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THE MECHANISM OF CREEP IN POLYCRYSTALLINE MAGNESIUM OXIDE

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ABSTRACT

July 1969

Polycrystalline magnesium oxide, nominally fully-dense (>99.8% theoretical), was tested in compressive creep at a temperature of 1200°C and under initial applied stresses of $3.4 \times 10^8 - 13.76 \times 10^8$ dyne/cm^2. The observed steady-state creep rates were independent of grain size in the range ∿12-52μm, and proportional to $\sigma^{3.3}$, where σ is the applied stress. The activation energy was determined at 51 ±5 kcal/mole.

The results show that the creep behavior of polycrystalline MgO is similar to that observed in many metals. An analysis of the data in terms of various creep theories suggests that a dislocation mechanism is rate-controlling, such as the climb of dislocations without accompanying glide.

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1. INTRODUCTION

A major problem retarding the growth of knowledge of creep in ceramic systems has been the difficulty of obtaining polycrystalline materials not only of controlled porosity but also of purity. Both of these factors are of prime importance in any critical evaluation of the creep characteristics, since, in MgO at least, there is evidence that the size and distribution of pores is an important factor in the high temperature mechanical behavior,[1] and that impurities, even when present in amounts as small as 30 ppm, may segregate to the grain boundaries.[2] This latter effect may be especially important if grain boundary sliding is a significant mode of deformation at high temperatures, particularly if the boundary phase has a low melting point.

Many of the creep results published on ceramic polycrystals have suggested that the rate-controlling mechanism at high temperatures is the stress-directed diffusion of vacancies through the lattice, in the manner first suggested by Nabarro.[3] Under these conditions, Herring[4] has shown that the creep rate, $\dot{\varepsilon}$, is given by

$$\dot{\varepsilon} = \frac{B}{d^2} \frac{\Omega\sigma}{kT} D_L \qquad (1)$$

where B is a constant ($\sim$10 for equi-axed polycrystals), Ω is the atomic volume, σ is the applied stress, d is the average grain diameter, k is Boltzmann's constant, T is the absolute temperature, and D_L is the lattice self-diffusion coefficient ($= D_{o(L)} \exp(-Q_L/kT)$ where $D_{o(L)}$ is a frequency factor and Q_L is the activation energy for lattice self-

diffusion). If the diffusional path is along the grain boundaries, Coble[5] has shown that the creep rate is then given by

$$\dot{\varepsilon} = \frac{150}{\pi} \frac{\sigma W \Omega}{d^3 kT} D_{GB} \qquad (2)$$

where W is the width of the boundary and D_{GB} is the grain boundary self-diffusion coefficient ($=D_{o(GB)} \exp(-Q_{GB}/kT)$ where Q_{GB} is the activation energy for boundary diffusion).

In several instances where Nabarro-Herring diffusional creep appears to occur, Eq. (1) has been used to directly calculate D_L from the measured creep rates, and tentative agreement has been found between the calculated values and those for cation diffusion (e.g. in Al_2O_3,[6-9] BeO,[10,11] MgO[12]). This result is unexpected, however, since the anions are known to be the slower moving ions in these materials and should therefore be rate-controlling.* To overcome this anomaly in Al_2O_3, it was suggested that oxygen migrates via grain boundaries at a faster rate than cation diffusion through the lattice.[15]

In contrast, some other creep studies on ceramic polycrystals (FeO,[16] LiF,[17] MgO,[18] NaCl[19]) have shown that the observed creep rates obey a power relationship rather than a linear stress dependence, thereby suggesting a mechanism such as the glide and climb of dislocations.

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* In some oxides, probably those that can easily become oxygen-deficient, the cation appears to diffuse more slowly than the anion, and the apparent equivalence between the activation energy for creep and that for cation diffusion is then reasonable (e.g. ThO_2,[13] UO_2[14]).

Under these conditions, Weertman[20] has shown that the creep rate is given by

$$\dot{\varepsilon} = \frac{A\sigma^n}{kT} \exp\left(-Q_c/kT\right) \qquad (3)$$

where Q_c is the activation energy for creep, n is a constant (4.5) and A is approximately constant but contains a slight dependence on temperature.

Although a large volume of creep data is currently available on MgO, in both single crystal and polycrystalline form, wide variations exist in the published results.[21] The present investigation was therefore undertaken specifically to determine the rate-controlling mechanism in non-porous high-purity MgO at a temperature of 1200°C ($\sim$0.5 T_m, where T_m is the melting point in degrees Kelvin). From Eqs. (1-3) it follows that if diffusional creep is rate-controlling, rather than a dislocation mechanism, two criteria must be satisfied:

(i) $\dot{\varepsilon}$ must be a linear function of σ

(ii) $\dot{\varepsilon}$ must be proportional to d, either as $1/d^2$ (Eq. 1) or $1/d^3$ (Eq. 2) respectively.

The purpose of this paper is to show that, under the present experimental conditions, neither of these criteria is fulfilled.

2. MATERIALS AND TECHNIQUES

Several discs of polycrystalline MgO, 1.5" in diameter and 0.25" thick, were produced by hot-pressing high-purity MgO powder in vacuum in a graphite die with an additive of 3 wt % LiF, using the technique and materials described by Benecke et al.[22] A pressure of 1200 p.s.i. was

applied at room temperature, maintained constant whilst the temperature was raised to 1000°C over a period of ∿3 hr, and held at temperature for 3 hr. The specimens were then furnace cooled. All discs were subsequently heat-treated for 3 hr at 1300°C, to give an average grain size, determined by the linear intercept method, of 11.8μm; some were also given additional heat treatments for 3 hr at 1500°C or 1750°C, to yield average grain sizes of 33μm and 52μm respectively.

The discs were transparent after heat treatment, with no visible porosity,[23] and the density was determined as 3.577 g/cc using the displacement in alcohol technique. This density is better than 99.8% theoretical in comparison with an MgO single crystal value of 3.584 g/cc, based on a lattice parameter determination.[24] A spectrographic analysis revealed the following impurities in ppm: Si:30, Fe:<20, Al:10, Cu:5, Ni:<10, Sr:<50, Ca:40, Li:75; all constituents are reported as oxides, with the exception of lithium which, although primarily removed during densification, was probably present as a fluoride due to the use of an LiF additive.

Specimens were cut from the hot-pressed discs with a diamond saw, the majority to dimensions of ∿0.38" x 0.25" x 0.25". Since there is evidence that specimen dimensions, particularly the length:width ratio, may affect the observed behavior when testing in compression,[25] some specimens were also cut to the same cross-section but with a length of ∿1.0". Prior to testing, the ends of each specimen were ground perpendicular to the longitudinal axis using a special jig, and one longitudinal face was polished to allow subsequent microscopic examination.

All specimens were tested in air inside a furnace with $MoSi_2$ heating elements, under conditions of constant load calculated on their initial cross-sections. For testing, the specimens were placed upright between small (∿0.7" diameter x 0.3" thickness) alumina buttons, protected from the Al_2O_3 by a thin (0.001") sheet of platinum, and then placed between two alumina loading rams within the furnace. For the long (∿1.0") specimens, the strain was continuously monitored by two sapphire rods which impinged into small holes, initially 0.5" apart, on one face of the specimen, and were connected externally to a linear voltage differential transformer and recorder. With the shorter (∿0.38") specimens, the sapphire rods were inserted in holes on the sides, and near the innermost edges, of the alumina buttons. In both cases, the total strains were in good agreement with those obtained by measuring the lengths of the specimens after testing. Strains were recorded to a sensitivity of $\pm 5 \times 10^{-5}$, and the specimen temperature was maintained constant during each test to an estimated ±5°C.

3. EXPERIMENTAL RESULTS

Specimens of the smaller length (∿0.38"), and of the three different grain sizes, were tested under various constant loads in the range 5,000-20,000 p.s.i. (3.44×10^8-13.76×10^8 dyne/cm^2), and the strain-time curves recorded in each case. Typical results, for the smallest grain size (11.8μm), are given in Fig. 1, and show that polycrystalline MgO exhibits both primary and secondary stages of creep when tested in compression. All tests were discontinued before fracture, the majority at total strains of <0.05; a few specimens were taken to higher strains, typically ∿0.1, but all were unloaded before there was any

evidence of a third, or accelerating, stage of creep.

For each specimen, the steady-state creep rate was plotted as a function of stress, as shown in Fig. 2. In addition, four specimens of the smallest grain size but of the longer length (∿1.0") were tested under similar conditions, and these results are also plotted. It can be seen that all points for the small specimens lie on the same line, independent of grain size, with a slope of n = 3.3. The results for the longer specimens show a similar stress dependence, although the creep rates in this case were slightly faster.

To determine the activation energy for creep, Q_c, additional tests were carried out in which the temperature was cyclically changed by ∿25°C, either in the range 1175-1200°C or 1200-1225°C, at regular strain increments of ∿0.02. By this procedure, it was assumed that the structure remained constant during the change,[26] and Q_c was calculated from the relationship

$$Q_c = \frac{\partial \ln \dot{\varepsilon}}{\partial(-1/RT)} \sim \frac{R \ln (\dot{\varepsilon}_2/\dot{\varepsilon}_1)}{(T_2-T_1)/T_1T_2} \qquad (4)$$

where $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ were the instantaneous creep rates immediately preceding and following a change in temperature from T_1 to T_2, and R is the gas constant. Some scatter was observed in the experimental data, since the equipment used in this work did not permit extremely rapid temperature changes; typical times to reach thermal equilibrium for decreases and increases of ∿25°C were 12 and 8 minutes respectively. The average value obtained for Q_c was 51 ±5 kcal/mole, and this was independent of the total strain.

4. DISCUSSION

The results in Fig. 2 show clearly that the two criteria required for diffusional creep are not satisfied under the present experimental conditions. Firstly, the slope of the line is given by $n = 3.3$, rather than unity; secondly, there is no apparent dependence on grain size in the range 11.8 - 52μm. However, since there is evidence of diffusional creep in polycrystalline MgO tested under similar conditions,[27] it is important to examine these results more closely.

a. Comparison with Diffusional Creep Mechanisms

From Eq. (1) and (2), it follows that, at constant stress and temperature, the strain rates, $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$, for two different grain sizes, d_1 and d_2, are related by

$$\dot{\varepsilon}_1/\dot{\varepsilon}_2 = d_2^2/d_1^2 \qquad \text{(lattice diffusion)} \qquad (5)$$

or

$$\dot{\varepsilon}_1/\dot{\varepsilon}_2 = d_2^3/d_1^3 \qquad \text{(grain boundary diffusion)} \qquad (6)$$

Taking the experimentally observed value of $\dot{\varepsilon}_1 = 1.83 \times 10^{-6}\ \text{sec}^{-1}$ for $d_1 = 11.8\mu\text{m}$ at $\sigma = 2 \times 10^4$ p.s.i., the calculated values of $\dot{\varepsilon}_2$ for $d_2 = 52\mu\text{m}$ are $9.3 \times 10^{-8}\ \text{sec}^{-1}$ and $2.1 \times 10^{-8}\ \text{sec}^{-1}$ from Eq. (5) and (6) respectively. Lines of slope $n = 1$ have been drawn through these points in Fig. 2, and illustrate the marked discrepancy between the experimental data and the two diffusional models.

The overall deviation from diffusional creep may be calculated by using Eq. (1) to determine the anticipated value of $\dot{\varepsilon}$ if creep takes place by the stress-directed diffusion of vacancies through the lattice.

With the smallest grain size ($d = 11.8\mu m$), taking $\Omega = 1.87 \times 10^{-23}$ cm^3, $\sigma = 10^4$ p.s.i., and D_L for extrinsic lattice diffusion of O^{2-} (2.5×10^{-6} exp-(62,400/RT)),[28] the value calculated for $\dot{\varepsilon}$ is $\sim 5.8 \times 10^{-10}$ sec^{-1}, almost three orders of magnitude slower than the experimentally observed creep rate of $\sim 1.9 \times 10^{-7}$ sec^{-1}. Since $\dot{\varepsilon} \propto 1/d^2$ for lattice diffusional creep, it follows that an even larger discrepancy occurs for the larger grain sizes (e.g. for $d = 33\mu m$, $\sigma = 10^4$ p.s.i., $\dot{\varepsilon}$ is calculated as $\sim 7.4 \times 10^{-11}$ sec^{-1}).

An alternative possibility is to take D_L for Mg^{2+}, although the only value available is for intrinsic diffusion (2.49×10^{-1} exp-(79,000/RT)).[29] Again taking $\sigma = 10^4$ p.s.i., $\dot{\varepsilon}$ is calculated as $\sim 1.8 \times 10^{-7}$ sec^{-1} or $\sim 2.2 \times 10^{-8}$ sec^{-1} for $d = 11.8\mu m$ or $33\mu m$ respectively. However, whilst the calculated value for $\dot{\varepsilon}$ is in close agreement with that observed experimentally for the smallest grain size, there are two reasons for not accepting this lattice diffusion coefficient. Firstly, the anion is the slower moving species and should therefore be rate-controlling; secondly, the observed activation energy for creep (51 ±5 kcal/mole) is closer to that for extrinsic diffusion of O^{2-} (62.4 kcal/mole)[28] than intrinsic diffusion of Mg^{2+} (79.0 kcal/mole).[29]

It is also of interest to make a similar calculation for grain boundary diffusional creep, particularly since, as Eq. (2) has a stronger grain size dependence than Eq.(1) and $Q_{GB} < Q_L$, there is a tendency for vacancy diffusion to become more favorable via the grain boundaries in specimens of very fine grain size at lower temperatures of deformation ($\lesssim 0.6\ T_m$; see, for example, Jones[30]). Unfortunately, D_{GB} for MgO is not known and an accurate determination of $\dot{\varepsilon}$ is therefore not possible.

However, for d = 11.8μm and $\sigma = 10^4$ p.s.i., Eq. (2) gives $\dot{\varepsilon} \sim 1.8 \times 10^9$ WD_{GB}. Taking, as a first approximation, $Q_{GB} \sim 0.6\ Q_L$,[31] $D_{o(GB)} = D_{o(L)}$, and an upper limit for W of $\sim 10^{-6}$ cm,[15,32] the value obtained for $\dot{\varepsilon}$ is $\sim 1.1 \times 10^{-8}$ sec^{-1} for boundary diffusion of O^{2-}. This is an order of magnitude slower than that observed experimentally, and, whilst depending strongly on the value assumed for WD_{GB}, again suggests that even the smallest grain size used in this investigation is too large to exhibit diffusional creep under the present experimental conditions.

b. Comparison with Dislocation Creep Mechanisms

The stress dependence of n = 3.3 shown in Fig. 2 suggests that a dislocation mechanism is rate-controlling, such as dislocation climb which requires that n = 4.5 in the glide and climb model developed by Weertman.[20] Using this model, a direct calculation of the anticipated value of $\dot{\varepsilon}/D$, where D is the diffusion coefficient, may be obtained from the equation developed by Weertman for high stresses:

$$\frac{\dot{\varepsilon}}{D} = \frac{3\pi^2\ \sigma^2}{2(2)^{0.5}G^2b^2}\ \sinh\left(\frac{(3)^{0.5}\ \sigma^{2.5}\ b^{1.5}}{8G^{1.5}M^{0.5}kT}\right) \qquad (7)$$

where G is the shear modulus ($\sim$0.4E, where E is Young's modulus), b is the Burgers vector, and M is the number of Frank-Read sources per cm^3 ($M^{0.5} = 0.526\ \rho^{0.75}$, where ρ is the dislocation density).[33] The value of G at 1200°C was obtained from the reported Young's modulus for non-porous polycrystalline MgO at room temperature,[34] and the temperature dependence of the elastic moduli.[35] Taking $\rho = 10^8$ dislocations/cm^2 and $b = 3 \times 10^{-8}$ cm, $\dot{\varepsilon}/D$ was calculated for the stress range used in this

work. The result is shown in Fig. 3, together with the experimental points calculated by taking the extrinsic lattice diffusivity of O^{2-}, and the predicted dependence for diffusional creep of the Nabarro-Herring type for d = 11.8μm and 33μm.

The dislocation glide and climb model breaks down at high stresses because of the sinh term in Eq. (7), and an estimate of the breakdown stress may be obtained by putting the expression within this term equal to unity. When this is done, it appears that Weertman's model is valid in polycrystalline MgO up to stresses of $\sim 1.5 \times 10^9$ dyne/cm^2, which is higher than the highest stress used in the present work.

Whilst the magnitude of the experimental points shown in Fig. 3 are in better agreement with the predictions arising from the dislocation glide/climb model than those of diffusional creep, this model predicts n = 4.5 whereas the experimental results show n = 3.3. Nabarro[36] has recently developed a theory of steady-state creep in which dislocation climb takes place without accompanying glide, and climb is a deformation rather than a recovery mechanism. The stress dependence is then 3 or 5 depending on whether the dislocations are sources and sinks for vacancies or the rate-controlling process is diffusion along the dislocation cores. Since the former model predicts a similar stress dependence to that observed experimentally, the value of $\dot{\varepsilon}/D$ was calculated for this mechanism from the equation:

$$\frac{\dot{\varepsilon}}{D} = \frac{b\,\sigma^3}{\pi k T\, G^2} \Big/ \ln\left(\frac{4G}{\pi\sigma}\right) \qquad (8)$$

Weertman[37] has recently re-examined this model, and obtained a slightly different expression, given by

$$\frac{\dot{\varepsilon}}{D} = \frac{\pi \beta^2 \Omega \sigma^3}{10 b^2 G^2 kT} \tag{9}$$

where $\beta \sim 1$. The predictions arising from Eq. (8) and (9) are shown in Fig. 3, and differ by almost an order of magnitude.

It is evident from Fig. 3 that, by making the assumption that lattice diffusion of O^{2-} is rate-controlling, the experimental results show better agreement with several dislocation mechanisms than with the predictions arising from diffusional creep. The value of $\dot{\varepsilon}/D$ obtained experimentally at any given stress is intermediate between that predicted by models of dislocation climb and a glide/climb process, but the stress dependence is in better agreement with the former mechanism. It is interesting to note that Groves and Kelly[38] have also concluded, from an analysis of published data, that the creep of Al_2O_3 may take place by Nabarro's dislocation climb model. However, as in the present results for MgO, taking a stress of 10^9 dyne/cm^2 and the coefficient of self-diffusion for oxygen, the calculated strain rate was over an order of magnitude slower than that obtained experimentally.

c. Comparison with Other MgO Creep Data

The observed activation energy of 51 ±5 kcal/mole is lower than the accepted value of 62.4 kcal/mole for extrinsic O^{2-} diffusion,[28] although the difference may not be significant because of experimental difficulties in both types of experiments. In addition, the experimental value

here is intermediate between that reported for 98% dense MgO tested in torsion at 1100-1300°C (46.4 kcal/mole)[18] and that for >99.5% dense MgO tested in bending at 1107-1527°C (54.1 kcal/mole).[27]

The lack of any dependence on grain size conflicts with reports of creep rates proportional to $d^{-2.5}$ and d^{-2} respectively.[27,39] In both of these investigations, however, the tests were conducted in four-point bending, resulting in a complex deformation pattern, and the creep rate was found to continuously decrease with increasing strain; in one instance this was ascribed to simultaneous grain growth.[39] No discernible grain growth took place in the present work due to the preliminary heat treatment at 1300°C; furthermore, measurements showed that the spread of grain diameters for specimens of each of the three grain sizes exhibited similar Gaussian distributions prior to testing,[40] thereby suggesting that the grain configurations were relatively stable.

5. SUMMARY AND CONCLUSIONS

1. The compressive creep behavior of nominally fully-dense (>99.8%) polycrystalline MgO was investigated at 1200°C for grain sizes in the range ∿12-52μm and for initial stresses of 3.4×10^8-13.76×10^8 dyne/cm^2.

2. The material exhibited primary and secondary regions of creep, and the steady-state creep rate was independent of grain size and proportional to $\sigma^{3.3}$, where σ is the applied stress.

3. The activation energy was determined as 51 ±5 kcal/mole, which is slightly lower than the reported value of 62.4 kcal/mole for extrinsic O^{2-} diffusion.

4. Slight differences were observed in the overall creep rates of two sets of specimens having different length:width ratios, although

the stress dependence was the same in each case.

5. Contrary to some previous investigations, the results suggest that the creep behavior of polycrystalline MgO is essentially similar to that observed in many metals. It is concluded that the rate-controlling mechanism is some form of dislocation motion, such as the climb of dislocations in the absence of glide, rather than the stress-directed diffusion of vacancies.

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REFERENCES

1. T. G. Langdon and J. A. Pask, to be published.
2. M. H. Leipold, J. Am. Ceram. Soc. 49, 498 (1966).
3. F. R. N. Nabarro, "Report of a Conference on the Strength of Solids," p. 75, The Physical Society, London (1948).
4. C. Herring, J. Appl. Phys. 21, 437 (1950).
5. R. L. Coble, J. Appl. Phys. 34, 1679 (1963).
6. S. I. Warshaw and F. H. Norton, J. Am. Ceram. Soc. 45, 479 (1962).
7. R. L. Coble and Y. H. Guerard, J. Am. Ceram. Soc. 46, 353 (1963).
8. C. W. Hewson and W. D. Kingery, J. Am. Ceram. Soc. 50, 218 (1967).
9. E. M. Passmore and T. Vasilos, J. Am. Ceram. Soc. 49, 166 (1966).
10. R. R. Vandervoort and W. L. Barmore, J. Am. Ceram. Soc. 46, 180 (1963).
11. W. L. Barmore and R. R. Vandervoort, J. Am. Ceram. Soc. 48, 499 (1965).
12. T. Vasilos, J. B. Mitchell and R. M. Spriggs, J. Am. Ceram. Soc. 47, 203 (1964).
13. L. E. Poteat and C. S. Yust, J. Am. Ceram. Soc. 49, 410 (1966).
14. W. M. Armstrong and W. R. Irvine, J. Nucl. Mat. 12, 261 (1964).
15. A. E. Paladino and R. L. Coble, J. Am. Ceram. Soc. 46, 133 (1963).
16. B. Ilschner, B. Reppich and E. Riecke, Discs. Faraday Soc. 38, 243 (1964).
17. D. R. Cropper and T. G. Langdon, Phil. Mag. 18, 1181 (1968).
18. J. F. Wygant, J. Am. Ceram. Soc. 34, 374 (1951).
19. P. M. Burke and O. D. Sherby, Progress Report on NASA Research Grant SC-NGR-05-020-084, Stanford University, Stanford, California (1967).

20. J. Weertman, J. Appl. Phys. 28, 362 (1957).

21. T. G. Langdon and J. A. Pask, Report No. UCRL-18250, Lawrence Radiation Laboratory, University of California, Berkeley (1968). (To be published in "Refractory Oxides," Academic Press.)

22. M. W. Benecke, N. E. Olson and J. A. Pask, J. Am. Ceram. Soc. 50, 365 (1967).

23. T. G. Langdon and J. A. Pask, "Ceramic Microstructures," p. 594, John Wiley and Sons, New York (1968).

24. B. J. Skinner, Am. Mineral. 42, 39 (1957).

25. D. R. Cropper and J. A. Pask, Bull. Am. Ceram. Soc. 48, 555 (1969).

26. T. E. Tietz and J. E. Dorn, Trans. Am. Inst. Min. Engrs. 206, 156 (1956).

27. E. M. Passmore, R. H. Duff and T. Vasilos, J. Am. Ceram. Soc. 49, 594 (1966).

28. Y. Oishi and W. D. Kingery, J. Chem. Phys. 33, 905 (1960).

29. R. Lindner and G. D. Parfitt, J. Chem. Phys. 26, 182 (1957).

30. R. B. Jones, J. Nucl. Mat. 19, 204 (1966).

31. D. W. James and G. M. Leak, Phil. Mag. 12, 491 (1965).

32. L. W. Barr, I. M. Hoodless, J. A. Morrison and R. Rudham, Trans. Faraday Soc. 56, 697 (1960).

33. J. Weertman, Trans. Am. Inst. Min. Engrs. 218, 207 (1960).

34. D. H. Chung, Phil. Mag. 8, 833 (1963).

35. D. H. Chung and W. G. Lawrence, J. Am. Ceram. Soc. 47, 448 (1964).

36. F. R. N. Nabarro, Phil. Mag. 16, 231 (1967).

37. J. Weertman, Trans. Am. Soc. Metals 61, 681 (1968).

38. G. W. Groves and A. Kelly, Phil. Mag. 19, 977 (1969).

39. G. R. Terwilliger and R. S. Gordon, J. Am. Ceram. Soc (in press).

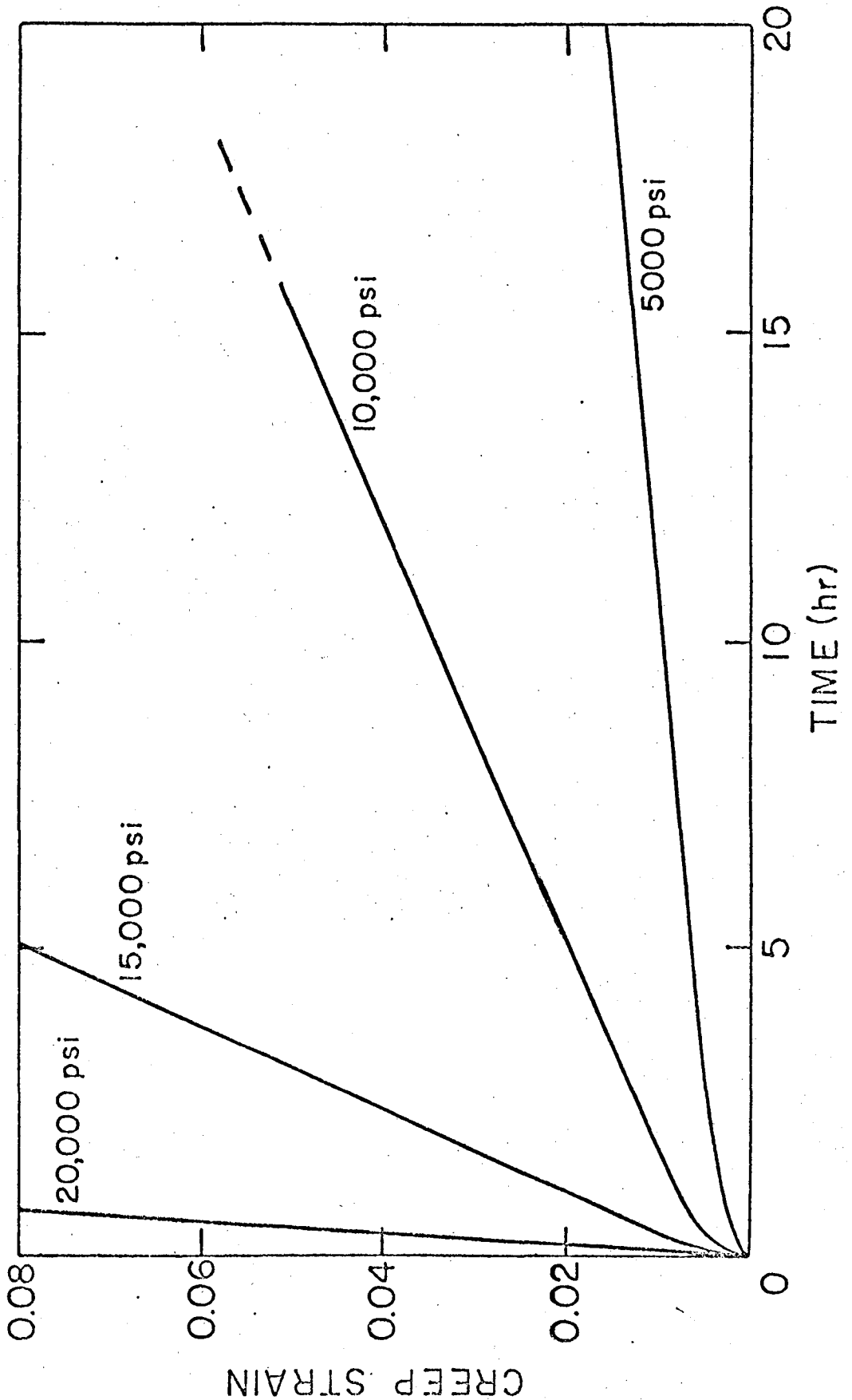
40. D. A. Aboav and T. G. Langdon, Metallography 1, 333 (1969).

FIGURE CAPTIONS

Fig. 1. Creep curves for specimens of smallest grain size (11.8μm), tested in compression at 1200°C.

Fig. 2. Steady-state creep rate versus stress for specimens of grain size = 11.8μm (□ : length l/width w = 4; ∇ : l/w = 1.52), 33μm (Δ :l/w = 1.52) and 52μm (O : l/w = 1.52). The lines of slope n = 1 indicate predictions arising from theories of diffusional creep via the lattice and grain boundaries respectively.

Fig. 3. Diffusion compensated creep rate ($\dot{\varepsilon}/D$) versus stress, taking D for extrinsic lattice diffusion of O^{2-} to calculate the experimental points. The dashed lines represent predictions arising from Nabarro-Herring diffusional creep (for d = 11.8μm and 33μm respectively), a dislocation glide/climb mechanism, and the dislocation climb model formulated by Nabarro (N) and reanalyzed by Weertman (W).


20,000 psi
15,000 psi
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CREEP STRAIN
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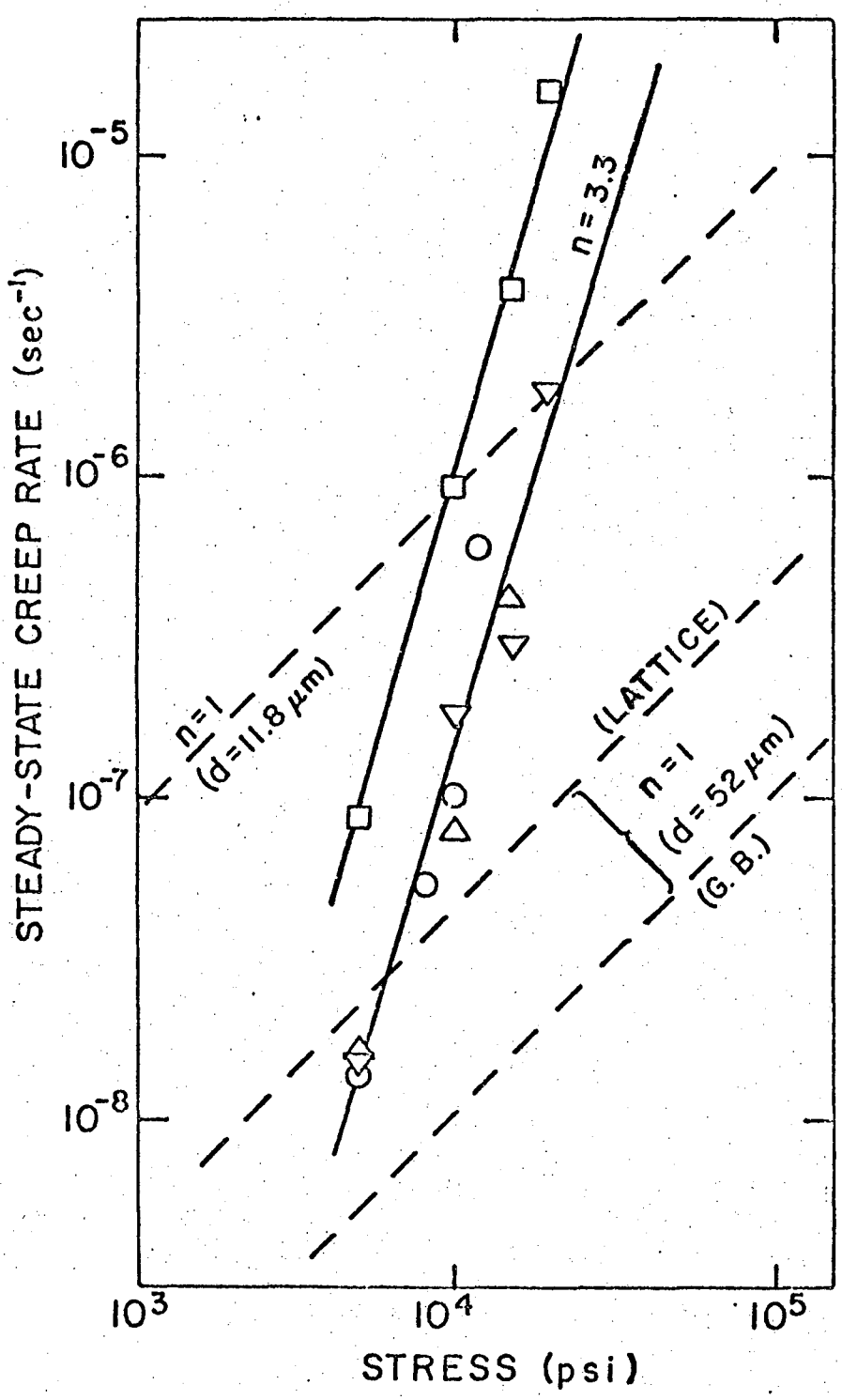
20
TIME (hr)

Fig. 1


STEADY-STATE CREEP RATE (sec^{-1})
10^{-5}
10^{-6}
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10^{-8}
10^{3}
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10^{5}
STRESS (psi)
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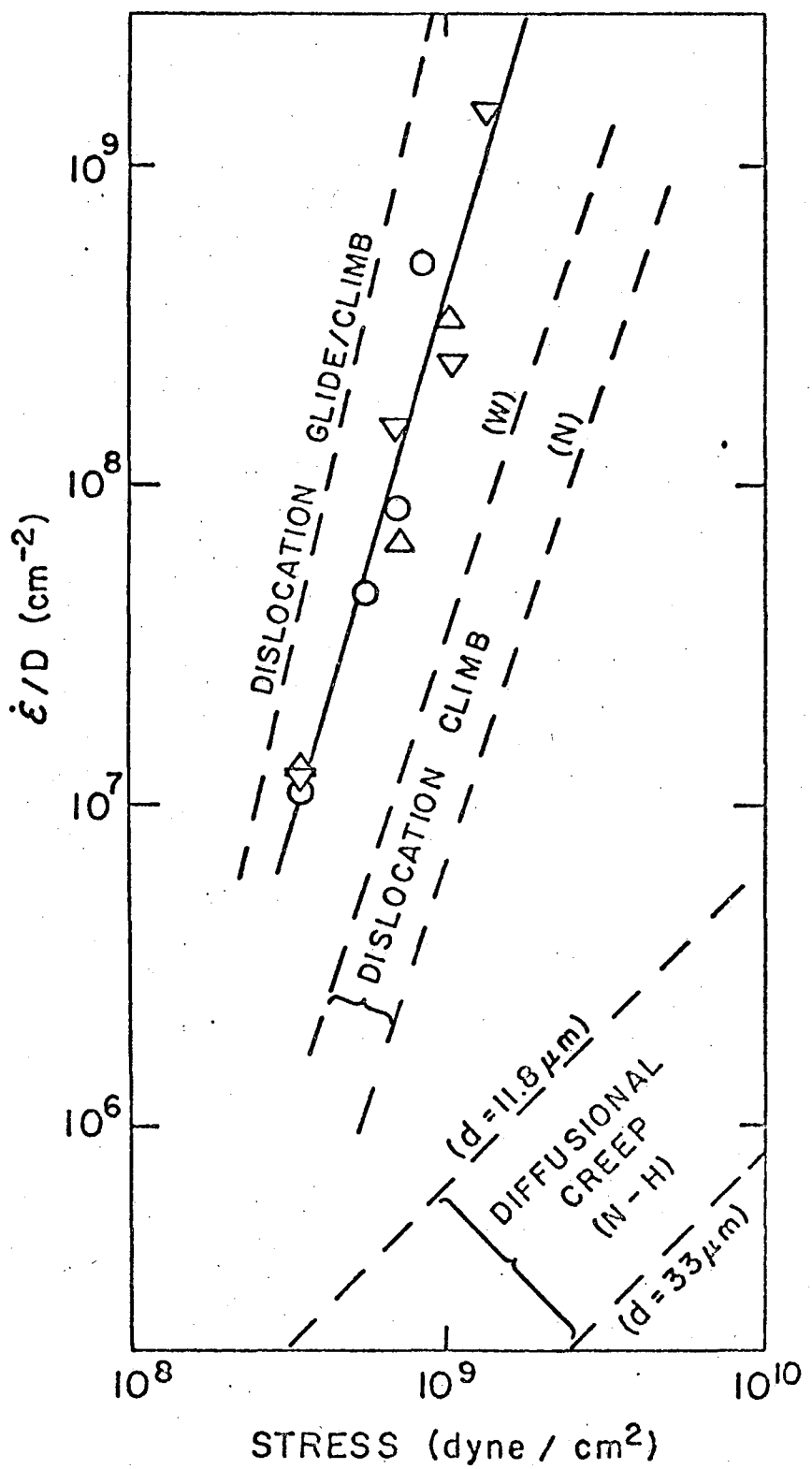
n=1
(d=11.8 μm)
(LATTICE)
n=1
(d=52 μm)
(G.B.)

Fig. 2


10^9
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$\dot{\varepsilon}/D$ (cm^{-2})
DISLOCATION GLIDE/CLIMB
DISLOCATION CLIMB
(W)
(N)
(d = 11.8 μm)
DIFFUSIONAL CREEP (N - H)
(d = 33 μm)
10^8
10^9
10^{10}
STRESS (dyne / cm^2)

Fig. 3

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