Intercrystalline cracking, grain-boundary sliding, and delayed elasticity at high temperatures

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The hypothesis of an interrelation between grain-boundary sliding and delayed elasticity in polycrystalline materials at high homologous temperatures is used to investigate the conditions conducive to microcracking. It is known that a material may exhibit cracking activity on attaining a critical delayed-elastic strain corresponding to a critical grain-boundary sliding displacement. Experimental data on ice at temperatures $> 0.9\,T_m$ are used to verify this concept. The new criterion is then extended to develop simple, self-consistent equations describing the interdependence of stress, strain, time, temperature, and grain size in predicting the onset of structural degradation due to microcracking and hence possible failure by fracture or rupture. The merit of the theory lies in its ability to forecast explicitly a large number of commonly observed high-temperature phenomena, including superplasticity, brittle–ductile transition, and the stress and temperature dependence of the apparent activation energy for fracture. One derivation makes it clear that cracking occurs when a critical stress depending only on temperature (and independent of grain size) is exceeded. The near constancy of fracture strain in the quasi brittle range can also be predicted

1. Introduction

Intergranular fracture in polycrystalline materials is commonly observed at homologous temperatures greater than about 0.3 to 0.4 T_m, where T_m is the melting point [1–4]. This mode of failure is characterized by low ductility, as measured by the strain at failure, and is often referred to as high-temperature brittleness. Initiation, growth and coalescence of intergranular cavitives or cracks are the three predominant stages leading to intergranular fracture at elevated temperatures [5–9]. Nucleation of cavities or cracks has frequently been observed at triple points, grain-boundary junctions and irregularities, or at inclusions on grain boundaries.

Several mechanisms have been proposed in the last four decades as the cause of various types of failure [10], many involving the phenomenon of grain-boundary shear or sliding [11–13]. An early proposal was that grain-boundary sliding results in elastic stress concentrations at grain interface junctions or triple points, producing wedge-type cracks [11]. Grain-boundary sliding in conjunction with intragranular slip, forming ledges or steps in grain boundaries, may also nucleate cavities [12, 13]. That it plays an important role in cracking activity during high-temperature deformation has already been established by Intrater and Machlin [14].

Sinha [15] has related the strain from grain-boundary sliding to delayed elastic effect. The present paper explores this possibility further and examines the link, if any, between microcracking at elevated temperature and delayed elasticity. The term "microcrack" is defined as an opening in the material, one dimension being much less than the other two, and the largest dimension comparable to the size of the grains of the material. There are three major points to keep in mind: (a) because of the limited number of slip systems in hexagonal materials such as ice, sliding and subsequent cracking are thought to play an important

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role in their deformation behaviour; (b) the transparency of ice and the usually large grain size facilitate direct visual observation of the formation of microcracks; (c) the fact that extremely high homologous temperatures can be reached with relative ease and that pertinent information is available makes ice an attractive material for study of the failure process.

To simplify analysis and subsequent presentation, discussion is restricted to the development of the first axial microcracks under uniaxial loading conditions and the beginning of cracking activity during constant load deformation. This article links microstructural observations to the larger scale manifestations of deformation behaviour and fracture. In this way the phenomenological approach that has been described as a "black box" by McLean *et al.* [16] may, for ice, be more of a "grey box."

2. Preliminary considerations

Where grain-boundary diffusion processes do not make a relatively significant contribution, grain-boundary sliding could result in elastic stress concentrations at grain-boundary junctions or triple points [17, 18]. This mechanism was originally proposed by Zener [11] to explain wedge-type cracks. Stroh [19] showed that the minimum shear stress, τ_m, needed to produce a crack at the end of a sliding interface of length L is

$$\tau_m = \left(\frac{12\gamma G}{\pi L}\right)^{1/2} \qquad (1)$$

where γ is the surface free energy and G the shear modulus. McLean [20] used the above relation for intercrystalline cracking by identifying L with the length of the grain boundary.

γ should actually be the effective fracture surface energy; and the appropriate energy to be used for boundary crack produced in a brittle manner, as pointed out by McLean [20], is the difference between the surface free energy with respect to vapour, γ_{vs}, and half of the grain-boundary free energy, γ_{gb}, giving

$$\tau_m = \frac{\sigma_m}{2} = \left[\frac{12G(\gamma_{vs} - \gamma_{gb}/2)}{\pi L}\right]^{1/2} \qquad (2)$$

where σ_m is the equivalent normal stress.

Usually Equation 1, known as the Stroh–McLean equation, is used because information on γ_{gb} is not available for most materials. There is experimental evidence for many materials of a critical stress below which cracks do not form readily and this supports the Stroh–McLean relation.

If for ice, $\gamma_{vs} = 0.109\,\mathrm{J\,m^{-2}}$, $\gamma_{gb} = 0.065\,\mathrm{J\,m^{-2}}$ at 0° C [21], and $G = 3.8\,\mathrm{GN\,m^{-2}}$ (derived from $G = E/2\ (1+\nu)$ using $E = 9.5\,\mathrm{GN\,m^{-2}}$ Poisson's ratio $\nu = 0.3$), then for $L = 4$ mm, Equation 2 gives $\tau_m = 0.53\,\mathrm{MN\,m^{-2}}$ or a uniaxial normal stress σ_m of $1\,\mathrm{MN\,m^{-2}}$. This agrees well with Gold's [22] observation of minimum stress of about $0.6\,\mathrm{MN\,m^{-2}}$ at which cracks form in uniaxial compressive creep for ice of similar average grain diameter at -5° C. γ_{gb} should, of course, depend on relative crystallographic orientation of the interfaces and should affect τ_m accordingly, but this information is not yet available.

Microseismic activities in ice during strength and deformation tests were correlated with cracking activity in ice by Sinha [23] using a locator system of acoustic emission. Following loading, there is a long period of silence before the initiation of cracking activity. The rise times of the discrete bursts of acoustic signals, corresponding to visible cracks, were very short, in the order of a few milliseconds. The cracks appear to form suddenly (to the naked eye) and grow to their full size in a very short time. Under these conditions, dissipation of energy due to local plasticity or creep at the crack tip may not be significant. If this is so, then the effective fracture surface energies for the formation of the cracks may not be different from the surface energy. Such a possibility is supported by measurements of fracture toughness (K_{Ic}) for polycrystalline ice by Goodman [24], who found that the strain energy release rate was not much greater than twice the surface energy for $\dot{K}_{Ic} > 600\,\mathrm{kN\,m^{-3/2}\,sec^{-1}}$ or fracture times less than about 20 sec. This is more than a thousand times larger than the rise times of the visible cracks described above. It may explain the fair agreement between the experimental observation of Gold [22] and the calculations shown above. The disadvantage of Equations 1 or 2 is that neither gives information on time of fracture.

The model of a wedge crack formed by dislocations on two intersecting planes, proposed by Cottrell [25], describes the stable crack length, a, normal, to the applied stress as

$$a = \frac{4G\gamma}{\pi(1-\nu)\sigma^2}\left[1 - \frac{\sigma w}{4\gamma} - \left(1 - \frac{\sigma w}{2\gamma}\right)^{1/2}\right]. \qquad (3)$$

The condition for instability is given by

$$\sigma w \geqslant 2\gamma, \quad (4)$$

where σ is the applied stress and w is the wedge displacement or opening. A crack will therefore grow in a stable manner until w reaches a critical value,

$$w' = 2\gamma/\sigma. \quad (5)$$

Williams [26] suggested the use of Equation 5 for intercrystalline cracks. Using an aluminium–20% zinc alloy, grain size of 1 mm at 200° C, he noticed that a triple-point crack grew steadily at first and then accelerated in an unstable manner before it reached the adjacent grain-boundary junction. Williams estimated γ from Equation 5 and the observed values of w', and obtained a consistent value nearly independent of applied stress. He also measured the sliding of the grain boundaries, x, directly associated with the wedges in question and noticed a direct relation between x and wedge height

$$x = w. \quad (6)$$

Williams' observations bear close resemblance to those of Gold [22] and Sinha [23] on the nature of the formations of cracks. The long period of silence noted in ice experiments before the initiation of cracks large enough to be visible to the naked eye could be linked to the period before unstable crack growth occurred in Williams' experiments. This similarity, together with the important observations given in Equation 6, encourages one to explore the possibilities of the following analysis.

From Equations 5 and 6 and replacing γ by $(\gamma_{vs} - \gamma_{gb}/2)$, the critical grain-boundary sliding for instability of intergranular crack is

$$x' = 2(\gamma_{vs} - \gamma_{gb}/2)/\sigma. \quad (7)$$

The strain, ϵ_{gbs}, induced by grain-boundary sliding (gbs) is usually given by [2, 27]

$$\epsilon_{gbs} = K\bar{x}/d \quad (8)$$

in which $\bar{x}$ is the average grain-boundary displacement, d the average grain diameter, and K the averaging factor nearly equal to unity.

If $\bar{x}'$ is the average critical gbs for instability of cracks, then from Equation 8 the corresponding critical ϵ'_{gbs} is

$$\epsilon'_{gbs} = K\bar{x}'/d. \quad (9)$$

From Equations 7 and 9,

$$\epsilon'_{gbs} = 2K(\gamma_{vs} - \gamma_{gb}/2)/\sigma d. \quad (10)$$

TABLE I Creep parameters for ice obtained independently from earlier creep experiments and other necessary information

E	$= 9.5\,\mathrm{GN\,m^{-2}}$; $G = 3.8\,\mathrm{GN\,m^{-2}}$
Q	$= 67\,\mathrm{kJ\,mol^{-1}}$ ($16\,\mathrm{kcal\,mol^{-1}}$)
c_1	$= 9$
d_1	$= 1\,\mathrm{mm}$
s	$= 1$
n	$= 3$
b	$= 0.34$
a_T	$(T = 263\,\mathrm{K}) = 2.5 \times 10^{-4}\,\mathrm{sec^{-1}}$
$\dot{\epsilon}_{v_1}$	$= 1.76 \times 10^{-7}\,\mathrm{sec^{-1}}$; $\sigma_1 = 1\,\mathrm{MN\,m^{-2}}$, $T = 263\,\mathrm{K}$
M_1'	$= (1.67 \pm 0.10)10^{-3}$
m_1	$= (4.55 \pm 0.41)10^{-6}\,\mathrm{K^{-1}}$
γ_{vs}	$= 0.109\,\mathrm{J\,m^{-2}}$ at 273 K
γ_{gb}	$= 0.065\,\mathrm{J\,m^{-2}}$ at 273 K

Equation 2 gives τ_m or σ_m in terms of L or grain size, assuming L is equivalent to grain diameter, d. Equation 10, on the other hand, gives ϵ'_{gbs} in terms of stress and grain size. Equation 10 and Table I give, as an example, $\epsilon'_{gbs} = 3.8 \times 10^{-5}$ for $\sigma = 1\,\mathrm{MN\,m^{-2}}$ at 0° C and $d = 4\,\mathrm{mm}$. This, according to Equation 9, gives $\bar{x}' = 0.15\,\mu\mathrm{m}$. Although Equations 9 and 10 do not give any information on time of cracking, it is inherent in the derivation that an incubation time is involved before the critical gbs displacement or strain is reached.

Sliding is a complex process depending markedly on external conditions of stress and temperature and on internal properties such as the crystalline structure of the matrix and the defects, fabric of the material, grain size and its distribution, impurities in the material and inclusions at the grain boundaries. As resistance to boundary sliding increases with the onset of serrations in the grain boundaries, i.e. because of intragranular slip, the duration of load application and hence the total deformation also play an important role. So far, there is no substantive evidence in the literature that there is a critical gbs strain for the initiation of cracking activity, but the above analysis gives some incentive to examine the possibility. An indirect approach will be taken, but first a series of experiments and the conclusions drawn from them will be described.

3. Experimental details

Using large transparent specimens (5 cm × 10 cm × 25 cm) of transversely isotropic, columnar-grained ice subjected to a constant compressive load applied perpendicular to the long direction of the grains, Gold [28] noted the formation of the first

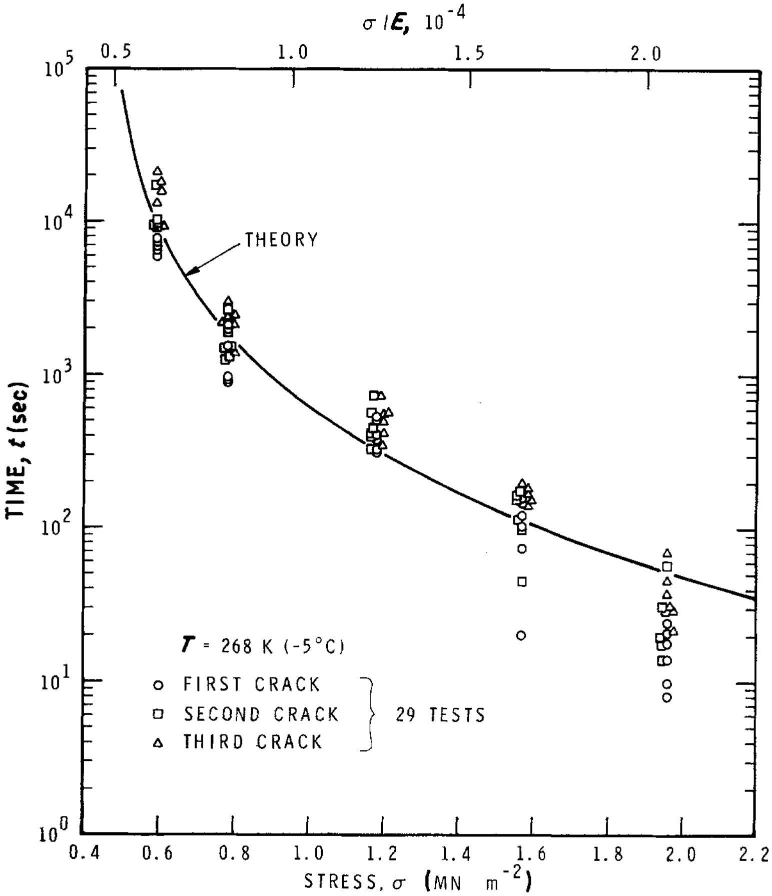

Figure 1 Dependence of time to the formation of first cracks on stress, $T = 268$ K ($-5°$ C). Experimental results are from Gold [28, 29].

large cracks at -10 and $-31°$ C to be a reasonably well defined event in previously undeformed specimens. As formation of cracks was observed visually in a cold room, it was decided to record the time of formation of the first three cracks to obtain a better measure of the beginning of cracking activity; further experiments were carried out at -5, -10, -15 and $-31°$ C [22, 29]. These cracks were said to be greater than 1 to 2 mm wide and 2 cm long. The cross-sectional diameters of the grains perpendicular to their long axis were reported by Gold to be in the range of 1 to 6 mm, the longer dimensions of the cracks being parallel to the axis of the columnar grains. The cracks therefore fall into the category of "microcracks," defined earlier. Experimental results are shown in Figs. 1 to 4, and are similar to the Zhurkov type dependence of tensile failure time on stress for metals and alloys [30]. Gold [22, 29] noted this similarity and presented the dependence of first crack time, t_f, on stress σ as

$$t_f = t_0 \exp\left(\frac{Q_f - \alpha\sigma}{kT}\right) = A(T) \exp(-B\alpha\sigma) \tag{11}$$

where t_0, A, B and α are constants and Q_f is the apparent activation energy for crack formation at zero stress. The temperature is T (Kelvin) and the Boltzmann's constant, k. Considerable deviation from the above relations was noted for the results at the lower end of the stress and temperature ranges. Cracks were noted to form only when stress exceeded a limiting level of about 0.6 MN m^{-2}.

Gold [22, 29] discussed the nucleation of the cracks on the basis of stress concentrations produced by pile-up of dislocations against grain boundaries. Using a relation proposed by Smith and Barnby [31] he obtained good agreement between the computed minimum stress for crack initiation and experimental observations. To obtain agreement in predicted strain, however, it was necessary to take into account the fact that stress must exceed a critical value before cracks form. Gold [29] also pointed out that the deviation from that predicted by the simple dislocation model became significant the lower the stress and temperature. It should be remembered that the observed minimum stress for cracking can also be predicted by the Stroh–McLean

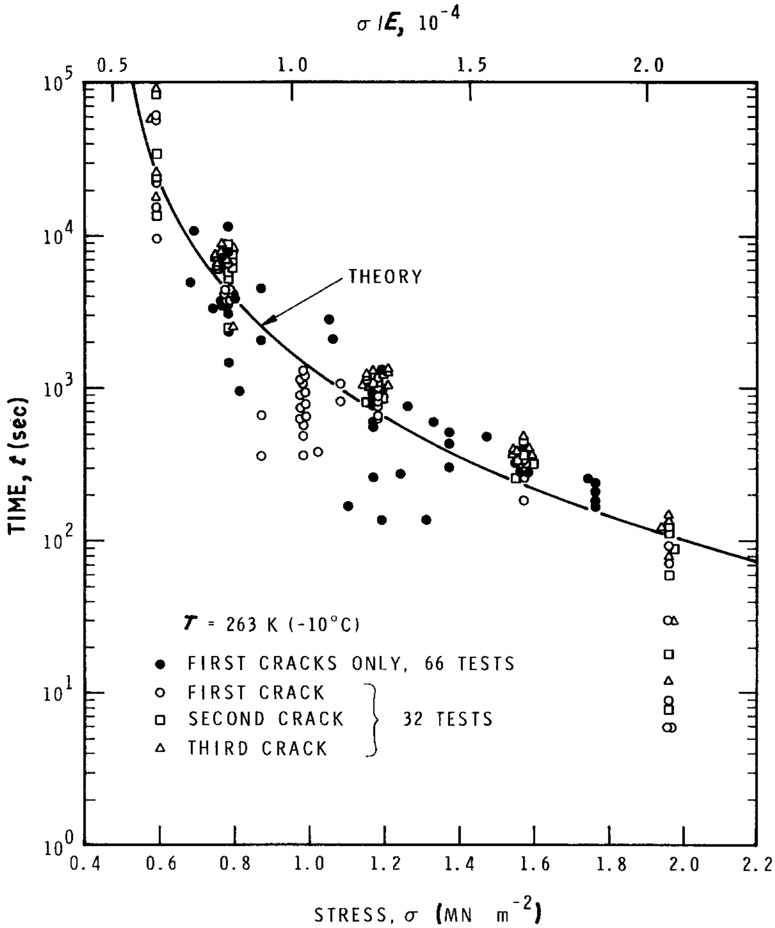

Figure 2 Dependence of time to the formation of first cracks on stress, $T = 263$ K ($-10°$ C). Experimental results are from Gold [28, 29].

equation, so that the relative importance of the two mechanisms of crack initiation is not clear.

3.1. Microstructural analysis

A few creep experiments were conducted on large specimens (5 cm x 10 cm x 25 cm) of columnar-grained ice following procedures already described [32]. The ice was completely transparent, with a density of 917.7 kg m^{-3} (at $-9°$ C). The specimens were unloaded as soon as possible after the formation of the first large cracks completely inside the specimen (i.e. cracks that did not propagate to the specimen surface). This was to ensure that the observations would be made on cracks not produced by surface effect. The specimens were then sectioned through the crack, perpendicular to it, and large replicas (10 cm x 10 cm) were made by the method described earlier [33].

A particularly interesting intercrystalline crack is shown in Fig. 5. It and a boundary associated with it were nearly parallel to the applied compressive load axis. Extension of the crack inside the neighbouring grains was mainly caused by the compatibility of the crystallographic axes of these grains with respect to applied load and crack propagation. The crack propagated in a direction parallel to the basal plane in the grain, on the left side (as indicated by the dislocation etch pits), but it extended in the plane parallel to the [0 0 0 1] axis on the right. These preferences of planes for crack propagation are consistent with those observed by Gold [34, 35]. The absence of any pattern in the distribution of dislocations in the crack tip area, as indicated by the elongated etch pits, seems to provide yet another example of cracking without a damage zone [36].

As the chosen loads were sufficient to produce cracks during this series of creep tests, it would be expected that various stages of pile-up would be apparent. The absence of any such dislocation pile-ups around the cracks in hundreds of grains is evidence that first cracks may not be produced by the stress concentration resulting from pile-up.

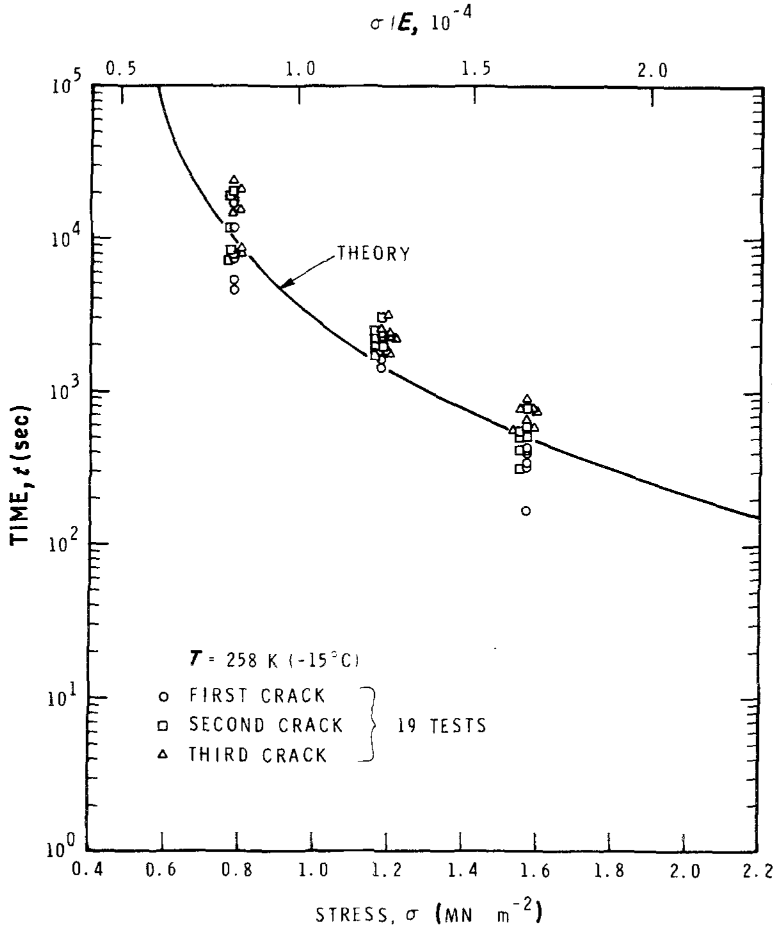

Figure 3 Dependence of time to the formation of first cracks on stress, $T = 258$ K ($-15°$ C). Experimental results are from Gold [28, 29].

3.2. Critical delayed elastic criterion

A non-linear viscoelastic model incorporating grain-size effect was proposed by Sinha [15] to describe the initial high-temperature creep of polycrystalline materials. The recoverable portion of the creep strain was assumed to be a delayed elastic effect associated with grain-boundary sliding. Assuming, for simplicity, that delayed elastic strain (des), ϵ_d, is related to gbs strain by

$$\epsilon_d = \epsilon_{gbs}, \tag{12}$$

it was shown that

$$\epsilon_d = c_1 \left(\frac{d_1}{d}\right)\left(\frac{\sigma}{E}\right)^s \{1 - \exp\left[-(a_T t)^b\right]\} \tag{13}$$

where E is Young's modulus, t is time, d is grain size, and b and s are temperature independent constants. The constant c_1 corresponds to the unit or reference grain size d_1. The modulus, E, associated with primary bond distortion or average of lattice deformation was found to be practically independent of time and only slightly dependent on temperature [32]. Temperature dependence of delayed elasticity was found to be governed by the inverse relaxation time, a_T, given by

$$a_{T_2} = a_{T_1} \exp\left[\frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right)\right] \tag{14}$$

where Q and R are activation energy and gas constant, respectively, and T_1 and T_2 are temperatures in Kelvin.

Equations 13 and 14 have a number of empirical constants. One can raise the objection that suitable numerical values can be assigned to fit any result. It was decided, therefore, to use values determined from a single set of constant load creep tests on ice of a known grain size [32] in applying the equations to Gold's [22, 28, 29] results for the present analyses (to be discussed later). These values were also used in examining the formulations developed to predict the deformation characteristics under widely varying, monotonically increasing stress history during testing

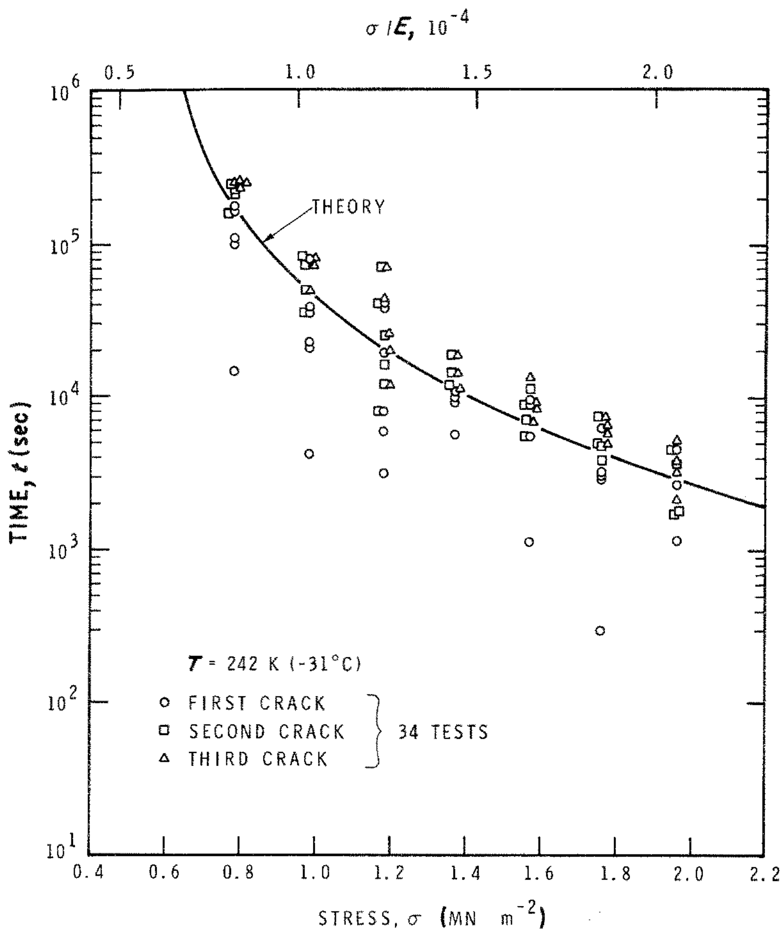

Figure 4 Dependence of time to the formation of first cracks on stress, $T = 242$ K ($-31°$ C). Experimental results are from Gold [28, 29].

for strength of ice at different temperatures [37] and conducted with different test machines in different laboratories [38]. The good agreement in all these cases supports the creep model and the concept behind it. The values of the quantities are given in Table I and have been tabulated in this form also by Sinha [15].

If there is a critical gbs strain for initiation of cracking activity and the assumption expressed by Equation 12 is not far from reality, one would expect a critical delayed elastic strain (des) for cracking. This hypothesis can be examined by applying Equation 13 to all the experimental data in Figs. 1 to 4 and computing the corresponding ϵ_d.

Results shown in Figs. 6 to 9 are calculated on the basis of Table I, assuming (to be clarified later) grain size $d = 4.5$ mm. The figures show clearly that the first large cracks form when (in contradiction of the stress dependency expected from Equation 10) delayed elastic strain reaches a critical value, irrespective of the applied level of stress in the load range studied. Although the scatter is large (± 10%), the average critical des, ϵ_d^c, for the formation of first cracks increases almost linearly with decrease in temperature (Fig. 10) and may be written as

$$\epsilon_d^c = M_d - m_d T \tag{15}$$

where m_d is slope and M_d the critical des at absolute zero. Regressional analysis of all the data points gave M_d ($d = 4.5$ mm) $= (3.70 \pm 0.23) \times 10^{-4}$ and $m_d = (1.01 \pm 0.09) \times 10^{-6}\,K^{-1}$. Convenient alternative forms of Equation 15, with the above values for slope and intercept, are shown in Fig. 10. It is understood, however, that the relation is applicable only for temperatures a few degrees below the freezing point.

As the calculations for ϵ_d^c were made with assumed d of 4.5 mm, the values of M_d and m_d are appropriate for this grain size. The numerical value of ϵ_d^c for the reference grain size of d_1 (= 1 mm) will be given, by virtue of inverse proportionality of des on grain size in Equation 13, as

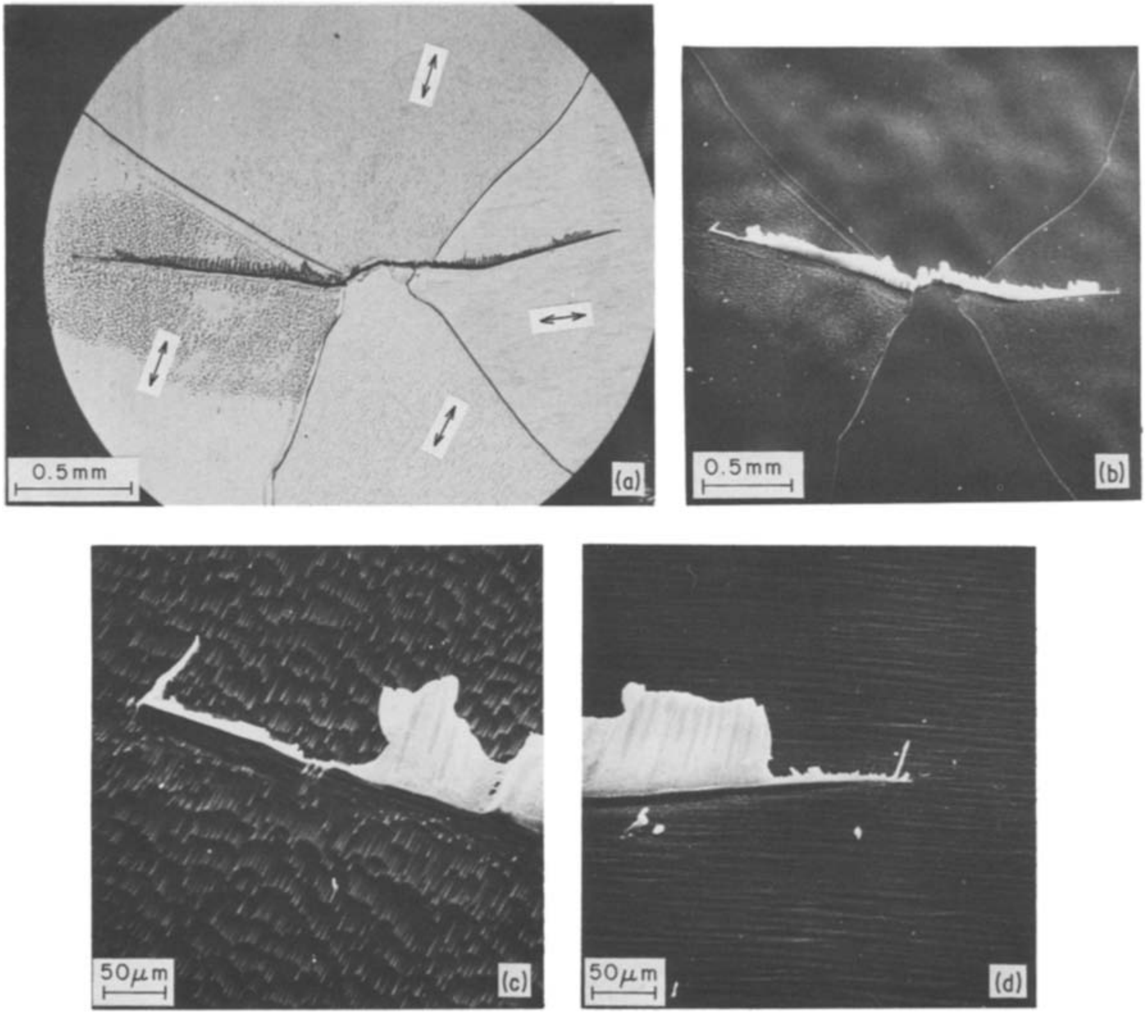

Figure 5 First crack produced in ice at $-10°$ C under a stress of 1 MN m^{-2}. (a) optical micrograph of the replica. Arrows indicate direction of [0 0 0 1] axis. (b) Scanning electron micrograph of the same replica. (c) and (d) SEM of the crack tip areas.

$$\epsilon^{c}_{d_1} = \epsilon^{c}_{d} d/d_1 = (M_1 - m_1 T) = (M_d - m_d T)d/d_1 \qquad (16)$$

so that

$$M_1 = M_d d/d_1 \quad \text{and} \quad m_1 = m_d d/d_1$$

or

$$M_1 = (1.67 \pm 0.10) \times 10^{-3}$$

and

$$m_1 = (4.55 \pm 0.41) \times 10^{-6}\,\text{K}^{-1}.$$

Equations 8, 12, 15 and 16 give the critical grain boundary displacement for the first cracks as

$$\bar{x}_c = (M_d - m_d T)d/K = (M_1 - m_1 T)d_1/K. \qquad (17)$$

The critical sliding, therefore, is independent of grain size and stress level.

Equation 15 gives ϵ^c_d of 9.5×10^{-5} at 0° C for $d = 4.5$ mm; correspondingly $\bar{x}_c$, according to Equation 17 and with $K = 1$, is 0.43 μm. These numerical values are, incidentally, comparable to the values of ϵ'_{gbs} ($= 3.8 \times 10^{-5}$) and x' (= 0.15 μm) calculated earlier from Equations 10 and 9, respectively.

3.3. Critical time for fracture

If t_{fc} is the time for the formation of first cracks, then the application of the concept of critical des criteria to Equation 13 and readjustment gives

$$t_{fc} = a_T^{-1}\left\{-\ln\left[1 - \frac{\epsilon^c_d}{c_1}\left(\frac{d}{d_1}\right)\left(\frac{E}{\sigma}\right)^s\right]\right\}^{1/b}. \qquad (18)$$

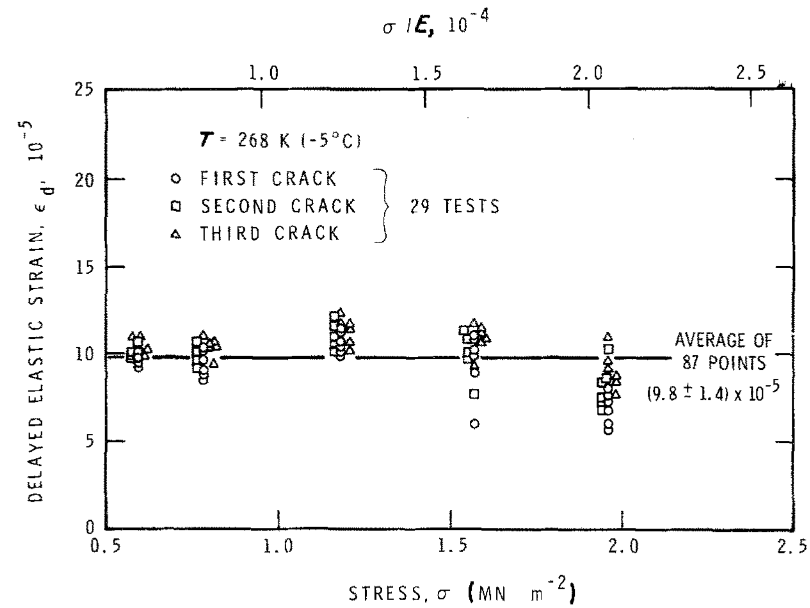

Figure 6 Computed delayed elastic strain for the formation of first three large cracks during creep in S-2 ice of average grain diameter of 4.5 mm at 268 K (– 5° C).

From Equations 16 and 18

$$t_{fc} = a_T^{-1}\left\{-\ln\left[1-\frac{M_1-m_1T}{c_1}\left(\frac{E}{\sigma}\right)^s\right]\right\}^{1/b}. \tag{19}$$

This is independent of grain size. The arbitrary choice made earlier for $d = 4.5$ mm did not, therefore, affect the calculations and subsequent estimation of M_1 and m_1. This choice was made because of the extensive grain size determinations carried out by the author during strength tests [39] on ice produced essentially by the method used by Gold [29]. Most of the ice had average cross-sectional grain diameters in the range of 4 to 5 mm.

Dependence of t_{fc} on stress and temperature,

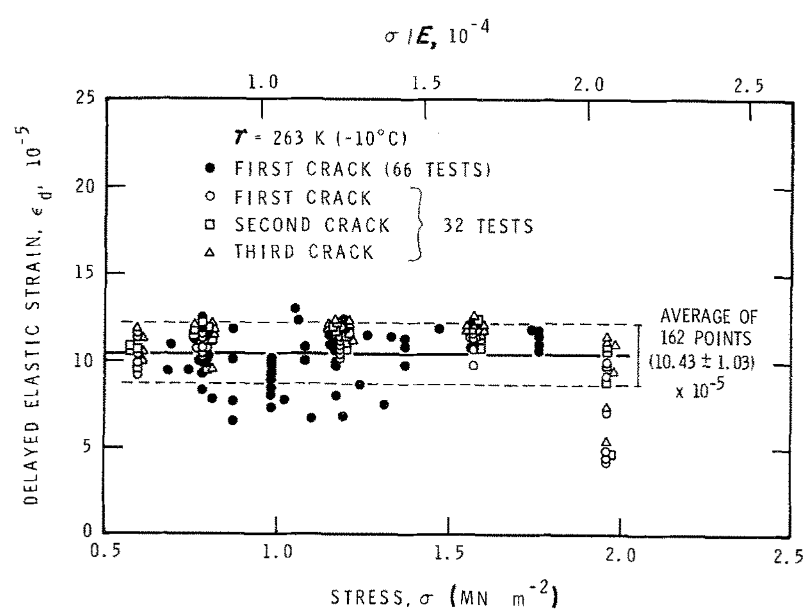

Figure 7 Computed delayed elastic strain for the formation of first and first three large cracks in S-2 ice of average grain diameter of 4.5 mm at 263 K (– 10° C).

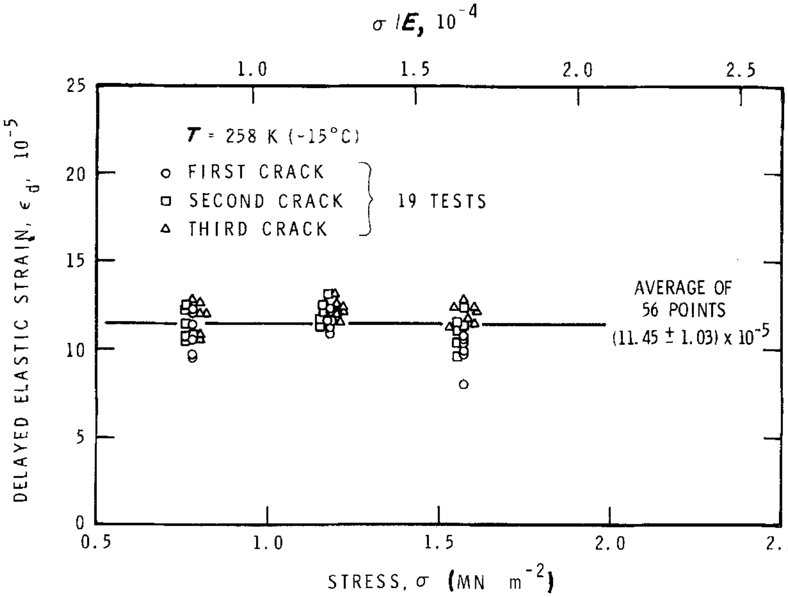

Figure 8 Computed delayed elastic strain for the formation of first three large cracks in S-2 ice of average grain diameter of 4.5 mm at 258 K (– 15° C).

calculated on the basis of Equation 19 and Table I, is shown in Figs. 1 to 4. These calculations seem to describe the experimental results better than the straight lines, on log–linear plot, given by expressions of the type of Equation 11, used by Gold [22, 29] and common in describing tensile failures in metals and alloys. The applicability of the present formulation, particularly at the lower end of the stress level, is of considerable interest because of the possibility of using it to predict a minimum stress for cracking and failure by fracture and therefore for the condition for superplasticity [40].

3.4. Minimum stress for fracture

The minimum stress level, σ_{min}, required for cracking activity is the level of load for which

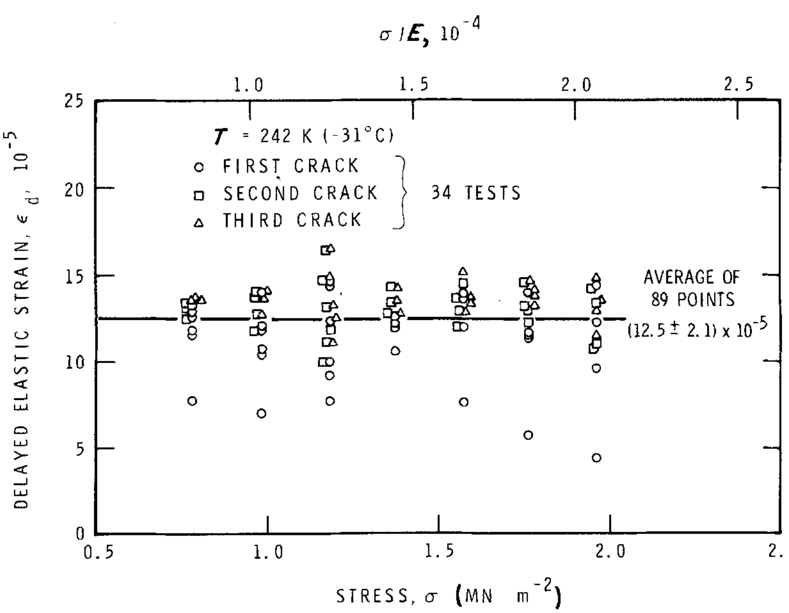

Figure 9 Computed delayed elastic strain for the formation of first three large cracks in S-2 ice of average grain diameter of 4.5 mm at 242 K (– 31° C).

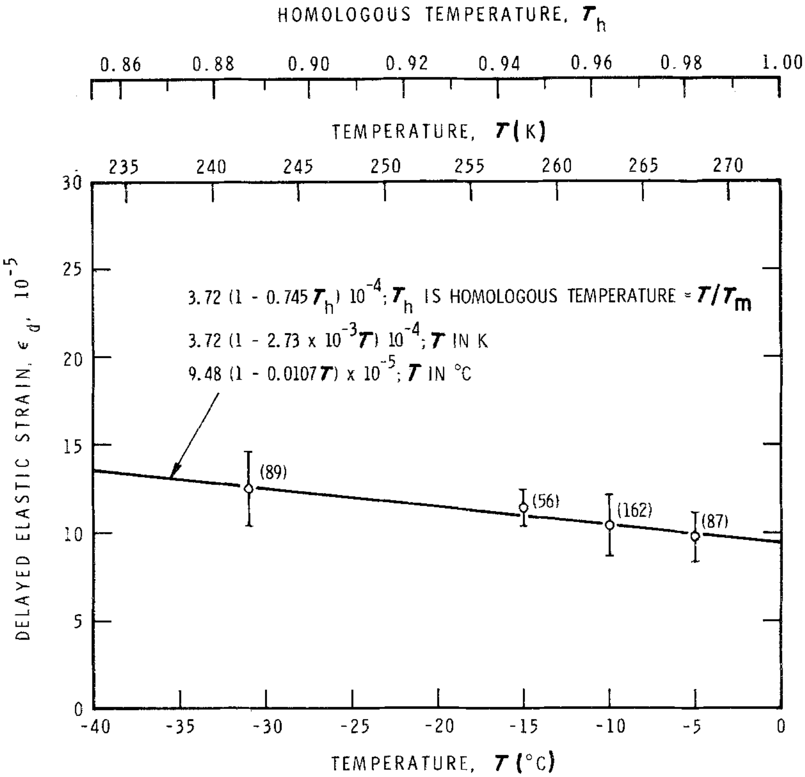

Figure 10 Temperature dependence of critical delayed strain for the formation of first large cracks in S-2 ice of average grain diameter of 4.5 mm (numbers in parenthesis indicate number of data points).

$t_{fc} = \infty$. Equation 19 gives, for this condition,

$$\sigma_{\min} = E\left(\frac{M_1 - m_1 T}{c_1}\right)^{1/s}. \qquad (20)$$

This indicates that $\sigma_{\min}$ is sensitive to temperature but independent of grain size. Elastic moduli for single crystal ice increase about 5% in the temperature range 0 to $-30°$ C [41], indicative of the additional influence of temperature on $\sigma_{\min}$ through the temperature dependence of the elastic modulus, E. According to Equation 13, increase in E with decrease in temperature will decrease M_1 and m_1, counteracting the effect of the temperature dependence of E on $\sigma_{\min}$. The temperature dependence of E (if known) must be taken into account in the calculations.

Equation 20, with information in Table I, gives $\sigma_{\min}$ of $0.47\,\text{MN m}^{-2}$ ($\sigma_{\min}/E = 4.9 \times 10^{-5}$) and $0.59\,\text{MN m}^{-2}$ ($\sigma_{\min}/E = 6.2 \times 10^{-5}$) at -5 and $-30°$ C, respectively. This explains why Gold [28, 29] did not observe cracking below $0.5\,\text{MN m}^{-2}$, but did observe cracking activity in ice within the experimental times used for stresses at or greater than about $0.6\,\text{MN m}^{-2}$. It is interesting that Zaretsky *et al.* [42] estimated this minimum stress to be $0.5\,\text{MN m}^{-2}$ in the temperature range -4 to $-13°$ C for columnar-grained ice containing grains varying in size from 2 to 12 mm. They used the technique of acoustic emission for detecting cracking activity and called the minimum stress the "creep limit", meaning that for stresses higher than this level creep develops to an accelerating stage (commonly known as the tertiary stage), whereas for lower stresses creep rate tends to a limiting or steady-state value.

The rate sensitivity of the level of stress for the onset of cracking activity in transparent ice has been examined by Sinha [23], who did not observe any effect of grain size in the range of 2 to 5 mm. Strongest support for Equation 20, which indicates that $\sigma_{\min}$ is sensitive to temperature but independent of grain size, comes from Currier [43]. When he conducted tensile strength tests on granular ice with average grain size varying from 1 to 7.3 mm at $-10°$ C, he observed no grain size effect on the level of stress at onset of acoustic emission. Furthermore, the stress level was 0.44 ± 0.17 MN m^{-2} for average strain rates during testing in the range of about $1 \times 10^{-6}\,\text{sec}^{-1}$.

Eliminating E from Equation 19 by substituting its value from Equation 20,

$$t_{fc} = a_T^{-1} \left\{ -\ln \left[1 - \left(\frac{\sigma_{min}}{\sigma} \right)^s \right] \right\}^{1/b}. \quad (21)$$

Equation 21 readily shows whether a given stress will produce any cracking activity during a given period of loading if stress exceeds σ_{min}. This may be preferable to Equation 19 in presenting the dependence of t_{fc} on σ.

3.5. Strain at first crack

Total strain, ϵ_{fc} at the time of formation of first cracks, t_{fc}, will be given by

$$\epsilon_{fc} = \frac{\sigma}{E} + \epsilon_d^c + \dot{\epsilon}_{v_1} \left(\frac{\sigma}{\sigma_1} \right)^n t_{fc} \quad (22)$$

where the first term is the elastic strain ϵ_e, the second the critical des, and the third describes the viscous component, ϵ_v. This is according to the three-component creep model described in detail by Sinha [15]. In this, $\dot{\epsilon}_{v_1}$ is the viscous creep rate corresponding to the unit or reference stress σ_1, and n is the stress exponent. Activation energy for viscous creep rate was found to be the same as that of des [32], and its temperature variation is also described by Equation 14, with $\dot{\epsilon}_{v_1}$ replacing a_T. Values obtained for $\dot{\epsilon}_{v_1}$ and n from previous experiments and discussed by Sinha [15] are given in Table I.

Viscous creep rate was assumed to be independent of grain size [15] for conditions where the intergranular accommodation is not governed by the grain-boundary diffusional process and where the microstructure has not deteriorated because of voids, cracks or recrystallization, that is, small strain. The insensitivity of viscous strain to grain size of 1 to 10 mm of ice has since been reported by Duval and LeGac [44]. Under these conditions the grain-size influence on fracture strain is determined mainly by the dependence of ϵ_d^c on d.

Calculations based on Equation 22 are shown in Figs. 11a and b for grain size of 4.5 mm. Finer grained ice would have a larger contribution from ϵ_d^c and the curves would be shifted in the direction of larger strain. Sharp decrease in total strain is mainly caused by decrease in viscous strain (Fig. 11a). As the viscous strain approaches a negligible value and the elastic strain increases with stress, the predicted total strain actually goes through a minimum (as may be seen in Fig. 11a), although it might appear to reach a constant value (Fig. 11b). The number of experimental results shown are not considered significant, but trends in the theoretical prediction agree well with measurements. Fig. 11a indicates that cracking activity involves some energy dissipation through viscous flow even at the highest stress level examined because of small but finite values (about 1×10^{-4}) for viscous strain.

4. Discussion

Formation of the first crack in compression does not determine the ultimate failure strength. It indicates, however, the beginning of internal damage that might lead to cleavage failure in tension if the applied load is sufficient to propagate the crack beyond the boundaries of the grains. An estimation of the applied load for such failures was made by Gold [45] using Griffith's criterion

$$\sigma_f = \left(\frac{2E\gamma_{vs}}{\pi l} \right)^{1/2} \quad (23)$$

where $2l$ is the crack length.

Using the values for γ_{vs} and E in Table I and crack length of 1 mm (the width reported by Gold [28, 29], $\sigma_f = 1.2\,\mathrm{MN\,m^{-2}}$ ($\sigma_f/E = 1.2 \times 10^{-4}$). This level of stress may therefore be sufficient to initiate a crack in 10 min at $-10°$ C (Fig. 2) and propagate it to failure if applied in tensile mode at the high temperatures considered. No tensile creep rupture data on columnar-grained ice are available for examining this aspect of the analysis. Burdick's observations [46] on randomly oriented, fine-grained ice conform well, however, with the above predictions. He examined the tensile creep failure of dumb-bell-shaped snow ice with average grain diameter of 0.7 mm at $-9°$ C ($0.97\,T_m$) in the stress range of 0.7 to $1.56\,\mathrm{MN\,m^{-2}}$. He noted a specimen with an initial stress of $0.7\,\mathrm{MN\,m^{-2}}$ that lasted up to four days, whereas specimens with initial stress of $1.56\,\mathrm{MN\,m^{-2}}$ fractured in less than a minute. Burdick stated that no specimens with an initial stress of less than $1.2\,\mathrm{MN\,m^{-2}}$ failed before 100 min and that some were eventually extended to 100% of their original length. He also noted that "visual inspection did not reveal any significant flaws in the early failure samples", which may be interpreted as evidence that the first few cracks led to failure.

According to the calculations in Fig. 1, a stress level of $1.2\,\mathrm{MN\,m^{-2}}$ generates a crack at $t_{fc} = 3 \times 10^2$ sec for $-5°$ C and $\epsilon_{fc} = 3.9 \times 10^{-4}$ (Fig. 11) for the coarse grain size of 4.5 mm. The average

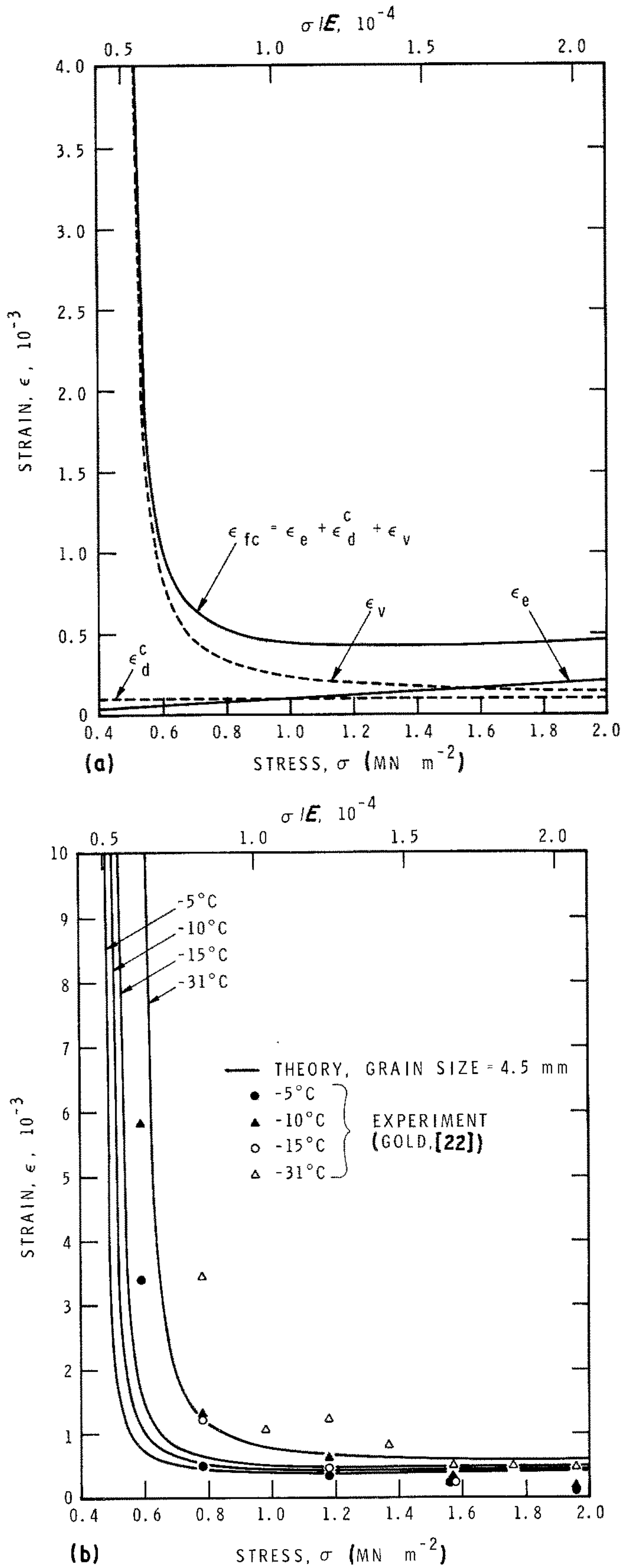

Figure 11 (a) Theoretical total strain and its three components for the formation of the first cracks at 263 K (− 10° C) for grain diameter of 4.5 mm. (b) Stress and temperature dependence of creep strain to the formation of the first large cracks.

strain rate to fracture is $\dot{\epsilon}_{fc} = \epsilon_{fc}/t_{fc} = 1.3 \times 10^{-6}$ sec^{-1}. Fig. 11 shows that higher stresses do not affect ϵ_{fc} appreciably, although corresponding reduction in t_{fc} would greatly increase $\dot{\epsilon}_{fc}$. Finer grain size would result in a larger contribution of delayed elastic strain to the total strain and would tend to increase $\dot{\epsilon}_{fc}$ further. Calculations give, for example, $\epsilon_{fc} = 8.6 \times 10^{-4}$ and $t_{fc} = 48$ sec (Fig. 1) for the initial stress of 2 MN m^{-2} at $-5°$ C for the grain size of 0.7 mm, giving $\dot{\epsilon}_{fc} = 1.8 \times 10^{-5}$ sec^{-1}.

Strain-rate sensitivity, or rather relative insensitivity, of tensile strength of coarse-grained (3 to 4 mm) columnar-grained ice observed by Carter and Michel [47] and of fine-grained (0.7 mm) granular ice examined at high strain rates by Hawkes and Mellor [48] corroborates these values. One example from the latter investigation is an average tensile strength of 2.1 MN m^{-2} (ten specimens) tested under a reported average strain rate to peak stress of 1.3×10^{-5} sec^{-1}. The samples failed at an average fracture time of 49.7 sec, with an average failure strain of 6.6×10^{-4}. These tests were carried out on dumb-bell-shaped specimens at $-7°$ C under constant crosshead rates using a conventional screw-driven machine. The observations compare favourably with calculations given in the previous paragraph.

A few aspects of the calculations shown in Fig. 11 are worth mentioning.

1. For a given temperature and grain size, ϵ_{fc} decreases sharply over a narrow range of stress, indicating an apparent transition from ductile to so-called "brittle" behaviour (the term commonly, and often ambiguously, used for failures at low strains).

2. The transition stress range and corresponding range of strain decrease with increase in temperature, indicating a shift in apparent brittleness (usually with respect to failure strain) to lower stress and higher temperatures.

3. Not shown but visualized is the increase in strain (particularly noticeable at higher stresses) with decrease in grain size, an indication of increasing brittleness with increasing grain size.

4. There is a level of stress below which the material will flow to a large strain without producing intergranular cracks by the process discussed here. The conditions favourable for this "stress controlled" superplastic flow are high temperatures and low stresses, as illustrated, and fine grain sizes.

5. At stresses greater than the transition range, the strain to the formation of the first crack approaches nearly constant value, and for this condition "strain-controlled" fracture may appear to be nearly independent of temperature in a small range of temperatures.

These observations have actually been made in polycrystalline materials in general, and failures have been characterized as "cracking" or "rupture" processes [49]; they could be described as "strain controlled" or "stress controlled".

A quick survey of fracture maps compiled by Ashby *et al.* [10] and Gandhi and Ashby [50] reveals that failure by intergranular cracking commonly occurs above $\sigma/E = 5 \times 10^{-5}$ and is therefore similar to that in ice. At these low stress levels (still too high for pure diffusional creep to dominate) formation of several cracks or some degree of damage may be required before a specimen will fail in tension. Nonetheless, failure strain, as indicated in Fig. 11, could be large, consisting mainly of viscous strain, i.e. the contribution of the third term in Equation 22, as can be seen in Fig. 11a. Phenomenologically, one might conclude that such failures are controlled by power-law creep. Relations of the type, $t_f \dot{\epsilon}_v$ = constant, where t_f is failure time and $\dot{\epsilon}_v$ viscous creep rate, are common [8]. Another important aspect to be noted from Fig. 11a is the significant contribution of delayed elastic strain and viscous strain to total strain at or higher than the so-called ductile–brittle transition stress range. This points out that such failures do not indicate pure elastic loading conditions and hence might be far from truly brittle failures.

The present discussion would be incomplete if other derivations from the present formulations on the subject of activation energy for fracture or of deformation processes involving cracks were not included. Suppose t_{fc}^1 and t_{fc}^2 are the times for the first cracks at temperatures T_1 and T_2, respectively, for a given stress σ. The apparent activation energy, Q_{fc}, for the first cracks will be given by;

$$Q_{fc} = \frac{R}{[(1/T_1) - (1/T_2)]} \ln\left(\frac{t_{fc}^1}{t_{fc}^2}\right). \quad (24)$$

Substituting t_{fc}^1 and t_{fc}^2 from Equation 19 into Equation 24 gives

$$Q_{fc} = \frac{R}{[(1/T_1) - (1/T_2)]} \times \ln\left\{\frac{a_{T_2}}{a_{T_1}}\left(\frac{\ln\{1 - [(M_1 - m_1 T_1)/c_1](E/\sigma)^s\}}{\ln\{1 - [(M_1 - m_1 T_2)/c_1](E/\sigma)^s\}}\right)^{1/b}\right\}. \quad (25)$$

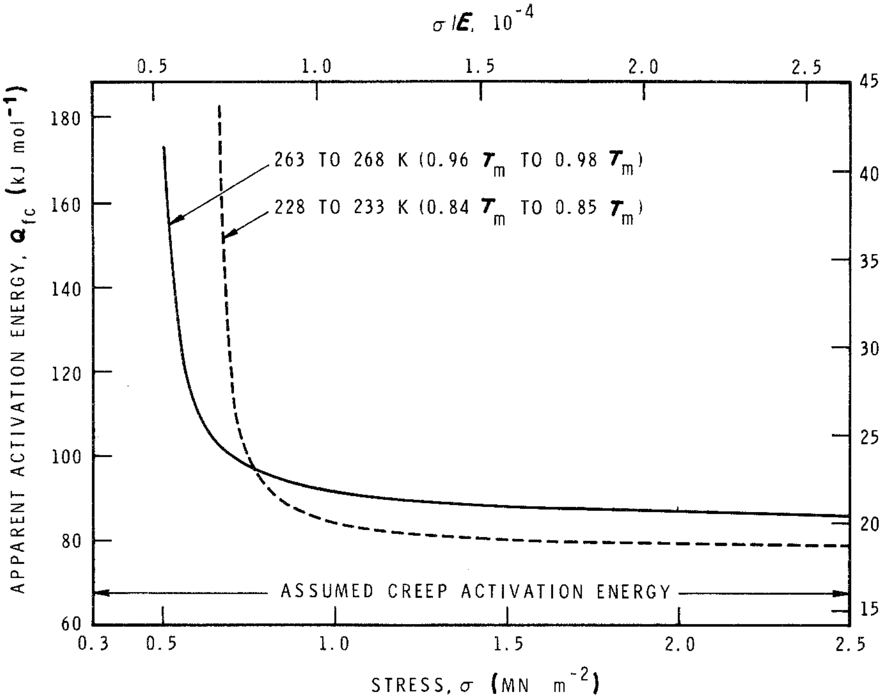

Figure 12 Predicted stress and temperature dependence of apparent activation energy associated with the formation of first crack.

Substituting Q from Equation 14 into Equation 25 gives

$$Q_{\mathrm{fc}} = Q + \frac{R}{b[(1/T_1)-(1/T_2)]} \times \ln\left(\frac{\ln\{1-[(M_1-m_1T_1)/c_1](E/\sigma)^s\}}{\ln\{1-[(M_1-m_1T_2)/c_1](E/\sigma)^s\}}\right). \tag{26}$$

Calculations made on the basis of Equation 26 and Table I are shown in Fig. 12 for two ranges of temperature, 228 to 233 K (-45 to $-40°$ C) and 263 to 268 K (-10 to $-5°$ C). Fig. 12 shows that Q_{fc} could be greater than the creep activation energy, Q, involving no cracks and furthermore could depend on stress and temperature. It must be pointed out that the numerical value for Q assumed here is not different from the activation energy for self-diffusion [22, 51, 52], although creep in ice may not be diffusion-controlled but glide-controlled [53], and was determined by Sinha [32] for the range -44 to $-10°$ C ($0.84\,T_{\mathrm{m}}$ to $0.96\,T_{\mathrm{m}}$) using $\sigma = 0.5$ MN m^{-2} ($\sigma/E = 5 \times 10^{-5}$) to avoid any cracking activity. Gold's original observations [22] on the dependence of apparent crack activation energy on stress and temperature and Ramseier's [52] observations on creep activation energies involving cracks are therefore consistent with the present calculations. Introduction of temperature correction in E would, of course, alter the calculated results slightly.

In discussing the activation energy for steady-state, or rather minimum, creep rate of polycrystalline ice experimentally determined high values above $-10°$ C ($0.96\,T_{\mathrm{m}}$) were treated by Weertman [51] as unrealistic. Examinations of experimental conditions used, for example, by Glen [54] and Barnes *et al.* [55] indicate that the load level ($\sigma/E > 5 \times 10^{-5}$) must certainly have caused the formation of cracks during experiments. Activation energies (see Fig. 12) in the range of 130 kJ mol^{-1} (30 kcal mol^{-1}) obtained by the investigators could very well be due to degradation of the structure as a result of internal voids. The response of ice during strength testing [39, 56], involving a mixed-mode deformation process, can be properly analysed only after clarifying the effects of cracking activity and cumulative damage on the texture and fabric of the ice, due to both constant and variable stress-loading conditions. These will be considered in a future publication. It should, however, be mentioned that the prediction made by Equation 26 and illustrated in Fig. 12 seems to be applicable, in general, to other materials also [30].

For example, the decrease in apparent activation energy with increase in stress at higher temperatures and its greater value at higher temperature, reported by Vagarali and Langdon [57] for polycrystalline magnesium, bear close resemblance to that of Fig. 12.

If $\sigma_{\min, T_1}$ and $\sigma_{\min, T_2}$ are, respectively, the minimum stresses for cracking at T_1 and T_2, then Equations 20 and 26 give

$$Q_{fc} = Q + \frac{R}{b[(1/T_1) - (1/T_2)]} \times \ln \left\{ \frac{\ln [1 - (\sigma_{\min, T_1}/\sigma)^s]}{\ln [1 - (\sigma_{\min, T_2}/\sigma)^s]} \right\}. \quad (27)$$

Variation of Q_{fc} with stress and temperature is therefore linked with the temperature dependence of $\sigma_{\min}$. According to Equation 20, $\sigma_{\min}$ varies from $0.47\,\mathrm{MN\,m^{-2}}$ at $-5°$ C to $0.59\,\mathrm{MN\,m^{-2}}$ at $-30°$ C. This 25% increase is directly associated with a similar increase in ϵ_d^c, as may be seen from Equation 16 or Fig. 10. But a 5% increase in E in this temperature range will only account for about one-fifth of the 25% increase in $\sigma_{\min}$ or ϵ_d^c (and therefore in the minimum grain-boundary displacement for cracking). The remaining increase could very well be explained by the increase in $(\gamma_{vs} - \gamma_{gb})/2$ in the temperature range considered [41].

5. Summary and conclusions

1. The high temperature grain-boundary embrittlement phenomenon in polycrystalline materials has been analysed to show the possibility of the onset of cracking activity on attaining a critical grain-boundary sliding displacement, $\bar{x}'$. This is correlated with a critcal grain-boundary sliding strain, ϵ'_{gbs}, and hence with a critical delayed-elastic strain, ϵ_d^c.

2. Constant stress creep cracking observations on ice in conjunction with a previously developed rheological equation have been used to test the above hypothesis in the temperature range of 0.89 T_m to 0.98 T_m and stress range, σ, of $6 \times 10^{-5} E$ to $2 \times 10^{-4} E$. It is found that ϵ_d^c for crack initiation is independent of stress in the range studied, but increases with decrease in temperature and is inversely proportional to grain size. The critical grain-boundary displacement, $\bar{x}'$ ($= 0.47\,\mu$m at $0.96\,T_m$), increases with decrease in temperature but is independent of stress and grain size.

3. The above criterion was applied in formulating explicit interrelations for σ, t, d, cracking time t_{fc} and the corresponding strain ϵ_{fc}. The equation developed for the dependence of t_{fc} on σ has been shown to represent the experimental observations better than a purely empirical Zhurkov-type relation. In addition, it predicts that cracking activity occurs on exceeding a critical stress, $\sigma_{\min}$, at a constant temperature and that t_{fc} is independent of grain size.

4. Stress, temperature, and grain size dependence of strain to the formation of cracks, ϵ_{fc}, show the existence of a region of brittle-to-ductile transition and predict superplasticity for stresses below $\sigma_{\min}$. It is shown that both stress and strain at transition decrease (hence a measure of increase in brittleness) with increase in temperature for a constant grain size. There is also an increase in brittleness with increase in grain size. the conditions predicted to be favourable for "stress controlled" superplastic flow are high temperatures, low stresses and fine grains. The formulations also predict a commonly observed phenomenon – a nearly constant strain at fracture in the so-called brittle range.

5. Formulations have been developed to show the commonly observed dependence of the apparent activation energy for cracks (or fracture) on stress and temperature. This can be predicted from the creep activation energy (which is close to the activation energy for self-diffusion or glide) and the temperature dependence of $\sigma_{\min}$, the minimum stress for cracking. The temperature dependence of $\sigma_{\min}$, on the other hand, is determined by the temperature dependence of E, Young's modulus, and the surface free energy.

6. It is shown again [56] that ice can serve as a good model material for strength and deformation studies at high homologous temperatures.

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References

1. D. McLEAN, "Grain Boundaries in Metals" (Clarendon Press, Oxford, 1957).
2. R. C. GIFKINS, in "Fracture", edited by B. C. Averbach, D. K. Felbeck, G. T. Hahn and D. A. Thomas (Wiley Interscience, New York, 1959)

p. 579.
3. H. CONRAD, in "Mechanical Behavior of Materials at Elevated Temperatures", edited by J. E. Dorn (McGraw-Hill, New York, 1961) p. 218.
4. A. H. COTTRELL, Intercrystalline Creep Fractures, in "Structural Processes in Creep" (Iron and Steel Institute, London, 1961) p. 1.
5. F. GAROFALO, "Fundamentals of Creep and Creep-Rupture in Metals" (Macmillan Company, New York, 1965).
6. G. W. GREENWOOD, in "Interfaces", edited by R. C. Gifkins (Butterworths, London, 1969) p. 223.
7. N. J. GRANT, in "Fracture", Vol. III, edited by H. Liebowitz (Academic Press, New York, 1971), p. 483.
8. A. J. PERRY, *J. Mater. Sci.* **9** (1974) 1016.
9. A. G. EVANS and T. G. LANGDON, *Prog. Mater. Sci.* **21** (314) (1976) 171.
10. M. F. ASHBY, C. GANDHI and D. M. R. TAPLIN, *Acta Metall.* **27** (5) (1979) 699.
11. C. ZENER, The micro-mechanism of fracture, in "Fracturing of Metals" (American Society for Metals, Cleveland, Ohio, 1948) p. 3.
12. R. C. GIFKINS, *Acta Metall.* **4** (1956) 98.
13. C. W. CHEN and E. S. MACHLIN, *ibid.* **4** (1956) 655.
14. J. INTRATER and E. S. MACHLIN, *ibid.* **7** (1959) 140.
15. N. K. SINHA, *Phil. Mag.* **40** (1979) 825.
16. D. McLEAN, B. F. DYSON and D. M. R. TAPLIN, The prediction of creep fracture in engineering alloys, in "Fracture", Vol. I, edited by D. M. R. Taplin (University of Waterloo Press, Waterloo, Canada, 1977) pp. 325–362.
17. C. W. LAU and A. S. ARGON, in "Fracture", Vol. II, edited by D. M. R. Taplin (University of Waterloo Press, Waterloo, Canada 1977) p. 595.
18. A. G. EVANS, J. R. RICE and J. P. HIRTH, *J. Amer. Ceram. Soc.* **63** (1980) 368.
19. A. N. STROH, *Proc. Roy. Soc. A.* **223** (1954) 404.
20. D. McLEAN, Point defects and the mechanical properties of metals and alloys at high temperatures, in "Vacancies and Other Point Defects in Metals and Alloys", Monograph and Report Series No. 23 (Institute of Metals, London, 1958) pp. 159–98.
21. W. M. KETCHUM and P. V. HOBBS, *Phil. Mag.* **19** (1969) 1161.
22. L. W. GOLD, "The failure process in columnar-grained ice". National Research Council of Canada, Division of Building Research, NRC 12637, 1972.
23. N. K. SINHA, in Proceedings of the Joint Conference on Experimental Mechanics, Soc. Expt. Stress Analysis and Japanese Soc. Mech. Eng., Hawaii, 1982, Part 2, pp. 767–772.
24. D. J. GOODMAN, Workshop on Ice Physics, Hokkaido University, Japan (1979) (also private communications).
25. A. H. COTTRELL, *Trans. Amer. Inst. Min. Engr.* **212** (1958) 192.
26. J. A. WILLIAMS, *Acta Metall.* **15** (1967) 1559.
27. R. L. BELL and T. G. LANGDON, *J. Mater. Sci.* **2** (1967) 313.
28. L. W. GOLD, "Physics of Snow and Ice", edited by H. Oura (Institute of Low Temperature Science, Hokkaido University, Japan, Part 1, 1976) pp. 359–70.
29. L. W. GOLD, PhD thesis, McGill University, Montreal, Canada (1970).
30. G. M. BARTENEV and YU. S. ZUYEV, "Strength and Failure of Visco-elastic Materials" (Pergamon Press, Oxford, 1968) p. 164.
31. E. SMITH and J. T. BARNBY, *Met. Sci. J.* **1** (1967) 56.
32. N. K. SINHA, *Exp. Mech.* **18** (12) (1978) 464–70.
33. N. K. SINHA, *Phil. Mag.* **36** (1977) 1385.
34. L. W. GOLD, *Nature* **192** (1961) 130.
35. L. W. GOLD, *Can. J. Phys.* **41** (1963) 1712.
36. B. R. LAWN, B. J. HOCKEY and S. M. WIEDERHORN, *J. Mater. Sci.* **15** (1980) 1207.
37. N. K. SINHA, *J. Cold Region Sci. Technol.* 8 (1983) pp. 25–33.
38. N. K. SINHA, in "Mechanics of Structured Media, Part A", edited by A. P. S. Selvadurai (Elsevier Scientific Publishing Co., Amsterdam, 1981) pp. 419–30.
39. N. K. SINHA, *Exper. Mech.* **21** (1981) 209.
40. P. M. HAZZLEDINE and D. E. NEWBURY, Role of grain boundaries in superplasticity, in "Grain Boundary Structure and Properties", edited by G. A. Chadwick and D. A. Smith (Academic Press, London, 1976) pp. 235–64.
41. P. HOBBS, "Ice Physics" (Clarendon Press, Oxford, 1974).
42. YU. K. ZARETSKY, B. D. CHUMICHEV and V. I. SOLOMATIN, *Eng. Geol.* **13** (1979) 299.
43. J. H. CURRIER, MSc thesis, Dartmouth College, Hanover, NH, USA (1981).
44. P. DUVAL and H. LeGAC, *J. Glaciol.* **25** (1980) 151.
45. L. W. GOLD, *ibid.* **19** (1977) 197.
46. J. L. BURDICK, Tensile creep-rupture of polycrystalline ice, in Proceedings of the 3rd International Conference on Port and Ocean Engineering under Arctic Conditions (POAC), Institute of Marine Science, University of Alaska, Vol. 1 (1975) pp. 235–246.
47. D. CARTER and B. MICHEL, Lois et mécanismes de l'apparente fracture fragile de la glace de rivière et du lac. Department of Civil Engineering Laval University, Report S-22 (1971).
48. I. HAWKES and M. MELLOR, *J. Glaciol.* **11** (1972) 103.
49. J. F. KNOTT, in "Fracture", Vol. 1, edited by D. M. R. Taplin (University of Waterloo Press, Waterloo, Canada 1977) pp. 61–92.
50. C. GANDHI and M. F. ASHBY, *Acta Metall.* **27** (1979) 1565.
51. J. WEERTMAN, Creep of ice, in "Physics and Chemistry of Ice", edited by E. Whalley, S. J. Jones and L. W. Gold (Royal Society of Canada, Ottawa, 1973) pp. 320–37.
52. R. O. RAMSEIER, PhD thesis, Laval University, Quebec, Canada (1976).
53. D. J. GOODMAN, H. J. FROST and M. F. ASHBY, *Phil. Mag. A* **43** (1981) 665.
54. J. W. GLEN, *Proc. Roy. Soc. London* **A228** (1955) 519.

55. P. BARNES, D. TABOR and J. C. F. WALKER, *ibid.* **A324** (1971) 127.
56. N. K. SINHA, *J. Mater. Sci.* **17** (1982) 785.
57. S. S. VAGARALI and T. G. LANGDON, *Acta Metall.* **29** (1982) 1969.

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