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by N.K. Sinha

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MICROCRACK-ENHANCED CREEP IN POLYCRYSTALLINE MATERIAL AT ELEVATED TEMPERATURE

N. K. SINHA

Institute for Research in Construction, National Research Council of Canada, Ottawa, Canada K1A 0R6

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Abstract—A grain size dependent rheological model is presented for high temperature creep that is capable of predicting both primary and tertiary as well as secondary creep. Primary and tertiary creep rate are shown to be strongly influenced by grain size, whereas secondary or minimum creep rate is rather insensitive to it. During the primary creep stage creep rate increases with decreasing grain size, but the reverse is true in the tertiary or accelerating range. An increase in grain size also dictates decrease in the time to reach minimum creep rate concomitant with a decrease in strain. The model is based on intragranular dislocation creep enhanced by grain-facet size cracks produced during deformation by the embrittlement process that is caused by an intergranular sliding mechanism. Incorporation of the kinetics of microcracking activity is the foundation of the theory.

Résumé—On présente un modèle rhéologique dépendant de la taille des grains pour le fluage à haute température; ce modèle est capable de prédire les stades primaire et tertiaire, ainsi que le stade secondaire. On montre que les vitesses de fluage primaire et tertiaire sont fortement influencées par la taille des grains, ce qui n'est pas le cas de la vitesse de fluage secondaire ou fluage minimal. Pendant le stade primaire, la vitesse de fluage augmente quand la taille des grains diminue, mais l'inverse se produit pendant le stade tertiaire ou stade d'accélération. Une augmentation de la taille des grains conduit également à une diminution du temps nécessaire pour atteindre la vitesse de fluage minimale, concomitante avec une diminution de la déformation. Le modèle est basé sur le fluage des dislocations intragranulaires, favorisé par des fissures de la taille d'une facette de grain, produites pendant la déformation par la fragilisation due au glissement intergranulaire. L'incorporation de la cinétique de la microfissuration constitue le fondement de la théorie.

Zusammenfassung—Es wird ein von der Korngröße abhängiges rheologisches Modell des Hochtemperaturkriechens vorgelegt. Dieses Modell kann sowohl primäres und tertiäres wie auch sekundäres Kriechen voraussagen. Die Rate des primären und tertiären Kriechens wird, wie gezeigt wird, von der Korngröße stark beeinflusst, wohingegen die minimale Kriechrate des sekundären Kriechens ziemlich unempfindlich dagegen ist. Im Bereich des primären Kriechens nimmt die Kriechrate mit abnehmender Korngröße zu; das Gegenteil ist im tertiären oder beschleunigten Bereich der Fall. Zunehmende Korngröße bedingt auch eine Abnahme in der Zeit bis zur minimalen Kriechrate, welches einhergeht mit einer abnehmenden Dehnung. Das Modell baut auf dem Versetzungskriechen im Korn auf, welche verstärkt wird von Rissen in Größe von Korngrenzfacetten; diese Risse werden während der Verformung durch den Versprödungsprozeß erzeugt, der durch einen intergranularen Gleitmechanismus hervorgerufen wird. Grundlage der Theorie ist die Berücksichtigung der Kinetik der Mikrorißaktivität.

INTRODUCTION

Engineering design of structural components for high temperature applications can be severely constrained by the creep properties of the materials in use. Creep properties, in turn, are dictated by microstructural changes that occur during deformation. A common change is the formation of grain-boundary cavities and cracks leading to brittle response, known generally as elevated temperature embrittlement. As a consequence, designs for high temperature applications must give due consideration to the life expectancy, under operational conditions, of the material to be used. This is commonly achieved by examining constant stress deformation properties.

A typical creep curve is shown for total strain ϵ , curve (b), in Fig. 1, for uniaxial constant stress history σ , curve (a). The strain rate history $\dot{\epsilon}$, curve (c), shows a decelerating or primary creep stage up to

about time t_1 , followed by a secondary or quasi steady state from t_1 to t_2 and an accelerating or tertiary stage beyond t_2 . Failure occurs at t_3 by rupture or fracture. A frequently observed phenomenon, often used as the basis of engineering designs, is the inverse proportionality of failure time and strain rate [1]. This relation is given by the constancy of the product of time t_m , to reach minimum creep rate (hence the first stage of tertiary creep) and the minimum creep rate, $\dot{\epsilon}_m$.

This phenomenologically established relation has had a profound influence on the development of creep fracture models in the last thirty years. Engineering design, however, incorporates some type of safety factor that is usually based on the allowable deformation or damage state under actual operational conditions rather than an ultimate failure. The success of a creep fracture model must therefore

Fig. 1. Strain and strain rate histories for a constant stress loading condition (calculated using theory developed).

be judged not only on its compatibility with the Monkman–Grant [1] type relation but also on its ability to describe the entire creep curve, preferably the transient range and the beginning of the tertiary stage.

For moderate stresses relevant to many engineering applications, $\sigma > 1 \times 10^{-5} E$, where E is the Young's modulus, the deformation properties at temperatures greater than about $0.4 T_m$, where T_m is the melting point in Kelvin, are affected by a grain-boundary sliding mechanism and wedge type microcracks [2, 3] that develop at or are associated with the grain boundaries (Fig. 2). The enhancement of creep rate by these micromechanisms will be considered. It will be shown that the failure time based on time t_m to reach $\dot{\epsilon}_m$ and the above criteria can be modelled quite simply. In addition to the usual external variables of stress, time and temperature, the theory considers two internal variables: grain size, and the microcracks that develop during loading.

BASIS OF ANALYSIS

For a material with a given texture and fabric, deformation is macroscopically dependent on strain rate. Creep rate has been observed to depend on applied stress, time (or strain) and temperature. A consequence of this dependency is the development of constitutive equations of the general form

$$\dot{\epsilon} = \dot{\epsilon}(\sigma, t \text{ or } \epsilon, T). \quad (1)$$

A more refined constitutive equation would be one that incorporates the state of the material [4] and the history of loading. Ideally, the state of a material body is characterized by "all" the properties of the body, but this is impracticable because the processes under discussion are all thermodynamically irreversible [5]. In addition, understanding of material

response at elevated temperatures is still in its infancy. It is extremely difficult to measure at high temperatures even the few basic properties that are known to play dominant roles [6]. Macroscale strain and microscale defects such as dislocations, spheroidal voids or cavities, cracks and solid or liquid inclusions fall into this category, and these are only a few of the microstructural variables that describe the state of the body as the process of deformation progresses [7, 8]. Microdefects may already exist in the body and multiply, or they may form during further deformation. Efforts to develop constitutive equations based on the state variables [5, 9–12] have met with limited success. A more refined system of equations would be required, in principle, to account for the various microstructural parameters for such a presentation. Thus

$$\dot{\epsilon} = \dot{\epsilon}(\sigma, t, T, V_i) \quad (2)$$

$$V_i = V_i(\sigma, t, T, \dots) \quad (3)$$

where V_i represents other internal and external variables that could be influenced by σ , t and T . Expressions giving the dependence of creep rate on microstructural variables and equations of state are required. This paper will focus specifically on how creep rate is affected by grain size and microcracking activity during deformation. A generalization of the above relations to multiaxial stresses can only be made if one has a good understanding of general behaviour under uniaxial stress conditions.

Close examination of Fig. 2(a–d) reveals that crack size is directly dependent on grain size. Grain size must therefore be incorporated into the constitutive equation if the effect of damage due to microcracks (either present initially or nucleated during deformation) and variation in the grain structure are to be considered

$$\dot{\epsilon} = \dot{\epsilon}(\sigma, t, T, d, N) \quad (4a)$$

Fig. 2. Intergranular cracks in fine-grained (a) and coarse-grained (b) ice at $0.96 T_m$ (c) intergranular and triple point cracks and cavities in test specimen L138 (see Appendix A). Straight lines are small-angle tilt boundaries developed during deformation (d) intracrystalline crack and smaller cracks associated with the grain-boundary irregularities (e) multi-facet length cracks, parallel to compression axis, and large angle cracks formed during tertiary creep regime.

$$d = d(\sigma, t, T, N) \quad (4b)$$

$$N = N(\sigma, t, T, d) \quad (4c)$$

where N represents the crack or void density. Equation (4b) reflects the fact that the initial grain size, d , could change during deformation because of the formation of tilt and small-angle boundaries [Fig. 2(c)], grain-boundary migrations, cell or sub-grain formation [Fig. 2(d, e)] and dynamic recrystallization. This change in the texture depends not only on σ , t and T (and the initial grain-size distribution, grain orientation, pre-existing flaws, dislocation density, etc.), but also on subsequent cracking activities and the associated effect of dilatation that induces local triaxial situations and heterogeneous deformation. In order to simplify, grain size, d , will be assumed constant and equal to the initial size in the theory to be presented.

The question remains however: how to formulate equation (4c) for N , and how to introduce the effect of grain size and grain-size dependent cracks and their density on the creep rate in equation (4a)? Simple explicit forms of equations (4a) and (4c) will be developed to this end.

HIGH TEMPERATURE CREEP RATE

Most investigations, both experimental and theoretical, of high temperature creep phenomena have concentrated on quantifying the steady-state condition that occurs during period t_1 to t_2 (Fig. 1). A fair degree of understanding of steady-state creep rate (also referred to as secondary or minimum creep rate)—to be called “viscous creep rate,” $\dot{\epsilon}_v$, in this paper—has been developed for low and intermediate stress levels, $\sigma < 1 \times 10^{-4} E$. The strain rate is dominated by grain-boundary diffusion for $\sigma < 1 \times 10^{-5} E$. It is controlled by the mobility of dislocations in the range $1 \times 10^{-5} E \leq \sigma < 1 \times 10^{-3} E$ and may be described by a quasi-empirical equation of the form [13, 14]

$$\dot{\epsilon}_v = A \frac{EbD_v}{kT} \left(\frac{\sigma}{E} \right)^n \quad (5)$$

where A and n are constants, b is the Burger's vector, D_v is the lattice self-diffusion coefficient, and k is Boltzman's constant. An improved version of equation (5) includes an additional term for pipe diffusion along the dislocation cores, invoking a low temperature mechanism [15]. It fits the experimental results on minimum creep rate better in the moderate stress range up to $1 \times 10^{-3} E$. Equation (5) bears close resemblance to the following empirical relation, known popularly as “the power law”

$$\dot{\epsilon}_v = \dot{\epsilon}_{v_0} \left(\frac{\sigma}{\sigma_0} \right)^n \exp(-\Delta H/kT) \quad (6)$$

where $\dot{\epsilon}_{v_0}$ is the viscous creep rate for the reference or unit stress, σ_0 , and ΔH is the activation energy for creep.

In spite of the well established Hall [16] and Petch [17] relation for low temperature, which gives the lower yield point as inversely proportional to the square root of grain size, equations (5) and (6) contain no grain size effect. The intragranular slip mechanism, which is grain-size independent, is the basis of equations like type (5), and the value of the constants can be adjusted to fit well with the empirical relations of type (6) or its improved version, expressed as a sinh function [18]. The argument against any grain size effect is the observation that creep in the grain matrix occurs at all stress levels at high temperatures (primarily because non-elastic strain can readily be detected) and the concept of a yield stress does not apply. This low-temperature concept of a yield stress, however, is now undergoing re-examination [19]. An example of the non-applicability of the Hall-Petch relation to the tensile strength of an austenitic stainless steel at high temperatures was discussed by Crussard and Tamhankar [20].

Equations (5) and (6), moreover, provide neither information on creep rate during the period $t = 0$ to $t = t_1$ (Fig. 1), an important range in the service life of structural components, nor any information on the duration, t_1 to t_2 , of the steady-state period that is known to depend on σ and T , and therefore no indication of the onset of accelerating creep. Additionally, these equations do not clarify the kinetics of damage or the effect of damage due to the microcracking activities acknowledged at these stress levels. The effect of grain size is significant, since grain boundaries act as barriers to dislocations, whether gliding or climbing, and there are more boundaries in fine-grained than in coarse-grained material.

At low stresses viscous flow occurs in the grain matrix, inducing no macroscopic volumetric strain; a continuity in the microstructure is maintained during deformation without any development of voids. While this occurs at stresses $< 1 \times 10^{-5} E$ and small strains (where grain-boundary diffusion also plays an important role), it cannot be assumed valid at moderate to high stresses. Cavities and cracks induce volumetric change, and pure (macroscopically speaking) viscous flow does not occur without being accompanied by the strain caused by the voids (change in volume). Experimental data compiled on metals by Miller and Langdon [21] indicate that the total cavity volume fraction rarely exceeds 1%. Volumetric strain induced by the formation of voids is indeed very small (Appendix A), and contributions (due to voids) to the total strain may not be noticed or measured readily in most cases [22]. Cavities and cracks, however, have a profound effect on the redistribution of internal stresses and subsequent creep in the matrix (see Appendix A). In fact, even in materials that exhibit superplasticity, the earlier theory that cavities do not form during superplastic deformation [23] is no longer valid; the problem of cavitation failure in superplastic materials has become significant [24].

Consequently, steady-state flow (in the classical sense) does not occur at levels of stress of engineering concern; there is only a minimum creep rate period. It is therefore important to make a clear distinction between viscous creep rate, which occurs all the time (along with other mechanisms) during creep loading, and minimum creep rate, which occurs at a specific time:

1. *Viscous creep rate*, $\dot{\epsilon}_v$, is the "minimum" value of the grain matrix creep rate, but the "minimum" rate, $\dot{\epsilon}_m$, may not be equal to the viscous creep rate.
2. *Viscous creep* occurs continuously during creep, but the minimum creep rate occurs at a specific time for a given loading condition.

PRIMARY CREEP

Experimental observations by Crussard and Tamhankar [20] on the creep rate of austenitic stainless steel at temperatures of 923–1173 K, those of Garofalo *et al.* [25] on an iron-base austenitic alloy at 977 K, plus those of Barrett *et al.* [26] on copper, and of Malakondaiah and Rama Rao [27] on titanium provide ample evidence that creep properties during the transient period ($t < t_1$) must depend on grain size; further, they indicate that fine-grained material creeps more rapidly than its coarse-grained counterpart during this early creep period. Bird *et al.* [13] suggested, though qualitatively, that the observed increase in creep rate with decreasing grain size could be due to the increasing contributions of grain-boundary sliding to the overall creep rate. Quantitative support for this idea has been provided by Sinha [28]. After extensive review of experimental data on metals, alloys, and non-metals such as ceramics and ice, Sinha [28] hypothesized that the transient creep is associated with the delayed-elastic effect. He showed, further, that the delayed-elastic effect is associated with the grain-boundary sliding phenomenon and that both mechanisms have the same activation energy as the overall creep response. For conditions where Nabarro–Herring, Coble, or Ashby–Verrall [29] types of diffusion creep are not dominant and where the dislocation motion is the rate controlling mechanism, the overall creep rate (during the early primary creep period) is given by the sum of the delayed-elastic strain rate, $\dot{\epsilon}_d$, and the viscous strain rate, $\dot{\epsilon}_v$ [28]

$$\dot{\epsilon} = \dot{\epsilon}(\sigma, t, T, d) = \dot{\epsilon}_d(\sigma, t, T, d) + \dot{\epsilon}_v(\sigma, T). \quad (7)$$

At $T = \text{constant}$, equation (7) may be given explicitly as

$$\dot{\epsilon} = \frac{c_1 d_1 b}{td} (a_T t)^b \left(\frac{\sigma}{E}\right)^s \exp[-(a_T t)^b] + \dot{\epsilon}_v \left(\frac{\sigma}{\sigma_0}\right)^n \quad (8)$$

where c_1 is a constant corresponding to the unit grain size, $d_1 (= 1 \text{ m})$, E is the Young's modulus given by the lattice elastic constants, a_T is the inverse relaxation time corresponding to the temperature T , b is

a constant ($\approx 1/n$) and s is the stress exponent for grain-boundary sliding displacement.

Equation (8) has been shown by Ashby and Duval [30] to agree closely, up to the minimum creep rate, with recent experimental observations on polycrystalline ice carried out by Jacka [31]. It describes the overall creep rate as a decay function of time and the temperature effect as obtained from the activation energy [28]. It does not show, however, any accelerating creep.

CRACKING ACTIVITY DURING CREEP

It has been shown that the first grain-facet size cracks form in a polycrystalline material when the average grain-boundary sliding (gbs) displacement, $\bar{x}$, reaches a critical value, $\bar{x}_c$ [32, 33]. Direct experimental support for this concept comes from Morris and Harries [34], who studied type 316 austenitic stainless steel at 898 K. Experimental results on copper bicrystals by Watanabe [35] also provide support, although bicrystal results cannot, strictly speaking, be directly applied to polycrystalline materials. The formation of subsequent cracks is predicted on the basis of grain-boundary sliding displacement in excess of the critical gbs displacement [36], giving

$$N = N_c [\exp\{\psi(\bar{x} - \bar{x}_c)\} - 1] \quad (9)$$

where N_c is the crack density for $\bar{x}_c$ (the first cracks) and ψ is a constant; negative values of N are considered to be equivalent to no cracks. In equation (9) $\bar{x}$ is given as

$$\begin{aligned} \bar{x} &= \frac{\epsilon_d d}{K} = \frac{d}{K} \int_0^t \dot{\epsilon}_d dt \\ &= \frac{c_1 d_1}{K} \left(\frac{\sigma}{E}\right)^s \{1 - \exp[-(a_T t)^b]\} \end{aligned} \quad (10)$$

where $\dot{\epsilon}_d$ is given by the first term in equation (8) and K (assumed to be 1) is a constant arising from the equation of proportionality between gbs strain and average gbs displacement. Equation (10) gives a minimum stress, σ_m , for creep cracking as the stress for which $\bar{x}_c$ is reached at $t = \infty$

$$\sigma_m = E(K\bar{x}_c/c_1 d_1)^{1/s}. \quad (11)$$

As the lattice elastic constants and the fracture surface energy depend on temperature, $\bar{x}_c$ and hence σ_m and N depend on temperature. Substitution of $\bar{x}$ from equation (10) in equation (9) gives

$$\begin{aligned} N &= N_c \left\{ \exp \left[\psi \left(\frac{c_1 d_1}{K} \left(\frac{\sigma}{E}\right)^s \right. \right. \right. \\ &\quad \times \left. \left. \left. \{1 - \exp[-(a_T t)^b]\} - \bar{x}_c \right) \right] - 1 \right\}. \end{aligned} \quad (12)$$

CRACK ENHANCED CREEP

Each crack intensifies the matrix creep rate and the overall effect depends a great deal on the geometry of

these openings. The simplest form of cracks are planar in geometry, having one dimension infinitely larger than the other. For dilute concentrations of non-interacting, long narrow cracks oriented with their major planes perpendicular to a uniaxially-applied tensile load, and for materials obeying power law viscous flow, the enhanced creep rate at a constant temperature, $\dot{\epsilon}_{vc}$, following Weertman [37] and equation (6), is given by

$$\dot{\epsilon}_{vc} = \dot{\epsilon}_0 (1 + 2\pi N a^2 n^{1/2}) \left(\frac{\sigma}{\sigma_0} \right)^n \quad (13)$$

where N is the number of cracks that cross a unit cross-sectional area oriented perpendicular to the plane of the cracks and all the cracks are assumed to have the same size, with width of $2a$. The condition for non-interaction is defined by $a^2 N \ll 1$.

If the cracks are assumed to be the same size as those of the grain facets and if the cross-sectional geometry of the grains is assumed to be hexagonal [Fig. 2(a, b)], then the crack size, $2a$, can be expressed in terms of the grain diameter, d , according to

$$a^2 = \frac{\pi}{24\sqrt{3}} d^2 \quad (14)$$

where d is the diameter of a circle having the same area as the average cross-sectional area of the grains. Substitution of a^2 from equation (14) in equation (13) gives

$$\dot{\epsilon}_{vc} = \dot{\epsilon}_0 \left(1 + \frac{\pi^2}{12\sqrt{3}} N d^2 n^{1/2} \right) \left(\frac{\sigma}{\sigma_0} \right)^n. \quad (15)$$

Replacing the second term in equation (8) by the right side of equation (15), creep rate incorporating crack enhancement is obtained

$$\dot{\epsilon} = \frac{c_1 d_1 b}{t d} (a_T t)^b \left(\frac{\sigma}{E} \right)^s \exp[-(a_T t)^b] + \dot{\epsilon}_0 \left(1 + \frac{\pi^2}{12\sqrt{3}} N d^2 n^{1/2} \right) \left(\frac{\sigma}{\sigma_0} \right)^n. \quad (16)$$

As the first term is a decay function of time, equation (16) predicts a decelerating creep rate leading to a steady-state or viscous creep rate in a virgin material, provided $\sigma < \sigma_m$. For $\sigma > \sigma_m$ where cracks form and multiply during deformation, as given by equation (12), the second term in equation (16) contributes an increasing creep rate with time after the onset of cracking activities. The opposite effects of the two terms in equation (16) lead to a minimum creep rate, followed by an accelerating or tertiary stage. Equations (16) and (12), therefore, describe both strain rate and damage (crack density) in a polycrystalline material undergoing creep. It should be mentioned that further refinements can be made by introducing the effect of the cracks on the elastic components, the effect of cracks in the dilatation, grain size and hence crack size distribution, the change in grain size and formation of subgrains, etc.

DISCUSSION

An essential element of the above presentation is the emphasis assigned to mechanisms that are generally believed to play an important but not dominant role at elevated temperatures. Grain-boundary sliding and the associated accommodation processes have been shown to generate grain-size dependent and decreasing creep rate with time during the pre-minimum creep rate period. It is this mechanism again that is shown to be responsible for generating intergranular cracks that enhance the matrix creep rate and lead to the tertiary stage. The general characteristics of a typical creep curve are shown to be governed by the gbs mechanism. In view of this, the overall creep curve may be thought of as a response curve generated by modulating the effect of the processes associated with the intergranular mechanisms. Questions remain however: How well do equations (16) and (12) describe the usual macroscopically observed creep rates? Can they be used in a predictive manner?

In addressing these problems it is necessary that the material constants are known or that they can be estimated from the available experimental data, but it is not an easy task owing to the numbers involved. Leaving aside the unit dimensions, namely d_1 , σ_0 , N_c and K ($=1$, assumed), there are nine unknown material constants in equations (16) and (12) to be determined.

The value of E is known for most materials, and in any case can be obtained easily using seismic methods. As grain-boundary shearing is, strictly speaking, not independent of intragranular processes (because of the accommodations required at grain corners and imperfections at the boundaries), b is related to n . It has been shown by Sinha [38] that for ice $b = 1/n$, a value that has also been used recently by Ashby and Duval [30]. Whether this simple relation applies to other materials remains to be seen. In any case, it may be assumed that b can be estimated from n . This leaves seven unknown constants to be determined from creep tests.

The values of $\dot{\epsilon}_0$ and n can be determined from the steady-state creep rates or preferably from the measured permanent strain [38] after unloading, provided (1) that the tests are carried out for $\sigma < \sigma_m$ to avoid any effect of microcracking, and (2) that some consideration has been given to avoiding test conditions (not too low a stress or too fine-grained a material) dominated by other mechanisms such as the formation of rounded cavities [39] or grain-boundary diffusion [27]. The value of a_T can readily be determined at the required temperature by obtaining the relaxation time from the strain recovery on unloading after constant stress tests, provided conditions (1) and (2) are met or that loading times are less than those for the onset of cracking activity for $\sigma > \sigma_m$.

Once a_T is known, c_1 and s can be estimated from either the strain recovery curve or the transient part

Fig. 3. Computed strain rates and cracking activity during creep for the strain history given in Fig. 1.

of the forward creep. Alternatively, grain-boundary sliding can be measured for the determination of c_1 and s . Determination of x_c and ψ entails considerable difficulties, requiring metallographical studies [33–35]. Acoustic emission (AE) techniques are now well developed and can be used, provided interpretation of AE data is made in conjunction with microstructural investigations (see Appendix A).

Considerable progress has been made in the field of ice mechanics with regard to determining material constants at high temperatures. Those available for ice provide sufficient information to permit calculations and to demonstrate the applicability of the theory to polycrystalline materials in general. The following calculations make use of equations (16) and (12) and the creep constants for ice given by Sinha [28] plus values of x_c , N_c and ψ [33, 36].

The dependence of the components and the total

strain rates in ice on time at $0.96 T_m$ (-10°C) for a given grain size is shown in Fig. 3; creep strain for this example is indicated in Fig. 1. Note that $\dot{\epsilon}_d$ dominates the overall creep rate during the transient period, whereas crack-enhanced viscous creep dominates the tertiary regime. Although the cracks start to form rather early in the transient regime, their enhancement effects on the average matrix creep rate can be detected only after some time has elapsed following the onset of cracking activity. Note also in Fig. 3 the approach of a steady cracking rate as the minimum creep rate is approached.

Figure 4 shows the effect of stress on the time dependence of overall creep rate. It should perhaps be pointed out that all the characteristics shown in this figure have been observed in other materials. For $\sigma < \sigma_m$ ($\approx 5 \times 10^{-5} E$ at $0.96 T_m$), creep rate decreases with time and approaches a steady-state condition.

Fig. 4. Theoretical stress and time dependence of strain rate.

Fig. 5. Stress and grain size dependence of the time to reach minimum creep rate.

With increase in stress level, the strain rate passes through a minimum. The duration of the “secondary stage”, at which the strain rate maintains a quasi steady-state value, decreases with increase in stress. The locus of the minima within the family of curves (the broken line in Fig. 4) indicates the dependence of t_m on $\dot{\epsilon}_m$ and hence σ (Fig. 5). This locus is curved, but can be represented approximately, for phenomenological purposes, by two straight lines: one for the higher stresses and one for the lower ones. These, again, have been reported widely and are popularly known as the “Brittle” and “Ductile” ranges, respectively [40]. Each line may be represented as

$$\log t_m = \text{constant} - \log \dot{\epsilon}_m$$

giving

$$t_m \dot{\epsilon}_m = \text{constant}. \quad (17)$$

The inverse proportionality between t_m and $\dot{\epsilon}_m$ indicated by equation (17) bears a striking resemblance to the Monkman and Grant [1] equation for brittle fracture and Hoff's equation for ductile rupture in pure metals and alloys [41].

Since the minimum strain rate, $\dot{\epsilon}_m$, occurs at some finite time when strain rate due to delayed elastic effect is still having a finite value (Fig. 3). $\dot{\epsilon}_m$ must be greater than $\dot{\epsilon}_v$. The difference between $\dot{\epsilon}_m$ and $\dot{\epsilon}_v$, however, depends on stress and is illustrated in Fig. 6, which indicates the increasing deviation of $\dot{\epsilon}_m$ from the straight line (on log-log ordinates) for $\dot{\epsilon}_v$ [equation (6)] as stress increases. This characteristic increase in $\dot{\epsilon}_m$ with stress is known as “power-law breakdown” and has been widely reported and empirically presented in terms of equation (6) by increasing the stress exponent n [42, 43] or, alternatively, by the sinh function suggested by Garofalo [18]. In ice with grain size of about 1 mm and stress up to $9 \times 10^{-4} E$, a sinh function was used by Barnes *et al.* [44] to fit their data. They attributed the effect to

dynamic recrystallization without, however, providing any microstructural evidence. Perhaps it should be mentioned, however, that the theoretical curve for fine-grained ice (based on crack-enhanced creep) in Fig. 6 agrees extremely well with their experimental data [44].

Although the minimum strain rate does not show any dependence on grain size at intermediate stresses,

Fig. 6. Stress and grain size dependence of the minimum creep rate and the viscous creep rate without crack enhancement.

Fig. 7. Theoretical time, stress and grain size dependence of strain rate.

Fig. 6 indicates a pronounced grain size effect at higher stresses, due entirely to higher cracking activity. The minimum strain rate is predicted here as increasing with decrease in grain size. This could therefore provide an alternative explanation for the reappearance of grain-size effect in creep rate at high stresses and temperatures, reported by many investigators. Experimental data on an austenitic stainless steel by Garofalo *et al.* [25] and by Takahashi and Yamane [42] are of particular interest. An increase in $\dot{\epsilon}_m$ with decrease in grain size and concomitant increase in $\dot{\epsilon}_m$ with σ , requiring a sinh function or change in n , indicate that cracking activity must have played an important role in these tests.

A better appreciation of the effect of grain size on creep processes can be made if the time aspect of creep is examined, since time can be measured more accurately than any other variable. A few calculations are given in Fig. 7 to illustrate the effect of grain size and stress on the time dependence of the overall creep rate; it, along with Fig. 5, shows that the time required to reach the minimum creep rate decreases significantly with increase in grain size. For a given stress in the intermediate-to-high stress range the tertiary creep regime and the effect of microcracks should, therefore, become more pronounced as grain size increases. In other words, the theory predicts a decrease in life time with increase in grain size. Creep curves obtained by Mannan and Rodriguez [45] for Type 316 stainless steel certainly support these predictions. A comparative study of the creep response of ice and steel at elevated temperatures is presented in [46].

Experiments were conducted on ice to test various aspects of the theory. Although a detailed description of this series is outside the scope of the present paper, examples are shown in Fig. 8 for transversely isotropic (S-2) columnar-grained ice with load ap-

plied in the plane of isotropy. In this material, the deformation is essentially two dimensional, $\epsilon_1, \epsilon_2 > \epsilon_3$ (see Appendix A). Note the dilatation effect indicated by the transverse strain, ϵ_2 , during tertiary creep. The transverse strain rate, $\dot{\epsilon}_2$, increased more rapidly with time than the axial strain rate, $\dot{\epsilon}_1$, as the crack density increased.

CONCLUSIONS

1. To avoid much of the confusion that abounds in the literature, a clear distinction has been drawn between viscous creep rate, which occurs continuously during load application and is associated with no volumetric strain, and the "secondary" or "minimum creep" rate that occurs at a specific time and depends on loading condition.

2. For conditions where grain matrix deformation is dominated by the mobility of dislocations, minimum creep rate at lower stresses or strains where no voids or cracks form is practically independent of grain size and equal to viscous creep rate.

3. Minimum creep rate for deformation caused by microcracking is greater than viscous creep rate. The deviation increases with stress, leading to breakdown in the power law. In the regime of power law breakdown, the minimum creep rate exhibits significant grain size sensitivity and increases with decrease in grain size.

4. Most obvious changes in creep rate that can be observed macroscopically during creep, for $1 \times 10^{-5} E < \sigma < 10^{-3} E$, are governed by the intergranular sliding mechanism and its role in forming cracks and hence the grain matrix creep. The contribution of grain-boundary sliding strain to total strain adds to strain rate during the transient period, whereas grain-boundary sliding induced cracks enhance the matrix deformation and dominate the creep

Fig. 8. Constant stress tertiary creep in laboratory grown fine- and coarse-grained ice.

process during tertiary creep. The minimum strain rate occurs as a result of the two effects on the intragranular creep mechanism.

5. Brittle fracture failure (Monkman-Grant equation), ductile rupture failure (Hoff equation), and the transition zone can be formulated on a micromechanically based pair of simple relations showing that the time to the onset of tertiary creep decreases with increase in grain size. A loss in ductility for coarse grained material at higher stresses is predicted.

6. Only after considering micromechanics and performing specific tests should values be assigned to the material constants. Creep tests should be conducted in conjunction with the measurements of volumetric strain, microcracking activities, creep recovery and metallographic studies.

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APPENDIX A

Uniaxial constant-load creep tests were carried out on rectangular specimens of polycrystalline ice $250 \times 100 \times 50$ mm; load was applied parallel to the length of the specimens. The ice was made from distilled, de-aerated and de-ionized water using a special seeding and selection technique [38]. It was pure, bubble-free, transparent, transversely isotropic and columnar-grained. The long dimensions of the specimens were normal to the length of the grains. A closed-loop servohydraulic test machine (MTS) situated inside a cold room was used to apply load, using the feed-back load control system. To avoid any cracking activity due to load application, the load was applied at a rather low stress rate of $0.5 \text{ MN} \cdot \text{m}^{-2} \text{ s}^{-1}$. After the desired full load was reached (in a few seconds), it was maintained constant until the end of the test period. Axial as well as lateral strains in two mutually perpendicular directions were recorded using three commercially available displacement gauges (MTS) mounted directly on the specimen by means of special brackets (made at NRCC) frozen to the specimen surfaces (Fig. A1). Microcracking activity during creep was recorded by a microprocessor-based acoustic emission system (AET-5000) and a detector mounted on the specimen. Cracking activity was also recorded visually using an event marker primarily to identify the acoustic emission (A.E.) characteristics associated with the formation of grain-facet size cracks large enough to be visible.

An example of test results at $-10^\circ\text{C} \pm 0.1^\circ\text{C}$ is shown in Fig. A2. This test was continued for a period long enough to ensure that the tertiary creep regime was well established before the specimen was unloaded. Load was removed quickly, and creep recovery was continuously recorded (for a long time) until no further recovery could be detected.

As the final strain was small, the test results presented can be assumed to be those for a constant stress test, although strictly speaking it was the load that was maintained constant. The main purpose of citing this example is to show

Fig. A1. Specimen ($50 \times 100 \times 250$ mm) before (a) and after (b) the creep test (No. L138) at: $\sigma = 1.26 \times 10^{-4} E$ ($1.2 \text{ MN} \cdot \text{m}^{-2}$), $T = 0.96 T_m$, $d = 6.0$ mm.

the extent of damage due to microcracking, as may be seen in Fig. A1(b), a photograph taken after the test and following full recovery. The quantitative measure of damage within the specimen may be seen in Figs A2 and A3. A volumetric increase of slightly over 1% was recorded. This is certainly due to the dilatation effect of cracking activity that occurred during the creep period.

A detailed presentation of the three-dimensional aspect of the strain field is outside the scope of this paper. It is sufficient to say that microcracking activity plays a major role during tertiary creep at elevated temperatures for loading conditions conducive to the production of cracks.

Fig. A2. Dependence of axial, lateral and volumetric strain on time in transversely isotropic (S-2) ice sample ($50 \times 100 \times 250$ mm).

Fig. A3. Time dependence of acoustic emissions with event durations greater than 3 ms (visible or grain facet size).

This is also an opportune moment to mention that the use of columnar-grained ice (which is one of the most common types of ice in nature) adds to the usefulness of the measurements; its transparency assists in clarifying the interpretation of the effects of cracks. As the ice consists of long, pencil-like grains, deformation is essentially two-dimensional during most of the period and the strain is small along x_3 , the direction parallel to the length of the grains (Fig. A2). As for the cracks, they are long and narrow, with their major planes tending to be in the x_1 - x_3 plane; their lengths parallel to the length of the grains along the x_3 axis; and their widths parallel to the x_1 axis. A consequence of this geometry is the fact that the dilatation effect of the cracks should be noticed primarily in the lateral strain, ϵ_2 . An increase in ϵ_2 and its rate in relation to ϵ_1 , in conjunction with the rapid change in the volumetric strain, can be seen clearly during the later part of the tertiary creep period, greater than about 3000 s. Figure A3 shows that the theory estimates well the number of visible (grain-facet size) cracks up to about this time. Visible cracks were identified in the specimen, with the A.E. events having event durations greater than about 3 ms.

Analysis of a mid-plane, double-microtomed, thin section at right angles to the length of the columnar grains indicated the average cross-sectional grain diameter (area basis) to be 6.0 mm. Microstructural studies made on this thin section are illustrated in Fig. 2(c, d, e). Figure 2(c) shows intergranular and triple point (boundary) cracks and cavities. Grain boundaries and larger defects, such as tilt boundaries (straight lines) associated with the cracks, rounded cavities and kinks or jogs at the grain boundaries, are revealed by thermal etching. Figure 2(d) illustrates an intra-crystalline crack and two smaller cracks associated with the formation of irregularities at the grain boundaries (double lines correspond to the top and bottom surfaces of the thin section). Large transcrystalline cracks, parallel to the compression axis, and smaller cracks at large angles to the load axis [Fig. 2(e)] are indicative of crack interactions and gross non-uniformity in the stress or strain field developed during the later stages of creep. Smooth grain boundaries and lack of deformation features on the right-hand side of this micrograph show that the formation of the multiple-facet length crack was an efficient means of relieving the local stress concentration sites.