DEFORMATION OF ICE SINGLE CRYSTALS CLOSE TO THE MELTING POINT

By STEPHEN J. JONES *and* JEAN-GUY BRUNET

(Glaciology Division, Inland Waters Directorate, Department of Fisheries and Environment, Ottawa, Ontario K1A 0E7, Canada)

ABSTRACT. Constant strain-rate compression tests on ice single crystals at temperatures between $-20°$C and $-0.2°$C are described. The power-law dependence of yield stress on strain-rate gives a value of n which varies from 1.95 ± 0.04 at $-0.2°$C to 2.07 ± 0.08 at $-20°$C. The activation energy of deformation varies with strain-rate, but a mean value of 70 ± 2 kJ mol^{-1} is obtained, with no indication of any increase close to the melting point, as has been found in polycrystalline ice. An apparent work-hardening effect, at strains greater than about 15%, is explained as being due to bending of the crystal changing the orientation of the basal planes.

RÉSUMÉ. *Déformation des monocristaux de glace vers le point de fusion.* Nous décrivons les essais de compression effectués sur des monocristaux de glace à vitesse de déformation constante et à des températures comprises entre -20 et $-0.2°$C. La relation en puissance entre la limite élastique et la vitesse de déformation conduit à une valeur de n qui varie de 1.95 ± 0.04 à $-0.2°$C, à 2.07 ± 0.08 à $-20°$C. L'énergie d'activation de la déformation varie avec la vitesse de déformation mais la valeur moyenne de 70 ± 2 kJ mol^{-1} peut être retenue sans que l'on puisse noter une augmentation quelconque à proximité du point de fusion comme cela a été observé dans le cas de la glace polycristalline. Pour des déformations supérieures à 15%, un effet apparent de consolidation est observé et peut être expliqué par la courbure du cristal entrainant une variation de l'orientation du plan basal.

ZUSAMMENFASSUNG. *Verformung von Eis-Einkristallen dicht am Schmelzpunkt.* Es werden Druckversuche mit konstanter Verformungsgeschwindigkeit an Eis-Einkristallen bei Temperaturen zwischen $-20°$C und $-0.2°$C beschrieben. Die Abhängigkeit der Streckgrenze von der Verformungsgeschwindigkeit nach einem Potenzgesetz liefert einen Wert n zwischen 1.95 ± 0.04 bei $-0.2°$C und 2.07 ± 0.08 bei $-20°$C. Die Aktivierungsenergie der Verformung ändert sich mit der Verformungsgeschwindigkeit, aber man erhält einen Mittelwert von 70 ± 2 kJ mol^{-1} ohne Anzeichen irgendeines Anstiegs dicht am Schmelzpunkt, wie er in vielkristallinem Eis gefunden wurde. Ein scheinbarer Verfestigungseffekt bei Verformungen über ungefähr 15% hinaus wird als Folge der Biegung des Kristalls erklärt, welche die Orientierung der Basisebene ändert.

INTRODUCTION

Barnes and others (1971) studied the creep of polycrystalline ice and showed that below $-8°$C the activation energy for secondary creep was 78 kJ mol^{-1} but above $-8°$C the activation energy rose to 120 kJ mol^{-1}. They attributed this rise both to a liquid phase at the grain boundaries, and to grain-boundary sliding. If this is so, equivalent experiments on single crystals will not show such an increase in activation energy, and this was the primary reason for doing the work described here. We wished to study, also, the deformation of single crystals at large strains, where work hardening has been reported (Jones and Glen, 1969; Parameswaran, 1975).

EXPERIMENTAL METHOD

The experiment consisted of deforming single crystals of ice in uniaxial compression at a constant strain-rate on a table-model Instron mechanical tester.

The ice samples were grown by a technique described by Glen and Jones (1967) but modified to allow about 50 single crystals to be grown at one time. The water used to grow the ice was deionized and doubly-distilled. This method of growth produced right circular cylinders approximately 9 mm in diameter and 30 mm in length. They were each oriented under crossed polaroids and only samples in which the initial angle θ_0 between the c-axis and the compressive axis was $35° \leqslant \theta_0 \leqslant 55°$ were used. Usually, two stainless-steel end caps were frozen onto the samples before testing. These end caps eliminated any sideways movement of the ends of the samples. We tested some samples with only one end cap to see if there was any effect on the stress–strain curve. This method of growth provided crystals with a

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dislocation density of $\approx 10^6$ cm^{-2} and with some low-angle boundaries, as shown by X-ray topography. The surface of the ice was chemically polished in dilute methyl alcohol followed by rinsing in n-hexane or carbon tetrachloride.

After preparation, a sample was transferred to the testing bath on a table-model Instron tester in a cold room. This kerosene bath prevented evaporation of the sample and maintained the sample at a constant temperature, to within ± 0.01 deg. throughout a test. The bath was stirred constantly to obtain this degree of temperature control, the temperature of the cold room was kept several degrees colder than the test temperature, and the bath was heated to the required temperature. A mercury-in-glass thermoregulator switched the heat on or off as required. The temperature was independently monitored with a digital HP 2802 A platinum resistance thermometer capable of reading to ± 0.01 deg. The zero point of this thermometer was regularly checked against an ice bath.

The load applied to the ice was measured with a standard Instron load cell. This load F was converted to resolved shear stress τ by the formula (Reid, [c1973], p. 130)

$$\tau = (F/A_0)(L/l_0) \cos \phi_0 \cos \lambda_0, \tag{1}$$

where A_0 is the cross-sectional area of the specimen, L is the deformed length of the specimen, and l_0 is the original length of the specimen. ϕ_0 is the initial angle between the normal to the slip plane and the compressive axis. This angle is always obtuse in Reid's [c1973] definition, i.e. $\phi_0 = 180 - \theta_0$, where θ_0 was defined above. λ_0 is the initial, acute angle between the slip direction and the compressive axis. For ice, which slips along the direction of maximum shear stress (Kamb, 1961), $\lambda_0 = \phi_0 - \pi/2$ and so Equation (1) reduces to

$$\tau = (F/2A_0)(L/l_0) \sin 2\phi_0. \tag{2}$$

The deformation of the ice was not measured directly, but calculated from the known speed of deformation and elapsed time. This change in length was converted to resolved shear strain ϵ using the equation (Reid, [c1973], p. 117)

$$\epsilon = [\{(L/l_0)^2 - \sin^2 \lambda_0\}^{\frac{1}{2}} - \cos \lambda_0]/\cos \phi_0, \tag{3}$$

which, because $\lambda_0 = \phi_0 - \pi/2$, reduces to

$$\epsilon = -\tan \phi_0 + \{(L/l_0 \cos \phi_0)^2 - 1\}^{\frac{1}{2}}. \tag{4}$$

About 25 different samples were tested at each of the following temperatures: -20, -10, -5, -2, and $-0.2°$C. The strain-rates used varied from 10^{-7} to 10^{-3} s^{-1}.

RESULTS

Some typical stress–strain curves obtained for various strain-rates at $-5°$C are shown in Figure 1. Their shape is similar to that obtained by previous workers (Higashi and others, 1964; Readey and Kingery, 1964; Jones and Glen, 1969). A rapid initial rise in stress, mostly elastic in origin, is followed by a yield point and a rapid drop in stress. After a strain of a few per cent the stress levels off to an almost constant, slightly decreasing, value. At strains greater than about 15%, the stress nearly always started to rise again, a fact which will be discussed later in connection with Figure 2. The yield point is thought to represent the stress at which dislocations can multiply rapidly, thus allowing the ice to deform and the stress to drop. It is a convenient and reproducible measure of the strength of the crystals. The yield stress depends on the number of initially mobile dislocations but we assume our crystals are reasonably uniform in this respect and the reproducibility of our results confirms this. In any event, a different initial number of dislocations should not affect our conclusions concerning the activation energy.
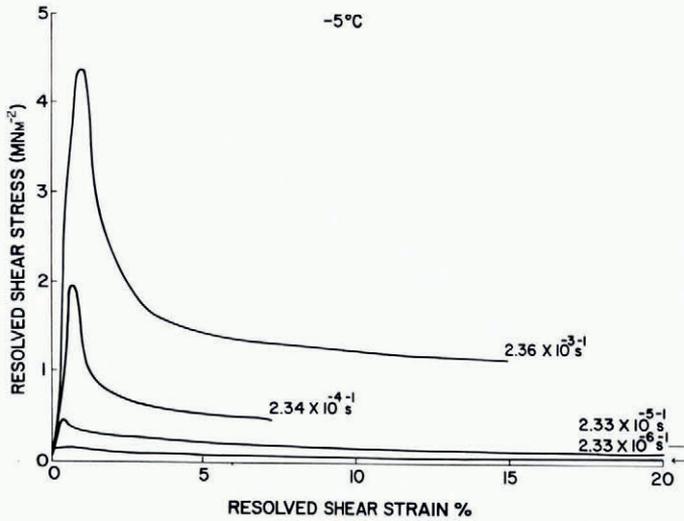
Fig. 1. *Typical stress–strain curves obtained at* $-5°C$ *at the various strain-rates shown.*
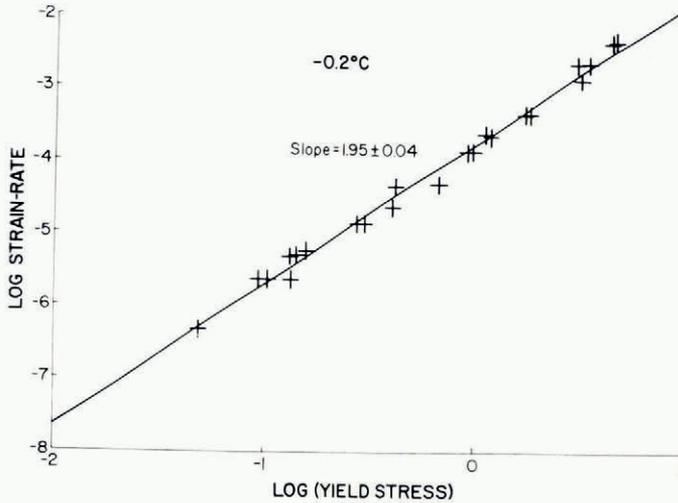

Fig. 2. *The dependence of yield stress on strain-rate at* $-0.2°C$. *In Figures 2–6, each point usually represents one sample, a number in parenthesis beside a point indicates more than one sample gave an identical result. A least-squares straight line is drawn through the points.*

The dependence of the yield stress on strain-rate is shown in Figures 2–6 for the five different temperatures. A good straight-line fit is obtained for these double logarithmic graphs, showing that the yield stress τ_y depends on strain-rate ϵ according to the equation

$$\epsilon \propto \tau_y{}^n, \tag{5}$$

where n, the slope of the straight lines in Figures 2–6, varies from 1.95 ± 0.04 at $-0.2°C$ to 2.07 ± 0.08 at $-20°C$. Figure 7 is a plot of the values of n against temperature, and shows that, in spite of the large errors in individual values, n depends on temperature in a linear manner. The straight line of Figure 7 extrapolates to give $n = 2.3$ at $-60°C$, a value in agreement with that of 2.6 ± 0.4 obtained by Jones and Glen (1968) from a linear analysis of their creep tests at $-60°C$.
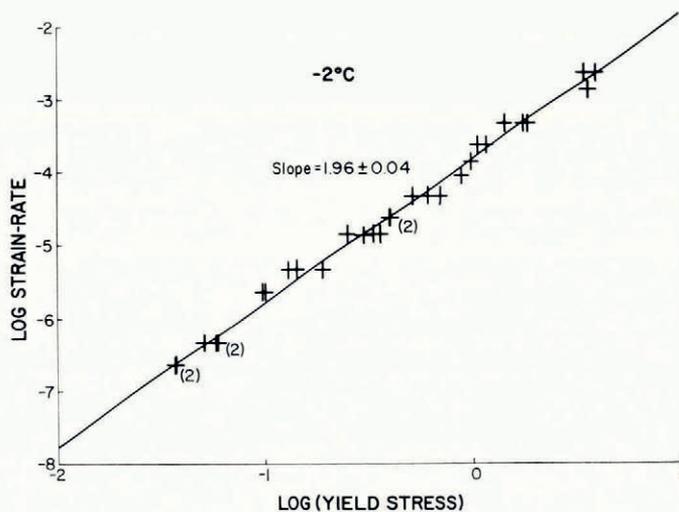
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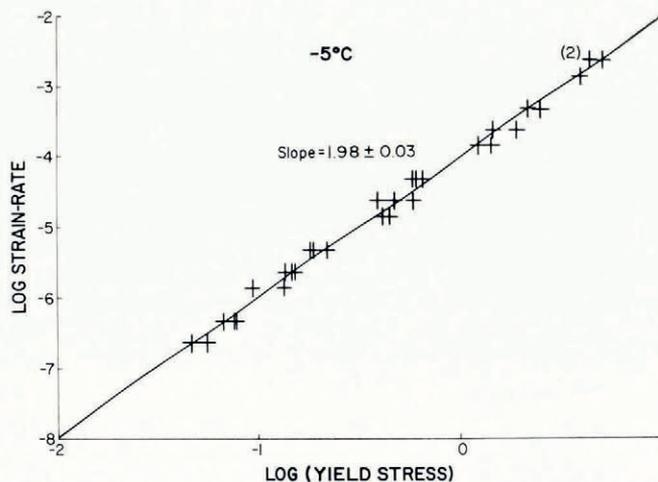Fig. 3. *The dependence of yield stress on strain-rate at* −2°C.

Fig. 4. *The dependence of yield stress on strain-rate at* −5°C.

Figure 8 shows our results at all five temperatures plotted on one graph; only the least-squares straight lines are shown for clarity. It can be seen from this figure that the temperature dependence at high strain-rates is less than at low strain-rates, thus the calculated activation energy will decrease with increasing strain-rate. Therefore, in order to calculate a mean activation energy over the entire range of strain-rates used we adopted the following procedure: The data of Figures 2–6 were re-analyzed by first assuming that n did not vary with temperature at all but maintained a constant value of 2.0. Least-squares straight lines of this slope were then plotted through the data, and the result is seen in Figure 9. From this figure, the values of $\log_{10}$ (strain-rate) for each temperature at a yield stress of 1 MN m^{-2} were re-plotted as natural logarithms against the reciprocal of the absolute temperature, as shown in Figure 10. The slope of such a curve gives an activation energy E according to the equation

$$\dot{\varepsilon} \propto \exp(-E/RT), \tag{6}$$
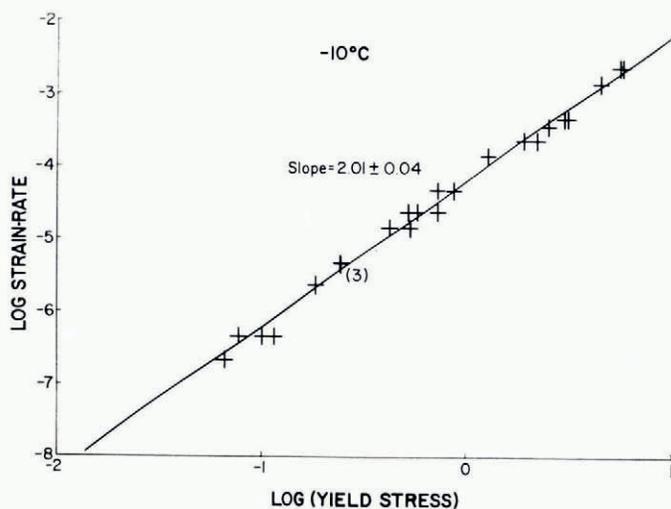
where R is the gas constant.

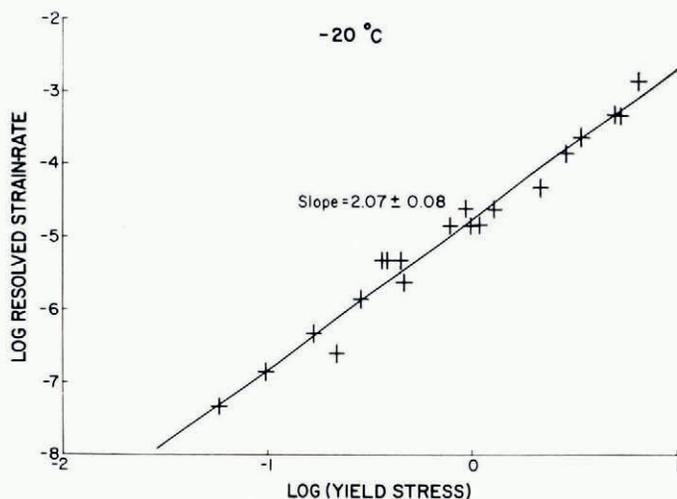Fig. 5. *The dependence of yield stress on strain-rate at* − 10°C.

Fig. 6. *The dependence of yield stress on strain-rate at* − 20°C.

The data of Figure 10 are best fitted by a straight line of a slope which gives a value for E of 70 ± 2 kJ mol⁻¹ over the entire range of temperature from −20°C to −0.2°C. There is no indication of any increase in activation energy close to the melting point, as was found by Barnes and others (1971) in polycrystalline ice above −8°C. We also calculated activation energies in a similar way directly from Figure 8. We obtained values of

$$E = 76 \text{ kJ mol}^{-1} \quad \text{at } \tau_y = 0.1 \text{ MN m}^{-2},$$

and

$$E = 62 \text{ kJ mol}^{-1} \quad \text{at } \tau_y = 10 \text{ MN m}^{-2}.$$

An independent experiment to determine activation energies was also used. Some samples were subjected to temperature-change tests in which the flow stresses before and after
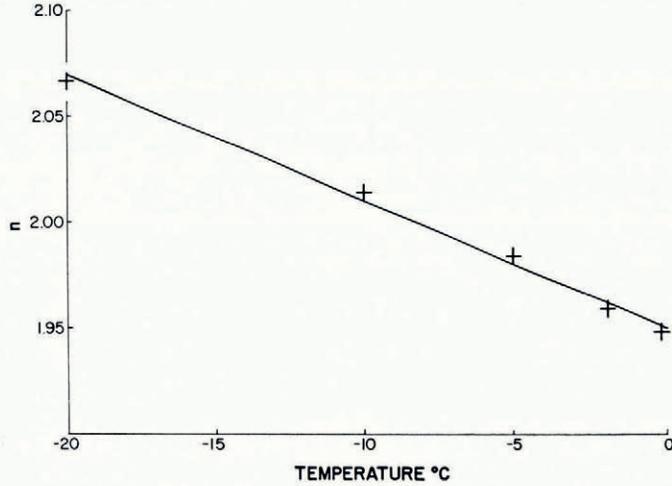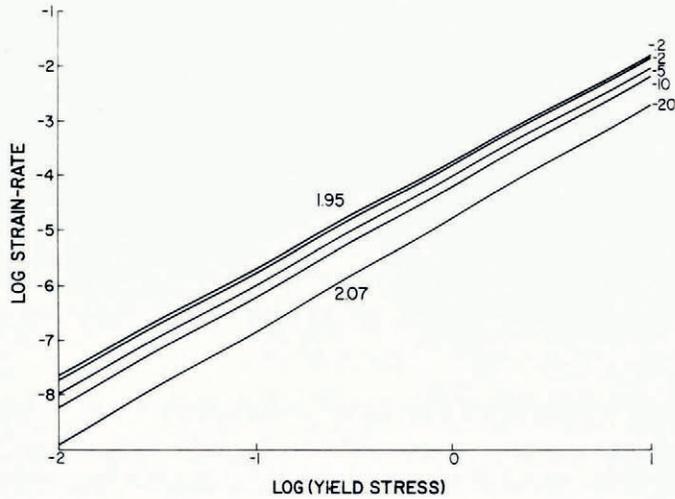
Fig. 7. *The dependence of n on temperature.*

Fig. 8. *The least-squares straight lines of Figures 2–6 drawn on one graph, showing their tendency to converge at high strain-rates.*

the temperature change were compared. With $n = 2.0$, an activation energy was calculated from the equation

$$\tau \propto \dot{\epsilon}^{1/n} \exp(-E/nRT).$$

The results, which were not so reproducible as the previous method, are:

82 ± 12 kJ mol^{-1}	between -20 and $-10°$C,
78 ± 9 kJ mol^{-1}	between -10 and $-5°$C,
73 ± 15 kJ mol^{-1}	between -5 and $-2°$C,
70 ± 15 kJ mol^{-1}	between -2 and $-0.2°$C.

Within the limits of error, there is again no indication here of an increase in activation energy close to the melting point; the results agree with the mean activation energy calculated above.
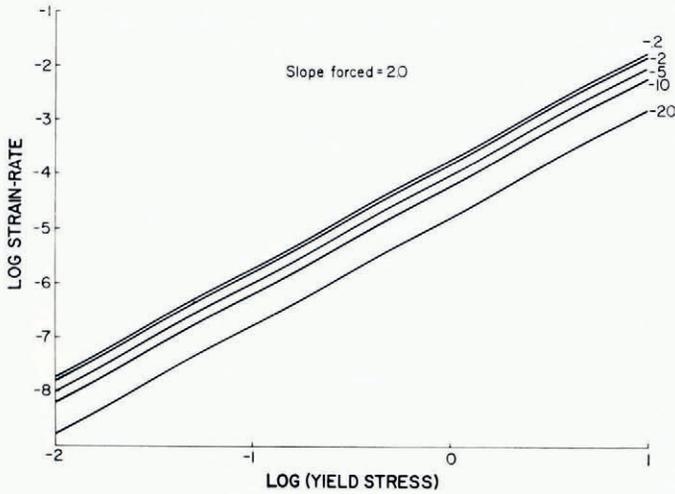
Fig. 9. *Least-squares straight lines of the data of Figures 2–6 but with a fixed slope of 2.0.*
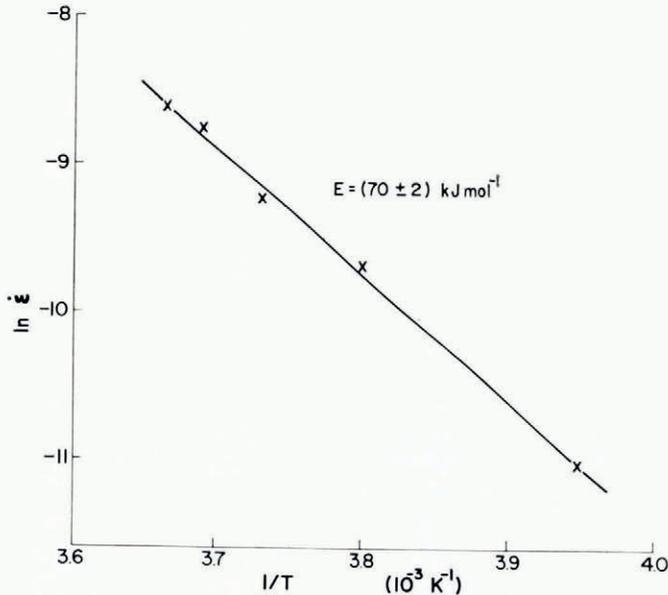

Fig. 10. *The natural logarithm of the strain-rate at a stress of 1 MN m^{-2} obtained from Figure 9, plotted against reciprocal absolute temperature. The slope gives an activation energy of 70 ± 2 kJ mol^{-1}.*

WORK HARDENING

It has generally been held that ice single crystals do not work harden. That is to say, after the yield point the stress continually decreases, even if very slowly, and does not rise again. However, Jones and Glen (1969) commented that one sample tested at −70°C showed an increase in stress at a strain of 18%, but, because it was the only sample tested to such a large strain, they could not conclude whether the result was a genuine work-hardening effect or whether it was due to distortion of the crystal at large strains. Parameswaran (1975) observed a similar effect beyond about 10% strain which he attributed to a rapid increase in

dislocation density. He was able to show that it was not due to any bulging of the specimen, because he remachined the sample after 10% strain and still observed the increase in stress.

We have also observed this effect in almost all our single crystals tested beyond 15% strain, as shown, for example, in Figure 11. The effect is much less marked if one end of the crystal is not constrained by an end cap but is free to move sideways. We believe, however, that this apparent work hardening is due to a bending of the crystal which causes a reduction in the angle θ and hence a reduction in the true resolved shear stress.

Figure 12(a) shows a sample photographed between crossed polaroids after 15% strain—the central portion of the ice has a distinctly different orientation from the end portions, which
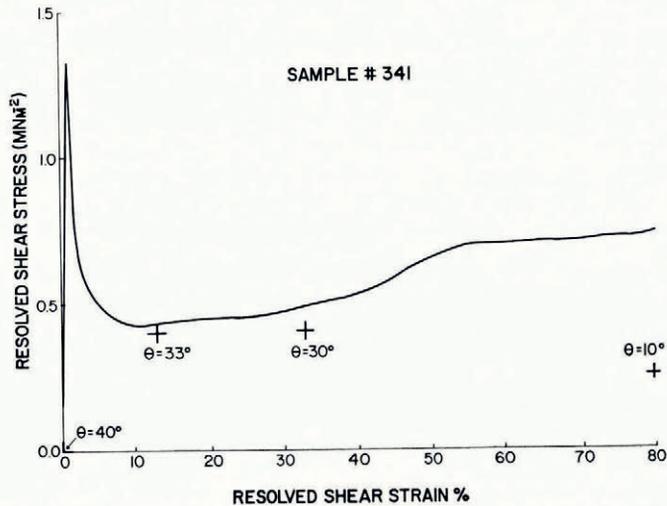

Fig. 11. An example of a crystal tested to large strains. The solid curve is the usual stress–strain curve, the points underneath are the values of stress for the different θ values as indicated.
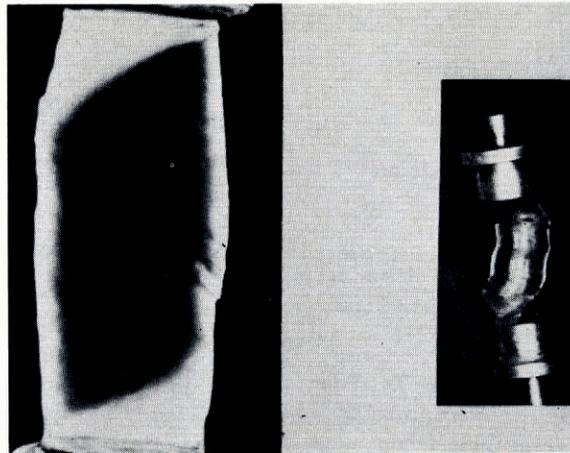

Fig. 12

a (left). A sample after 15% strain examined under crossed polaroids showing the different orientation of the central part of the crystal.
b (right). The sample of Figure 11 after 80% resolved shear strain. The sample had fractured close to both end caps and has been re-assembled for this photograph.

maintain the original orientation of the crystal because they are confined by the end caps. In the central portion, θ decreases because the crystal starts to bend as shown in Figure 13. This reduction of θ is not taken into account by Equation (2) which uses θ_0, the initial value, and so the stress rises, as seen in Figure 11. However, if the resolved shear stress is re-calculated using the new value of θ, it does not rise at all. This is shown in Figure 11 where the crosses below the full line are re-calculated shear stresses using the values of θ shown. These values were determined by removing the sample from the Instron at these points and measuring θ. Figure 12(b) shows this sample after fracture at 80% resolved shear strain when $\theta \approx 10°$.
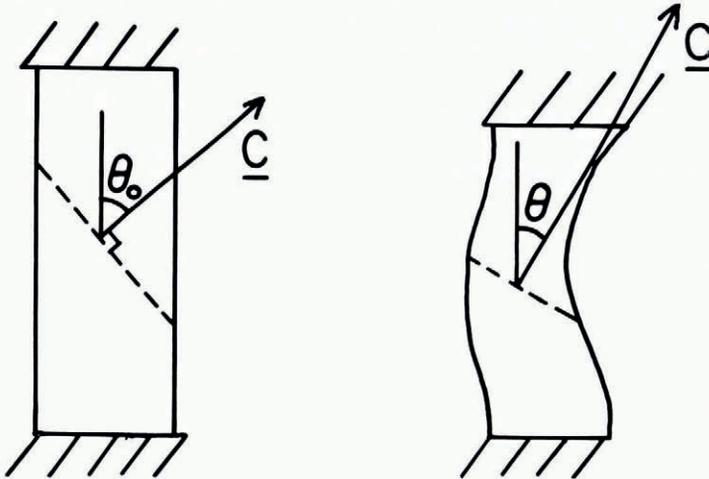

Fig. 13. A sketch showing how the crystal bends at large strains and causes a reduction in θ.

DISCUSSION AND CONCLUSIONS

The dependence of yield stress on strain-rate, $n \approx 2$, is similar to that found by others. Readey and Kingery (1964) obtained an average value of $n = 2.0$ for strain less than 25%, Higashi and others (1964) obtained 1.53 over a small strain-rate range. The dependence of n on temperature has not been found by others because they have not studied a sufficient number of samples.

Weertman (1973) has reviewed thoroughly previous studies on the deformation of ice and has tabulated the values of activation energy that have been found. For single crystals above $-50°C$, all workers have obtained values in the range 60–75 kJ mol^{-1}, except Muguruma (1969) who found a value of 46 kJ mol^{-1}. This seems anomalously low, but is probably a result of the low value of $n = 1.3$ which he found, and which was subsequently used in the calculation of the activation energy. Part of the reason for the scatter in values is probably the dependence of E on strain-rate which we have described here.

Our activation energy is constant throughout the temperature range $-20°C$ to $-0.2°C$. We conclude, therefore, that the rise in activation energy in polycrystalline ice (Barnes and others, 1971) is indeed due to grain-boundary effects as those authors surmised.

Although the resolved shear stress nearly always rises after 15% strain we do not believe this is a genuine work-hardening effect inherent to the crystals, but a result of the bending and re-orientation of the bulk of the crystal.

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DISCUSSION

D. R. Homer: Have you examined the stress dependence of n in the expression

$$\epsilon \propto \sigma^n ?$$

Our re-analysis of the data of Ramseier (see Homer and Glen, 1978, p. 439) indicates a change from $n = 2.0$ to $n = 2.5$ as the yield stress rises above 2 MN m^{-2}.

S. J. Jones: I see no stress dependence of n over the range I covered.

W. F. Budd: Why did you use constant-strain-rate tests rather than constant-load tests, and would you expect much difference for constant-load tests?

Jones: No real reason other than I had the machine available to use. I would not expect any significant differences in creep tests at comparable strain-rates and the results of Homer and Glen (1978) would seem to bear this out.

W. B. Kamb: What is the rationale for evaluating the flow law and activation energy by considering the yield stress rather than some other stress parameter derivable from your constant-strain-rate tests? Is not the yield stress rather sensitive to the initial condition of the crystals?

Jones: The yield stress represents the stress at which the dislocations can first move quickly through the crystal and multiply. Since there is no steady-state in the creep of single crystals of ice, it is a convenient parameter to use. It is sensitive to initial dislocation content at low dislocation densities, but much less sensitive when the density is about 10^5 to 10^6 cm^{-2} as we have in our samples.

G. Noll: In your constant-strain-rate tests under uniaxial compression which showed an apparent work hardening, the crystal was frozen into the grip heads. In my tests the crystal was deformed by uniaxial compression between plane metal plates and thin plastic foils were put between the crystal and plates and allowed the crystal to change its orientation during the deformation. With such a test arrangement I never found work hardening of a mono-crystal, even at large strains. This supports your explanation of the work hardening being only apparent.

Jones: We have also done tests with one free end. The rise in stress is less marked but some-times apparent.

P. Duval: Have you done experiments with ice single crystals which contain sub-boundaries?

Jones: I think that my samples probably do indeed contain sub-boundaries, if by that you mean parts of the crystal having orientation differences of the order of minutes.

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