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GRAIN BOUNDARY SLIDING IN POLYCRYSTALLINE MATERIALS

by N. K. Sinha

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SOMMAIRE

Le phénomène d'élasticité retardée durant le fluage haute température de matériaux polycristallins est mis en corrélation avec la déformation due au cisaillement intergranulaire. Cette corrélation est utilisée pour élaborer un modèle phénoménologique de la viscoélasticité qui comprenne l'effet de la grosseur des grains. En utilisant la glace comme matériau de référence, l'auteur montre que la contribution de la déformation due au cisaillement intergranulaire à la déformation totale, ainsi que sa dépendance par rapport à la contrainte, au temps, à la température et au diamètre des grains, peuvent être analysées systématiquement grâce au modèle proposé. Les résultats semblent s'accorder avec les tendances observées dans d'autres matériaux.

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Grain boundary sliding in polycrystalline materials

By N. K. SINHA

Geotechnical Section, Division of Building Research,
National Research Council of Canada, Ottawa, Canada

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ABSTRACT

Delayed elastic phenomenon during high-temperature creep of polycrystalline materials is correlated with strain due to grain boundary sliding. This correlation has been used to develop a phenomenological viscoelastic model that includes the grain-size effect. With ice as a reference material, it is shown that the contribution of the grain-boundary sliding strain to the total strain, and its dependence on stress, time, temperature and grain diameter can be systematically analysed by the proposed model. The results appear to agree with the observed trends in other materials.

§ 1. INTRODUCTION

Creep of single crystals at high temperature is a thermally activated process and is governed by the mobility of point or line defects. Shear or sliding in the grain boundary regions introduces additional complexities to the creep of polycrystalline materials at elevated temperatures (King and Chalmers 1949, McLean 1957). The structure of grain boundaries is complex (Chaudari and Matthews 1972, Chadwick and Smith 1976) and the microstructure model for sliding, including its accommodation, is in the developmental stage (Pond, Smith and Southerden 1978). The phenomenological aspects of the contribution of sliding to the creep strain are better known but still not well formulated (Conrad 1961, Stevens 1966, Bell and Langdon 1969, Bird, Mukherjee and Dorn 1969).

The purpose of this paper is to examine the possible role that grain boundary sliding plays in the creep of polycrystalline materials at elevated temperatures, and to develop a phenomenological model that will illustrate the dependence of the rheological behaviour on grain size and external variables. It is hoped that the model will help explain various experimental observations of the sliding phenomena. Ice has been used here as a reference material because of the availability of pertinent information required for this analysis. The term 'sliding' will be used here in a general sense to describe shear deformation localized in the grain boundary region.

§ 2. VISCOELASTICITY OF ICE

A programme of observation was undertaken on the uniaxial compressive creep and recovery of columnar-grained ice with an average grain size of 3 mm at $T > 0.84 T_m$, where T_m is the melting point in degrees Kelvin. The [0001] axis of the grains was randomly oriented in the plane normal to the columns and

the load was applied in this plane. It has been shown (Sinha 1978 a) that the time-dependent deformation, ϵ_t , can be expressed by

$$\epsilon_t = \epsilon_e + \epsilon_d + \epsilon_v, \quad (1)$$

where ϵ_e is the pure elastic deformation, ϵ_d the recoverable delayed elastic strain and ϵ_v the viscous (usually called plastic) or permanent deformation.

Relation (1) can be expressed in terms of stress, σ , and time, t , at a given temperature, T , by

$$\epsilon_{t(d_0)} = \frac{\sigma}{E} + c_0 \left(\frac{\sigma}{E} \right)^s \{1 - \exp [-(a_T t)^b]\} + \dot{\epsilon}_{v_1} t \left(\frac{\sigma}{\sigma_1} \right)^n, \quad (2)$$

where E is Young's modulus; $\dot{\epsilon}_{v_1}$ is the viscous strain rate for unit stress, $\sigma = \sigma_1$; and c_0 , b , a_T , n and s are constants as given in the table (Sinha 1978 a). The additional subscript for the total strain indicates an average grain size of d_0 .

Both $\dot{\epsilon}_{v_1}$ and a_T depend on temperature, such that

$$\dot{\epsilon}_{v_1}(T_1) = \frac{\dot{\epsilon}_{v_1}(T_2)}{S_{1,2}} \quad (3)$$

and

$$a_T(T_1) = \frac{a_T(T_2)}{S_{1,2}}, \quad (4)$$

where $S_{1,2}$ is a shift function (Sinha 1978 a) given by

$$S_{1,2} = \exp \left[\frac{Q}{R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \right], \quad (5)$$

Q and R being the activation energy and gas constant respectively; T_1 and T_2 are temperatures in degrees Kelvin.

The creep rate at any time t after loading is given by differentiating eqn. (2) with respect to time:

$$\dot{\epsilon}_{t(d_0)} = \frac{c_0 b}{t} \left(\frac{\sigma}{E} \right)^s (a_T t)^b \exp [-(a_T t)^b] + \dot{\epsilon}_{v_1} \left(\frac{\sigma}{\sigma_1} \right)^n. \quad (6)$$

The first two terms of eqn. (2) provide a model for the stress, time and temperature dependence of the effective elastic modulus of ice; eqn. (6) provides a relationship for the transient as well as the steady-state flow rate (Sinha 1978 a, b).

Although eqn. (6) was found to describe satisfactorily the deformation behaviour of ice with an average grain size of 3 mm, this was not the case for ice of different grain sizes (Sinha 1978 b). Equation (2) must incorporate the effect of grain size to be valid on a general basis.

§ 3. ANALYSIS OF THE RHEOLOGICAL RESPONSE

3.1. Elastic response

Ice belongs to the family of hexagonal crystals. Its elastic response is described by five independent elastic moduli (Hobbs 1974). Most polycrystalline ice is sufficiently isotropic to exhibit an average of the single-crystal

elastic response. The dynamic Young's modulus of high-density, low-salinity polycrystalline ice has been found by several investigators to lie in the range $9\text{--}10\text{ GN m}^{-2}$ for various types of ice. Variation with temperature, in the temperature range $0.80 T_m$ to $\approx 1.0 T_m$, is small (Sinha 1978 a). It is thus reasonable to consider E in eqn. (2) as independent of temperature and grain size without introducing any large error. However, careful consideration in choosing a value for E should be given for ice with low density or ice showing marked anisotropy in the crystallographic orientations of its grains.

3.2. Viscous response

The third term in eqn. (2) is the empirical steady-state flow, first introduced for ice by Glen (1955) and later supported by many investigations (see, for example, Steinemann 1958, Voitkovskii 1960, Dillon and Andersland 1967, Barnes, Tabor and Walker 1971, Gold 1970). In the limited stress range $0.1 < \sigma \leq 1.0\text{ MN m}^{-2}$ ($1.5 \times 10^{-5} < \tau'/G \leq 1.5 \times 10^{-4}$, where $\tau' = \sigma/2$ and G is the rigidity modulus), $n = 3$ satisfies the observations, but internal cracking activity alters the creep rate at higher stresses and steady state is not observed (Gold 1972). The applicability of this power-law relationship at lower stresses is still somewhat controversial. Weertman (1969) suggested that the apparent non-applicability of power-law creep in the stress range 0.1 MN m^{-2} or lower, reported by previous investigators, probably reflected inadequate time of loading and inaccuracy of measurements. This hypothesis appears to be justified directly by the experimental observations of Gold (1973) at 0.1 MN m^{-2} and an analysis by Sinha (1978 b) showed that a sharp transition in the stress exponent might be expected if the time of measurement is insufficient.

The previous experimental results on the creep rate of various types of ice in the intermediate stress range seem to indicate that the steady-state flow is not affected by grain size. No detailed investigations of the variation of creep rate with ice-crystal size have been conducted in the past. Baker (1978), however, reported an apparent reversal in the creep rate at a grain size of about 1 mm . His experimental conditions suggested that the increase in creep rate with the grain size could be attributed to the decrease in the number of grains in the cross-section and hence to a geometrical effect. However, increase in creep rate with decreasing grain size could be attributed to the greater relative contribution of the delayed elastic strain to the total strain if the time over which the observations are made is not sufficiently long, as will be clarified later. Thus Baker's observed reversal in the creep rate might be the result of these two conflicting effects. The results of careful experimental investigations into high-temperature creep in metals and alloys justify this hypothesis.

Evidence of the geometric effect can be seen in the investigations of Davies, Dennison and Evens (1964–65) on pure (99.997–99.999%) aluminium, nickel, copper and gold of grain size $\sim 1.8\text{ grains/mm}$. Using two sets of cylindrical specimens, one containing > 12 grains and the other < 4 grains across the diameter, they determined the stress-strain curves and observed that the latter specimens deformed more than the former ones for a given stress.

Bird *et al.* (1969) documented an extensive review on high-temperature creep and showed that the available data, excluding that on Nabarro creep, refutes the concept of the dependence of steady-state creep rate on grain size (Conrad 1961, Garofalo 1965). They used the results of Garofalo, Domis and

Gemmingen (1964) on austenitic stainless steels, and those of Barrett, Lytton and Sherby (1967) on copper, to show the invariance of the steady-state creep rate with grain size above a critical value of about 0.08 mm, but an increasing creep rate with decreasing grain size below this critical value. Bird *et al.* (1969) attributed the observed increase in creep rate with decreasing grain size to the increasing contributions of grain boundary sliding to the overall creep rate.

3.3. *Delayed elastic response*

The preceding discussion strongly indicates that if a grain-size effect exists, it should be incorporated into the delayed elastic term of eqn. (2). Qualitatively, at least, the inclusion of such an effect would help explain the pronounced variation in transient creep rate from one type of ice to another, and might provide additional insight into the grain-size dependence of the creep rate for polycrystalline materials in general.

While searching for an analytical expression to make the required modifications in the recoverable part of the creep strain, the age-old question arose as to why polycrystalline ice (and also other polycrystalline materials) shows a delayed elastic effect. The transient creep is usually explained by the development of more creep-resistant substructures. The delayed elasticity must therefore be associated with a degree of reversibility in these changes in the microstructure on unloading.

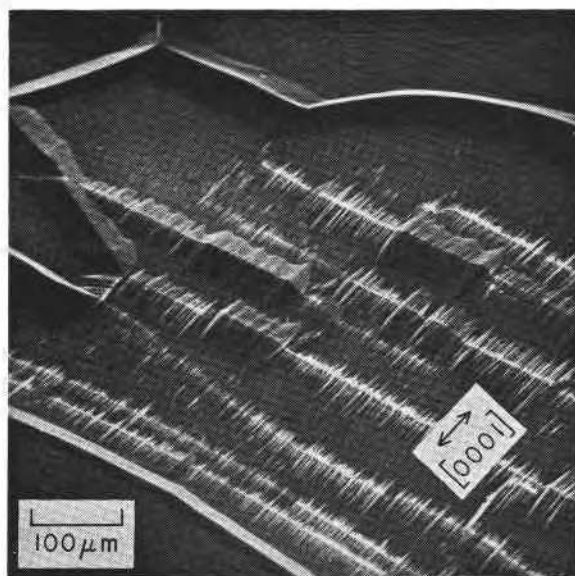
To develop a better understanding, microstructure analyses of polycrystalline ice at various stages of creep and subsequent recovery, under constant stress and temperature, were carried out by the dual process of etching and replicating (Sinha 1977). It was observed that during the very early period, when the creep strain was almost fully recoverable with no permanent deformation (i.e. less than the accuracy of measurements), the microstructure showed no detectable change from its undeformed state. A change in dislocation density could not be detected with any certainty because of the large variation in the initial dislocation density, which ranged from 10^6 to 10^7 cm⁻². No effort was made to perform the experiment on specially prepared ice with low initial dislocation density because the purpose was to examine what happens in natural polycrystalline ice.

With continued deformation, several substructural modifications were noticed: the formation of corrugations at the grain boundaries associated with slip planes in the grain, the formation of small-angle boundaries, the migration of grain boundaries, dislocation pile-ups (see fig. 1), the formation of cavities and cracks along the boundaries and triple points (see fig. 2) and the development of various substructures in the dislocation distributions. The appearance of these features depended strongly on temperature, stress, amount of strain and the crystallographic orientations of the neighbouring grains. This study thus confirms some of the observations made in the laboratory by Gold (1970). The present method, however, not only allowed the detection of these features at very early stages of creep, but also illustrated the role played by dislocations in the process of deformation. Many of these features are not reversible, even after a long period of storage in a room at $0.96 T_m$ (Sinha 1978 c). The formation of voids and cracks may require simultaneous grain boundary sliding in addition to the other modes of deformation (Chang and Grant 1956, McLean

1958, Mullendore and Grant 1961). The healing of some of these cracks, observed on unloading, could also be partly due to recovery in the sliding processes.

These observations suggested that the measurement of delayed elasticity during creep could give a measure of the reversible part of the grain boundary sliding mechanisms and associated complex interactions with the intragranular and intergranular substructures. This concept, therefore, maintains a continuity with the grain boundary sliding model suggested by Zener (1948) and his associates for low-stress, small-strain anelastic effects in polycrystalline materials, as can be seen from the microstructure analysis during very early creep. Zener's model formed the basis of the famous Kê-type of internal-friction measurements (Kê 1947, McLean 1957, Conrad 1961).

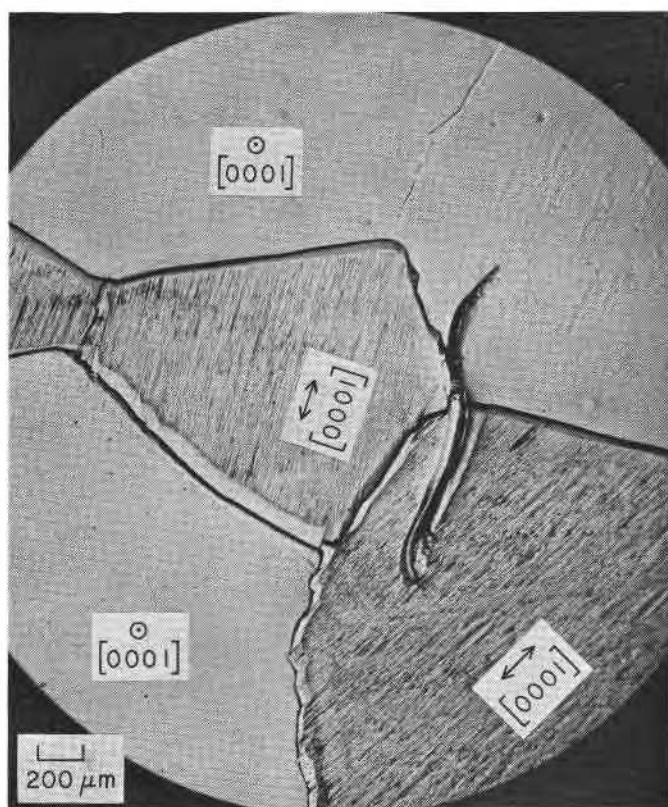
Fig. 1



Scanning electron micrograph of a replica of deformed ice exhibiting the pile-up of dislocations associated with grain boundary corners and step on the grain boundary.

Internal friction in ice, however, is not ascribed to grain boundary sliding alone, but also to the reorientation of the protons; which of the two effects predominates in polycrystalline ice is not clearly established (Hobbs 1974). The idea that grain boundary sliding and delayed elasticity are related is supported by the fact that fine-grained polycrystalline materials exhibit more recoverable strain than do coarse-grained ones for equivalent times (Garofalo 1965), whereas single crystals, including those of ice (Brill and Camp 1961),

Fig. 2



Optical micrograph of a replica showing a crack associated with a triple point and the development of steps on preferred grain boundaries; $\sigma = 1 \text{ MN m}^{-2}$, $\epsilon_t = 7 \times 10^{-4}$, -10°C ($0.96 T_m$). The compressive stress axis was normal to the plane of the photograph.

show negligible recoverable creep. The differences can be explained tentatively by the fact that slip systems of individual grains in polycrystalline aggregates are constrained, unlike single crystals, and should result in pronounced intragranular dislocation interactions. However, observations of large delayed elastic effects in ice immediately after loading, without any measurable (i.e. less than the accuracy of measurement) permanent deformation (Sinha 1978 a) and residual intragranular structural changes mentioned previously, indicate that the initial strain has little dependence on intragranular deformation process. The low dislocation velocities in ice (Fukuda and Higashi 1973) do not conflict with the above conclusion. The decay type of function obtained for the delayed elastic strain and the proposed eqn. (2) indicate, however, that this dependence increases with creep time. While examining the dependence of grain boundary sliding on intragranular deformation, Mullendore and Grant (1963) also came to a similar conclusion: "the grain boundary sliding which

occurs in the initial stage of deformation occurs independently of prior slip with only that amount of grain deformation necessary to accommodate the sliding." They observed that the initial sliding usually occurs in the immediate vicinity of the boundary, but sliding gradually takes place in a zone, with increased creep time, resulting in non-uniform distortions and the development of subgrains or cell formation in this zone.

Indirect (though inconclusive) support for the idea postulated above comes from the observation (Sinha 1978 a) that the apparent activation energy for delayed elasticity is equal to the creep activation energy in ice, which has been shown to be nearly equal to the activation energy for self-diffusion (Gold 1970, Weertman 1973). The activation energies for low-amplitude sliding in internal-friction measurements, macroscopic grain boundary sliding and creep have also been shown to be nearly equal to each other, as well as close to that of self-diffusion, in a number of metals and alloys (Conrad 1961, Garofalo 1965, Stevens 1966).

§ 4. GRAIN BOUNDARY SLIDING DISPLACEMENT FROM DELAYED ELASTIC STRAIN

Several attempts have been made in the past to devise methods of determining grain-boundary displacements at the specimen surface and inside the bulk of the material during creep, thereby to predict the contribution of grain boundary sliding to the total strain. Both the experimental procedure and the interpretation of the results are difficult (Gifkins 1959, Stevens 1966, Bell and Langdon 1967, Langdon 1972).

The strain induced by grain boundary sliding, ϵ_{gbs} , is usually given by

$$\epsilon_{\text{gbs}} = K\bar{x}d^{-1}, \quad (7)$$

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

Assuming that $\epsilon_{\text{gbs}} = f(\epsilon_d)$, the first simplification would be to postulate that

$$\epsilon_{\text{gbs}} = \epsilon_d. \quad (8)$$

With this assumption, ϵ_{gbs} , and hence \bar{x} , are time-dependent at a given temperature. From eqns. (7) and (8),

$$\bar{x}_t = \epsilon_d d / K. \quad (9)$$

From eqn. (9) and the second term, ϵ_d , of eqn. (2), and recalling that $d = d_0$,

$$\bar{x}_t = \frac{c_0 d_0}{K} \left(\frac{\sigma}{E} \right)^s \{1 - \exp [-(a_T t)^b]\}. \quad (10)$$

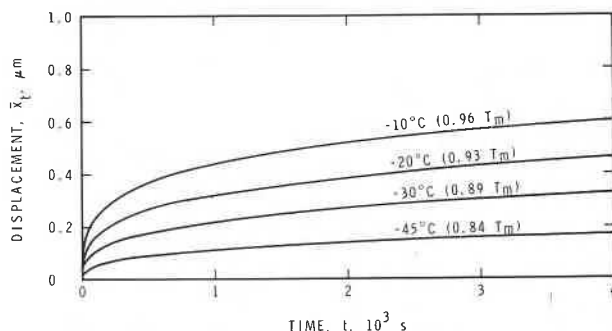
The rate of sliding is then given by the first derivative of eqn. (10),

$$\dot{\bar{x}}_t = \frac{c_0 d_0 b}{K t} \left(\frac{\sigma}{E} \right)^s (a_T t)^b \exp [-(a_T t)^b]. \quad (11)$$

Equation (10) gives the stress, time and temperature dependences of \bar{x}_t , and s is implied as the stress exponent for sliding.

Figure 3 illustrates the effect of temperature on \bar{x}_t for ice of average grain size, $d_0 = 3$ mm, computed for $\sigma = 1.0 \text{ MN m}^{-2}$ ($\tau'/G \approx 1.5 \times 10^{-4}$) using $K = 1$. The variation of \bar{x}_t with time and temperature appears to be similar to creep curves. Such similarities have been seen in a large number of investigations (McLean 1957, Conrad 1961, Gifkins 1959, Stevens 1966, Bell and Langdon 1967), providing sufficient encouragement for the present approach.

Fig. 3



The effect of temperature of \bar{x}_t /time curves for $\sigma = 1 \text{ MN m}^{-2}$ ($\tau'/G \approx 1.5 \times 10^{-4}$); grain diameter = 3 mm.

§ 5. GRAIN BOUNDARY SLIDING STRAIN AND DELAYED ELASTICITY

The contribution of grain boundary sliding strain to the total strain is usually denoted by γ and is expressed as a percentage :

$$\gamma = \frac{\epsilon_{\text{gbs}}}{\epsilon_t} \times 100. \quad (12)$$

Langdon (1973) carried out experiments on a magnesium alloy and high-purity aluminium to obtain more information on the dependence of sliding on grain size and stress at temperatures of $\approx 0.5 T_m$. He observed an initial linear dependence of \bar{v} , the average displacement normal to the specimen surface, on ϵ_t , and proposed the empirical relation

$$\frac{\epsilon_{\text{gbs}}}{\epsilon_t} = (1 + \psi d \sigma^p)^{-1}, \quad (13)$$

where ψ is a constant at constant temperature and p is the difference in stress exponent between that for steady-state creep rate and that for sliding. He did not, however, elaborate on how ψ varied with temperature.

To satisfy eqn. (13), Langdon (1973) found p to be 2 and 2.8, for aluminium and magnesium alloy respectively, for strains lower than 0.01; this led to a stress exponent of ~ 2.4 for the sliding rate of both materials.

The two sides of eqn. (13) are not consistent with each other. The right-hand side deals with creep strain rates only, whereas the left-hand side incorporates initial elastic strain. A self-consistent, dimensionally balanced form of eqn. (13) would be

$$\frac{\epsilon_{\text{gbs}}}{\epsilon_t - \epsilon_e} = \left[1 + \xi \frac{d}{d_1} \left(\frac{\sigma}{\sigma_1} \right)^p \right]^{-1}, \quad (14)$$

where ξ is a time- and temperature-dependent parameter, d_1 is the unit grain diameter and $p = n - s$ by definition and implication.

Substituting ϵ_d (from eqn. (8)) for ϵ_{gbs} in eqn. (14), with $\epsilon_t - \epsilon_e = \epsilon_d + \epsilon_v$ (from eqn. (1)), and rearranging, gives

$$\epsilon_d = \frac{\epsilon_v}{\xi} \frac{d_1}{d} \left(\frac{\sigma_1}{\sigma} \right)^p. \quad (15)$$

The observation that steady-state creep is independent of grain size (for conditions where Nabarro-Herring, Coble, or Ashby-Verrall (1973) types of diffusional creep do not play the dominating role) allows the substitution of ϵ_v from eqn. (2) in eqn. (15), without introducing any additional grain-size effect, and gives

$$\epsilon_d = \frac{\dot{\epsilon}_v t d_1}{\xi d} \left(\frac{\sigma}{\sigma_1} \right)^{n-p}. \quad (16)$$

Substituting the term ϵ_d of eqn. (2) in eqn. (16), and recalling that $d = d_0$ and $p = n - s$,

$$\xi = \frac{\dot{\epsilon}_v t d_1}{c_0 d_0} \left(\frac{E}{\sigma_1} \right)^s \frac{t}{\{1 - \exp[-(a_T t)^b]\}}, \quad (17)$$

which is independent of σ and gives ξ in terms of time and temperature. Substituting ξ from eqn. (17) in eqn. (16),

$$\epsilon_d = \frac{c_0 d_0}{d} \left(\frac{\sigma}{E} \right)^s \{1 - \exp[-(a_T t)^b]\}. \quad (18)$$

The above result could also be obtained by identifying ϵ_d and ϵ_{d_0} for d and d_0 respectively in eqn. (16), taking the ratio, and substituting the term ϵ_{d_0} of eqn. (2). This derivation implicitly assumes that a_T and b are independent of grain size.

§ 6. GENERALIZATION OF CREEP AND SLIDING EQUATIONS

Equation (17) or (18) implies that $c_0 d_0 = \text{constant} = c_1 d_1$, where c_1 corresponds to the unit grain diameter, d_1 , and can be determined from experimental observations (see the table for ice). This observation assists in presenting eqn. (18) in the form

$$\epsilon_d = \frac{c_1 d_1}{d} \left(\frac{\sigma}{E} \right)^s \{1 - \exp[-(a_T t)^b]\}, \quad (19)$$

Creep parameters for ice used in the calculations.

$$\begin{aligned}
 E &= 9.5 \text{ GN m}^{-2} \\
 Q &= 67 \text{ kJ/mol (16 kcal/mol)} \\
 c_0 &= 3, c_1 = 9 \\
 d_0 &= 3 \text{ mm}, d_1 = 1 \text{ mm} \\
 s &= 1 \\
 n &= 3 \\
 b &= 0.34 \\
 a_T(T = 263 \text{ K}) &= 2.5 \times 10^{-4} \text{ s}^{-1} \\
 \dot{\epsilon}_{v_1} &= 1.76 \times 10^{-7} \text{ s}^{-1}; \sigma_1 = 1 \text{ MN m}^{-2}, T = 263 \text{ K}
 \end{aligned}$$

which describes generalized delayed elastic strain exhibiting an inverse proportionality with grain size. The relation for the generalized internal grain boundary sliding displacement is given by combining eqns. (9) and (19) as

$$\bar{x}_t = \frac{c_1 d_1}{K} \left(\frac{\sigma}{E} \right)^s \{1 - \exp [-(a_T t)^b]\}. \quad (20)$$

Replacing the second term in eqn. (2) by eqn. (19), the generalized form of the creep equation is obtained as

$$\epsilon_t = \frac{\sigma}{E} + \frac{c_1 d_1}{d} \left(\frac{\sigma}{E} \right)^s \{1 - \exp [-(a_T t)^b]\} + \dot{\epsilon}_{v_1} t \left(\frac{\sigma}{\sigma_1} \right)^n, \quad (21)$$

which gives the creep rate as

$$\dot{\epsilon}_t = \frac{c_1 b}{t} (a_T t)^b \left(\frac{d_1}{d} \right) \left(\frac{\sigma}{E} \right)^s \exp [-(a_T t)^b] + \dot{\epsilon}_{v_1} \left(\frac{\sigma}{\sigma_1} \right)^n. \quad (22)$$

The contribution of the grain boundary sliding strain to the total strain, γ , is then given by

$$\gamma = \gamma_t = \frac{\epsilon_{gbs}}{\epsilon_t} = \frac{\epsilon_d \text{ (eqn. (19))}}{\epsilon_t \text{ (eqn. (21))}}, \quad \text{or} \quad = \frac{\text{second term of eqn. (21)}}{\epsilon_t \text{ (eqn. (21))}} \quad (23)$$

§ 7. DISCUSSION

The applicability of eqn. (21) in describing the various aspects of the deformation behaviour of ice has been examined and will be presented in the future. Since the basis of this analysis is the contribution of grain boundary sliding to creep, it seems appropriate to limit the discussion to this topic.

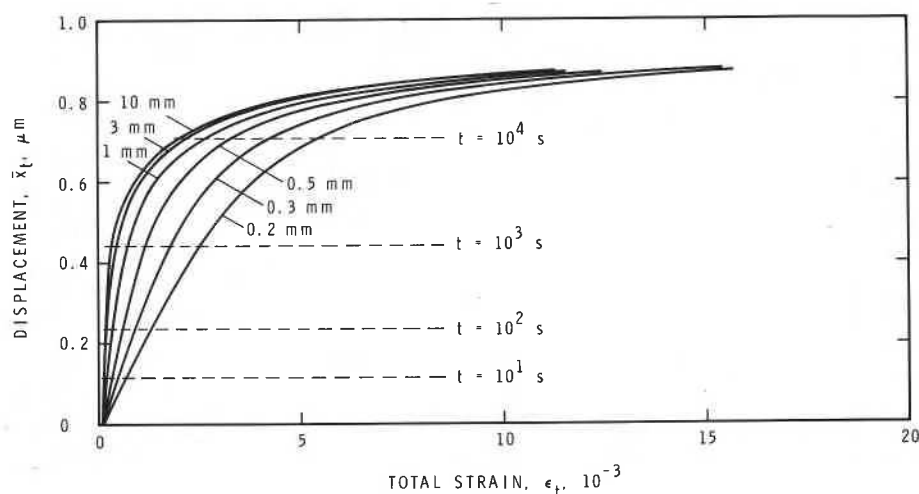
7.1. Grain-size effect on sliding

The stress, time and temperature dependence of \bar{x}_t is given by eqn. (20). It implies that, for a given time and constant stress and temperature, \bar{x}_t is independent of grain size, thus agreeing with Langdon (1973) and also providing more flexibility in presenting this important observation.

7.2. Dependence of sliding on strain

It is convenient to plot \bar{x}_t as a function of ϵ_t . Using eqns. (20) and (21), a set of calculations for ice at $0.96 T_m$ and $\sigma = 1.0 \text{ MN m}^{-2}$ ($\tau'/G \approx 1.5 \times 10^{-4}$) is presented in fig. 4. All the curves show an asymptotic approach to the same limiting \bar{x}_t , corresponding to the stress level used. The intercept of the curves on the strain axis is the corresponding elastic strain. Broken lines give the scale of time illustrating the invariance of \bar{x}_t on d at constant t , T and σ .

Fig. 4



Variation of \bar{x}_t with ϵ_t for ice of different grain size at -10°C ($0.96 T_m$); $\sigma = 1 \text{ MN m}^{-2}$ ($\tau'/G \approx 1.5 \times 10^{-4}$).

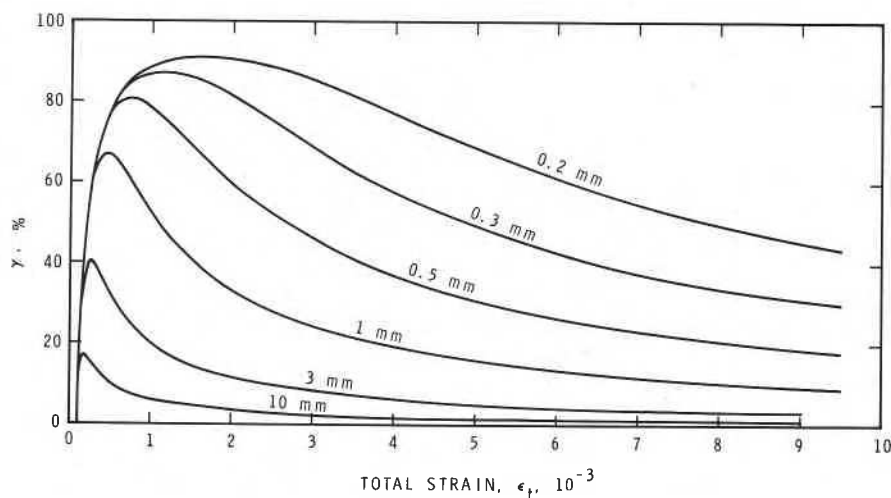
Since the apparent activation energy for sliding (as implied here) and for viscous flow are the same, eqns. (20) and (21) predict that the shape of the \bar{x}_t versus ϵ_t curves will remain the same, irrespective of temperature, if the temperature variation of E is neglected. The temperature will, however, affect the time-scale according to the shift function (5). Thus a creep time of 10 s at $0.96 T_m$ will be equivalent to 12 days at $0.7 T_m$, 36 years at $0.6 T_m$ and about half a million years at $0.5 T_m$, assuming Q to be constant in these temperature ranges. This is much too simplified an assumption (Garofalo 1965), but the calculations indicate that grain boundary sliding as a mode of deformation becomes insignificant at lower temperatures, and that even a relatively long creep time in the range $0.5 T_m$ – $0.7 T_m$ will probably give only the initial part of the family of \bar{x}_t versus ϵ_t curves shown in fig. 4. A striking example of this prediction is provided by the experimental observations of Langdon (1973) of \bar{v} , the average displacement normal to the specimen surface, with ϵ_t for a magnesium alloy having different grain sizes at $0.5 T_m$ and a stress of $\tau'/G \approx 9 \times 10^{-4}$. Langdon, however, accepted the initial linearity between \bar{v} and ϵ_t , but ascribed the non-linearity at larger ϵ_t entirely to surface phenomena.

Several examples of both linear and non-linear dependence of \bar{x}_t on ϵ_t have been discussed in detail by Garofalo (1965).

7.3. Dependence of γ on strain

The variation of γ with ϵ_t is shown in fig. 5 for the same set of calculations presented in the previous figure. Several important aspects are to be noticed in fig. 5: (1) the increasing value of γ with decreasing grain size, (2) the occurrence of the maximum γ during the very early part of the creep, (3) the gradual shift of the maxima towards larger strains with decreasing grain size, (4) the asymptotic decrease in γ with the increase in strain after reaching the maxima and (5) the decreasing dependence of γ on grain size at large strains.

Fig. 5



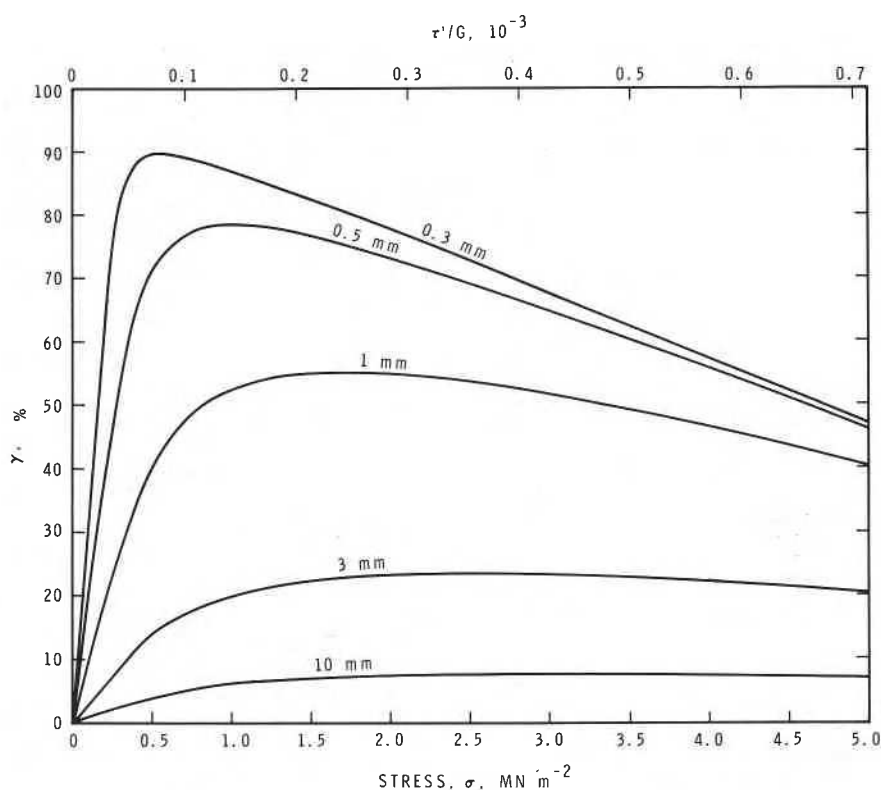
Strain dependence of the ratio of delayed elastic (grain boundary sliding) to total strain for various grain sizes; $\sigma = 1.0 \text{ MN m}^{-2}$ ($\tau'/G \approx 1.5 \times 10^{-4}$) at $0.96 T_m$.

The first aspect has been proved conclusively in several materials. Evidence of (2) and (5) can be found in Ishida, Mullendore and Grant (1964) and Mullendore and Grant (1963) respectively. Aspect (4) is analogous to several observations that γ is either nearly a constant, or a decreasing function of strain, for a given grain size, depending on the strain range over which the measurements had been made and the stress and temperature used (Conrad 1961, Bell and Langdon 1969, Stevens 1966). There is, however, no conclusive experimental evidence for (3), although the agreement of the observations of Langdon (1973) on the dependence of \bar{v} on ϵ_t with our prediction seem to suggest that the maxima do shift towards larger strains.

7.4. Dependence of γ on stress

The dependence of γ on stress can be examined, as indicated in fig. 5, by computing its value at a particular ϵ_t , while keeping d and T constant. Examples of such computations are shown in fig. 6. The exact shape of the curves and the value of the critical stress corresponding to the maximum γ depend strongly on the choice of ϵ_t . Four important general observations can be made, however, from this illustration: (1) the increase in γ with increasing σ below a critical stress, (2) the decrease in γ with increasing σ above the critical stress, (3) the increase in γ with decreasing grain size at a given stress, and (4) the shift of the maximum γ or critical stress towards lower stress with decreasing grain size.

Fig. 6



Stress dependence of the ratio of delayed elastic (grain boundary sliding) to total strain at a total strain of 1×10^{-3} .

Examples of (1) can be found in the experiments by Harper, Shepard and Dorn (1958) on aluminium and in experiments by H. Brunner and N. J. Grant, illustrated in Grant (1959), on aluminium and aluminium-magnesium alloy. They also provided support for (2), along with others (McLean 1952-53, McLean and Farmer 1956-57, Garofalo, Whitmore, Domis and Gemmingen

1961, Bell and Langdon 1967). The compilations of Garofalo (1965) of experimental results on a number of metals and alloys provide strong evidence for (2). The observations of Bell and Langdon (1967) on a magnesium alloy give a good example of (3), but to the author's knowledge there is no evidence to support (4) at present.

7.5. Temperature effect on γ

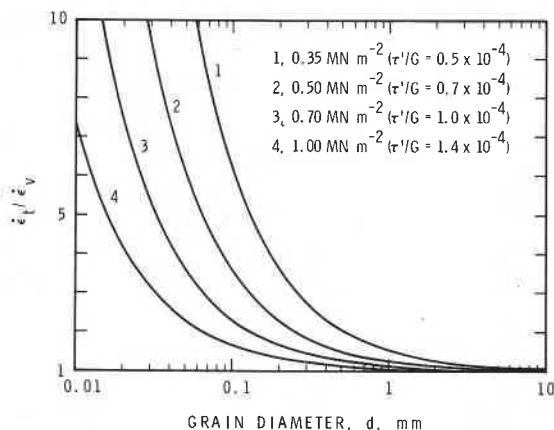
Since both the viscous flow and the delayed elasticity have the same activation energy, it can be shown from eqn. (21) that temperature will have no effect on the interrelationship between γ and ϵ_t , under a constant stress, if the variation of E with temperature is neglected. This might well be the case if the strain were relatively large compared to the initial elastic strain. Experimental evidence for metals and alloys (Fazan, Sherby and Dorn 1954, Martin, Herman and Brown 1957, Dorn 1956) does lend support to this prediction, but there are not sufficient data to prove it conclusively.

7.6. Creep rate

The problem of ascertaining the steady-state creep rate can be examined from eqn. (22). The time required to attain a quasi-steady state depends strongly on stress, temperature and grain size. The deviation in the observed creep rate, $\dot{\epsilon}_t$, from the corresponding steady-state strain rate, $\dot{\epsilon}_v$, (second term of eqn. (22)) would, in any case, depend largely on the conditions and accuracy of measurements.

Figure 7 illustrates the variation in $\dot{\epsilon}_t/\dot{\epsilon}_v$ with grain diameter for various stresses. Computations were made on the basis of eqn. (22) for $0.96 T_m$ at $t = 10^4$ s. The time was arbitrarily chosen and might appear to be very short, but this is equivalent to a creep period of about two months at $0.8 T_m$, or about two years at $0.75 T_m$, assuming a constant creep activation energy in this temperature range (Sherby, Lytton and Dorn 1957, Sinha 1978 a). A longer creep time or a higher temperature causes the family of curves to move

Fig. 7



Grain-size dependence of the ratio of creep rate at a loading time of 10^4 s with the steady-state creep rate for various stresses at -10°C ($0.96 T_m$).

towards smaller grain size, or vice versa. Evidence provided by Langdon (1973) suggests that $n-s$ in metals and alloys may not be much different from that found for ice; hence a similar type of deviation of $\dot{\epsilon}_t$ from $\dot{\epsilon}_v$ can be expected in these materials.

The characteristics of the creep rates presented in fig. 7 are similar to the experimental observations discussed earlier. The present analysis not only agrees conceptually with the idea proposed by Bird *et al.* (1969), mentioned previously, but appears to provide a stronger basis for future experiments. As far as ice is concerned, the first requirement—to examine the effect of grain size on creep rate—is to produce randomly oriented polycrystalline ice of varying grain sizes without any intergranular air or vapour bubbles. This is a challenge still to be met by glaciologists.

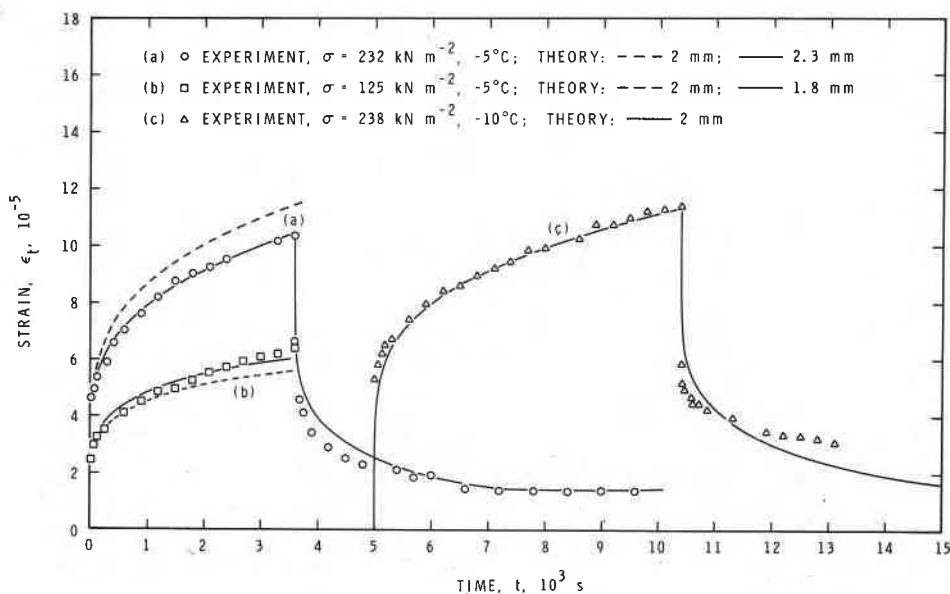
The present analysis is based on the rheological model (Sinha 1978 a), developed primarily to present the effect of external variables—stress, temperature and time—on the high-temperature creep response of a pure and randomly oriented polycrystalline material of a given grain size. The material used, ice, was columnar grained and had random crystallographic orientations of its grains in the plane containing the stress axis. Its density did not differ from that of a single crystal of ice by more than the error of measurements. The model, though empirical, was developed to provide a suitable and realistic framework into which the influences of internal variables (grain size, grain-size distribution, geometry of the grain structure, crystallographic orientation, impurities, inclusions, etc.) could be introduced. One such variable, grain size, has been introduced in this report. Several assumptions were made to simplify the procedures; the inverse proportionality shown between delayed elasticity and grain size was the result of these simplifications. Impurities and inclusions in the solid or liquid state at the grain-boundaries and at triple points will either retard or enhance the intergranular displacements and should influence the accommodational processes and, hence, the grain-size dependence. Morphological changes in the microstructure, such as the formation of internal cracks and recrystallization, should also influence the long-term creep properties.

7.7. An example of creep in ice

In an attempt to provide an example of the applicability of the proposed model within its present limitations, the literature on glaciology was scanned for independent transient creep and recovery measurements on ice of type and grain size different from the one used by this author (Sinha 1978 a). Most studies, however, have not emphasized the transient range. To the author's knowledge, the only data available in a usable form are those of Brill and Camp (1961). They used randomly oriented snow ice, but the reported lower density (886 kg/m³) indicates the presence of air or vapour bubbles in their ice. The tests were performed under uniaxial tension, rather than the compression used in most studies, including those of this author. The experimental results shown in fig. 8, marked (a), (b) and (c), were originally given by Brill and Camp (1961) as being typical of their observations.

The stresses and duration used in the above tests were such that internal cracks and recrystallization can be assumed not to have occurred (Barnes *et al.* 1971, Gold 1972). The temperatures were sufficiently below the melting point for the apparent activation energy to be assumed constant (Barnes *et al.* 1971).

Fig. 8



Comparison between the calculated and experimental creep and recovery of snow ice. Experimental data are taken from Brill and Camp (1961). The zero time for curve (c) has been shifted for clarity.

Brill and Camp (1961) did not, however, measure the grain sizes of their specimens but made the general statement that "the grains were irregular shape and 1 to 2 mm in diameter". Experience has shown that snow ice can have grains varying in sizes from less than 0.5 mm to 3 mm—a rather broad range. Calculations, based on eqn. (21) in conjunction with eqns. (3) to (5) and the data given in the table, were made first by arbitrarily choosing $d = 2$ mm. The results are presented in fig. 8. The computed strains after 60 min were 10% higher than those measured by Brill and Camp (1961) for (a) and 10% lower for (b), for tests at -5°C ($0.98 T_m$). The agreement with (c) at -10°C ($0.95 T_m$) can be considered acceptable. It was found that grain sizes of 2.3 mm for (a), 1.8 mm for (b) and 1.95 mm (not shown) for (c) resulted in better agreement with the experimental observations, as shown in fig. 8. Computed recovery curves are also shown for two tests along with the reported experimental results. The recovery curves were calculated by subtracting the total elastic strains (the first two terms of eqn. (21)) from the total strains at the time of unloading. This method is consistent with the method employed to develop the delayed elastic formulation (Sinha 1978 a). Recovery curves are more difficult to obtain, particularly if the elongations are measured from one end of the specimen, as done by Brill and Camp (1961). In spite of the uncertainties, the agreement between the experimental observations and the predicted recoveries can be considered as fair.

It is important to mention here that the above experimental results were the basis on which Brill and Camp (1961) came to the conclusion that snow ice was

a 'linear viscoelastic material'. They used conventional linear spring and dashpot combinations to describe these results, but had to use different sets of two elastic moduli and two viscosity coefficients (i.e. four parameters) to fit each curve. They noticed that the viscosities of (b) and (c) were less than those of (a), and ascribed the differences to the different storage time of the specimens. It was mentioned that (a) was a specimen from a batch of ice stored at -10°C ($0.96 T_m$) for about ten weeks prior to use, and (b) and (c) were from a second batch used immediately. Note that, according to our calculations, the grain size satisfying test (a) is larger than that for either (b) or (c) and, therefore, is indicative of possible grain growth in the ice of the first batch during storage.

§ 8. CONCLUSION

For conditions where grain boundary sliding with diffusional accommodation does not play the dominating role, the strain due to grain boundary sliding might be estimated from the delayed elastic effects. A non-linear visco-elastic model, incorporating the grain-size effect, has been developed on the basis of the above hypothesis to describe the transient and steady-state creep of polycrystalline materials at elevated temperatures. It is shown that the model is in agreement with observed effects of stress, strain, time, temperature and grain size on the contribution of grain boundary sliding strain to the total creep strain. The success of the proposed model lies in its ability to unify a large number of apparently conflicting experimental observations on grain boundary sliding and creep of polycrystalline materials at high homologous temperatures.

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