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EFFECTS OF SURFACE CONDITION ON THE MECHANICAL PROPERTIES OF ICE CRYSTALS

by

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Effects of surface condition on the mechanical properties of ice crystals

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Abstract. A comparative study of the deformation behaviour of mechanically and chemically polished ice crystals is described. For single crystals, mechanical disturbance of the surface layer greatly reduces the maximum stress for basal glide in constant strain rate tests in compression. Single crystals with sub-boundaries do not show any prominent surface effect, but exhibit quite a low maximum stress. The results indicate that mechanically disturbed surface layers and sub-boundaries do not appear to act as strong barriers to dislocation movement in ice crystals, but rather as sources of dislocations. Surface condition does not appear to affect the maximum stress for polycrystals. The influence of strain rate (range $2 \cdot 1 \times 10^{-5}$ to $1 \cdot 7 \times 10^{-4}\,\mathrm{s}^{-1}$) and temperature (range -10 to $-30\,^{\circ}\mathrm{c}$) upon the maximum stress was also investigated for both single crystals and polycrystals of ice.

1. Introduction

The deformation behaviour of ice single crystals in constant strain rate tests (Higashi *et al.* 1964, Readey and Kingery 1964, Jones 1967) indicates a wide variation in the shape of the stress–strain curve. This variation is considered to be the result of differences in the dislocation density of the crystals. It was observed by Higashi *et al.* (1965), however, that the steady-state strain rate in bending creep tests is influenced by the surface condition of the crystals. The possible influence of surface condition on the deformation behaviour of ice has not been studied experimentally.

Experiments have been conducted on the effect of surface condition on the plastic flow of crystals. Considerable attention has been given to the Rehbinder effect, i.e. how the mechanical properties of a crystal are affected by a thin film on its surface, and the Joffé effect, i.e. the effect on the mechanical properties of the dissolution of the surface layer of a crystal by a surrounding solution (Nabarro 1967). It has also been pointed out (Fisher 1952) that the stress necessary to operate a surface source is about one half that required to operate an interior source of the same length.

It was the object of the present experiments to determine whether the condition of the surface has any effect on the deformation behaviour of ice, and if it has, to establish a plausible explanation. Evidence is presented that not only the method of specimen preparation but also the quality of the crystals has a strong influence on the yield behaviour of ice single crystals in controlled strain rate tests. The results are discussed in terms of surface sources of dislocations. Observations of the effect of sub-boundaries and grain boundaries on plastic flow are also reported.

2. Experimental procedure

Ice single crystals and columnar-grained ice were made from de-aerated water using the technique described by Gold (1963). Crystals thus obtained were transparent and free of internal cracks and voids. Single-crystal specimens were obtained from very large grains (about 15 cm by 20 cm) formed in blocks about 15 cm thick. The columnar-grained

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ice specimen had an average grain diameter between 2 and 5 mm. The [0001] direction of each grain was almost normal to the long direction of the columns but had no preferred orientation in the plane perpendicular to that direction.

Rectangular specimens were cut from the ice with a band-saw. The single-crystal specimens were cut so that the basal plane, the plane of easy glide, was 45° to the long axis of the specimen and normal to its largest face. Examination with polarized light showed that most of the single crystals had no sub-boundaries. The boundaries were quite distinct in those that did, as can be seen in figure 4, and the change in crystallographic orientation across them was only a few degrees. For columnar-grained ice the axis of the columns was perpendicular to the largest face. The specimens were planed with a fine blade until they were 10 mm thick, 25 mm wide and 30 mm long. Each was given a final polish, either mechanically with a fine blade or emergy paper, or chemically with an alcohol-soaked silk cloth on a flat glass plate (Muguruma *et al.* 1966). The final dimensions were 5 mm thick, 20 mm wide and 25 mm gauge length. The specimens were annealed in a kerosene bath for at least 24 hours at −10 °C before testing, in order to remove any mechanical or thermal stress that might have been produced during preparation.

Specimens were deformed in compression in a special fixture with a Hounsfield tensometer. The rate of cross-head movement could be varied through the use of a gear train and was kept constant during each test. The load was applied to the 5 mm by 20 mm faces and measured with a load cell having an electric output.

3. Results

3.1. *Stress–displacement curves: single crystals*

Typical stress–displacement curves for single crystals are shown in figure 1 (curves A and B), where it may be seen that the maximum stress σ_{max} for chemically polished specimens is about twice that for mechanically polished specimens. Maximum stress, however, varied greatly from one crystal to another and comparative tests could only be carried out on specimens from the same crystal. The variation for mechanically and chemically polished specimens is shown in figure 2. Though the variation in yield stress is large, the difference in the behaviour of the mechanically and chemically polished crystal can be clearly seen.
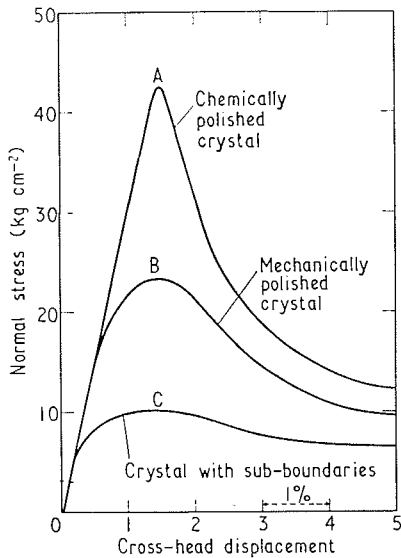

Figure 1. The effect of surface condition on the compressive stress–displacement curve of ice single crystals in basal glide at −10 °C (cross-head speed 0·063 mm min⁻¹, specimen length 25 mm).
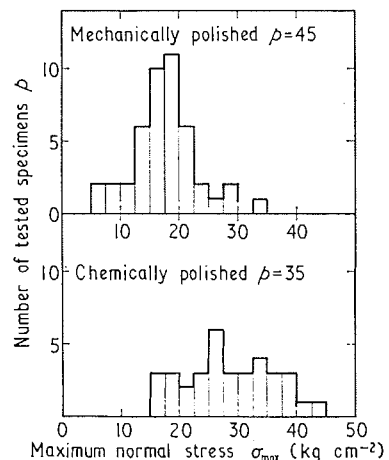
Figure 2. Variation of the maximum stress for chemically and mechanically polished single crystals.

It was necessary to remove chemically a layer about 100 μm thick in order to obtain the maximum increase in the yield point of mechanically polished specimens.

The nature of the slip bands just after yielding and their direction and the direction of the compressive stress are shown in figure 3 (plate). Specimens that had been chemically polished had sharply defined and widely spaced bands (figure 3(a)). Bands in mechanically polished specimens, on the other hand, were diffuse and the finer ones could not be resolved with the optical microscope (figure 3(b)). No influence of surface area on the change in yield stress could be detected when the cross-sectional area and length of specimen were kept nearly constant.

In order to establish whether the surface dislocation sources are of primary importance in deformation behaviour, mechanically polished specimens were annealed in a kerosene bath at $-3\,°$c for 120 hours. It was observed that not only the maximum stress but also the slip bands were almost the same as those obtained for chemically polished specimens, as may be seen in figure 3(c). This indicates that the dislocations generated at the surface layer by mechanical polishing are removed from the crystal during annealing.

Mechanically polished specimens were deformed about 3%, completely unloaded and subjected to the same annealing procedure. Upon reloading, the deformation curve coincided with the extension of the stress–strain curve previously obtained. The fact that no recovery took place indicates that dislocations in the interior of a specimen control deformation after yield, and are more stable than those in the surface layer.

Crystals with sub-boundaries, on the other hand, did not exhibit a marked yield point, and their maximum stress remained at quite a low level (figure 1, curve C). For these crystals the yield point did not depend significantly on the method used to polish the surface.

Well-developed slip-bands could be seen in the crystals with sub-boundaries after about 5% strain, as may be seen in figure 4. The slip traces are continuous across the boundaries, suggesting that such boundaries are not strong obstacles to the movement of dislocations.

3.2. *Stress–displacement curves: columnar-grained ice*

Figure 5 shows a typical stress–displacement curve for columnar-grained ice. Although the shape of the curve is almost the same as that for chemically polished single crystals, the decrease in stress after it reaches the maximum is much steeper.
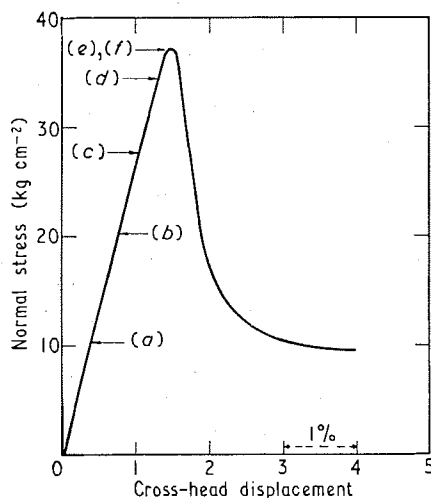
Figure 5. Stress–displacement curve at $-10\,°$c for a polycrystalline specimen at $-10\,°$c; cross-head speed 0.063 mm min^{-1} and specimen length 25 mm. Arrows (a)–(f) show the displacement $(4.2 \times 10^{-5}\ \text{sec}^{-1})$ at which the photographs in figure 6 were taken.

The method of surface polishing does not appear to affect the value of the maximum stress. This fact implies that the boundaries are a more prominent source of dislocations than the surface.

Crack formation was observed in the initial linear portion of the stress–displacement curve. Examples are shown in figure 6; the stage of deformation for each figure is indicated by arrows in figure 5. The number of cracks suddenly increased when the stress attained its maximum value (figure 6 (*e*) and (*f*)).

3.3. *Dependence of the maximum stress on strain rate and temperature*

At the yield point, the rate of cross-head movement is equal to the strain rate. A series of tests was undertaken to determine the dependence of the maximum stress σ_{max} on strain rate ϵ and temperature T. The dependence of σ_{max} on strain rate is shown in figure 7, where it can be seen that $\sigma_{max}{}^m \propto \dot{\epsilon}$.
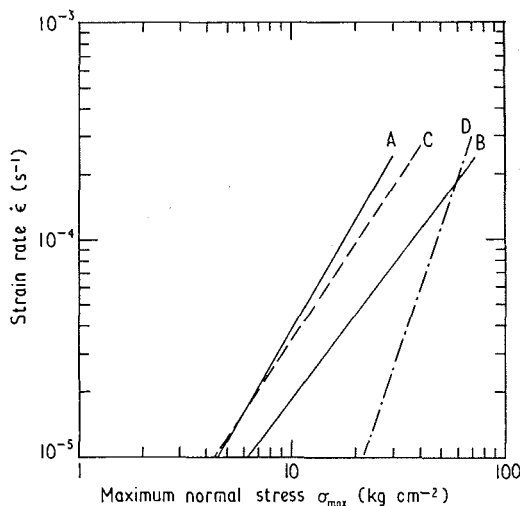

Figure 7. Strain-rate dependence of the maximum stress for single crystals, crystals with sub-boundaries and columnar grained crystals: A, mechanically polished single crystals; B, chemically polished single crystals; C, crystals with sub-boundaries; D, columnar grained crystals.

The values of m for single crystals calculated from figures 7 and 8 are: $m = 1 \cdot 3$ for chemically polished specimens; $m = 1 \cdot 7$ for mechanically polished specimens; $m = 1 \cdot 5$ for crystals with sub-boundaries. Figure 7 illustrates the difference in the maximum stress for chemically and mechanically polished specimens. It is of interest that the lines drawn through the observations intersect at the point $\dot{\epsilon} = 2 \times 10^{-6}$ s^{-1} and $\sigma_{max} = 1 \cdot 8$ kg cm^{-2}.

The dependence of σ_{max} on strain rate for columnar-grained ice is shown in figure 7. The value for m in this case is $3 \cdot 0$.

The temperature dependence of the maximum stress for single crystals and columnar-grained ice is shown in figure 8. For single crystals, the tests had to be made on specimens from the same crystal because the maximum stress varied greatly from one crystal to another, as is shown in figure 2. Although it was difficult to obtain a single crystal with uniform quality, the results do fall quite closely on a straight line when the logarithm of the maximum stress is plotted against the reciprocal of temperature. The dependence of the maximum stress on temperature for a strain rate of $2 \cdot 1 \times 10^{-5}$ s^{-1} is given by an expression of the form

$$\sigma_{max} \propto \exp{(A/T)} \tag{1}$$

with $A = 4 \times 10^4$ °K for single crystals, and

$A = 2 \cdot 5 \times 10^4$ °K for columnar-grained ice between -10 and -30 °C.
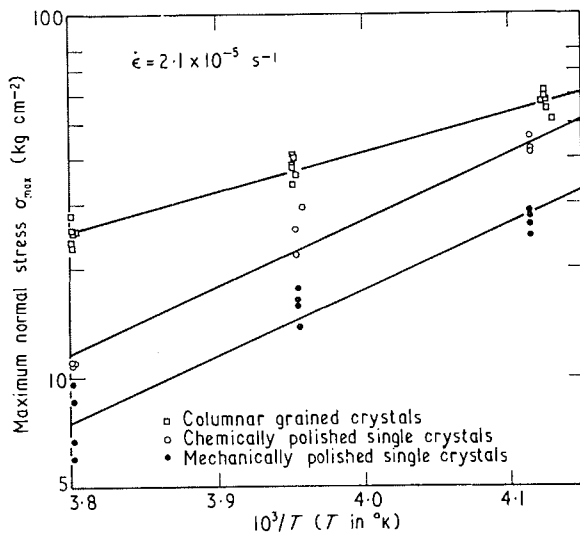
Figure 8. Temperature dependence of the maximum stress for chemically and mechanically polished single crystals and polycrystals with average grain diameter of 4 mm.

The equality of the coefficients for chemically and mechanically polished crystals suggests that the same thermally activated process is controlling the plastic deformation of both types of specimens. The lower value observed for the columnar-grained ice indicates that the process involved in the deformation of those specimens is different from that for the single crystals.

3.4. *Dependence of maximum stress on grain size*

The maximum stress σ_{max} was found to vary linearly with the inverse square root of the grain diameter d in columnar-grained ice, as shown in figure 9. The observations can be expressed by

$$\sigma_{max} = 0 \cdot 075 + 0 \cdot 31 \, d^{-1/2}.$$

It may be noted that the value of the maximum stress when extrapolated to $d^{-1/2} = 0$ coincides well with the value of the maximum stress for mechanically polished single crystals under the same condition of deformation.
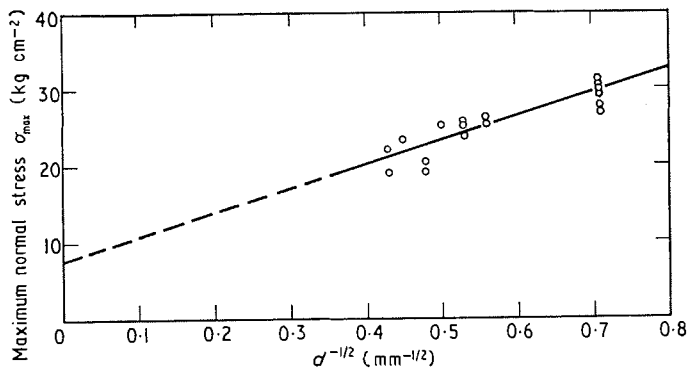

Figure 9. Relation between the maximum stress and grain diameter: strain rate $2 \cdot 1 \times 10^{-5} \, s^{-1}$; temperature $-10\,°\text{c}$.

4. Discussion

4.1. *Influence of surface condition on maximum stress*

The observations show that there is a clear difference in the maximum stress of chemically and mechanically polished specimens. Additional observations showed also that specimens prepared by melting the surface had a low maximum stress (about the same as mechanically polished specimens), and that specimens polished quite carefully by hand could have a high maximum stress, about equal to that of chemically polished specimens. These observations emphasize the significant influence of the surface layer on the plastic properties of ice crystals.

It is possible that surface roughness itself is a source of stress concentration that reduces the yield point. To test this possibility, chemically polished and mechanically polished specimens were etched with an ethylene-dichloride solution that made severe irregularities on the surface. This surface etching did not significantly change the maximum stress. This supports the hypothesis that it is only dislocations or dislocation sources in the surface layer, introduced by mechanical polishing, that influence yield behaviour.

A surface film, however, might be formed by the chemical polish and might restrict or prevent the operation of surface sources, thus causing an increased yield point. Surface films might also prevent the egress of dislocations from the crystal, so that they become piled up beneath the film. Chemically polished specimens were deformed in a dilute solution of alcohol or sodium chloride to check this possibility, but no noticeable change in the maximum stress was observed. This confirms that it is a disturbed surface layer produced by the mechanical polish that is responsible for the lowering of the yield point, and that chemical polishing establishes a higher yield point by removing this layer. It can be said, therefore, that the behaviour observed is not the result of a Rehbinder or Joffé effect.

If surface sources have a significance influence on yield point, it might be expected that slip bands in the mechanically polished specimens would be closely spaced and faint because many surface sources would be active and the displacement per slip-band not need to be so large to produce a given strain. The slip-bands of a chemically polished specimen should be more widely spaced and clear because fewer dislocation sources operate and a larger displacement per slip band would be required to produce the same strain. The observations are consistent with this behaviour and thus provide additional evidence that surface dislocation sources can play an important role in the deformation of ice crystals.

In order to increase the maximum stress of mechanically polished specimens to about the same level as that of chemically polished specimens, a layer almost 100 μm thick had to be removed chemically from the surface. It might be possible to estimate the minimum thickness of the disturbed surface layer formed by mechanical polishing by applying the theory of Fisher-type 'surface sources' which can be formed by the Frank–Read (1950) mechanism of slip. In figure 7 the curves for mechanically and chemically polished specimens intersect at $\sigma_{max} = 1 \cdot 8$ kg cm^{-2}, suggesting that the critical stress required to move a surface dislocation is about this value, and that the stress associated with the intersection of the two curves corresponds to the maximum length of dislocation sources generated by the mechanical disturbance.

Writing the effective length of surface sources as $2l_0$, the critical shear stress τ will be equal to $Gb/2l_0$, where G is the shear modulus and b is the Burgers' vector. Taking $\tau = 0 \cdot 9$ kg cm$^{-2} = 9 \times 10^5$ dyn cm^{-2}, $G = 3 \times 10^{10}$ dyn cm^{-2} and $b = 4 \cdot 5 \times 10^{-8}$ cm, one obtains $2l_0 \simeq 15$ μm.

In an early study of etch pits in ice (Muguruma 1961) the number of etch pits formed in a mechanically polished surface was found to be an order of magnitude greater than that formed when a layer about 100 μm thick had been removed by thermal etching. This difference was considered to be due to a disturbed surface layer less than 100 μm thick, formed by mechanical polishing. From the present experiments and the foregoing discussion, it would appear that the thickness of the disturbed surface layer formed by

mechanical polishing ranges between 15 and 100 μm, depending upon the severity of the polishing.

Crystal quality, in terms of initial density of mobile dislocation in the interior of a crystal, should also have a significant influence on plastic deformation. If the crystal does not show a high maximum stress or a significant difference of maximum stress for the two methods of polishing, it would be expected that the quality of crystal must be poor; that is, that the crystal has a high density of internal dislocations or dislocation sources. If the initial dislocation density or number of dislocation sources in the interior of the crystal is comparable to that produced by mechanical polishing, the removal of the surface-disturbed layer should not give rise to a significant change in deformation behaviour.

Although no attempt was made to obtain the dislocation density in the present experiments, the following facts support the view that the density of internal dislocations or sources is as important as surface sources. Crystals that showed a low maximum stress exhibited clear deformation bands an an early stage of deformation that were perpendicular to the slip planes, as is shown in figure 10. In figure 10(*a*) the direction of the slip planes is indicated by vapour figures produced by internal melting (Nakaya 1958). Distinct deformation bands lying normal to the slip plane are shown in figure 10(*b*). The crystals that had a high maximum stress did not develop distinct deformation bands during deformation. As a deformation band consists of a row of edge dislocations normal to the slip plane the formation of such a band must be greatly dependent upon the density of dislocations in the crystal and the number of active slip planes. The fact that crystals with sub-boundaries had quite a low maximum stress shows clearly that these boundaries do not act as barriers to dislocations, and may even be a source of dislocations.

In columnar-grained ice, surface condition had no effect on the maximum stress. This implies that grain boundaries act as sources of dislocations during the initial stage of deformation.

4.2. *Strain rate and temperature dependence of the maximum stress*

In their investigations of the creep properties of ice, Glen (1955) Steinemann (1956) and Higashi *et al.* (1964) conclude that the behaviour of ice resembles that of metals, and that the usual steady-state creep rate $\dot{\epsilon}$ can be expressed by

$$\dot{\epsilon} = C_1 \sigma^m \exp\left(\frac{-Q}{RT}\right) \tag{2}$$

where σ is the applied stress, T is the absolute temperature and C_1, m and Q are constants. Rewriting this equation in terms of the stress

$$\sigma = C_2 \dot{\epsilon}^{1/m} \exp\left(\frac{+Q}{mRT}\right). \tag{3}$$

It may be seen that equation (3) has the same form as equation (1), so that one can calculate a value for Q from the present results. The values of m and Q calculated from figures 7 and 8 are summarized in table I, together with the results obtained by the other investigators.

4.3. *Grain size dependence of the maximum stress*

Higher maximum stresses for polycrystalline ice than for single crystals imply that grain boundaries act as a barrier to the motion of dislocations. It has been found experimentally that the relation between the yield point and the grain size can be expressed in the form

$$\sigma = \sigma_0 + k d^{-1/2}$$

where σ is the applied stress, σ_0 a constant, which can be related to the frictional force acting on moving dislocations, d the average grain diameter and k is a constant. The coefficient of the reciprocal square root of the grain size has been interpreted according to

Table 1. **Values of stress exponent** m **and activation energy** Q

Investigator	m		Q (Kcal/mole)	Remarks
Single crystals				
Readey and Kingery (1964)	2·0		14·3	Tension (0 ∼ − 42 °c) Artificial crystals
Higashi *et al.* (1964)	1·53		15·9	Tension (− 15 ∼ − 40 °c) Natural crystals
Jones and Glen (1968)	2·2 (creep)		18	Compression (− 10 ∼ − 50 °c)
			10·3	Compression (− 55 ∼ − 80 °c) Artificial crystals
Muguruma	1·3		11	Chemically polished Compression (− 10 ∼ − 30 °c)
	1·7		15	Mechanically polished Compression (− 10 ∼ − 30 °c) Artificial crystals
Polycrystals				
Glen (1955)	3·2	4·2	31·8	Creep (0 ∼ − 13 °c)
Landauer (1955)			14	Creep (− 5 ∼ − 20 °c)
Bromer and Kingery (1968)			12	Creep (− 3 ∼ − 13 °c)
Muguruma		3·0	16	Compression (− 10 ∼ − 30 °c)

several different mechanisms, all of which involve the pile-up or running together of dislocations.

The ice crystal has only one primary slip system so that one can apply the simple mechanism proposed by Stroh (1957). According to his model, k is given by

$$k = G\left(\frac{b}{2\pi(1-\nu)}\right)^{1/2}$$

where G is the shear modulus, b Burgers' vector and ν Poisson's ratio. Taking $G = 3 \times 10^{10}$ dyn cm^{-2}, $b = 4\cdot5 \times 10^{-8}$ cm and $\nu = 0\cdot3$, then $k = 0\cdot9$ kg mm$^{-3/2}$, which is in fairly good agreement with the observed value of $k = 0\cdot3$.

When the stress–strain curve reaches its maximum value, the number of cracks increases suddenly, and at the same time some of the cracks propagate into the neighbouring grain (figure 6 (*e*) and (*f*)). This fact suggests that large and isolated piled-up groups of dislocations are formed during deformation, and supports the assumption that the theory of blocked dislocations can be applied to the yield behaviour of polycrystalline ice.

5. Conclusions

The following conclusions have been reached.

1. Surface imperfections introduced by mechanical polishing of single-crystal specimens can reduce the maximum yield point to half that of chemically polished specimens.

2. Fisher-type 'surface sources' of dislocations in a layer between 15 and 100 μm thick in mechanically polished single crystals can explain the observations.

3. The mechanically disturbed surface layer and sub-boundaries in single crystals do not act as barriers to the movement of dislocations, but rather as a source of dislocations.

4. Because surface sources as well as internal sources of dislocations play an important role in the plastic deformation of single crystals, the maximum stress for a constant rate of movement of the cross-head would depend not only on the quality of the crystals but also on the method of preparation of the specimens.

5. Mechanically disturbing the surface does not alter the value of the maximum stress for columnar-grained ice. This implies that grain boundaries are a prominent source of dislocations in the early stage of deformation.

6. Observations on crack formation and the dependence of the yield point on grain size are consistent with the theory of blocked dislocations.

Acknowledgments

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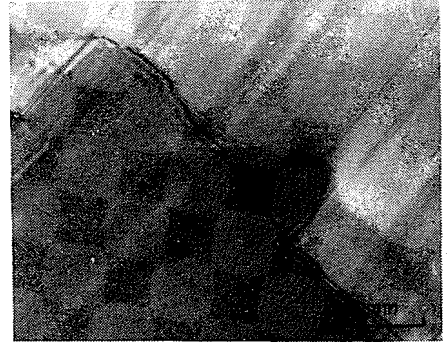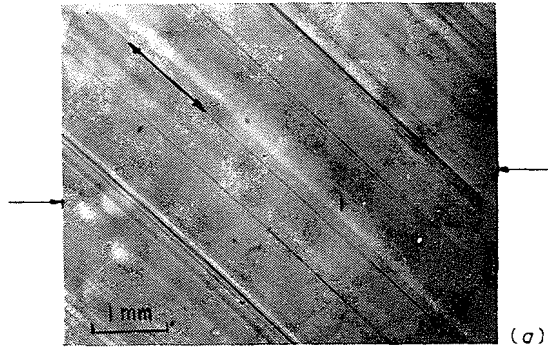STROH, A. N., 1957, *Adv. Phys.*, **6**, 418–65.

(a)

Figure 4. Well-developed slip bands in crystals with sub-boundaries after 5% strain (under crossed polaroids).
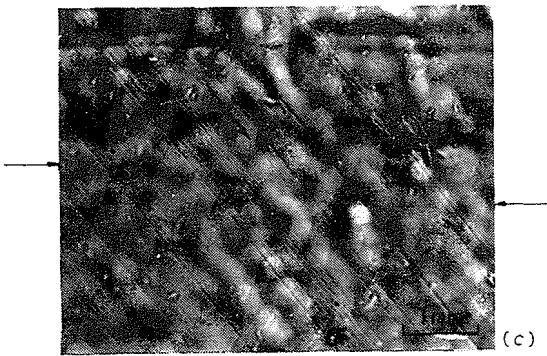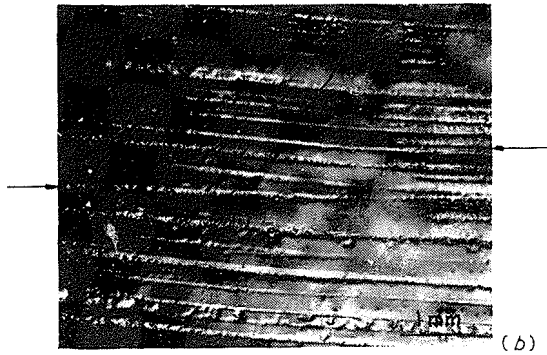

(b)

(c)

Figure 3. Appearance of the slip bands just after yielding: (a) chemically polished specimen; (b) mechanically polished specimen; (c) mechanically polished specimen annealed in kerosene bath at −3 °c for 120 hours. The direction of the slip lines is shown by the arrows on the photograph, and the direction of the stress by the arrows at the side.
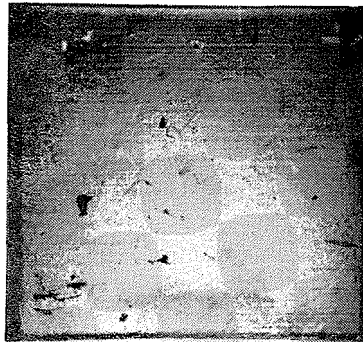
(a) Under crossed polaroids
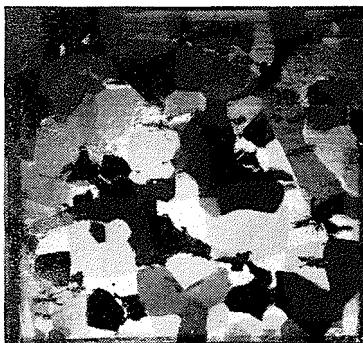
(b) Crack   1 cm
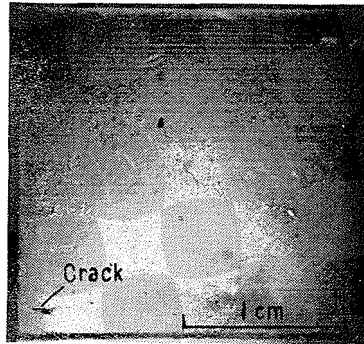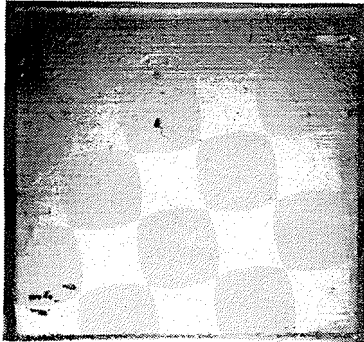
(c)

(d)

(e)

(f) Under crossed polaroids

Figure 6.   Successive photographs of crack formation in polycrystal of ice.   The deformation
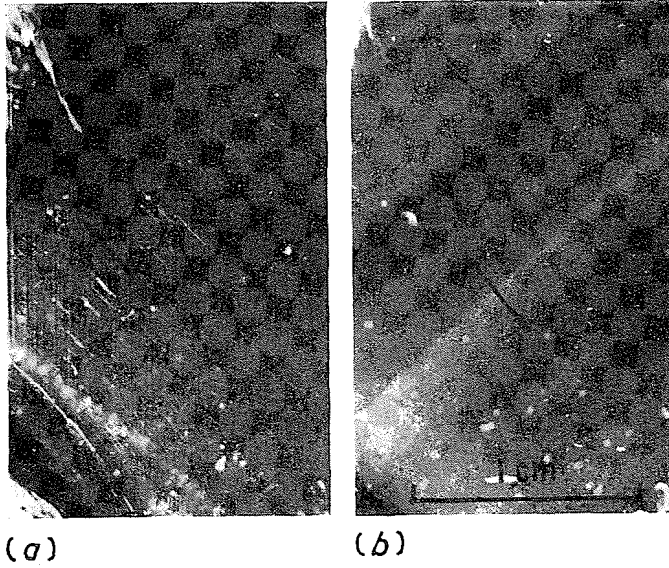at which the photographs were taken are indicated by arrows in figure 5.

(a) (b)

Figure 10. (a) Vapour figures produced by internal melting indicate the direction of slip planes. (b) Distinct deformation bands lying normal to the slip plane.   The double-headed arrows indicate the slip planes and the single-headed arrow the vapour figure.

Effet de L'Etat de la Surface de Cristaux de Glace sur leurs Proprietes Mécaniques

SOMMAIRE

L'auteur fait la description d'études comparatives du comportement de cristaux de glace polis par des moyens mécaniques ou chimiques, lorsqu'ils sont soumis à des efforts de déformation. Les mono-cristaux dont la couche superficielle a été polie mécaniquement ont une résistance maximale très réduite pour les glissements sur le plan de base, lorsqu'ils sont soumis à des efforts de déformation à vitesse constante. Les monocristaux possédant des sous-joints ne montrent pas d'effet de surface marqué mais ne possèdent qu'une faible résistance maximale. Les résultats indiquent que les couches superficielles et les sous-joints modifiés mécanique-ment n'agissent pas comme de puissantes barrières aux mouvements de dislocation des cristaux de glace mais plutot comme des sources de dislocations.

L'état de la surface ne semble pas affecter la résistance maximale des polycristaux. L'influence de la vitesse de déformation (de 2.1×10^{-5} a $1.7 \times 10^{-4}\,s^{-1}$) et de la température (de -10 a $-30^{o}C$) sur la résistance maximale a aussi été étudiée autant pour les monocristaux que pour les polycristaux de glace.