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CREEP MECHANISMS IN CERAMIC MATERIALS AT ELEVATED TEMPERATURES

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ABSTRACT

The creep of ceramic materials at elevated temperatures may take

place by the movement of dislocations within the lattice, by grain

boundary sliding, and/or stress-directed diffusion either through the

lattice or along the grain boundaries. Other accommodation mechanisms,

such as grain boundary separations, may also occur. Some indication of

the significant creep mechanism may be obtained by determining the depen-

dence of steady-state creep rate on stress, grain size, and temperature.

A comparison is made between the predictions arising from the theoretical

models and recent experimental data obtained on several materials in both

single crystal and polycrystalline form.

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I. INTRODUCTION

An attraction of ceramic materials is the possibility of their use as structural components at high temperatures, but some knowledge is then required of the plastic deformation, or creep, which occurs when the materials are subjected to a constant load or stress. Creep tests in the laboratory are primarily concerned with a determination of the rate-controlling mechanism under a given set of experimental conditions; but it is also necessary to know whether these results may be extrapolated to predict the behavior of the material under the much slower creep rates usually required in practice. Although it is not normally possible at the present time to meaningfully extrapolate existing data over wide ranges of stress or temperature, recent creep studies on ceramics have added substantially to our knowledge of creep mechanisms and is providing a foundation on which further work may be based.

This report is divided into two parts. Firstly, possible deformation mechanisms occurring during high temperature steady-state creep are reviewed; secondly, these mechanisms are examined with particular reference to the experimental results obtained from the research program on the compressive creep of ceramic materials conducted at the University of California at Berkeley.

II. THE MECHANISMS OF CREEP

In reviewing the mechanisms occurring during high temperature creep, it is convenient to make a division according to whether they are controlled by the movement of point or line defects. Furthermore, a direct comparison is made easier by using one basic creep equation for all mechanism types.

Creep is a thermally-activated process, and the secondary or steady-state creep rate, $\dot\varepsilon$, may be generally formalized under a given set of experimental conditions by an equation of the form

$$\dot\varepsilon = \frac{A}{kT} \left(\frac{\ell}{d}\right)^m \frac{\sigma^n}{G^{n-1}} D \qquad (1)$$

where A is a constant independent of temperature and having dimensions of length, k is Boltzmann's constant, T is the absolute temperature, ℓ is a length, d is the average grain size, σ is the applied stress, G is the shear modulus, D is the coefficient of diffusion $(=D_o \exp(-\Delta H/kT)$, where D_o is a frequency factor and ΔH is the activation energy for creep, and m $(= -(\partial\ln\dot\varepsilon/\partial\ln d)_{\sigma,T})$ and n $(= (\partial\ln\dot\varepsilon/\partial\ln\sigma)_{d,T})$ are constants. The significance of the length ℓ depends on the particular model under consideration, but it may be equated with $\Omega^{1/3}$, where Ω is the atomic volume and is equivalent to $\sim0.7b^3$ where b is the Burgers vector.

In general, each creep mechanism predicts a specific value for the constants m and n, but the constant A is usually not too well defined and depends on such unknown factors as the height of grain boundary ledges or the density of mobile dislocations. The mechanisms are therefore compared in Table I by presenting the predicted values for m and n and also noting the relevant value for D, where D_ℓ is the coefficient for

lattice self-diffusion, D_{gb} is the coefficient for grain boundary diffu-
sion, D_p is the coefficient for pipe diffusion along the dislocation
cores, and D^* is a diffusion coefficient related to the second phase
rather than to the host lattice.

With the exception of mechanisms V, VIII and IX, these processes were
developed for the creep of metals, and, whilst the major deformation
modes are probably included, the list is not exhaustive. For example,
Table I does not include the possibility of lattice diffusional creep
between sub-grain boundaries, due to the lack of experimental evidence
for such a process; this would give a creep rate faster than that pre-
dicted by the Nabarro-Herring equation[1,2] (because m = 0 since the con-
trolling sub-grain size is independent of grain size) and, if the size of
the sub-grains decreases with increasing applied stress, n > 1. Similarly,
the theory of creep due to the motion of jogged screw dislocations is
not included because of the problems associated with the interpretation
of this model,[10] although a recent analysis provides some evidence in favor
of jog-limited behavior.[11]

It is convenient to briefly examine these mechanisms before considering
in more detail the methods of determining the rate-controlling process.

A. Point Defect Mechanisms

Mechanisms I-IV are illustrated schematically in Fig. 1 for tests
performed under an applied stress, σ.

In Fig. 1(a), the grain boundaries perpendicular to the stress axis
experience a tensile stress and thus have an excess vacancy concentra-
tion, whereas boundaries parallel to the stress axis have a depletion of
vacancies. This gives rise to a vacancy flow, as indicated by the arrows,

and a consequent elongation of the grain. In the original theory[1,2] the flow of vacancies was assumed to occur through the lattice (I), but a later modification considered flow along the grain boundaries (II)[3], as indicated in Fig. 1(b). Under a compressive stress, the direction for vacancy flow is reversed and the grains become flattened. Mechanism II predicts a higher value for m and a diffusion coefficient of D_{gb} rather than D_ℓ; since the activation energy for diffusion along the grain boundaries is lower than that for diffusion through the lattice, it follows that this mechanism becomes dominant under conditions of lower temperatures (typically $\lesssim 0.6\ T_m$, where T_m is the absolute melting point) and smaller grain sizes. This diffusional flow is associated with some relative grain movement as an accommodation process to maintain coherency across the grain boundaries[12]; this movement gives rise to offsets in surface marker lines which are similar in appearance to those due to high-temperature grain boundary sliding[13] (c.f. mechanisms XII and XIII).

Ashby et al[4] have developed a model for diffusion-controlled sliding in a grain boundary containing a periodic array of ledge-pairs, such as AB and CD in Fig. 1(c), where sliding occurs due to the vacancy flow arising from the shear stress, τ, acting on the ledges. In this theory, which represents an improvement on the earlier model of Gifkins and Snowden[14], the rate of sliding on any boundary is controlled by the ledge-pair having the greatest height and length, and vacancy flow may again take place either through the lattice (AB, III) or along the grain boundaries (CD, IV). It has been shown that mechanism III is dominant at the higher temperatures (vide mechanisms I and II) and when the ledge-

pairs are widely separated (λ large in Fig. 1(c)).

Mechanism V refers specifically to a phenomenon associated with the presence of a liquid phase along the grain boundaries at the creep temperature, which is of greater importance in ceramic materials rather than metals. It is probably particularly significant in hot-pressed polycrystalline ceramics where an additive is used to aid densification; for example, this may account for the results obtained at high temperatures or hot-pressed BeO when there was an MgF_2 phase at the boundaries.[15] In addition, tests on ceramics have shown that impurities may segregate at the grain boundaries, even when only present in amounts as small as ~30 ppm,[16] but their effect on the nature of the creep mechanism is not clear.

B. Line Defect Mechanisms

Mechanisms VI to XIII refer specifically to models involving the movement of dislocations. In the original theory of climb-controlled creep (VI), it was assumed that the dislocations glide on their slip planes from Frank-Read sources, until the motion of the leading edge dislocations was blocked by dislocations of opposite sign on the adjacent planes.[5] A pile-up then occurred, and the back stress was relieved by the climbing together, and consequent annihilation, of the leading edge dislocations in each pair. A recent modification of this theory, involving groups of dislocation dipoles, yields the same values for m, n and D.[17] If some form of interaction occurs on the glide plane, such as solute drag, the rate of glide may be slower than the rate of recovery by climb, and the value of n is reduced from 4.5 to 3 (VII)[6]; an example of the latter is provided by the experimental results obtained on NaCl-KCl solid-solution alloys.[18]

Whilst mechanism VI predicts a stress dependence in reasonable agreement with that often observed experimentally in metals (n ~ 4.5), a disadvantage of the original theory was that it depended on the formation of piled-up arrays of edge dislocations. However, these arrays are not usually observed with the electron microscope, either in metals or in ceramics such as MgO, and it has also been shown that they are unstable. Since dislocation dipoles and loops are readily observed in deformed crystals, including MgO, an alternative procedure was suggested in which creep is controlled by dipole (VIII) or loop (IX) dissolution and n is 5 or 4, respectively.[7]

A modified form of diffusional creep is represented by mechanisms X and XI, for the situation where vacancies flow between dislocations within the lattice and the resultant climb occurs without glide. A constant internal stress was assumed in the original theory, and this gives values for n of 3 or 5 depending on whether the dislocations are sources and sinks for vacancies or, at lower temperatures, pipe diffusion occurs along the dislocation cores.[8] It has been pointed out that the introduction of an effective stress for climb and a periodic internal stress field gives values for n which can be very large.[19]

Grain boundary sliding is often overlooked as a possible deformation mechanism, although there is good evidence that it can be of considerable importance in polycrystalline ceramics with small grain sizes.[20] A possible mechanism for sliding is by the glide/climb movement of dislocations either in a zone adjacent to the boundary (XII) or along the boundary (XIII).[9] An alternative possibility is that sliding occurs by the pure glide of grain boundary dislocations having their Burgers vectors in the

plane of the boundary, but this seems unable to account for the experimental observations of sliding on a macroscopic scale.[21] Some accomodation process would also be necessary along the grain boundaries.

C. Identifying the Rate-Controlling Mechanism

Creep investigations are usually concerned with an identification of the principal deformation mechanism, although this determination may be difficult if the tests are conducted in a stress or temperature range which is a transition region where more than one process is significant. In some instances, such as for diffusional creep (I or II) and dislocation climb (VI), the mechanisms may contribute strain rates which are essentially additive; but in other cases, as when grain boundary sliding occurs, the interaction between the individual processes is less clear. The rate controlling mechanism is usually investigated by determining the values of ΔH, m and n.

A measure of ΔH may be of only limited use in ceramic systems at the present time, because the diffusion coefficients whether intrinsic, extrinsic or grain boundary, are usually not known with a high degree of accuracy. Furthermore, the atomistic nature of the deformation mechanism is poorly understood so that it is not clear which of the two atomic species controls the behavior; whilst the slower moving ion should be rate-controlling, such as the anion in stoichiometric oxides, there is some contrary evidence in materials of very fine grain size.

More information is obtained from a measure of the constants m and n, since some generalizations are then possible. Firstly, mechanisms involving point defects give Newtonian viscous behavior with n=1, whereas line defect mechanisms give n > 1. However, it should be noted that the

Nabarro-Herring model of diffusional creep assumes the grain boundaries to act as perfect sinks and sources for vacancies[1,2]; if this is not the case, so that the absorption or emission of vacancies influences the process rate, there may exist a threshold stress to give Bingham flow.[22] Secondly, line defect mechanisms occurring within the lattice show no dependence on grain size (i.e. m=0, mechanisms VI to XI). The constant m is only finite when the rate-controlling mechanism involves either (i) movement of vacancies along a diffusional path governed by the separation between the boundaries (as in I), or (ii) a deformation process occurring in the boundary zone (as in II-V, XII and XIII).

The diffusional path, for mechanisms I and II, whether through the lattice (m = 2) or along the grain boundaries (m=3), is identified at low stress levels by testing specimens covering a range of grain sizes; although even here the result may appear ambiguous, as shown by the value of m=2.5 reported for polycrystalline MgO obtained in bending.[23] In some ceramic materials there is a change in the value of n from unity at low stress to ~4.5 at high stress levels, suggesting a change in mechanism from diffusional creep to a dislocation process within the lattice. In these cases, an indication of the diffusional path may be obtained, even when tests are conducted on specimens of only one grain size, from the temperature dependence of the transition stress between the two mechanisms.[24]

Some information on the rate-controlling mechanism is also provided by the shape of the creep curves: two examples are shown in Fig. 2. In A, there is an instantaneous strain, ϵ_o, a primary stage I in which the creep rate is decreasing, a secondary, or steady-state, stage II in which the creep

rate is constant with increasing strain, and a tertiary stage III (observed in tensile creep) in which the creep rate increases to fracture. Stage I is associated with the formation of an intragranular substructure which then remains essentially constant during stage II; this behavior typically relates to mechanisms controlled by dislocation movement (with the exception of mechanism VII). In contrast, creep processes dependent on diffusion-controlled mechanisms exhibit no primary stage, and the deformation proceeds immediately in "steady-state" (curve B); this statement should be qualified however, since, as the average path length in mechanisms such as I is changing with increasing strain, the concept of a continuous steady-state is not strictly valid. For polygonal grains tested in axial tension, the strain rate tends to diminish with increasing strain; for axial compression, a quasi-steady-state may be maintained to a shortening of ~50%.[25]

III. COMPARISON WITH EXPERIMENTAL RESULTS

All of the experimental results discussed here were obtained from tests conducted in compression, thereby avoiding the problems associated with the correlation and interpretation of data from bending tests. For brevity, details of the various experimental procedures are not given, but full particulars are included in the appropriate references.

A. Lithium Fluoride

Lithium fluoride single crystals of $\langle 100 \rangle$ and $\langle 111 \rangle$ orientations were creep tested at temperatures from 650° to 750°C ($\sim$0.8-0.9 T_m)[26]. Figure 3 shows the steady-state creep rates obtained from isothermal tests plotted as a function of applied stress for $\langle 100 \rangle$ crystals; the points marked with an asterisk represent cube specimens. Values of n calculated from the three principal sets of data (open symbols) average 3.9. The effect of crystal orientation, asperity ratio, and purity appears to be minimal, at least in the high temperature range of this investigation; crystals of lower and higher purities (half shaded symbols) as well as crystals in $\langle 111 \rangle$ orientation exhibited similar behavior with n ranging from 3.4 to 3.9. The activation energy in all cases was $\sim$55 kcal/mole, which is similar to the value for intrinsic diffusion of F ions.

Tests were also carried out on polycrstalline LiF of two different grain sizes which differed by more than an order of magnitude ($\sim$ 160 μm and 3000 μm, respectively), and at temperatures in the range 300- 550°C ($\sim$0.50-0.72 T_m)[27]. Steady state creep rates, determined by change in stress experiments under isothermal conditions, are plotted as a function of the applied stress in Fig. 4. The results show no dependence on grain

size (m=0) so that, from Table I, the possibility of a point defect

mechanism is excluded. This is further proven by a plot of the ratios of

the steady-state creep rates ($\dot{\epsilon}_1$ and $\dot{\epsilon}_2$) and the stresses (σ_1 and σ_2) be-

fore and after a change in stress, as shown in Fig. 5. In this plot, a

least squares analysis yields n=7.6, and the experimental points are clearly

far removed from the expected trend for mechanisms is which n=1, as indicated

by the line for Nabarro-Herring creep through the point $\dot{\epsilon}_2/\dot{\epsilon}_1=1=\sigma_2/\sigma_1$. Since

the creep curves also exhibited both primary and secondary stages (type A

of Fig. 2), it appears that creep is controlled by dislocation motion.

B. Magnesium Oxide

Transparent polycrystalline MgO, produced with the aid of a process-

ing addition of LiF, was tested at 1200°C ($\sim$0.5 T_m), and the steady-state

creep rates were found to be independent of grain size (m=0) in the range

11.8-52 μm.[28] The results are shown in Fig. 6 for specimens having length:

width (ℓ/w) ratios of 4.0 and 1.52, respectively. The value obtained for

n was 3.3 and the deviation from theories of diffusional creep may be

appreciated by a comparison of the line of slope n=1 drawn through the point

for d=11.8 μm at the highest stress and the predicted lines for d=52 μm for

lattice (I, m=2) and grain boundary (II, m=3) mechanisms, respectively.

Figure 7 shows the data for the specimens having ℓ/w=1.52 replotted

as $\dot{\epsilon}$/D versus σ, where D is calculated from the extrinsic lattice diffusi-

vity of O^{2-}. Also shown are the theoretical predictions arising from

Nabarro-Herring diffusional creep (I) for d=11.8 μm and 33 μm, respectively,

the dislocation glide/climb mechanism controlled by climb (VI), and dis-

location climb (X) as formulated by Nabarro (N)[8] and reanalyzed by Weertman

(W)[17]. The experimental values for $\dot{\epsilon}$/D are intermediate between those

predicted by mechanisms VI and X, but the stress dependence is in better agreement with the latter.

C. Doped Polycrystalline Aluminum Oxide

Polycrystalline Al_2O_3, doped with 0.23 wt.% MgO and 0.22 wt. % NiO, was creep tested at temperatures in the range 1300-1470°C ($\sim$0.68-0.75 T_m) both after hot-pressing at 1450°C ("hot-pressed") and after a subsequent long anneal at 1430°C ("annealed").[29] The results are shown in Fig. 8 for experiments where the stress was changed periodically during the test: the numbers represent the order of changing. Due to accompanying grain growth, the notation indicates both the initial and final average grain sizes. The results show n$\sim$1.1 for "hot-pressed" specimens and $\sim$1.3 for "annealed", with no apparent dependence on grain size (compare points 1 and 6 for the "hot-pressed" material at 1470°C). The activation energy for the creep of former specimens was $\sim$95 kcal/mole, and for the latter $\sim$125 kcal/mole.

The observation of m=0 suggests that deformation does not proceed by the usual mechanisms of diffusional creep, and the low value of n precludes the normal dislocation models. The results suggest instead the presence of localized plastic deformation within the grains at stress concentration points (such as triple edges) with accommodation by grain boundary sliding and then separation resulting from the low grain boundary strength.

D. Polycrystalline Magnesium Oxide With Lithium Fluoride

An unannealed specimen of polycrystalline MgO containing $\sim$0.95 wt.% LiF, and having $\sim$99.4% theoretical density relative to MgO, was placed under a compressive stress of 750 psi and heated at a rate of $\sim$250°C/hour.[30] Under these conditions, the specimen started straining at $\sim$630°C, and

showed a rapid increase in strain rate at ~830°C (Fig. 9). This corresponds closely to the melting point of LiF (848°C) and the lower temperature activity may be due to the presence of hydroxyl and carbonate anion impurities. True strains in excess of 55% were achieved without fracture, thus demonstrating a behavior similar to superplasticity. In contrast, a specimen showed only thermal expansion under equivalent conditions. Values for n of 1.2 and 1.1 were obtained for the unannealed specimen from changes in stress tests at 770°C and 850°C suggesting that primarily viscous flow occurs in a liquid phase at the grain boundaries, possibly as in mechanism V. The dependence on grain size was not evaluated.

IV. DISCUSSION AND CONCLUSIONS

The creep mechanisms summarized in Table I were developed on the
basis of the existance of a theoretically dense continuum which maintains
coherent grain boundaries during deformation. Some of these relationships
may need to be modified for use with ceramic materials, due to problems
associated with such features as preferential impurity segregation at the
grain boundaries (either in solution or as a separate phase), the presence
of significant porosity, and the limited number of active slip systems in
some crystal structures.

The effect of porosity on creep is not covered here, but the need
for some modification in existing theories is seen by the experimental
observation that the steady-state creep rate is markedly affected by the
degree of porosity within the system, even for theoretical densities
exceeding ∿95% as reported for BeO.[31] This sensitivity could arise if
dislocations were absorbed or emitted at pores within the lattice, although
the size and location of the pores may be of more importance than the total
volume of porosity per se.[32]

An indication of the rate-controlling mechanism is at present most
readily obtained from a measure of the grain size sensitivity, -m, or the
stress sensitivity, n. Intragranular deformation mechanisms, such as the
motion of lattice dislocations, are independent of grain size (m=0); those
processes directly controlled by the presence of a grain boundary or free
surface are dependent on grain size (m $\geq$ 1). Similarly, the mechanisms con-
trolled by the motion of the line defects have n > 1, whereas those controlled
by diffusion of point defects have n=1. The indicated variations of the

experimentally determined values of n from those indicated by model

systems are not completely clear.

It is also apparent that the observed behavior may be dependent

upon the method of testing. For example, an examination of the published

creep data on polycrystalline MgO indicates some values of m greater than

0 and large variations in n. A direct comparison of data obtained in

bending and compression is therefore difficult, especially if grain boun-

dary separation is important. Furthermore, it is expected that problems

may arise with the occurrence of barrelling at high strains in compressive

creep tests; this factor has not as yet been completely evaluated.

In conclusion, it appears that the complexity of the creep behavior

of ceramic systems has not always been fully appreciated. However, the

large volume of creep data now available is providing a background of

knowledge and some understanding upon which future meaningful studies can

be designed and performed.

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TABLE I. Creep Mechanisms at Elevated Temperatures

Mechanism (Point Defect Mechanisms)	M	n	D	Reference
(I) Vacancy diffusion through the lattice	2	1	D_1	1,2
(II) Vacancy diffusion along the grain boundaries	3	1	D_{gb}	3
Vacancy diffusion around grain boundary ledges:				
(III) (a) through the lattice	1	1	D_1	4
(IV) (b) along the grain boundaries	1	1	D_{gb}	4
(V) Grain rearrangement by viscous flow due to second phase at boundaries	1	1	D^*	-
Mechanism (Line Defect Mechanisms)				
(VI) Dislocation glide/climb controlled by climb	0	4.5	D_1	5
(VII) Dislocation glide/climb controlled by glide	0	3	D_1	6
(VIII) Dissolution of dislocation dipoles	0	5	D_p	7
(IX) Dissolution of dislocation loops	0	4	D_1	7
Dislocation climb without glide:				
(X) (a) dislocations are sources and sinks for vacancies	0	3	D_1	8
(XI) (b) pipe diffusion occurs along dislocation cores	0	5	D_p	8
Grains boundary sliding by dislocation glide/climb:				
(XII) (a) in zone near boundary	1	2	D_1	9
(XIII) (b) along boundary	1	2	D_{gb}	9
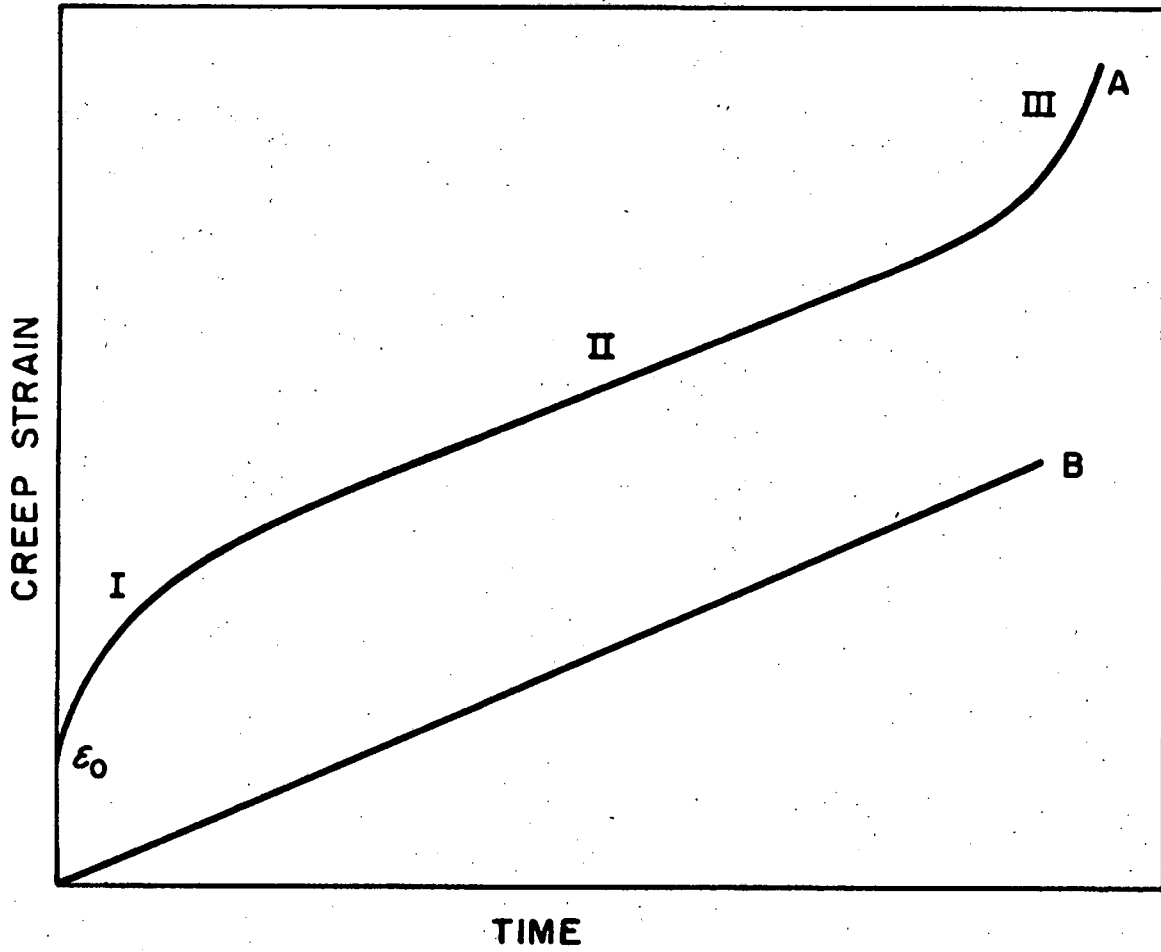
Fig. 2
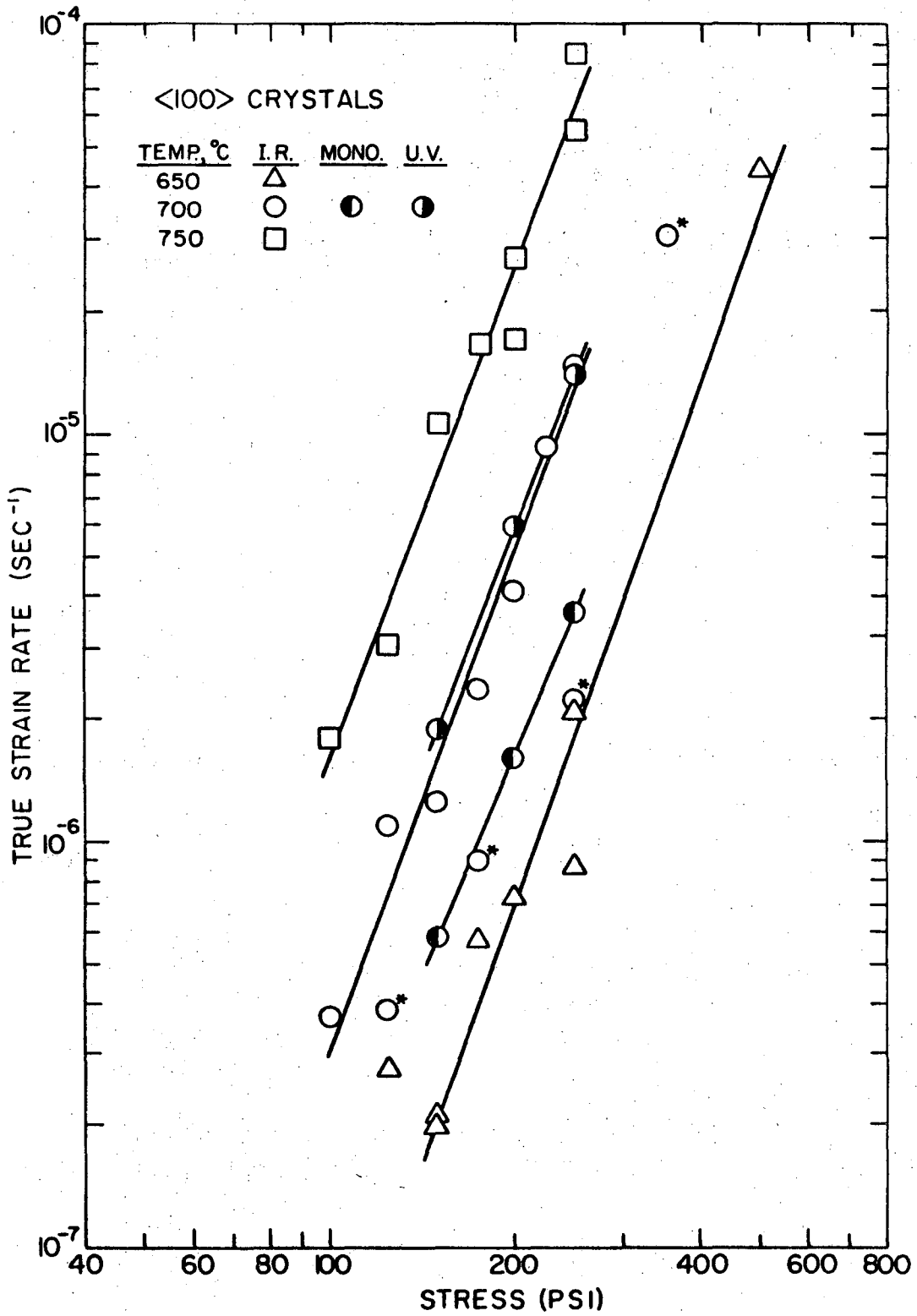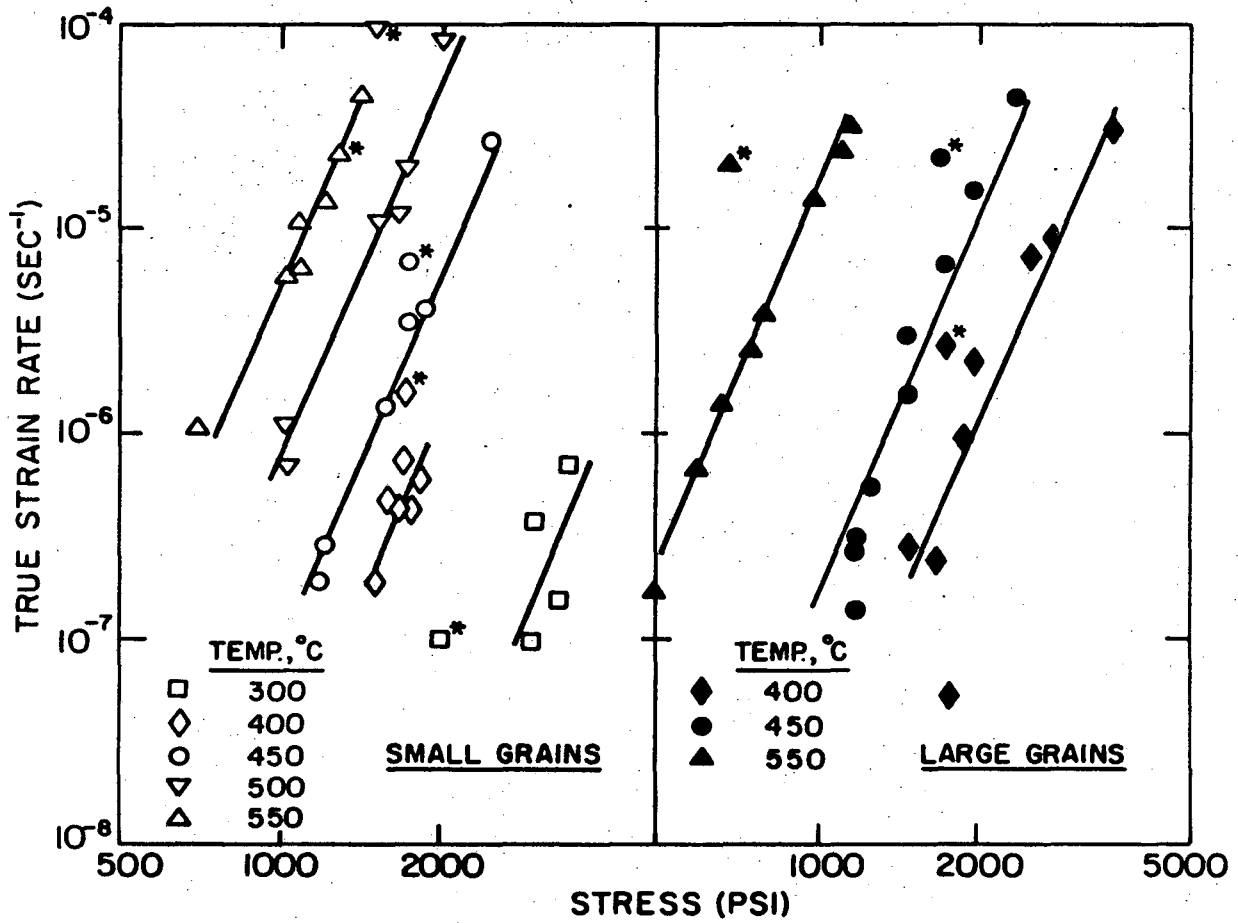
XBL 7011-7077

XBL 7011-7085

Fig. 4
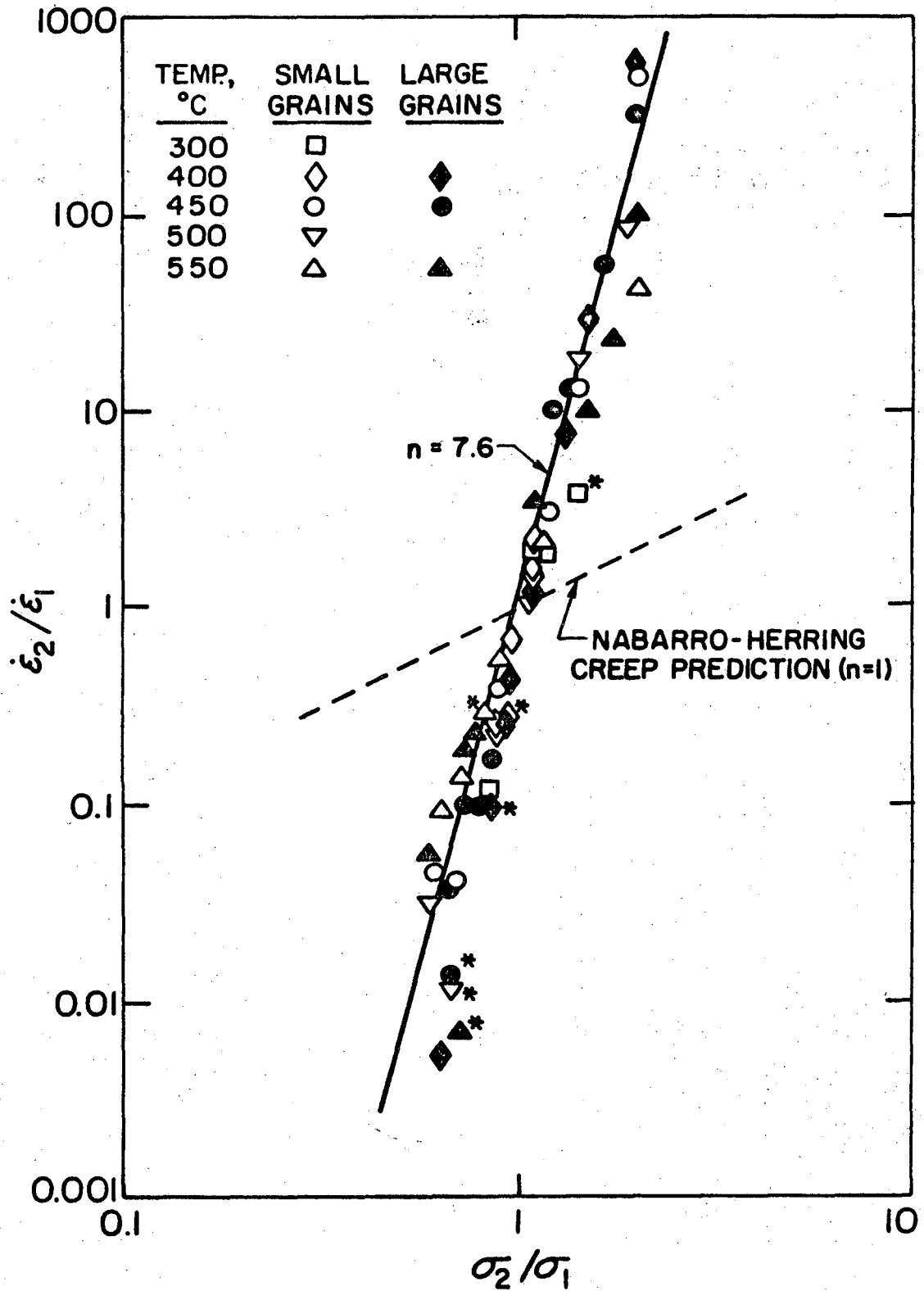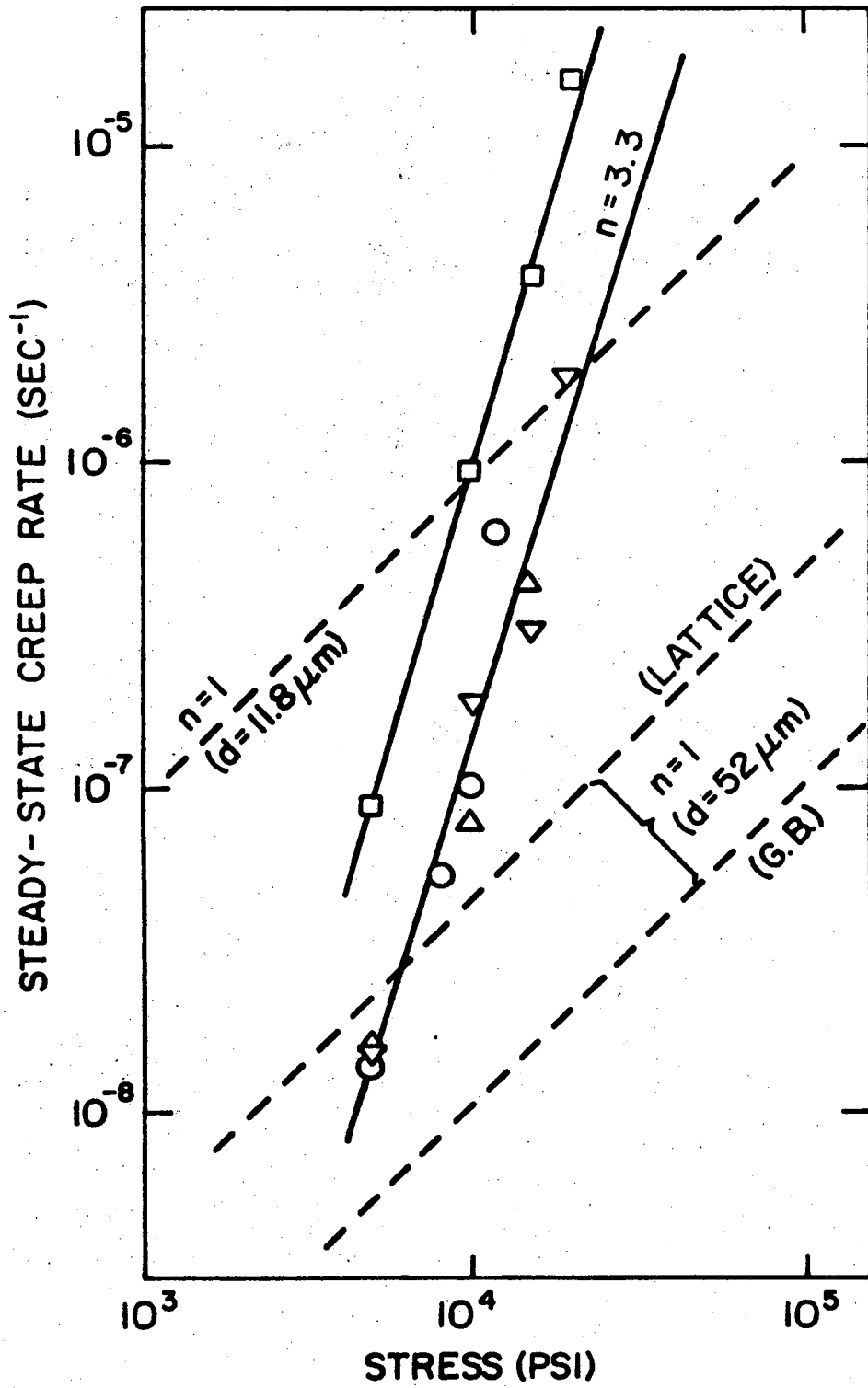
XBL 7011-7086

XBL 7011-7082

Fig. 6

XBL 7011-7083

Fig. 7

Fig. 8

XBL 696-801

Fig. 9

XBL 6712-1984