesses. This system is analysed in Appendix IIIC and it can be seen that the output intensity depends on the optical path

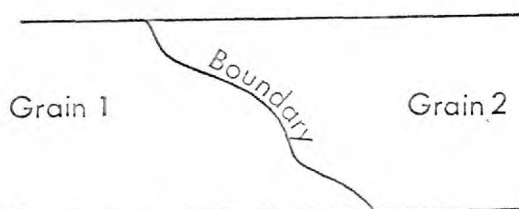
(a) Interference bands at a grain boundary



(b) Cross-section of a regular grain-boundary



(c) Cross-section of a irregular grain-boundary



lengths in the two crystals, their relative orientations and on the setting of the crossed polaroids. Thus, because the optical path lengths vary as one crosses the grain boundary, an interference pattern will be observed and this will change as the polaroids are rotated. Furthermore, such bands will only be seen if the overlap of the two grains is visible in the microscope (i.e. $h \neq 90^\circ$) and so the failure to observe them at all grain boundaries is quite understandable.

This explanation of the interference bands was checked quantitatively with boundaries which only involved kinking in the plane of the specimens. In such instances the value of " g_2 " did not change for either grain and so the principal refractive indices could be calculated. By using these, the experimental values of " t " and " h ", and equations (4) and (5) of Appendix IIIC, theoretical values for the spacing of the fringes could be calculated and compared with the measured average spacings obtained with one or other of the two grains blacked-out. The results of such exercises are shown in Table 4.1 and it can be seen that the agreement is quite satisfactory.

The last two columns of this table give the differences in the orientations of the two grains as measured from their extinction positions and from the change in direction of the slip bands in crossing the boundary. When kinking occurs in

TABLE 4.1 SPACINGS OF INTERFERENCE FRINGES AT GRAIN BOUNDARIES.

Type of Ice	Theoretical spacings	Measured spacings	Difference in extinction positions	Change in Direction of slip bands
Contaminated	8.9×10^{-3} cms.	8.3×10^{-3} cms.	10°	$(7 \pm 4)^\circ$
with alcohol	5.3×10^{-3} cms.	5.3×10^{-3} cms.	$3\frac{1}{2}^\circ$	$(4 \pm 2)^\circ$
"	3.0×10^{-2} cms.	3.2×10^{-2} cms.	11°	$(6 \pm 1)^\circ$
Contaminated				
with paraffin	6.2×10^{-2} cms.	6.2×10^{-2} cms.	1°	$(7 \pm 1)^\circ$
Pure	1.7×10^{-2} cms.	1.9×10^{-2} cms.	5°	$(4 \pm 1)^\circ$
"	2×10^{-2} cms.	$(2 \text{ to } 3) \times 10^{-2}$ cms.	5°	$(6 \pm 1)^\circ$

the plane of the specimen, these will agree; ~~otherwise~~
~~they will not.~~ However, provided the discrepancy is small,
 this will not affect the agreement between the two values for
 the band spacings, as " n' " is not a very sensitive function
 of g_2 (where " n' " is the extra-ordinary refractive index).

The analysis shows that when one of the grains is
 blacked out, the spacing of the bands depends only on the
 variation in the optical path length in the other grains.
 Thus a boundary such as that shown in Fig. 20(b) will have
 uniformly spaced bands associated with it because " h " is
 constant, while that shown in Fig. 20(c) will exhibit
 irregularly spaced ones. This means that when the observed
 bands were erratically spaced, the value of " h " must have
 varied from point to point.

4.5 THE INTERFERENCE BANDS.

As it may provide some valuable information on the
 mechanism by which ice deforms, the effect described in this
 section is considered to be the most important one to emerge
 from this investigation. For the sake of clarity, the
 section is divided into two parts - the first describes the
 phenomenon; while the second discusses its possible
 explanation.

4.5.1 THE PHENOMENON.

Some dark bands can be seen in Fig. 21(a) which are

Fig. 21



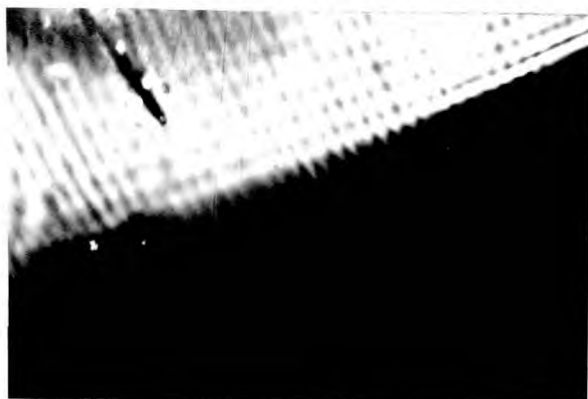
(a) Interference bands

— $\frac{1}{2}$ mm.



(b) Change in direction of interference bands when the surface is contained.

— $\frac{1}{2}$ mm.



(c) Interference bands crossing a grain boundary.

— $\frac{1}{2}$ mm.

approximately parallel to the basal plane. As these disappeared when the polaroids were uncrossed and changed to faint coloured fringes if a white light source was used, they must have been an interference effect of some type. Furthermore, these "Interference Bands" moved as the crossed polaroids were rotated and were only visible when the specimen was nearly blacked-out.

In addition to those just mentioned, the Interference Bands exhibited the following qualitative properties:-

(1) The Interference Bands appeared after a finite deformation, which varied from specimen to specimen - but was usually smaller than that required for slip bands to be observed.

(2) Though their visibility varied, the Bands tended to become more distinct as the deformation of the specimen increased.

(3) The Bands were not distributed uniformly across a specimen - being most prominent in regions where slip bands finally developed.

(4) Though no instantaneous change could be detected if the load was removed, as time passed the Bands became less distinct and on occasions they actually disappeared.

(5) Sometimes the spacing of the Bands was rather erratic and on two occasions forking was observed. However, these effects seem to have been due to variations in the

thickness of the specimen as by deliberately contouring the ice, the Bands could be made to change direction. (Fig. 21(b)).

(6) On crossing a grain boundary, the Bands changed direction and, if the interference effect described in Section 4.4 was also present, a curious cross-hatching ensued. (Fig. 21 (c)).

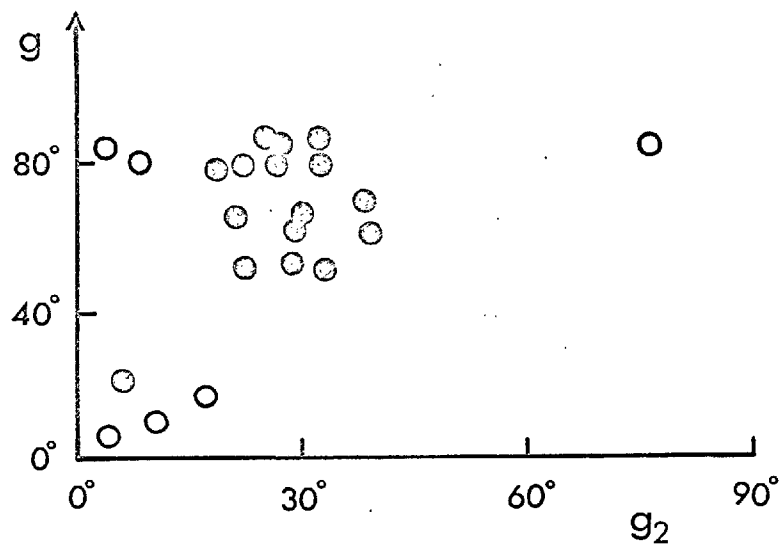
(7) The Bands were present even if the specimen was not immersed in paraffin.

By photographing them, the spacings of the Bands were measured and it was found that for any specimen there was quite a spread in values (i.e. the Bands were not equally spaced). Within the limitations imposed by this, it would appear that the Interference Bands have the following quantitative properties:-

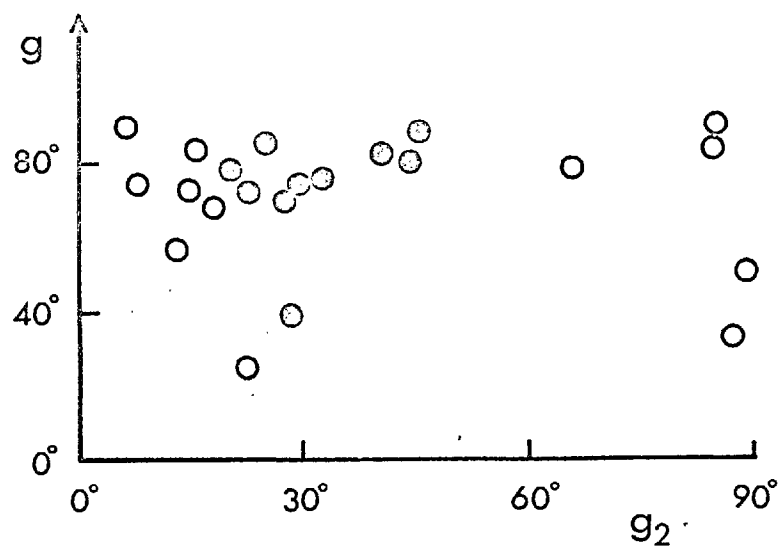
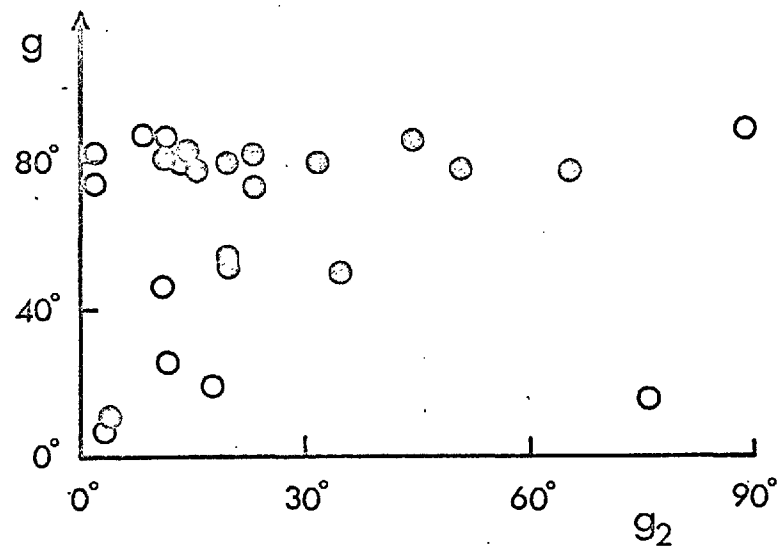
(1) For a given specimen, the spacing of the Bands was independent of time, strain, orientation of the polaroids and the wave length of the light used for their observation.

(2) Altering the stress during a run produced no detectable change in the spacing of the Bands.

(3) The probability of observing the Bands seemed to depend on the angle θ_2 that the optic axis made with the surface of the specimen. Thus it can be seen from Fig. 22 that they were not likely to be detected when θ_2 was near 0° or 90° . (Table 4.2). It can be seen from this Table



Pure ice

Ice contaminated
with paraffinIce contaminated
with alcohol

($\circ$ No bands
 $\circ$ Bands observed)

that the minimum value of g_2 depended on the purity of the ice and could not be fixed very accurately.

TABLE 4.2 The smallest values of g_2 at which Interference Bands were usually seen.

Type of Ice	Values of $g_2 = g_2)_d$
Pure	$15^\circ - 20^\circ$
Ice contaminated with alcohol	$10^\circ - 15^\circ$
Ice contaminated with paraffin	$20^\circ - 22^\circ$

However direct experimental evidence for the existence of such a minimum angle was provided by certain specimens which kinked and in so doing caused g_2 to change from a value that was larger than $g_2)_d$ to one that was smaller (or vice versa) - with the result that the Interference Bands either appeared or disappeared.

(4) The spacing of the Bands did not seem to depend much on the applied stress (Fig. 23).

(5) The spacings of the Bands tended to decrease with decreasing g_2 (Fig. 23).

4.5.2 POSSIBLE EXPLANATIONS.

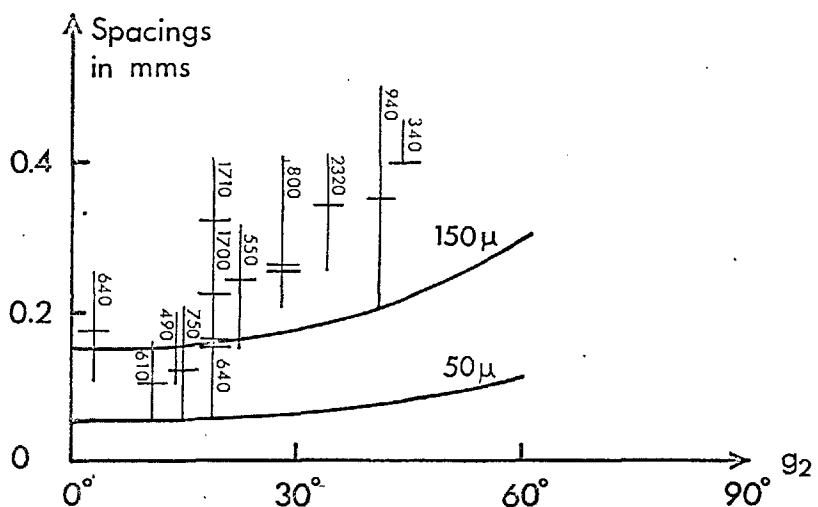
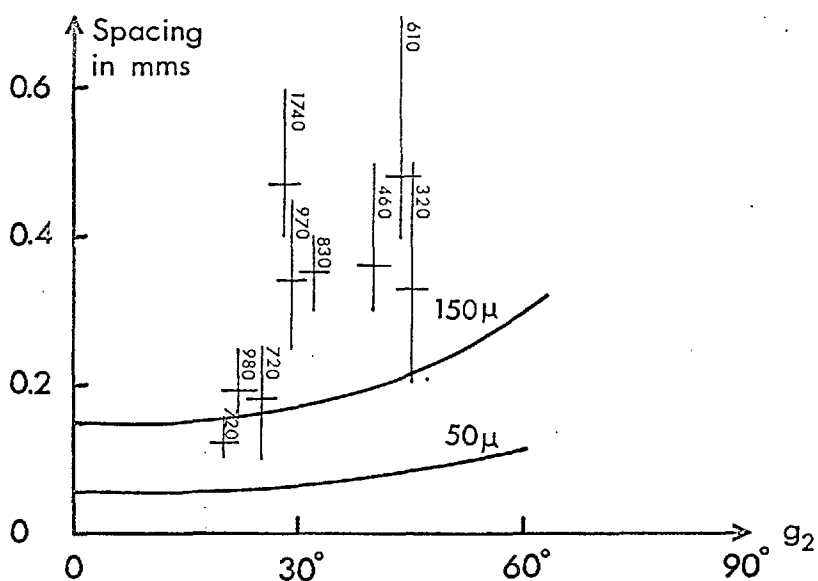
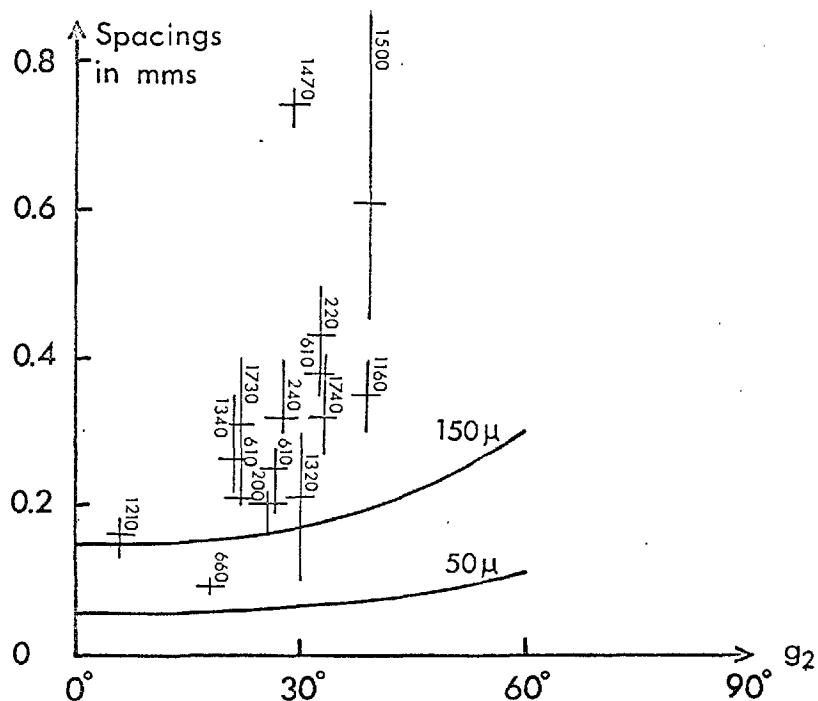
This phenomenon appears to have two possible explanations and these will now be considered in turn:-

(a) Grain Boundary Hypothesis.

As the similarity between these Bands and those

Fig. 23 The dependence of the spacings of the interference bands on g_2 .

(The two curves show the variation of the maximum and minimum slip band spacings with " g_2 ". Shear stresses are marked beside each point.)



encountered at some grain boundaries (see Section 4.4) was quite marked, it seemed logical to try and explain them in terms of lattice disorientations arising from the deformation of the crystal. Having accepted this as a premise, the next step was to decide on the orientation of the boundary across which such a change occurred, and as the Interference Bands were invariably parallel to the basal plane, this seemed to be the only reasonable choice. However, for interference bands to be seen at ordinary grain boundaries, the grains had to have different extinction positions, which means that in this case the elements must be rotated with respect to each other about an axis lying in the basal plane. As the lattice will be considerably distorted by this, the actual angle of twist will be small and so, assuming that this was true, the resulting optical system is analysed for a simple case in Appendix IIID.

The model chosen consists of a pile of ice plates with successive disorientations of $\pm \rho/2$ with respect to the mean extinction position and though very simple, exhibits most of the features associated with the Interference Bands. (See Tables A.2 and A.3 for the relevant equations and Fig. 27(a) for a schematic diagram of the model):-

(1) It can be seen from Table A.2, that apart from the usual extinction positions and thickness contours, each point on the surface blacks-out at an angle Θ_0 (see Table

A.3 and Fig. 27(b)) which is nearly at the extinction position (because ρ is assumed to be small). As the value of this angle is different at different places on the surface, an observer will see a series of bands, parallel to the basal plane, which move as the polaroids are rotated and are only visible near an extinction position.

(2) The larger the value of Θ_0 , the easier it will be to detect these bands as the rest of the surface will appear lighter. Now the values of Θ_0 are proportional to ρ (see Table A.3) and, as this increases as deformation proceeds, the Bands will appear after a finite deformation - possibly before any slip bands can be seen.

(3) Although the Bands will first be seen at places where Θ_0 is largest (i.e. at points such as X and Y in Fig. 27(b)), the extinction positions corresponding to X' , X'' , Y' , Y'' etc. will become detectable as deformation proceeds. The Bands at X' and X'' etc. will have finite widths and so will probably overlap - thus improving the visibility of the Interference Bands.

(4) All the values of Θ_0 depend on the total retardation, Δ (See Table A.3), and so any thickness changes will alter the x - value corresponding to a given Θ_0 . Thus forking, erratic spacing and changes in direction when the surface is contoured, are quite naturally explained.

(5) If the direction of the basal plane changes across a grain boundary, the Interference Bands will also change direction and may have the interference bands associated with such boundaries superimposed on them - if these are present (i.e. "Cross-hatching" will occur).

(6) As paraffin is not an optically active liquid, the Bands will not be affected by its presence.

(7) The work of Higashi and Sakai (1961) has shown that grain boundaries in ice do not break up into sub-boundaries unless the disorientation is about 10° . As ρ never attained values of this order, the original boundaries are quite capable of containing any deformation that occurred and so the spacing of the Interference Bands (which in the case of the model is equal to twice the thickness of the constituent plates) would not be expected to depend on such factors as time, strain, or stress changes - especially as the ordinary grain boundaries would form long before this.

(8) If it is assumed that the double bands at X' , X'' etc. cannot be resolved, either because they overlap or because the corresponding value of Θ_0 is too close to the extinction position, then, for the simple model chosen, the spacing of the Interference Bands will be $2s'$. Thus by extending this argument it can be seen that an ice specimen with a periodicity of " n " (the simple model had one of 2) will have bands spaced at intervals of " ns' ".

It can be seen from Fig. 24(a) that the angle the c - axis makes with the surface " g_2 ", will determine the observed spacing to some extent, and so if this spacing is " nd " when $g_2 = 0$ then for other values of g_2 it will be given by:-

$$ns' = \frac{nd}{\cos g_2}$$

Thus the spacing of the Interference Bands will decrease as " g_2 " $\rightarrow 0$.

(9) As " g_2 " $\rightarrow 90^\circ$ the ice becomes increasingly isotropic and so all the retardances tend to zero (i.e. $d_o \rightarrow 0$ etc). Thus the Interference Bands will disappear.

(10) The disappearance of the Interference Bands at small angles of " g_2 ", though not entirely unexpected, cannot be explained so satisfactorily. With the model chosen, the Bands will disappear when the number of plates is less than two. Unfortunately, for the thickness of ice used in the experiments, this gives a cut off at about 5° compared with the experimental values of about 15° .

Increasing the periodicity of the model would not improve this situation as the effective value of " n " would only be reduced as " g_2 " $\rightarrow 0$. This means that although the positions of the Bands might change, they would disappear no sooner.

Now the actual Interference Bands were found to be about 0.1 mm in width and, as " ns " must be greater than

this for them to be resolved, this is probably the reason for their disappearance at small values of " g_2 ". The exceptions to the disappearance rule would then have been due to the bands having been finer or further apart.

(11) It can be seen from Figs. 22 and 23 that the spacings of the Interference Bands were greater than those of the slip bands and seem to be independent of the stress. The model predicts that the Interference Bands will be further apart than the slip bands, but as they were not an integral number of slip band spacings and did not vary regularly with the resolved shear stress, the boundaries cannot have formed across every active slip band. Without more data, it is impossible to make any more comments except to point out that their formation would seem to have depended on the purity of the ice.

(12) During the optical experiments, two runners were used to keep the ice holders aligned and so the applied stress was not purely compressive but of the form shown in Fig. 24(b). This system is quite capable of causing rotation to occur about an axis lying in the basal plane and so the formation of the boundaries, required by the model, could have occurred. Furthermore the degree of rotation at each boundary has only to vary about a mean value for the Interference Bands to be formed.

Experimental evidence for this rotation was provided

by the change in the extinction position as deformation proceeded - it became less sharply defined and the extinction positions at the top and bottom often differed by a degree or two.

(13) Finally the formation of coloured fringes in white light is to be expected as all the retardances depend on the wave length of the light used. Thus for a given Θ_0 , the value of x will vary with wave length (see Fig. 27(b)).

This hypothesis is quite capable of predicting all the experimental features of the Interference Bands and it is therefore reasonable to suppose that tilt boundaries must form during the deformation of ice.

If the existence of the tilt boundaries is accepted, the question of what dislocation networks are required for their formation arises. This question is answered in Appendix IV and it can be seen that two are necessary. The first is hexagonal in form and consists of ordinary basal dislocation lines with a Burger's vectors - this gives the twist component. However, the tilt component requires basal dislocation lines with Burger's vectors having a g component. Furthermore these must be active as ρ changes during the course of deformation, and this can only be achieved by moving them closer together. In practice they will probably be zig-zag in form (not straight as shown in Fig. 28(d)) as they will have to compromise between a decrease in energy

when they lie in potential energy wells and an increase if their length increases as a result of this (see Read (1953)).

Such networks inevitably involve a considerable distortion of the lattice and so it is hardly surprising that the Interference Bands tended to disappear when the stress was removed. Incidentally this sometimes occurred after a conventional grain boundary formed - probably due to this relieving most of the stress.

(b) Stress-Optical Explanation.

Although the explanation just given seems able to explain all the experimental observations, it might be argued that some form of stress-optical effect could also do this. However it will be shown below that this is not the case.

The inhomogeneous distribution of internal stress that results when a crystal is plastically deformed, may be studied directly if it is transparent and exhibits a photo-elastic effect (Nye (1949, 1950)). If this is the origin of the Interference Bands then, as no part of the crystal normally remained dark for all orientations of the polaroids, they must be isoclinics. (N.B. Each isoclinic traces the locus of all points with a particular extinction position). However this is not compatible with certain experimental results:-

- (1) No matter what the value of the applied stress,

the Interference Bands could only be detected near extinction positions. Thus, if they are isoclinics, this means that the directions of the principal stresses were always very close to the natural ones in ice. This does not seem very likely.

(2) The stress distribution would be expected to exhibit the same periodicity as the slip bands, but Fig. 23 shows that the Interference Bands were invariably further apart than the active glide planes.

(3) As inhomogeneous internal stresses must exist near a grain boundary, it is surprising that the Interference Bands only appear to change direction in crossing such regions - isoclinics would be modified.

(4) Nye states in his papers that the stress-optical effect is most easily seen when the active glide planes are viewed edge on (adjacent planes not overlapping). However the Interference Bands were not observed when " g_2 " became small.

(5) When the surface of the specimen was contoured, the Interference Bands changed direction whereas isoclinics would not have been affected.

In view of these facts it seems reasonable to conclude that the stress-optical effect is incapable of explaining the experimental results and so cannot have been the cause of the Interference Bands. However, it must be emphasised

that ice does exhibit a stress-optical effect - though under entirely different conditions (see next Section).

4.6 THE STRESS -OPTICAL EFFECT.

4.6.1 THE PHENOMENON.

One of the aims of this investigation was to study the "stress-optical" effect reported by Nakaya (1956). This phenomenon proved to be extremely elusive but was finally observed when the specimen was viewed along the optic axis and subjected to an inhomogeneous stress. The crystals either had holes drilled in them or else they were notched on one side. No effect was detected when this was not done.

In this orientation the specimens exhibited very poorly defined extinction positions and the effect manifested itself as dark "wipers" which moved as the crossed polaroids were rotated. On some occasions dark regions formed which remained dark as the polaroids were rotated and which could not have been thickness contours as ice is almost optically isotropic when viewed in this direction. They were probably Isochromatics and the "wipers" could well be Isoclinics. (Examples of the experimental features of this effect may be found in Fig. 25).

When a load was applied to a suitable specimen nothing usually occurred at the moment of application - though on two occasions "wipers" appeared which pivoted about points

Fig. 25 Examples of the stress - optical effect



Load off



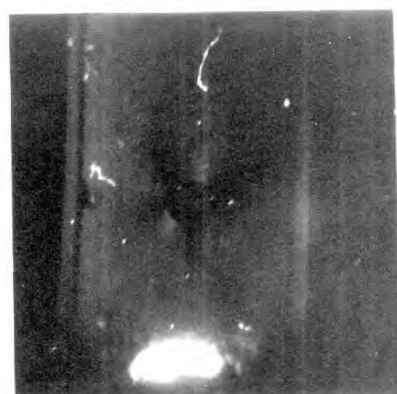
Load on



$\theta = 60^\circ$



$\theta = 27^\circ$



0 hrs



32 hrs

of stress concentration as the polaroids were rotated. These were probably due to the relatively high stress applied in these particular cases.

Then, as the specimen deformed, the variation in the extinction position across it became quite large and, when the specimen had a hole drilled in it, bore a marked resemblance to that observed with a comparable piece of perspex. If the load was removed, the pattern changed instantaneously to a form it would have attained had the load been left on and, if it was left off, this trend continued - albeit at a slower rate. Reapplying the load caused an instantaneous change in the opposite direction, though after this the pattern continued to change in the conventional direction. Repeating this procedure several times produced the same results.

4.6.2 DISCUSSION.

It would be incorrect to describe this phenomenon as a photo-elastic effect because the stresses that caused it were only partially elastic. (A pattern was observed even when the applied stress was zero). However the stress distribution must be relevant as an inhomogeneous stress was required for any patterns to be observed and so the term "stress-optical" effect seems the most appropriate.

As the patterns bore marked similarities to the expected photo-elastic ones, they must have depended on

both the magnitudes and directions of the principal stresses at various points. This probably means that as the patterns changed during the course of deformation, the stress distribution on the basal plane also changed. This is quite reasonable, as owing to the motion of the dislocations, internal stresses must arise, which, when added to the original elastic ones, change the actual stress at any point. The instantaneous change in the pattern when the load was removed confirms that only part of the stress was elastic and its continuing change after this shows that some form of stress relaxation occurred.

If it is assumed that the birefringence at any point is related to the stress present there, it is possible to come to some conclusions about the stress-optical coefficients. (The following treatment uses the methods and techniques of Nye (1960): the σ_i are the components of the stress tensor given in matrix notation).

Referring to Fig. 24(c) and using Nye's notation, it can be seen that the stresses acting on the various planes when a compressive stress, σ_T , is applied along OZ , are given by:-

$$\begin{aligned}
 \sigma_1 &= (\cos \alpha \sin g)^2 \sigma_T \\
 \sigma_2 &= (\sin \alpha \sin g)^2 \sigma_T \\
 \sigma_3 &= \cos^2 g \sigma_T \\
 \sigma_4 &= (\sin g \cos g \sin \alpha) \sigma_T \\
 \sigma_5 &= (\sin g \cos g \cos \alpha) \sigma_T \\
 \sigma_6 &= (\sin^2 g \sin \alpha \cos \alpha) \sigma_T
 \end{aligned}
 \left. \begin{array}{l} \\ \\ \\ \\ \\ \end{array} \right\} \begin{array}{l} \text{Compressive stresses} \\ \\ \\ \text{Shear stresses} \end{array} \quad \dots (1)$$

If B_i are the components of the relative dielectric impermeability tensor, then in the case of ice (i.e. Hexagonal symmetry is assumed) the changes that occur in their values when a stress is applied, are:-

$$\begin{pmatrix} \Delta B_1 \\ \Delta B_2 \\ \Delta B_3 \\ \Delta B_4 \\ \Delta B_5 \\ \Delta B_6 \end{pmatrix} = \begin{pmatrix} \pi_{11} & \pi_{12} & \pi_{13} & . & . & . \\ \pi_{12} & \pi_{11} & \pi_{13} & . & . & . \\ \pi_{31} & \pi_{31} & \pi_{33} & . & . & . \\ . & . & . & \pi_{44} & . & . \\ . & . & . & . & \pi_{44} & . \\ . & . & . & . & . & (\pi_{11} - \pi_{12}) \end{pmatrix} \begin{pmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \sigma_4 \\ \sigma_5 \\ \sigma_6 \end{pmatrix} ;$$

Now the stress-optical effect has only been positively identified when the observer was looking along the optic axis (or very near this direction) and so the relevant parts of the impermeability tensor are ΔB_1 , ΔB_2 and ΔB_6 . These are given by:-

$$\begin{aligned}
 \Delta B_1 &= \pi_{11} \sigma_1 + \pi_{12} \sigma_2 + \pi_{13} \sigma_3 \\
 \Delta B_2 &= \pi_{12} \sigma_1 + \pi_{11} \sigma_2 + \pi_{13} \sigma_3 \\
 \Delta B_6 &= (\pi_{11} - \pi_{12}) \sigma_6
 \end{aligned} \quad \dots (2)$$

In this orientation $g \approx 90^\circ$ and so substituting (1) into (2) gives:-

$$\begin{aligned}\Delta B_1 &= (\pi_{11} \cos^2 \alpha + \pi_{12} \sin^2 \alpha) \delta_T \\ \Delta B_2 &= (\pi_{12} \cos^2 \alpha + \pi_{11} \sin^2 \alpha) \delta_T \quad \dots (3) \\ \Delta B_6 &= (\pi_{11} - \pi_{12})(\sin \alpha \cos \alpha) \delta_T\end{aligned}$$

Now initially $B_1 = B_2 = 1/n_o^2$ (where n_o is the ordinary refractive index of ice) and these have to change for the specimen to exhibit a sharp extinction position, as opposed to remaining uniformly dark as the polaroids are rotated. Thus it seems that neither π_{11} nor π_{12} can be zero - especially as isochromatics were observed.

ΔB_6 is a measure of the degree to which the extinction position rotates (i.e. it follows the isoclinics) and as it was quite large, $\pi_{11} \neq \pi_{12}$.

It is more difficult to detect changes in other directions as B_1 and B_2 are not equal to B_3 and so the crystal already exhibits a sharp extinction position. Although rotations in the extinction position should have been detectable, no unambiguous instances of it were ever observed, and so it seems reasonable to conclude that π_{44} is very small.

This conclusion could be in error as on two occasions inexplicable phenomena were seen. Thus Fig. 24(d) shows part of a single crystal which had a different extinction position to the rest of the specimen and, as it had

developed during creep, this could well be an example of an applied stressing causing a rotation in the extinction position. (N.B. In this case the optic axis lay almost in the plane of the specimen). However it could also be an example of badly distorted Interference Bands (see Section 4.5).

CHAPTER 5.CONCLUDING DISCUSSION.

The aim of this rather short Chapter is to collect and collate the main results reported in this thesis in order to decide what the next steps in the investigation should be. This discussion is intended to supplement, not duplicate, those already given in various parts of this work.

Although a great deal of time and effort was expended in perfecting the production and perfection of single crystals, there is still room for improvement. This might be achieved by replacing the nylon tubing used in the construction of the moulds with some other synthetic material that does not "lose" its softness so easily. A suitable substance does not appear to be available at present, but if in the future one should be invented, it would be possible to reduce the organic carbon content of the ice.

The gas content of the ice could be reduced by setting up the growth frame in an inert atmosphere. In this way it should be possible to eliminate gases such as carbon dioxide or ammonia and this would make the ice more suitable for electrical studies.

In principle the most satisfactory way of shaping ice is by thermal cutting. However the procedure described in this thesis will have to be refined before the specimens are "true"

enough for creep studies. This could be done with a series of guides.

It seems reasonable to conclude that, although the techniques described in Chapter 2 are the best available at present, it may be possible to improve on them later, especially with regard to the points discussed above. When this is done it may become desirable to determine the orientations of the specimens more accurately, and X-rays will probably be required.

Although many of the results on the surface features developed during deformation have been described by other workers, the relatively high magnifications used have enabled the features to be studied in greater detail than has been possible hitherto and, as a result, some interesting questions have arisen.

For example, are the finely spaced slip bands always present? This question may be answered by refining the replica technique described in this thesis - the thickness of the layer of paraffin being more carefully controlled. However, an electron microscope will be required and it may well be that Muguruma has already acquired the relevant data.

Secondly, what is the significance of the so-called "critical" stress of 200 gm wt/cm^2 ? The work described in this thesis has shown that ice is quite capable of creeping at resolved shear stresses that are smaller than this. Thus

it may well be that compressive stresses can alter the value of the critical shear stress (cf. Tamman and Salge (1928)). Alternatively, this could be the minimum stress required for the formation of the coarse slip bands and that there is an increase in the rate of deformation associated with them.

Thirdly, does slip start on all the planes at once, or consecutively? As ice does not exhibit any hardening, a metallurgist would probably argue in favour of the former (see Barrett (1952)). However as Taylor's formula is only applicable to the finely spaced slip bands, this author is of the opinion that although these may all be activated at once, the coarse slip bands develop at various stages of the deformation. This would explain their lack of uniformity.

The work described in this thesis has confirmed that two types of non-basal slip lines may form on the surface of a deforming ice crystal - cross-slip lines and polygonisation boundaries. This distinction agrees with that of Gold (1962), and the X-ray studies of Glen and Perutz (1954) provide additional evidence that polygonisation boundaries form as a result of deformation.

Gold was of the opinion that cross-slip was a very unusual occurrence. However, the results quoted in this thesis seem to show that this is not the case - a large stress being all that is required. The phenomenon could also be due to the motion of a dislocation with a non-basal

Burger's vector. If more work is done on this, it may be possible to identify the actual slip planes involved by measuring the angles between the basal and non-basal slip lines for various orientations of the specimen and so differentiate between the two possibilities. Further, if it is cross-slip, it should be possible to detect some form of work hardening.

Apart from illustrating the distortion that occurs at a grain boundary, this thesis has shown how its three dimensional shape may be studied with the aid of polarised light. It might be interesting to observe how this changes with deformation - especially in a polycrystal.

The "flow contours" seem to show that during the course of creep ice is transferred from one part of the surface to another. This may be extremely relevant in the deformation of polycrystals. However, it must be emphasised that the same optical effect could be produced by Isochromatics arising from the stress field associated with the coarse slip bands present in the dark region.

The most unexpected result reported in this thesis is the formation of Interference Bands. Their explanation raises such important issues that much more work must be done on the way their form and spacing varies with temperature, applied stress, impurities and the orientation of the specimen. In this way it may be possible to relate

their spacing to that of the coarse slip bands.

However, if it is assumed that the explanation given is correct, then the existence of active dislocations with non-basal Burger's vectors follows automatically. In this connection, it must be emphasised that although the $\underline{g}$ vector given in the analysis seems the most probable, the only essential feature is the non-basal component and that there could be dislocations with $(\underline{a} + \underline{g})$ vectors for example. The existence of active non-basal dislocations is in direct contradiction with Wakahama's model but may well be able to explain some of the obscure results obtained in creep experiments.

Although the stress-optical effect was a trifle disappointing, it does provide a means of studying the way stresses on the basal plane change during deformation and the associated relaxation phenomena.

Unfortunately the author did not have sufficient time to pursue some of the topics raised in this Chapter further, especially the questions of the active dislocations with non-basal Burger's vectors and the motion of dislocations on non-basal planes. However, in time this will no doubt be remedied and the author would like to conclude by proposing two other topics that could usefully be studied:-

- (1) The effect of impurities on the deformation of

ice especially with regard to changes in its optical properties and the distribution of etch pits. A suitable contaminant would be caesium fluoride as this definitely affects the distribution of etch pits (see Truby (1955)).

(2) The deformation of polycrystals could be studied between crossed polaroids. This could be a rather fruitful study now that the basic phenomena have been elucidated.

APPENDIX I.

RELATION BETWEEN ORTHOGONAL ANGLES.

In Fig. 11 the required angle is "g" and the two orthogonal ones are "g₁" and "g₂". It can be seen from this figure that:-

$$OZ = OA \cos g \quad \dots \quad (1)$$

or:-

$$OZ = OB \cos g_{21} = (OA \cos g_2) \cos g_{21} \dots \quad (2)$$

Thus equating (1) and (2):-

$$\underline{\cos g = \cos g_1 \cos g_2}$$

A P P E N D I X II.

REFRACTION OF THE EXTRA-ORDINARY RAY.

It can be seen from Fig. 26(a) that two wavefronts emanate from a source of light situated in ice due to its being a uniaxial substance and so, when a beam of light enters it, two plane polarised beams are formed, which have their planes of polarisation at right angles to each other. Using Huyghen's Principle, the two rays may be found by constructing the corresponding wavefronts. The one polarised at right angles to the plane of incidence corresponds to the spherical wavefront and is called the Ordinary Ray; while the other is called the Extra-Ordinary Ray.

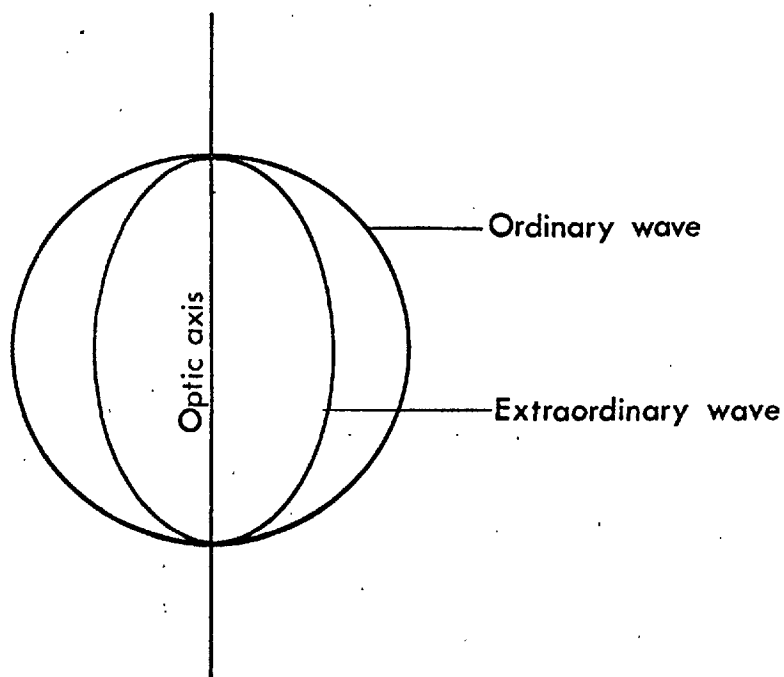
Having located the two rays, the wavefronts of the two refracted beams can be found by constructing the two planes which touch the wave surfaces where they are intersected by the rays. Now, though the Extra-Ordinary Ray will not normally lie in the Plane of Incidence, both the normals to the wavefronts always do (see Flügge) and so referring to Fig. 26(b):-

If radius of ordinary wavefront is "a" then

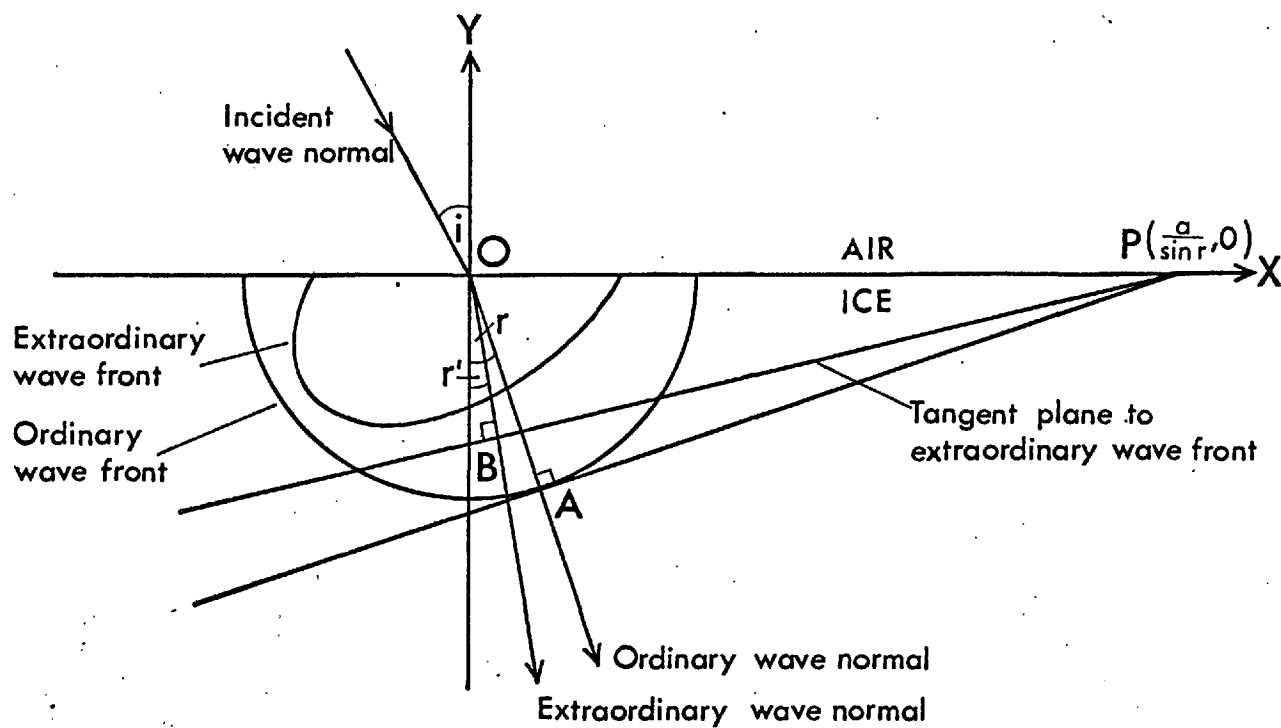
$$OB = p = \left(\frac{a}{\sin r} \right) \cdot \sin r' \quad \text{and so the equation of the tangent plane to the extra-ordinary wavefront is:-}$$

Fig. 26

(a) Wave surfaces in ice shown in two dimensions



(b) Refraction of light at an air - ice interface



$$\sin r'x - \cos r'y = p = \frac{a \sin r}{\sin r}$$

Or:-

$$\frac{\sin r}{a} x - \frac{\sin r}{a \tan r'} y = 1 \quad \dots \quad (1)$$

Now the equation of the extra-ordinary wavefront is:-

$$\frac{x'^2}{b^2} + \frac{y'^2}{b^2} + \frac{z'^2}{a^2} = 1 \quad \dots \quad (5)$$

when referred to the axes (OX', OY', OZ') and these are related to the former axes by:-

	x'	y'	z'
x	a_{11}	a_{12}	a_{13}
y	a_{21}	a_{22}	a_{23}
z	a_{31}	a_{32}	a_{33}

In this the "a's" represent the cosines of the angles between the axes, and so (1) may be transformed to:-

$$\left(\frac{a_{11} \sin r}{a} - \frac{a_{21} \sin r}{a \tan r'} \right) x' + \left(\frac{a_{12} \sin r}{a} - \frac{a_{22} \sin r}{a \tan r'} \right) y' + \left(\frac{a_{13} \sin r}{a} - \frac{a_{23} \sin r}{a \tan r'} \right) z' = 1 \quad \dots \quad (2)$$

Let the extra-ordinary ray cut the extra-ordinary wavefront at the point (l,m,n). The equation of the tangent plane at this point is:-

$$\frac{lx'}{b^2} + \frac{my'}{b^2} + \frac{nz'}{a^2} = 1 \quad \dots \quad (3)$$

However this is the same plane as that represented by (2) and so comparing them:-

$$\begin{aligned} l &= (a_{11} - a_{21} \cot r') \frac{b^2}{a} \sin r \\ m &= (a_{12} - a_{32} \cot r') \frac{b^2}{a} \sin r \quad \dots \quad (4) \\ n &= (a_{13} - a_{23} \cot r') \frac{a^2}{a} \sin r \end{aligned}$$

But the point (l,m,n) lies on the surface of the extra-ordinary wavefront and so putting (4) into (5):-

$$\begin{aligned} b^2(a_{11} - a_{21} \cot r')^2 + b^2(a_{12} - a_{32} \cot r')^2 \\ + a^2(a_{13} - a_{23} \cot r')^2 = a^2 \operatorname{Cosec}^2 r \quad \dots \quad (6) \end{aligned}$$

Or letting $\left(\frac{a}{b}\right)^2 = 1 + d$ and using the relationships that exist between the "a's" (see Nye (196)) this becomes:-

$$\begin{aligned} 1 + \cot^2 r' + d(a_{13} - a_{23} \cot r')^2 \\ = (1 + d) \operatorname{Cosec}^2 r \quad \dots \quad (7) \end{aligned}$$

Or:-

$$\begin{aligned} (1 + a_{23}^2 d) \cot^2 r' - 2d a_{13} a_{23} \cot r' \\ + (1 + d a_{13}^2 - (1 + d) \operatorname{Cosec}^2 r) = 0 \quad \dots \quad (8) \end{aligned}$$

But in the case of ice $d = 1.68 \times 10^{-5} \approx 2 \times 10^{-5}$ and so as the maximum values of a_{23} and a_{13} are unity, (8) becomes:-

$$\cot^2 r' - 2d a_{13} a_{23} \cot r' - \cot^2 r = 0 \quad \dots \quad (9)$$

Solving:-

$$\cot r' = a_{13} a_{23} d \pm \sqrt{(a_{13} a_{23})^2 d^2 + \cot^2 r} \dots (10)$$

Now the maximum value of r occurs when the incident beam just grazes the surface and in this case:-

$$n_o = \frac{\sin 90^\circ}{\sin r_{\max.}} \quad (n_o = \text{refractive index of ordinary ray})$$

Therefore:-

$$r_{\max.} = 50\frac{1}{2}^\circ$$

Thus (10) reduces to $r' = r$ as $\cot r_{\max.} \gg 10^{-3}$ and so it may be concluded that the ordinary and extraordinary waves follow the same path through blocks of ice.

APPENDIX III.THE INTERACTION OF POLARISED LIGHT WITH UNI-AXIAL CRYSTALS.A. Method of Analysis.

By using the conventional method of representing light waves by trigonometric functions, it is possible to calculate the output from various optical elements such as polarisers or linear retarders. However, except for the most simple systems, this method is extremely laborious and the answer almost impossible to interpret physically. Fortunately, more refined techniques have been developed which do not suffer from these shortcomings.

Of these modern methods, the one known as Jones Calculus was the most suitable for investigating the present problems. In it the incident light is described by a vector, the optical device by a matrix and the output is obtained by multiplying the vector by the matrix. If the optical device consists of various elements, the resultant matrix may be computed by multiplying the matrices of the individual optical components together in the order in which the light passes through them.

This method is discussed quite fully in Shurcliff (1962) and so no further introduction will be given here, as it is felt that it is adequately exemplified by the analyses that follow.

B. Thickness Contours.

To explain the phenomenon referred to in Section 4.3 as "Thickness Contours" it is necessary to consider a block of ice of thickness "t" situated between two crossed polaroids. If the X-axis is taken to be parallel to the transmission axis of the Polariser, then the Jones Vector of the plane polarised light entering the crystal is represented by:-

$$\begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

Thus, assuming that the ice acts as a homogeneous linear retarder of retardance "d" and that its fast axis makes an angle Θ with the X-axis, the Jones Vector of the output from the system is given by:-

$$JV)_{out} = \begin{bmatrix} 0 & 0 \\ 0 & 1 \end{bmatrix} \begin{bmatrix} (\cos^2 \Theta e^{id/2} + \sin^2 \Theta e^{-id/2}) & (i \sin 2\Theta \sin d/2) \\ (i \sin 2\Theta \sin d/2) & (\cos^2 \Theta e^{-id/2} + \sin^2 \Theta e^{id/2}) \end{bmatrix} \begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

(Analyser) (Ice) (Input)

If these are multiplied out, it will be found that:-

$$JV)_{out} = \begin{bmatrix} 0 \\ i \sin 2\Theta \sin d/2 \end{bmatrix} \quad \dots \quad (1)$$

Now the intensity of the light represented by a Jones Vector is equal to the sum of the squares of its two

components (using complex conjugates if necessary) and so from (1):-

$$I_{\text{out}} = 0 + \sin^2 2\Theta \sin^2 d/2 = 4 \sin^2 \Theta \cos^2 \Theta \sin^2 d/2 \dots (2)$$

This is zero when $\Theta = 0$ or $\pi/2$ and, of course, corresponds to the transmission axes of the polaroids being parallel to the transmission axes of the ice specimen. However it is also zero when $d/2 = m\pi$ (where "m" is an integer) and so as:-

$$d = \frac{2\pi t}{\lambda} (n' - n_0) \quad \text{(where } n' \text{ and } n_0 \text{ are the principal refractive indices of the specimen and } \lambda \text{ is the wavelength of the incident light)}$$

this gives:-

$$t = \frac{m\lambda}{n' - n_0} \dots (3)$$

If this relation is satisfied by any part of the specimen, then that part will remain dark even if the polaroids are rotated, because (3) does not depend on Θ .

C. Grain Boundaries.

A grain boundary which exhibits interference bands consists of two wedges of ice placed one behind the other and having different extinction positions. If the angle between the two fast axes is ρ then the matrices required to represent the system consisting of these two wedges placed between crossed polaroids will be:-

$$JV)_{in} = \begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

$$\text{First Wedge} = \begin{bmatrix} (\cos^2 \Theta e^{id_1} + \sin^2 \Theta e^{-id_1}) & i \sin 2\Theta \sin d_1 \\ i \sin 2\Theta \sin d_1 & (\cos^2 \Theta e^{-id_1} + \sin^2 \Theta e^{id_1}) \end{bmatrix}$$

$$= W_1$$

$$\text{Second Wedge} = \begin{bmatrix} (\cos^2 (\Theta + \rho) e^{id_2} + \sin^2 (\Theta + \rho) e^{-id_2}) & i \sin 2(\Theta + \rho) \sin d_2 \\ i \sin 2(\Theta + \rho) \sin d_2 & (\cos^2 (\Theta + \rho) e^{-id_2} + \sin^2 (\Theta + \rho) e^{id_2}) \end{bmatrix}$$

$$W_2$$

$$\text{Analyser} = \begin{bmatrix} 0 & 0 \\ 0 & 1 \end{bmatrix} = A$$

These matrices follow from what was said in Appendix IIIB provided that it is remembered that the two wedges have different thickness, t_1 and t_2 and so the retardances will in general be different. Furthermore if the fast axis of the first wedge makes an angle Θ with the analyser,

that of the second will make an angle of $(\Theta + \rho)$.

Thus the Jones Vector of the output is given by:-

$$JV)_{out} = A W_a W_1 \begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

Or:-

$$JV)_{out} = \begin{bmatrix} 0 \\ -\sin \frac{d_2}{2} \sin \frac{d_1}{2} \sin 2\rho \\ + 1(\sin \frac{d_2}{2} \cos \frac{d_1}{2} \sin 2(\Theta + \rho) + \cos \frac{d_2}{2} \sin \frac{d_1}{2} \sin 2\phi) \end{bmatrix}$$

Thus the output intensity is given by:-

$$I_{out} = \sin^2 2\rho \sin^2 \frac{d_2}{2} \sin^2 \frac{d_1}{2} \dots (1) \\ + (\sin \frac{d_2}{2} \cos \frac{d_1}{2} \sin 2(\Theta + \rho) + \cos \frac{d_2}{2} \sin \frac{d_1}{2} \sin 2\phi)^2$$

This is a rather complex expression and can be most easily compared with the experimental results by taking two special cases:-

1. The first wedge is blacked out.

In this case $\Theta = 0$ and if the second wedge was not present there would be no output. If this is substituted into (1) it becomes:-

$$I_{out} \Big|_{\Theta=0} = \sin^2 2\rho \sin^2 \frac{d_2}{2} \dots (2)$$

This is zero when either 2ρ or $\frac{d_2}{2}$ is equal to $m\lambda$ (where "m" is an integer). The first case is trivial as it means that there is no difference in the extinction positions of the two grains which make up the grain boundary and so it probably would not be detected. With the second case however:-

$$d_2 = m \frac{\lambda}{2} = \frac{2\pi t_2}{\lambda} (n'_2 - n_0) \quad \dots \quad (3)$$

$$t_2 = \frac{m \lambda}{(n'_2 - n_0)}$$

As t_2 will vary along the wedge, this means that a series of dark bands will be seen. Furthermore if the wedge has an angle of "h", the distance between adjacent bands will be:-

$$S_2 = \frac{\lambda \cot h}{(n'_2 - n_0)} \quad \dots \quad (4)$$

2. The second wedge is blacked out.

In this case $\Theta + \rho = 0$ and the results are the same as in section 1. except that the suffix "2" is replaced by a "1". Thus the distance between adjacent bands will be:-

$$S_1 = \frac{\lambda \cot h}{(n'_1 - n_o)} \dots \quad (5)$$

Thus in conclusion it may be stated that the interference bands seen at a grain boundary will usually move back and forth as the polaroids are rotated (see (1)) and their spacing may also vary (compare (4) with (5)).

D. Interference Bands.

Part I.

In previous sections of this Appendix some relatively simple optical systems were analysed in order to illustrate the use of Jones' Calculus. However, both the examples given could have been treated by conventional methods, though the results would have been rather unwieldy in the second case. In this section a system is considered which could not be analysed in this fashion, and even with the use of modern techniques, the mathematics is far from elementary. Thus the actual manipulation has been kept to a minimum so as not to obscure either the method or results.

This explanation of the Interference Bands is based on the premise that the single crystal breaks up into a series of plates which have slightly different extinction positions. In general this is an extremely complex system, because of the many permutations and combinations that are possible, and so the special case of a pile of $(n + 1)$ plates with alternate disorientations of $\pm \rho$ (i.e. The difference in the extinction positions of successive plates was either $+\rho$ or $-\rho$) was chosen. The plates are situated between crossed polaroids and are identical, apart from the first and last ones. Thus their retardances are:-

$d_0, d, d, d, \dots, d, d_n.$

The Jones' Matrix of a block of single crystalline ice may be written in an alternative form to that used previously:-

$$R = \begin{bmatrix} (\cos^2 \Theta e^{id_n} + \sin^2 \Theta e^{-id_n}) & i \sin 2\Theta \sin \frac{d_n}{2} \\ i \sin 2\Theta \sin \frac{d_n}{2} & (\cos^2 \Theta e^{-id_n} + \sin^2 \Theta e^{id_n}) \end{bmatrix}$$

$$= S(-\Theta) P_n S(\Theta).$$

$$(\text{where } P = \begin{bmatrix} e^{id_n} & 0 \\ 0 & e^{-id_n} \end{bmatrix})$$

$$\text{and } S(\Theta) = \begin{bmatrix} \cos \Theta & \sin \Theta \\ -\sin \Theta & \cos \Theta \end{bmatrix}$$

This form is more convenient for this particular analysis as the $S(\Theta)$ Matrices have an important property:-

$$S(\Theta_1) \times S(\Theta_2) = S(\Theta_1 + \Theta_2) \quad \dots \quad (1)$$

Thus if $(n+1)$ is an odd number and the first rotation is $+\rho$, the Jones' Vector of the output from the system may be written as:-

$$JV)_{out} = \begin{bmatrix} 0 & 0 \\ 0 & 1 \end{bmatrix} \begin{pmatrix} (S(-\Theta)P_n S(\Theta))(S(-\Theta-\rho)PS(\Theta+\rho)) \dots \\ \dots (S(-\Theta)P_0 S(\Theta)) \end{pmatrix} \begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

$$(\text{where } P = \begin{bmatrix} e^{id/a} & 0 \\ 0 & e^{-id/a} \end{bmatrix} \text{ and } P_0 = \begin{bmatrix} e^{id_0/a} & 0 \\ 0 & e^{-id_0/a} \end{bmatrix})$$

Or using (1):-

$$JV)_{out} = \begin{bmatrix} 0 & 0 \\ 0 & 1 \end{bmatrix} S(-\Theta) P_n S(-\rho) PS(\rho) [PS(-\rho)PS(\rho)]^{\frac{n-2}{2}} P_0 S(\Theta) \begin{bmatrix} 1 \\ 0 \end{bmatrix}$$

The term $|PS(-\rho)PS(\rho)|^{\frac{n-2}{2}}$ may be evaluated by diagonalising

the matrix as is shown in Jones (1941). Furthermore as ρ is a small quantity, terms in ρ^2 are negligible and so, as the real part of $JV)_{out}$ is multiplied by ρ it does not contribute to the output intensity which becomes:-

$$I_{out} = \frac{\sin \Delta}{\Delta} \sin 2\Theta \left(\frac{\sin \Delta}{\Delta} \sin 2\Theta + \frac{2\rho \sin nd/a \cos(d_n - d_0/a) \cos 2\Theta}{\cos d/a} \right) \dots (2)$$

(where Δ = Total retardance of the pile of plates).

This is zero when:-

$$\Theta_e = m\pi/a \quad - \text{Extinction position}$$

$$\Delta = 2m\pi \quad - \text{Thickness contour} \quad \dots$$

(3)

$$\tan 2\Theta_e = -2\rho \left(\frac{\sin nd/a}{\cos d/a} \right) \cdot \left(\frac{\cos d_n - d_0/a}{\sin \Delta/a} \right)$$

(where "m" is an integer).

If $(n + 1)$ is an even number the corresponding result is:-

$$I_{out} = \frac{\sin \Delta}{2} \sin 2\Theta \left(\frac{\sin \Delta}{2} \sin 2\Theta + 2 \left(\frac{\sin \Delta}{2} + \frac{\cos \frac{\Delta}{2} \sin(d_1 - d_0)}{\cos \frac{\Delta}{2}} \right) \cos 2\Theta \right) \dots (4.)$$

and this is zero when:-

$$\Theta_e = m\pi/2 \quad - \text{Extinction position}$$

$$\Delta = 2m\pi \quad - \text{Thickness contour}$$

$$\tan 2\Theta_e = -2\rho \left(1 + \frac{\cos \frac{\Delta}{2}}{\cos \frac{\Delta}{2}} \cdot \frac{\sin(d_1 - d_0)}{\sin \frac{\Delta}{2}} \right) \dots (5)$$

Part II.

Having derived expressions for the output intensity, the next step is to use them to predict how the appearance of a block of ice will change as the crossed polaroids are rotated.

Fig. 27(a) shows part of a block of ice that has a mean extinction position of $\Theta = m\pi/2$. It consists of a pile of "N" individual plates that are disorientated with respect to each other by $\pm\rho$ and have retardances of $d_1, d, \dots, d_N$. If all these are equal except for the first and last ones (i.e. d_1 and d_N) then the basic relations given in Table A.1 will hold. Furthermore, if "N" is assumed to be an ^{odd}~~even~~ number, then the variation of the output intensity between "A" and "E" (as a function of

Fig. 27

(a) Ice model for the production of interference bands

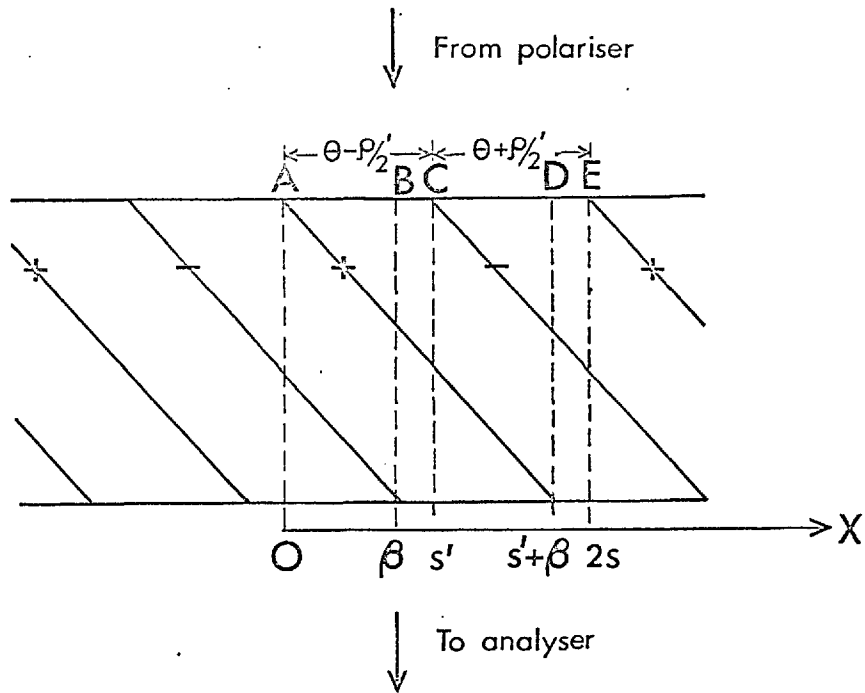
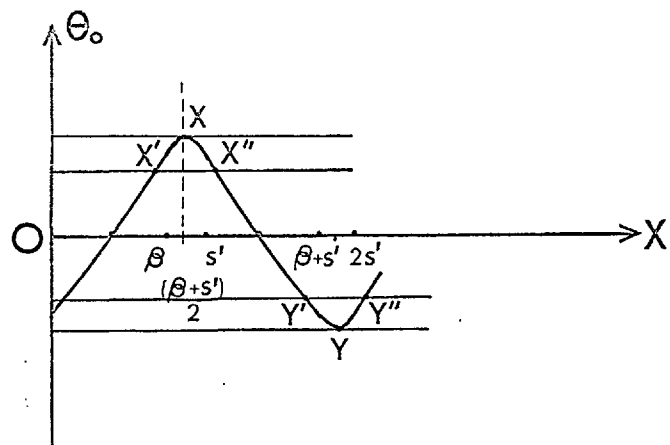
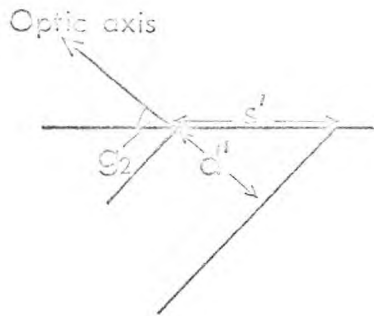
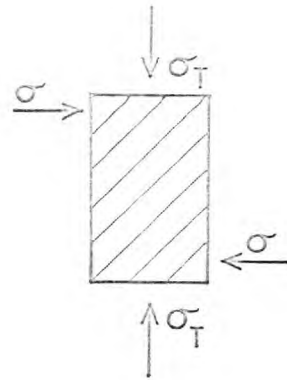
(b) Variation of extinction position with x 

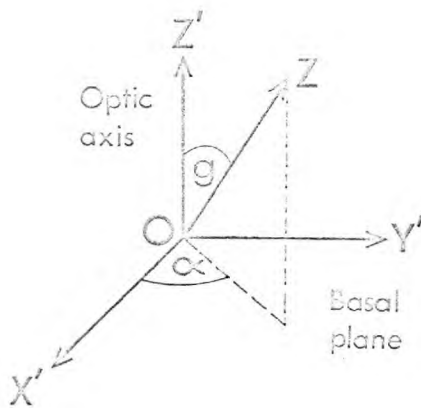
Fig. 24

(a) Dependence of s' on g_2 

(b) Stress systems acting during creep



(c) Relation between axis of compression and optic axis



(d) An unusual effect

 $\frac{1}{2}$ mm.


Θ , ρ , Δ , d and x) may be calculated by substituting the data given in Table A.1 into the appropriate formula given in Part I of this Appendix. The various Intensity functions are collected in Table A.2 and it can be seen that they give a continuous intensity distribution function. Apart from the usual zero's of intensity (i.e. Extinction Positions and Thickness Contours), there are a whole series of Θ_0 values. These are given in Table A.3 and the way these values vary with "x" is shown schematically in Fig. 27(b).

TABLE A.1 - Basic Data.

Region	Orient- ation of 1st plate	First change in Orient- ation	No. of plates	d_i	d_N	$d_N - d_i$	$d_N + d_i$
A(0) to B(p)	$\theta - p/2$	$+p$	$N + 1$	$\frac{dx}{s'}$	$\frac{d(p-x)}{s'}$	$\frac{d(p-2x)}{s'}$	$\frac{dp}{s'}$
B(p) to C(s')	$\theta - p/2$	$+p$	N	$\frac{dx}{s'}$	$\frac{d(p-(x-s'))}{s'}$	$\frac{d(p+s'-2x)}{s'}$	$\frac{dp + d}{s'}$
C(s') to D(s'+p)	$\theta + p/2$	$-p$	$N + 1$	$\frac{d(x-s')}{s'}$	$\frac{d(p-(x-s'))}{s'}$	$\frac{d(p-2(x-s'))}{s'}$	$\frac{dp}{s'}$
D(s'+p) to E(2s')	$\theta + p/2$	$-p$	N	$\frac{d(x-s')}{s'}$	$\frac{d(p+s' - (x-s'))}{s'}$	$\frac{d(p+s' - 2(x-s'))}{s'}$	$\frac{dp + d}{s'}$

TABLE A.2 - The Intensities.

Region	Intensity
A to B	$I_1 = \frac{\sin \Delta}{2} \sin 2\theta \left[\frac{\sin \Delta}{2} \sin 2\theta + 2\rho \frac{\cos N \frac{d}{2}}{\cos d/2} \sin \left(\frac{d\rho}{2S'} - \frac{dx}{S'} \right) \cos 2\theta \right]$
B to C	$I_2 = \frac{\sin \Delta}{2} \sin 2\theta \left[\frac{\sin \Delta}{2} \sin 2\theta - 2\rho \left(\frac{\sin \Delta}{2} - \frac{\sin(N-1)\frac{d}{2}}{\cos d/2} \cos \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + \frac{d}{2} \right) \right) \cos 2\theta \right]$
C to D	$I_3 = \frac{\sin \Delta}{2} \sin 2\theta \left[\frac{\sin \Delta}{2} \sin 2\theta - 2\rho \frac{\cos N \frac{d}{2}}{\cos d/2} \sin \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + d \right) \cos 2\theta \right]$
D to E	$I_4 = \frac{\sin \Delta}{2} \sin 2\theta \left[\frac{\sin \Delta}{2} \sin 2\theta + 2\rho \left(\frac{\sin \Delta}{2} - \frac{\sin(N-1)\frac{d}{2}}{\cos d/2} \cos \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + \frac{3d}{2} \right) \right) \cos 2\theta \right]$

TABLE A.3 - The Values of Θ_0 .

Intensity	Values of Θ_0
I_1	$\tan 2\Theta_0 = \frac{-2\rho \cos N \frac{d}{2}}{\cos d/2} \times \frac{\sin \left(\frac{d\rho}{2S'} - \frac{dx}{S'} \right)}{\sin \Delta/2}$
I_2	$\tan 2\Theta_0 = \frac{+2\rho \left[1 - \sin(N-1) \frac{d}{2} \cos \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + \frac{d}{2} \right) \right]}{\cos d/2 \sin \Delta/2}$
I_3	$\tan 2\Theta_0 = \frac{+2\rho \cos N \frac{d}{2} \sin \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + d \right)}{\cos d/2 \sin \Delta/2}$
I_4	$\tan 2\Theta_0 = \frac{-2\rho \left[1 - \sin(N-1) \frac{d}{2} \cos \left(\frac{d\rho}{2S'} - \frac{dx}{S'} + \frac{3d}{2} \right) \right]}{\cos d/2 \sin \Delta/2}$

A P P E N D I X IV.

DISLOCATION MODELS FOR GRAIN BOUNDARIES.

A. General Result.

The arrangements of dislocations, which are necessary to give any particular grain boundary, will depend on the orientation of its plane and also on the relative orientations of the two grains. A method of determining the most suitable dislocation model is given in Read (1953), and as it was decided to use this technique, an outline of it will be given here.

Let the normal to the plane of the boundary be $\underline{n}$ where:-

$$\underline{n} = n_1 \underline{i} + n_2 \underline{j} + n_3 \underline{k} \quad \dots \quad (1)$$

In this expression $\underline{i}$, $\underline{j}$ and $\underline{k}$ are unit vectors along the three Cartesian axes and n_1 , n_2 and n_3 are integers.

Then any vector $\underline{V}$ lying in the plane of this boundary must satisfy the following condition:-

$$\underline{V}_1 n_1 + \underline{V}_2 n_2 + \underline{V}_3 n_3 = 0 \quad \dots \quad (2)$$

since the vector dot product must be zero. By using this relation, two vectors $\underline{V}$, which lie in this plane and are perpendicular to each other, may be chosen. Then if $\underline{u}$ is a unit vector parallel to the axis of relative rotation of the two grains and "r" is the angle of rotation, it is shown in Read (1953) that the sum of the Burgers vectors, $\underline{S}$, cut

by $\underline{V}$ is:-

$$\underline{S} = (\underline{u} \times \underline{V})r$$

or if $\underline{W} = \underline{ur}$:-

$$\underline{S} = (\underline{W} \times \underline{V}) \quad \dots \quad (3)$$

By using this expression an appropriate model may be chosen, provided it is remembered that only two choices of $\underline{V}$ are necessary to find the possible models, and if more than one is possible, the arrangement with the lowest energy is the one required. A further point that should be borne in mind is that if for any $\underline{V}$, $\underline{S} = 0$, then $\underline{V}$ is parallel to dislocations making up the boundary.

B. The dislocation array necessary to explain the Interference Bands.

In explaining the interference bands, the formation of very small angled grain boundaries at the active basal glide planes was postulated. Thus if the coordinate system shown in Fig. 28(a) is used $\underline{n} = [0,0,1]$. Furthermore in traversing such a boundary g_1 must change by Δg_1 and as this rotation occurs about ON:-

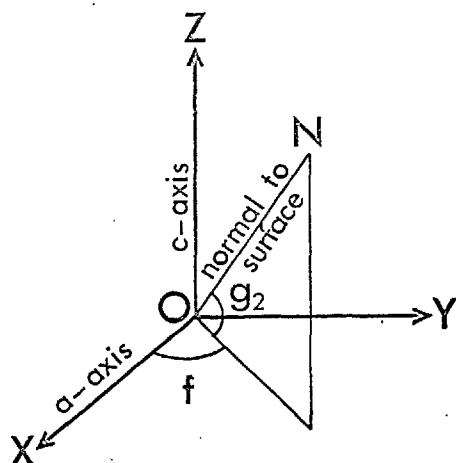
$$\underline{W} = [\cos g_2 \cos f, \sin f \cos g_2, \sin g_2] \Delta g_1 \dots \quad (4)$$

(See Appendix I).

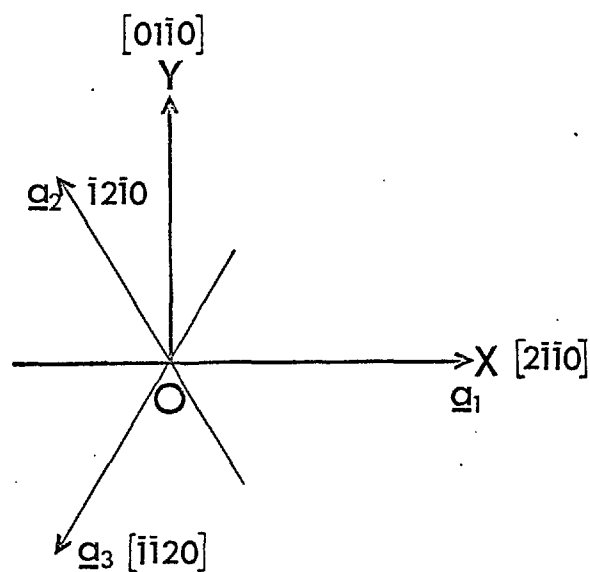
In choosing the "V's" relation (2) must be satisfied and so $V_3 = 0$. Thus (3) becomes:-

Fig 28

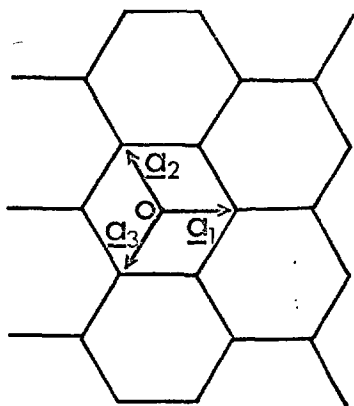
(a) Rectangular coordinate system



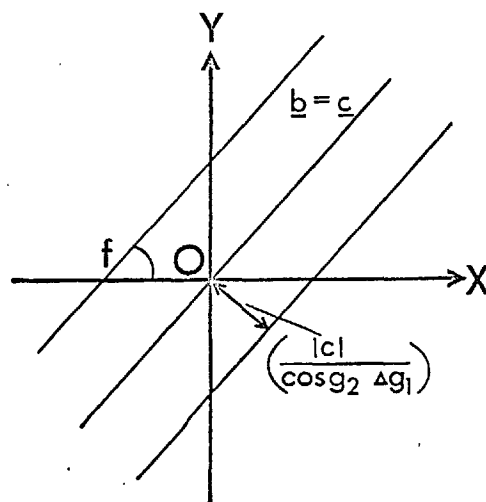
(b) Hexagonal coordinate system



(c) Dislocation array for twist component



(d) Dislocation array for tilt component



$$\underline{S} = \begin{pmatrix} \underline{l} & \underline{j} & \underline{k} \\ w_1 & w_2 & w_3 \\ v_1 & v_2 & 0 \end{pmatrix}$$

$$= \underline{l} (-v_2 w_3) + \underline{j} (v_1 w_3) + \underline{k} (v_2 w_1 - v_1 w_2) \dots (5)$$

If the directions of $\underline{V}$ are parallel to the X and Y axes, the corresponding values of $\underline{S}$ are those given in Table A.4. These results may be expressed in the hexagonal system of coordinates (See Barrett (1952)) - in which case Table A.5 is obtained. It can be seen that the $\underline{S}$ vectors have been divided into Tilt and Twist components. The latter result from dislocations whose Burgers vectors lie in the plane of the boundary and must therefore be $\underline{g}$ vectors. If the arrangement of screw dislocations shown in Fig. 28(c) is considered, it will be found to give the required twist components of $\underline{S}$.

In Fig. 28(d) an arrangement of edge dislocations with $\underline{g}$ Burgers vectors is shown which will give the tilt components of $\underline{S}$. It should be noted that in this case the Burgers vectors are perpendicular to the boundary and so the dislocations are non-basal. Another important point is that they are parallel to the projection of the normal to the surface of the specimen onto the basal plane.

TABLE A.4 - Corresponding values of V and S -
Rectangular Coordinates.

<u>V</u>	<u>S</u>
[100]	$w_{3j} = w_{2k}$
[010]	$-w_{3i} + w_{1k}$

TABLE A.5 - Corresponding values of V and S -
Hexagonal Coordinates.

<u>V</u>	<u>S</u>	
	Twist	Tilt
$[2\bar{1}10]$	$+\frac{\sin g_2 \Delta g_1}{\sqrt{3} a } (a_2 - a_3)$	$-\frac{\sin f \cos g_2 \Delta g_1}{ c } c$
$[01\bar{1}0]$	$-\frac{\sin g_2 \Delta g_1}{ a } a_1$	$+\frac{\cos f \cos g_2 \Delta g_1}{ c } c$

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