The Deformation of Ice Single Crystals at Low Temperatures

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Abstract

Creep tests in uniaxial tension have been performed on single crystals of ice at temperatures down to −70°C.

At −50°C continually increasing creep is observed in which creep strain ε varies with time t according to the relation

$$\varepsilon \propto t^m,$$

where m has the value 1.5 ± 0.2 for strains between 0.2 and 2% but increases for larger strains. The strain-rate $\dot{\varepsilon}$ at a given strain varies with stress τ as

$$\dot{\varepsilon} \propto \tau^n,$$

where the value of n is 4 ± 1. Creep strains of up to 5% have been obtained at −60°C. The temperature dependence of the strain-rate is discussed.

At all these temperatures deformation occurs by slip on the basal (0001) plane, sometimes with large offsets on a single slip band. The change of orientation of the tensile axis supports the view that $\langle 11\bar{2}0 \rangle$ is the slip direction.

I. Introduction

A number of people have investigated the mechanical properties of single crystal ice at temperatures close to its melting point, for example Glen and Perutz (1954), Steinemann (1954), Butkovich and Landauer (1958), Griggs and Coles (1954) and Jellinek and Brill (1956). Two groups of workers, Readey and Kingery (1964) and Higashi and others (1965) have made similar studies at various temperatures down to −42°C. One of the objects of the present work was to continue these investigations to even lower temperatures and this paper describes creep tests on single crystals of ice between −50 and −70°C. The shape of the creep curves is discussed and an expression giving the stress dependence of the creep rate is derived.

The mode of deformation at these temperatures is still found to be slip on the basal (0001) plane in the $\langle 11\bar{2}0 \rangle$ direction.

Data concerning the stress at which the crystals fractured are also presented and show that the fracture stress increases toward lower temperatures.

II. Experimental Procedure

II. 1. *Preparation of the ice crystals.* The single crystals used in this work were prepared in the same manner as that used by Glen and Perutz (1954). This method provides cylindrical crystals, several millimetres in diameter with any desired orientation of the c-axis.

Distilled water was boiled to remove air, cooled under reduced pressure, and then allowed to freeze in a bowl. The ice first forms as flat dendrites on the surface. The space between them fills in to give a flat plate of ice consisting of many crystals each with its optic axis perpendicular to the surface, and then these grow downwards to form columnar crystals. If a glass tube has been inserted in the water and held at an angle, some of the columnar crystals enter the top, and one grows down its length. Thus the ice in such a tube usually consists of a single crystal with its optic axis perpendicular to the surface of the water. By tilting the tube cylindrical crystals with any desired angle between cylinder and optic axes can be produced.

This simple method is shown in Fig. 1; it has proved very successful, yielding clear single crystals of the expected orientation. They can be removed by cutting or melting the ice to extract the tubes which are then gently warmed until the crystal slides out. Eight glass tubes were used in any one bowl of water, producing at most eight single crystals. A particular crystal is referred to in this paper by a roman number and a letter *e.g.* VII C. The number (VII) denotes the batch of crystals grown at any one time, and the letter (C) specifies a particular crystal.

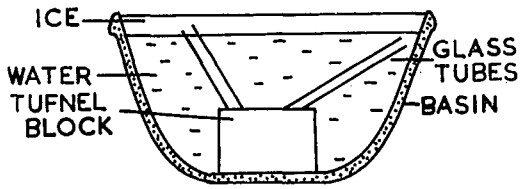


Fig. 1. Method of growing the ice single crystals

Before testing, the orientation of the crystals was checked either by using a polarized light technique or by taking an X-ray Laue picture.

In order to test these crystals, their ends were frozen into small cups made by turning down brass nuts and soldering eyelets into their ends. These cups were filled with ice-cold water, and the ends of the crystals were inserted; the surrounding water was then allowed to freeze into the threads of the cups.

II. 2. *The tensile testing machine.* The essential parts of this apparatus are shown diagrammatically in Fig. 2. To prevent evaporation, the ice crystal, A, was surrounded by toluene contained in a copper cell, B. The cell was surrounded by polyurethane foam insulation,

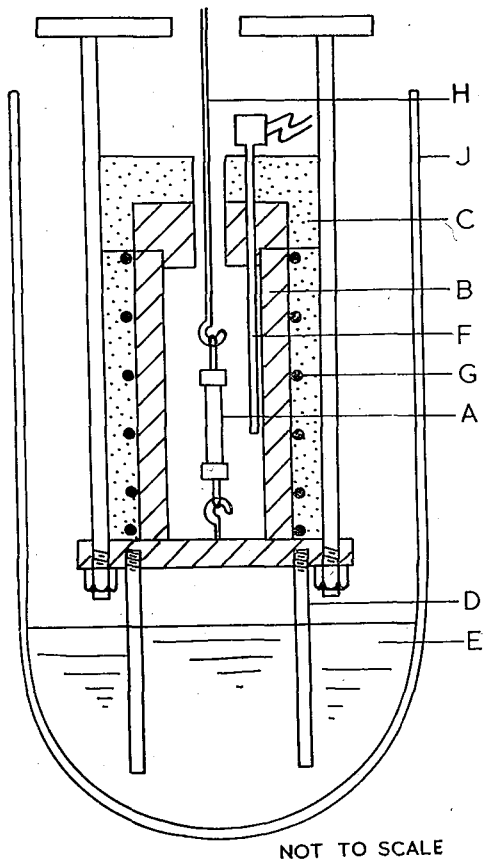


Fig. 2. Schematic diagram of the tensile testing machine

C. The stainless steel rods, D, attached to the base of the cell conducted heat to the liquid nitrogen, E, contained in the vacuum flask, J, thus cooling the cell. In order to maintain the temperature constant, a platinum resistance thermometer, F, was used in conjunction with a "Fielden" temperature-indicator/controller. The effect of this apparatus was to pass a current through the heating coil, G, when the temperature fell below the control point. This proved very satisfactory, the mean temperature of the crystal being maintained within ±0.05°C of the control point, with a temperature gradient along the cell of 0.1°C per cm.

One end of a steel wire was attached to the tensioning rod, H. The other end was passed over a pulley and attached to a pan containing the deadweight load. The tests were therefore creep tests under constant load rather than constant resolved shear stress. However, only small strains were measured during the experiment, usually up to about 2% and so the resolved shear stress remained essentially constant.

The elongation of the specimen was measured with an optical lever arrangement. A thin wire was attached to the main loading wire, passed around a pulley and then back to the main wire via a small tensioning spring. Situated on the axle of the pulley was a small mirror which reflected a beam of light onto a scale about two metres away. As the crystal extended and the wire moved upwards, the mirror rotated. The magnification, M, afforded by such an arrangement is given by

$$M = 2L/r \cos \phi \,, \tag{1}$$

where L is the distance of the scale from the mirror,
r is the radius of the pulley,
and ϕ is the angle between the main wire and the connecting wire.

This arrangement gave a magnification of 1 340. That is, an increase in length of the specimen of 1 mm gave a movement of the reflected beam of light on the scale of 1 340 mm.

III. Experimental Results

III. 1. *Tests at −50°C.* A typical creep curve obtained at −50°C is shown in Fig. 3 in which the shear strain resolved on the basal plane is plotted as a function of time. It is of the same form as that found at higher temperatures, namely a continually increasing creep rate is observed with no apparent strain hardening. The shape of the curves was found by plotting the logarithm of the strain ε against the logarithm of the time t as is done in Fig. 4. In all cases the result was a straight line showing that

$$\varepsilon \propto t^m \,, \tag{2}$$

where $m = 1.5 \pm 0.2$ for strains between 0.2 and 2%. Below 0.2% measurements of the strain were unreliable. Above 2% the value of m increased, and for crystal XV A, m equalled 2.2 between strains of 5 and 15%.

Since $\varepsilon \propto t^{1.5}$, it follows that $\dot{\varepsilon} \propto t^{0.5}$ and hence $\dot{\varepsilon} \propto \varepsilon^{1/3}$. This being so, $\dot{\varepsilon}/\varepsilon^{1/3}$ should be a constant for each crystal, the value of which depends on the resolved shear stress acting on the crystal. This was found to be the case and a graph of log $(\dot{\varepsilon}/\varepsilon^{1/3})$ against

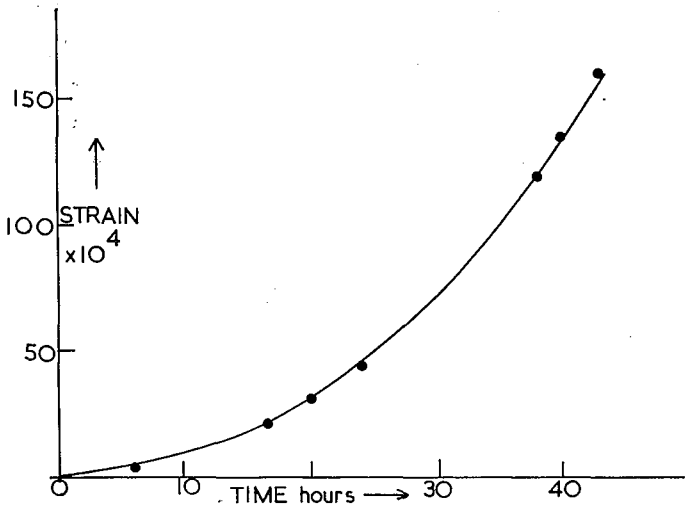


Fig. 3. Typical creep curve obtained at −50°C. The strain plotted is the maximum shear component of the strain tensor acting in the basal plane

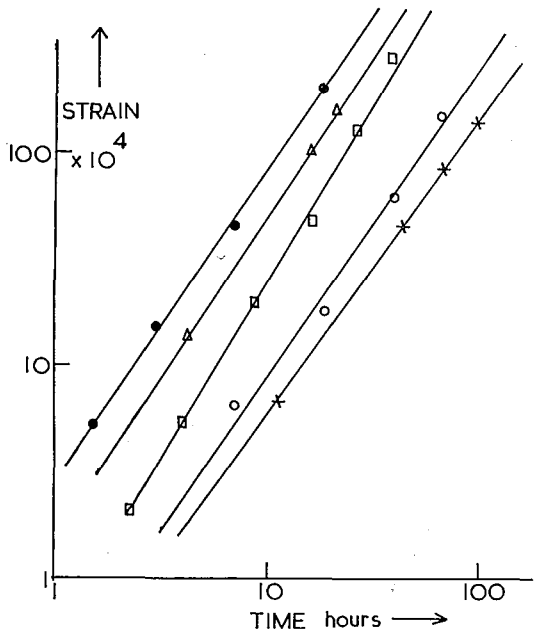


Fig. 4. *Creep curves for different crystals deformed at* −50°C plotted logarithmically

log τ was drawn as shown in Fig. 5. This was found to be approximately a straight line of slope n showing that

$$\left(\frac{\dot{\varepsilon}}{\varepsilon^{1/3}}\right)=\left(\frac{\tau}{\tau_0}\right)^n, \tag{3}$$

with $n=4\pm1$. The constant of proportionality, τ_0, was computed and found to be 18.6 bar·hour$^{1/4}$.

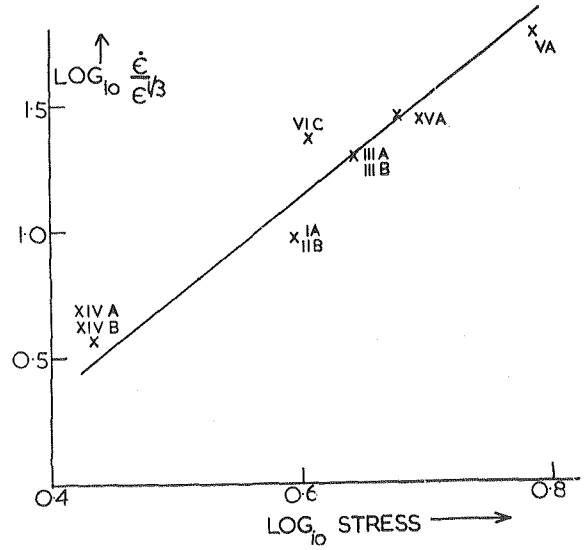
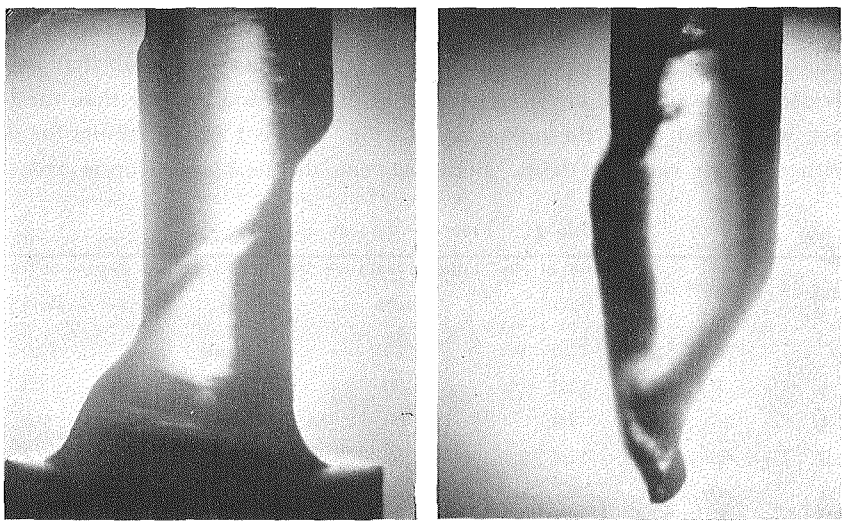


Fig. 5. Graph showing the stress dependence of the creep curves at −50°C. An arbitrary constant 3.67 has been added to the ordinates

The appearance of the crystals after straining was of interest. Normally after a large amount of strain (10–20%) the crystal had begun to develop a 'neck' or 'tape' at some point along its length. On one occasion, however, large offsets were observed at clearly defined glide bands which were parallel to the basal plane of the crystal. There were three such glide bands spaced about 0.5 cm apart along the crystal. The largest offset occurred very close to one end and a photograph of this is shown in Fig. 6 a. The crystal was returned to the machine and the load re-applied. The crystal continued



(a) (b)

Fig. 6. Crystal I A, deformed at −50°C, showing distinct glide bands (a) during deformation (b) after fracture. Magnification ×5

to strain by slip on the same glide bands and eventually fractured at the largest offset. A photograph taken after fracture is shown in Fig. 6 b.

An attempt was also made to observe a glide direction in ice, using the same method as Glen and Perutz (1954). From X-ray Laue pictures, the orientation of the tensile axis before and after straining, relative to the crystal directions, was plotted on a stereographic projection. This is shown in Fig. 7 for eight different crystals, all strained at −50°C. This figure shows that those crystals for which the initial maximum resolved shear stress was close to a $\langle 11\bar{2}0\rangle$ direction, (crystals III A, VI C, XI C, IV A, IV C), deformed with a $\langle 11\bar{2}0\rangle$ preferred glide direction. However, for those crystals in which the maximum resolved shear stress lay close to a $\langle 01\bar{1}0\rangle$ direction, (crystals V C, IX A, VIII A), no preferred crystallographic direction was observed.

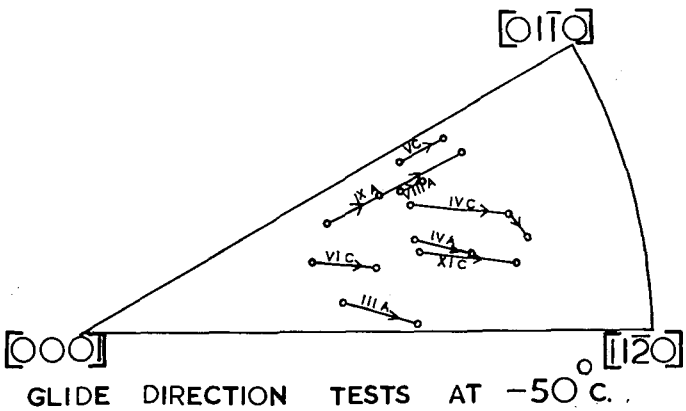


Fig. 7. Stereogram of the orientation of the tensile axis before and after straining

III. 2. *Fracture stress data at −50°C.* When the specimens fractured, about 75% of them did so on a plane perpendicular to the tensile axis and very often at a point close to one of the end grips. The nominal stress applied to the crystal, that is the load applied divided by the original cross-sectional area of the crystal, rather than the resolved shear stress, was found to be the influencing factor. Table 1 shows results for different crystals with different values of θ the angle between the tensile and optic axes. Large

Table 1. Fracture data at −50°C

Crystal	θ	Nominal stress	Time to fracture	Strain at fracture	Stress at fracture
		Bars	Hours	%	Bars
V C	65	12.0	<10	small	12.0
VII A	9.2	12.3	80	1.3	12.3
VI A	46	7.9	60	20	9.5
III B	68	12.4	55	5	12.8
III A	50	8.7	70	40	12.1
IV B	63	12.6	<17	small	12.6
II B	68	10.7	50	1.6	10.8
V A	52	15.8	2	0.1	15.8

strains were only obtained when the nominal stress on the crystal was about 8 bars. If this stress was around 12 bars, fracture occurred within 20–30 hours, after straining approximately 1%.

For very short times, however, a crystal could sustain larger stresses, for example crystal V A, which existed under a stress of 16 bars for 2 hours. The last column of Table 1 shows the tensile stress at the moment of fracture, assuming a uniform deformation. Since it is known that the crystals do not deform uniformly this column can only give a lower limit for the tensile stress at the moment of fracture. Thus the actual tensile stress acting at the tape of crystal VI A after it had strained overall by 20% may well have been much larger than 9.5 bars. It was concluded that fracture would occur at −50°C at a stress of 12.4±0.4 bars.

III. 3. *Deformation at −60 and −70°C.* At the time of writing, three crystals have been tested at −70°C and three at −60°C.

One of these crystals, II A, was first tested at −70°C for 120 hours under a stress of 3.6 bars, during which time it strained by less than 0.05%. Re-tested under the same load at −60°C, it strained by 0.7% in the same time. A crystal existing under the same conditions at −50°C would have strained by approximately 1.5% in this time. The shape of the creep curve, shown in Fig. 8, obtained for this crystal at −60°C, was interesting in that the strain rate first increased, then decreased, after about 60 hours, before continually increasing again. However, the two other crystals tested at this temperature, crystals VI B and II C, gave continually increasing creep curves similar to Fig. 4, but in both these cases the shear stress applied to the crystal was greater, 6.1 and 7.2 bars respectively. These two crystals obeyed a creep law of the form $\varepsilon \propto t^m$ with the average value of m equal to 1.6. This is very similar to the value of 1.5±0.2 found at −50°C, although more crystals need to be tested at −60°C before any firm conclusions can be drawn.

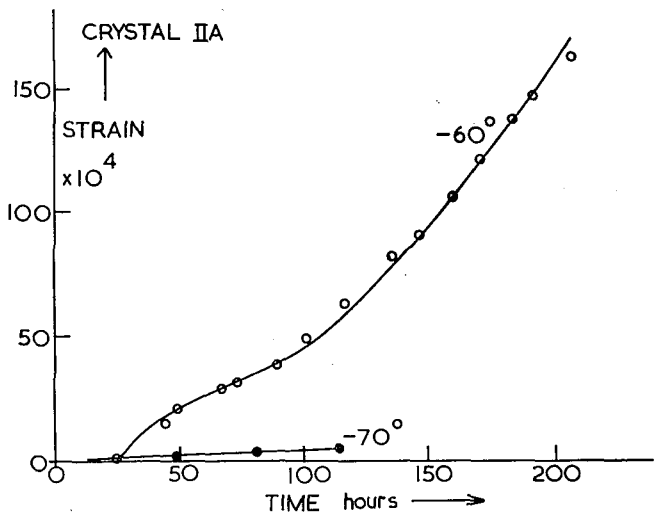


Fig. 8. Creep curve obtained for crystal II A at −60°C after previous deformation of 0.05% at −70°C

Apart from crystal II A, two other crystals, VI A and IX B, have been deformed at −70°C. Crystal VI A, under a stress of 8.4 bars deformed at a continually increasing strain-rate for 24 hours before fracturing and crystal IX B, at a stress of 6.3 bars deformed by 2% in 240 hours. The latter gave a creep curve similar to that of crystal II A shown in Fig. 8, namely a decrease in the strain rate about 50 hours after the start of the test, before it increased continually again.

III. 4. *Fracture data at −60 and −70°C.* The small amount of data so far collected at these temperatures is tabulated in Table 2. The stress at fracture is noticeably higher than the value of 12.4 bars found at −50°C, but there is no significant difference between the results for −60 and −70°C. More crystals are being tested at these temperatures.

Table 2. Fracture data at −60 and −70°C

Temp. °C	Crystal	θ	Nominal stress Bars	Time to fracture Hours	Strain at fracture %	Stress at fracture Bars
−60	VI B	50	16.1	<17	0.02	16.1
−60	II C	63	17.3	170	4	18.0
−70	VI A	50	16.7	24	0.3	16.7
−70	IX B	64	15.7	240	2	16.0

IV. Discussion

The results have shown that single crystals of ice remain plastic at a temperature of −70°C and that they still deform by slip on the basal plane.

The shape of the creep curves obtained is similar to that found by Glen and Perutz (1954), and other workers, at higher temperatures. Different workers have found different values of m in eq. (2); Butkovich and Landauer (1958) obtained a mean of 1.7, Jellinek and Brill (1956) found $m=2.0$ as did Griggs and Coles (1954). The present result of 1.5 at −50°C, which increased at higher strains, indicates that m is not very dependent on temperature.

The only other workers to have conducted creep tests at low temperatures are Higashi and others (1964). They conducted bending creep tests on glacier ice crystals at various temperatures down to −42°C and found their curves characterized by a gradual increase in the strain rate followed by a constant rate of strain. This difference in the shape of the curves may be due to differences in material, to the range of strain employed, or to inherent differences between tensile and bending tests.

Kamb (1961) has shown that the failure of Glen and Perutz (1954) and Steinemann (1954) to observe a glide direction in ice is due to the low stress dependence of the strain-rate in ice (*i.e.* the low value of n in eq. (3)). According to Kamb, "the response of a hexagonal crystal is entirely independent of a-axis orientation if $n=3$ [or 1] exactly. For $3<n<c.$ 5, the true glide direction should be weakly apparent, whereas for $1<n<3$ the crystal should show a response weakly suggestive of preferred glide in a direction perpendicular to the true glide direction". Previous values of n have

all been about 2; Butkovich and Landauer (1958) have reported a value of $n=2.5$, Higashi and others (1965) found $n=1.58$ down to $-42°C$ and Readey and Kingery (1964) quote $n=2.0$ for nominal strains less than 25%. Thus the failure to observe a glide direction for these values of n is satisfactorily explained by Kamb. For $n=4$, as found in this present work at $-50°C$, the maximum deviation of the glide direction from the resolved shear stress direction given by Kamb is 2.9° and would almost certainly be undetectable. However, we have observed deviations of about 9°. Those crystals which did not deviate were those which were oriented with two $\langle 11\bar{2}0\rangle$ directions very nearly equally stressed, and would only deviate by a very small amount if the two slip systems were both active. Despite the apparent disagreement with Kamb's theory, we believe that our evidence supports the view that $\langle 11\bar{2}0\rangle$ is the glide direction. This is also to be expected from structural considerations of the ice lattice and also from the X-ray topographical work of Hayes and Webb (1965). Using the Lang technique, they observed dislocations of Burgers vector $\langle 11\bar{2}0\rangle$ lying in the basal plane.

It is intended to continue collecting creep data at -60 and $-70°C$, and at lower temperatures.

Acknowledgments

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