JOURNAL OF MATERIALS SCIENCE 9 (1974) 794-800

Work hardening and recovery during compressive creep of polycrystalline MgO

J. M. BIRCH, B. WILSHIRE
Department of Metallurgy and Materials Technology, University College of Swansea, Singleton Park, Swansea, UK

High precision equipment has been used to study the effects of small stress changes during steady state creep of magnesia at 1596K. When the stress, σ, is reduced by a small amount, $\Delta\sigma$, the creep rate decreased to zero for a period, Δt, before accelerating to a new steady value. Calculating the rate of recovery, $r(=-\partial\sigma/\partial t)$ as $\Delta\sigma/\Delta t$ and the coefficient of strain hardening $h(=\partial\sigma/\partial\epsilon)$ as $\Delta\sigma/\Delta\epsilon$ (where $\Delta\epsilon$ is the instantaneous strain recorded on increasing the stress by $\Delta\sigma$) gave the ratio, r/h, which predicts accurately the observed steady creep rate, $\dot{\epsilon}_S$. It is proposed that when $\dot{\epsilon}_S \propto \sigma^3$, creep is recovery controlled. The results are explained in terms of a model for creep in which the rate controlling process is the growth of the 3-D dislocation network within subgrains, to form dislocation sources allowing slip to occur.

1. Introduction

At elevated temperatures, the stress dependence of the creep rate of magnesia can be described as

$$\dot{\epsilon}_S \propto \sigma^n , \qquad (1)$$

where $\dot{\epsilon}_S$ is the steady creep rate and σ is the applied stress. In the low stress range, the stress exponent, n, is approximately unity [1-4]. This linear stress dependence can be interpreted in terms of grain [5, 6] or grain-boundary [7] diffusion controlled creep. At higher stress levels, the n value for magnesia has been reported to be in the range of 2.3 to 4 [8-10]. This large stress exponent has usually been taken to indicate that dislocation movement is important under these conditions and several mechanisms have been suggested to account for this behaviour.

In general, creep theories based on dislocation movement can be classified into two main groups: those in which a dislocation glide process is considered to be rate controlling and those that depend on dynamic recovery by climb. The first category includes mechanisms such as the non-conservative motion of jogged screw dislocations [11], glide of dislocations dragging a cloud of charged defects [12, 13], etc. Theories in the second category are based on the idea that creep takes place because the strain hardening resulting from creep deformation is continuously annealed out by recovery [14-17]. Several investigators have used the concept that, during the steady state, a balance exists between recovery and strain hardening, to explain the creep behaviour of ceramics [10, 18].

A technique frequently used to study the mechanisms by which creep occurs, involves carrying out a sudden change in the applied stress during a creep test and examining the subsequent strain/time behaviour. This was suggested by the early work of Orowan [19] who proposed that, during the steady state condition eventually reached during high temperature creep, the strain hardening resulting from an increment of strain, $d\epsilon$, during a short time, dt, must be exactly balanced by recovery to maintain the flow stress, σ, constant as

$$d\sigma = \left(\frac{\partial\sigma}{\partial\epsilon}\right) d\epsilon + \left(\frac{\partial\sigma}{\partial t}\right) dt = 0$$

therefore

$$\dot{\epsilon}_S = d\epsilon/dt = r/h , \qquad (2)$$

where $r\ (=-\partial\sigma/\partial t)$ is the rate of recovery and $h\ (=\partial\sigma/\partial\epsilon)$ is the coefficient of strain hardening. If creep is recovery controlled, a small stress reduction ($\simeq 0.05\sigma$) during steady state creep should be followed by a period of zero creep rate, during which time the material recovers until creep can recommence at the reduced

stress. Such behaviour has been observed frequently for metals and alloys [16, 20, 21].

The present investigation was, therefore, undertaken to examine the behaviour of magnesia following small stress changes during steady state creep. This particular technique does, however, require high precision creep testing equipment. In many previous studies of the compression creep behaviour of ceramic materials, the specimen strain has been monitored by measurements of the displacement of the loading rams so that creep of the rams has to be allowed for in estimating the creep rate of the specimen [22, 23]. Accurate temperature control is necessary to prevent fluctuations in the specimen length [24]. Care must also be taken to avoid barrelling and buckling of the specimens which has been shown [25] to markedly affect the creep results obtained. In the present work, a high precision creep machine was developed to give the continuous accurate measurement of specimen strain required for a study of the effects of small stress changes during creep.

2. Experimental procedure

2.1. Fabrication of specimens

The magnesia used in this study was supplied in the form of sintered bars (6.4 × 6.4 × 32.0 mm^3) by the Steetley Co Ltd. Specially prepared $Mg(OH)_2$ was calcined at 1573 K, pressed into bars and sintered at 1923 K in air to produce samples of 94 to 96% theoretical density, 99.85% purity and having an average grain diameter of 10 to 14 μm [26].

Cylindrical test pieces (4.25 mm diameter and 6.4 mm long) were prepared, a polishing jig being used to ensure that the specimen ends were flat and exactly parallel.

2.2. The constant stress compressive creep machine

A schematic diagram of the equipment is shown in Fig. 1. The method of loading the specimens is similar to that used successfully for metals [27]. The load is applied through a 3:1 lever by flexible steel straps which follow the profile of a cam of the Andrade-Chalmers type [28] which compensates for the increase in the cross-sectional area of the specimen as it is compressed. The load is transmitted to the upper moving cross-head (a), through a length adjusting screw which enables the creep tests to begin with the cam in the correct position. The cross head (a) is rigidly connected to the lower moving cross head (b) by the two vertical steel rods (c) which pass through holes in the fixed cross head (d). Smooth axial movement of the cross head (b) is achieved by using two ball bushings (e) running on accurately ground, surface hardened, parallel pillars (f). Counter weights compensate exactly for the weights of the moving parts. Silicon nitride rams (g) attached to the fixed (d) and moving (b) cross head compresses the specimen (h) between two silicon nitride platens (i). The platens also position exactly a pair of sapphire extensometer rods on either side of the specimen. The extensometer rods are located, without contact in grooves machined along the lower ram, and pass through holes in the lower moving cross head (b). The specimen strain is then measured from the displacements of the extensometer rods using differential capacitance transducers (j) capable of measuring to 10 nm.

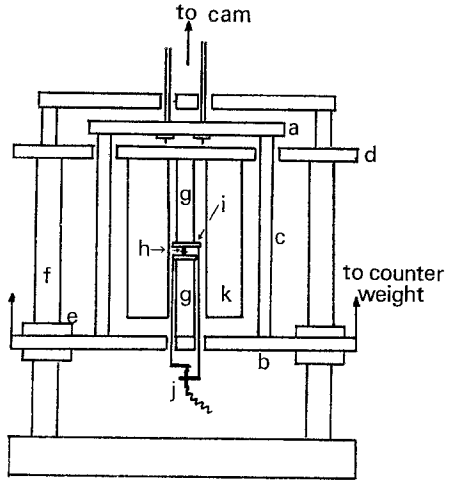

Figure 1 A schematic diagram of the compression creep machine.

During compression creep tests, the displacement measured on each pair of extensometer rods was identical on either side of the specimen, indicating that axial compression of the specimens was being achieved.

A platinum −40% rhodium alloy resistance furnace (k) is attached to the fixed cross head (d) and controlled using a saturable reactor controller with platinum alloy thermocouples located near the specimen. Measurement of the temperature distribution in the furnace during creep indicated that at 1650 K the temperature was ± 1 K over a 25 mm zone.

Although the present tests were carried out in air, provision has been made to allow tests to be performed under controlled atmosphere conditions.

2.3. Performance of creep machine

The accuracy with which the cam corrects for the change in the cross-sectional area of the specimen was examined using a procedure described elsewhere [27]. It was established that the stress remained constant up to 5% specimen strain and was constant to $\pm$ 2% up to 12% strain.

Frictional forces opposing cross head motion were equivalent to a load of less than 0.25 kg which is negligible compared to the applied loads.

The reproducibility of creep curves obtained at the same stress and temperature was assessed by carrying out a series of creep tests at 1596 K and 84 MN m^{-2}. The results obtained using specimens prepared from two different test bars (labelled M and N) are shown in Fig. 2. Although for the sake of clarity only three curves are presented in Fig. 2, six tests were carried out (Fig. 3). The steady creep rate, $\dot{\epsilon}_s$, was found to be 1.4×10^{-6} sec^{-1} ($\pm$ 15%). This reproducibility was considered to be good in view of the slight specimen-to-specimen variability usually encountered with ceramic samples.

Barrelling, due to frictional forces between the specimen and the platen, sets an upper limit to homogeneous deformation. It was only detectable at compressive strains above ~7% (Fig. 4), therefore, creep tests were discontinued at strains of ~ 6%. It should be also noted that, even after many creep tests, no impression was made on the platen by the specimens.

A problem allied to barrelling is the effect of the height to diameter (h/d) ratio of the specimen during compressive creep. Samples of a constant height (necessary because the cam was designed on the basis of a fixed initial height of the specimen) but with varying diameters were, therefore, tested at the same stress and temperature. The h/d ratios examined were 1.1, 1.5 and 2.0. The steady creep rates recorded were the same (within the spread expected due to specimen-to-specimen variation) supporting the view [29] that, within the range studied, the h/d ratio is not critical. All present work was then carried out with a h/d ratio of 1.5.

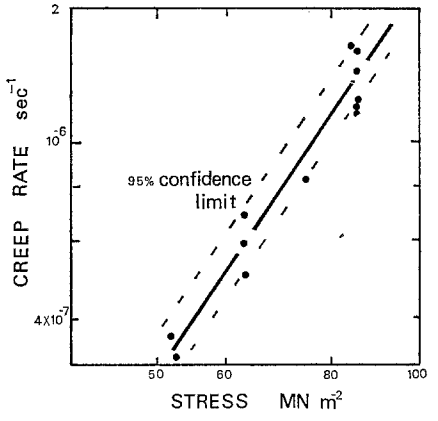

Figure 3 The stress dependence of the steady creep rate of polycrystalline magnesia at 1596 K.

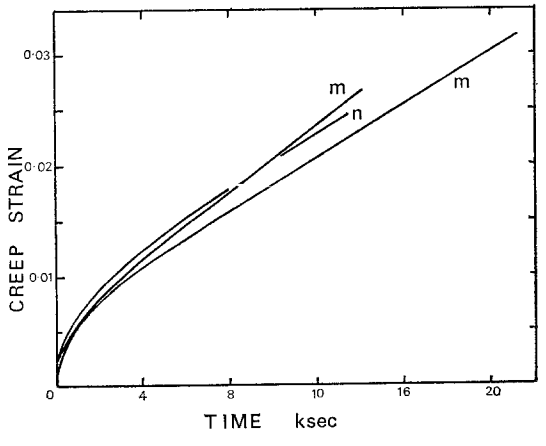

Figure 2 Creep curves recorded at 1596 K and 84 MN m^{-2} for specimens of polycrystalline magnesia produced from two bars, labelled M and N, of nominally identical material.

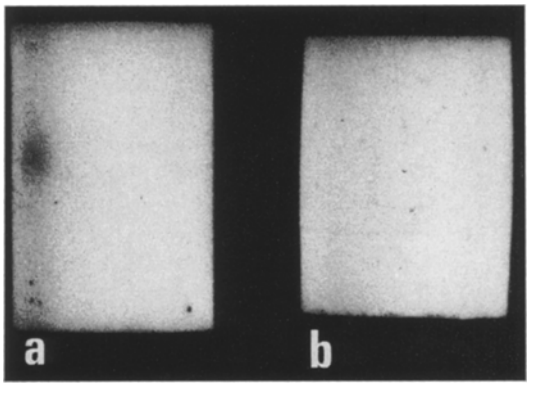

Figure 4 Cylindrical test specimens (6.4 mm long $\times$ 4.25 mm diameter), (a) before creep, (b) after 7% creep strain, illustrating slight barrelling which is detectable with compressive strains above ~ 6%.

3. Experimental results

3.1. The effect of small stress changes during creep

Duplicate creep tests were carried out at 1596 K (0.52 T_m, where T_m is the absolute melting point), and 62.6 MN m^{-2}. When the steady creep rate was attained, the applied stress was reduced carefully by a small amount, $\Delta\sigma$ ($\simeq 0.05\sigma$). It was established that after this small stress decrease, the creep rate was zero (Fig. 5). This incubation period of zero creep rate continued for some time before the creep rate increased to a new steady value. When this new steady creep rate was established at the reduced stress, the stress was increased again by $\Delta\sigma$ to the original stress level and the instantaneous strain ($\Delta\epsilon$) obtained on reloading was recorded (Fig. 5). On re-establishing the correct steady creep rate, $\dot{\epsilon}_s$, at the original applied stress, σ, the stress was again decreased by a slightly larger amount ($\sim 0.01\sigma$) and so on. The strain/time behaviour observed following a series of stress decreases are shown in Fig. 5. With small stress decreases (up to $\sim 0.15\sigma$), the duration of the incubation period, Δt, is linearly dependent on the extent of the stress reduction, $\Delta\sigma$, (Figs. 5 and 6) so that the ratio $\Delta\sigma/\Delta t$ is a constant. This ratio then gives a measure of the rate of recovery r ($= -\partial\sigma/\partial t$). Similarly, from the instantaneous specimen extension, $\Delta\epsilon$, obtained on increasing the applied stress by a small amount, $\Delta\sigma$, the ratio $\Delta\sigma/\Delta\epsilon$ can be obtained (Figs. 5 and 6), providing a measure of the coefficient of strain hardening, h ($= \partial\sigma/\partial\epsilon$). The strain, $\Delta\epsilon$, includes a small elastic contribution ($\sim 15\%$) which is comparable to the experimental error and thus has little effect on the value of h. In the present work, the ratio r/h predicts very closely the observed creep rate, $\dot{\epsilon}_s$, as required by Equation 2 (Table I).

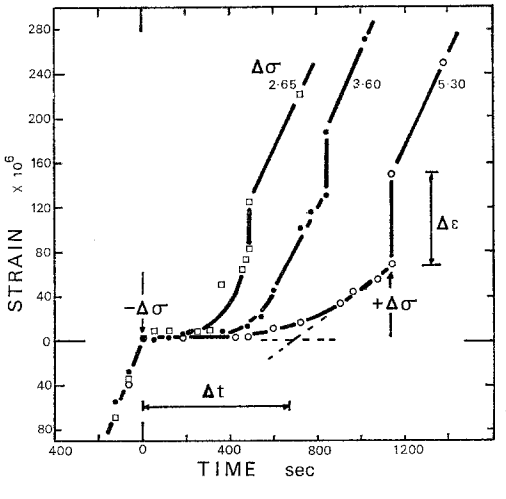

Figure 5 Strain/time relationships following stress changes during steady state creep of magnesia at 1596 K and 62.6 MN m^{-2}. The symbol □ denotes a stress decrease of 2.65 MN m^{-2} followed by a stress increase of 2.65 MN m^{-2} after a steady creep rate was established at the reduced stress. The symbols ● and ○ denote similar experiments with stress changes of 3.60 and 5.30 MN m^{-2} respectively.

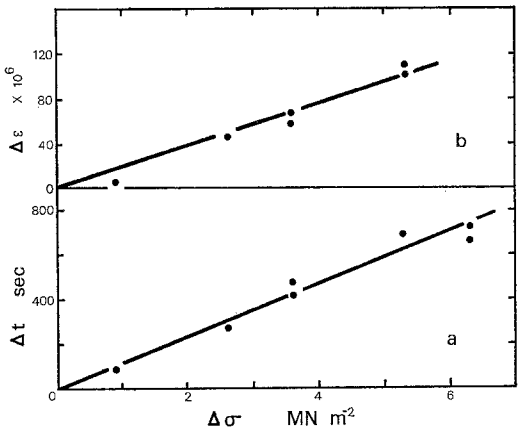

Figure 6 (a) The relationship between the duration of the incubation period of zero creep rate, Δt and the extent of the stress reduction, $\Delta\sigma$ during steady state creep. (b) The dependence of the instantaneous strain, $\Delta\epsilon$, observed on increasing the stress by $\Delta\sigma$ during steady state creep of magnesia at 1596 K and 62.6 MN m^{-2}.

3.2. Stress dependence of the rate of creep and recovery

A series of specimens were tested over a range of stresses at 1596 K. The creep tests were conducted at a temperature considerably below the sintering temperature employed to prepare the samples, in order to avoid grain growth during creep. Grain size measurement before and after creep indicated that no detectable growth had occurred. As previously found for magnesia [8-10], the steady state creep rate varies approximately as σ^3 (Fig. 3). Furthermore, the creep rates recorded are comparable with those of Hensler and Cullen [9]. At the same stress, the creep rates reported by Hensler and Cullen for magnesia of 97 to 99% density tested at 1573 K are approximately five times slower than the present results.

During each test, small stress changes were

TABLE I A comparison of the predicted steady creep rate (calculated as r/h) where r is the rate of recovery and h is the coefficient of strain hardening) and the measured steady creep rate, $\dot{\epsilon}_s$, over a range of stresses at 1596 K.

r (MN m^{-2} sec^{-1} × 10^2)	h (MN m^{-2} × 10^{-4})	Predicted creep rate, r/h (sec^{-1} × 10^7)	Observed creep rate, $\dot{\epsilon}_s$ (sec^{-1} × 10^7)
0.9	5.2	1.7	2.5
5.1	5.4	10.1	10.3
5.9	6.1	9.7	11.3
6.2	6.2	10.0	13.7
6.2	3.9	15.8	14.4
7.9	5.0	16.0	16.3
11.8	3.8	24.0	22.5
10.6	3.8	28.0	24.2
19.0	2.8	66.0	50.0
21.2	4.8	41.0	57.5

made when steady state creep was established so that, at each applied stress, the values of the rate of recovery, r, and the coefficient of strain hardening, h, were again determined as $\Delta\sigma/\Delta t$ and $\Delta\sigma/\Delta\epsilon$ respectively. Over the entire stress range examined, the steady creep rate was well predicted by the ratio r/h (Table I). Furthermore, the steady creep rate was found to be almost directly proportional to the rate of recovery (Fig. 7). Thus both the steady creep rate and the rate of recovery vary as σ^3.

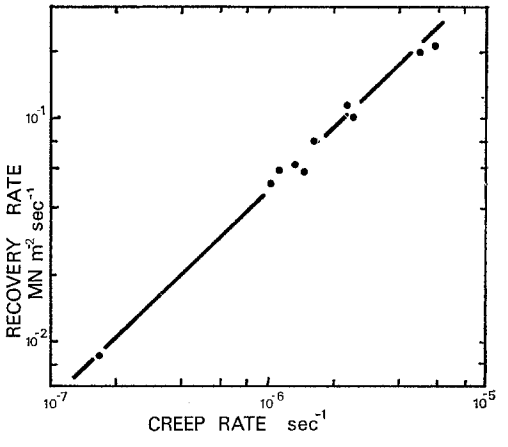

Figure 7 The relationship between the rate of recovery and the steady creep rate for magnesia at 1596 K.

4. Discussion

The present investigation confirmed that at high stresses [8, 10] the steady creep rate of magnesia varies approximately as σ^3. Since the stress exponent is greater than unity, deformation processes dependent upon stress directed diffusion through lattice [5, 6] or grain boundaries [7] are unlikely to be important. Similarly grain-boundary sliding is unlikely to contribute significantly to the overall creep rate, since the steady state creep rate of magnesia has been found [8] to be independent of the grain size over a range of stress and temperature similar to that of the present study. Furthermore, the results obtained indicate that glide processes, such as the motion of jogged screw dislocations [11], glide of dislocations dragging a cloud of charged defects [12, 13], etc. are not rate controlling since with these processes a small stress decrease should be followed immediately by a new slightly lower creep rate and not by an incubation period of zero creep rate. The occurrence of an incubation period (Fig. 5) indicates that the total internal stress opposing deformation during creep must be equal to or even slightly greater than the applied stress. Thus, when the stress is reduced, the structure must change by recovery processes before creep can recommence at the lower stress. This suggests that, under the present conditions, high temperature creep of magnesia is recovery controlled.

A recovery theory of creep by Weertman [14, 15] assumes that piled up groups of dislocation of opposite sign are generated on adjacent slip planes. The rate controlling process is then the climb and annihilation of edge dislocations from the piled up arrays allowing further deformation to occur. However, electron microscope studies have shown that piled up arrays are not found during creep of magnesia [10]. Instead, the dislocations are arranged in a three-dimensional network within subgrains [10]. A more general recovery theory of creep has been proposed by McLean [30, 16] based on the idea that deformation is considered to cause a

refinement of the dislocation network, while recovery results in a coarsening or growth of the network.

Recently, a modified network approach has been proposed by Davies and Wilshire [17] which suggests that the rate controlling process during high temperature creep is the growth of the dislocation network within the subgrains, to generate links of sufficient length to act as dislocation sources. Once a source is formed, the generation of dislocations increases the local dislocation density so that the next slip event is likely to occur elsewhere, i.e. the rate of deformation is controlled by the rate of recovery of the network and not, as in Weertman's theory, by climb of dislocations out of the piled up arrays. On this model, the existence of an incubation period on decreasing the stress would be expected since, at the reduced stress, the link length would be too small for sources to operate until the network size grows by recovery. Furthermore, the larger the stress reduction, the longer the incubation period as observed (Fig. 5).

Nabarro [31] has proposed a creep model in which deformation is considered to occur by operation of Bardeen-Herring sources. With this mechanism also, the rate controlling process is annealing of the dislocation network. However, with this model, climb is a deformation rather than a recovery process. In a detailed study of the dislocation arrangement developed during creep of magnesia, Bilde-Sorenson [10] concluded that the Nabarro model does not contribute significantly to the creep deformation. Instead the dislocation structures observed [10] were accounted for in terms of a recovery model similar to that proposed by Davies and Wilshire [17] namely that glide is the principal cause of deformation but that the creep rate is determined by diffusion controlled annealing of the dislocation network.

Friedel [32] has shown that the rate of network growth, $\mathrm{d}l/\mathrm{d}t$, is inversely proportional to l, the average length of the links in the network. The dislocation density, ρ, developed during creep of magnesia varies as [10]

$$\rho^{\frac{1}{2}} \propto \frac{1}{l} \propto \sigma . \tag{3}$$

The stress dependence of the rate of recovery is then given as [16]

$$r = \frac{\mathrm{d}\sigma}{\mathrm{d}t} = \frac{\mathrm{d}\sigma}{\mathrm{d}l} \cdot \frac{\mathrm{d}l}{\mathrm{d}t} \propto \sigma^3 . \tag{4}$$

The observed dependence of the steady creep rate and the rate of recovery (Figs. 3 and 7) on σ^3 can then be accounted for in terms of the network growth model for creep.

5. Conclusions

1. When the applied stress (σ) is reduced by a small amount, $\Delta\sigma$ ($\sim 0.05\sigma$) during steady state creep, an incubation period of zero creep rate is observed before the creep rate accelerates to a new steady value.

2. With the stress decreases up to $\sim 0.15\sigma$ the duration of the incubation period, Δt is proportional to the extent of the stress reduction, $\Delta\sigma$.

3. The rate of recovery, r ($\simeq -\partial\sigma/\partial t$), calculated as $\Delta\sigma/\Delta t$, is directly proportional to the steady creep rate, $\dot{\epsilon}_s$.

4. When the coefficient of strain hardening, h ($= \partial\sigma/\partial\epsilon$) is calculated as $\Delta\sigma/\Delta\epsilon$ (where $\Delta\epsilon$ is the instantaneous strain observed on increasing the stress by a small amount $\Delta\sigma$) the measured steady creep rate $\dot{\epsilon}_s$ is predicted accurately by the ratio r/h.

5. These observations show that high temperature creep of magnesia is recovery controlled.

6. The steady creep rate varies approximately as σ^3.

7. The results can be accounted for in terms of a model for creep in which the rate controlling process is the growth of the three-dimensional dislocation network within subgrains, to form dislocation sources, allowing slip to occur.

Acknowledgements

One of the authors, J. M. Birch, is indebted to the Science Research Council for a maintenance grant during the period of this research. The authors would also like to thank the Steetley Co Ltd for their co-operation in this work.

References

1. T. VASILOS, J. B. MITCHELL and R. M. SPRIGGS, *J. Amer. Ceram. Soc.* **47** (1964) 203.
2. E. M. PASSMORE, R. H. DUFF and T. VASILOS, *ibid* **49** (1966) 594.
3. T. ZISNER and H. TAGAI, *ibid* **51** (1968) 310.
4. R. S. GORDON and G. R. TERWILLIGER, *ibid* **55** (1972) 310.
5. F. R. N. NABARRO, "Report on Conference on The Strength of Solids", University of Bristol, July 1947 (The Physical Society, London, 1948) p. 75.
6. C. HERRING, *J. Appl. Phys.* **21** (1950) 437.
7. R. L. COBLE, *ibid* **34** (1963) 1679.

8. T. G. LANGDON and J. A. PASK, *Acta Metallurgica* **18** (1970) 505.
9. J. H. HENSLER and G. V. CULLEN, *J. Amer. Ceram. Soc.* **51** (1968) 557.
10. J. B. BILDE-SORENSON, *ibid* **55** (1972) 606.
11. N. F. MOTT, "Creep and Fracture of Metals at High Temperatures" (HMSO, London, 1956) p. 21.
12. J. WEERTMAN. *J. Appl. Phys.* **28** (1957) 1185.
13. W. R. CANNON and O. D. SHERBY, *J. Amer. Ceram. Soc.* **56** (1973) 157.
14. J. WEERTMAN, *J. Appl. Phys.* **26** (1955) 1213.
15. *Idem, ibid* **28** (1957) 362.
16. S. K. MITRA and D. MCLEAN, *Proc. Roy. Soc. A* **295** (1966) 288.
17. P. W. DAVIES and B. WILSHIRE, *Scripta Met.* **5** (1971) 475.
18. R. F. CANON, J. T. A. ROBERTS and R. J. BEALS, *J. Amer. Ceram. Soc.* **54** (1971) 105.
19. E. OROWAN, *J. West Scot. Iron and Steel Inst.* **54** (1946) 45.
20. C. K. L. DAVIES, P. W. DAVIES and B. WILSHIRE, *Phil. Mag.* **12** (1965) 827.
21. D. SIDEY and B. WILSHIRE, *Metal Sci. J.* **3** (1969) 56.
22. R. G. ST-JACQUES and R. ANGERS, *J. Amer. Ceram. Soc.* **55** (1972) 571.
23. R. L. BERTOLOTTI and W. D. SCOTT, *ibid* **54** (1971) 286.
24. G. M. FRYER and P. THOMPSON, *Trans. Brit. Ceram. Soc.* **71** (1972) 61.
25. R. D. CROPPER and J. A. PASK, *Ceram. Bull.* **48** (1969) 355.
26. D. R. F. SPENCER, *Trans. Brit. Ceram. Soc.* **11** (1972) 123.
27. P. W. DAVIES and R. DUTTON, *J. Sci. Instrum.* **43** (1966) 39.
28. E. M. da C. ANDRADE and B. CHALMERS, *Proc. Roy. Soc. A* **138** (1932) 348.
29. L. E. POTEAT, Ph.D. Thesis, North Carolinia State University, Raleigh, NC (1966).
30. D. MCLEAN, *Rep. Prog. Phys.* **29** (1966) 1.
31. F. R. N. NABARRO, *Phil. Mag.* **16** (1967) 231.
32. J. FRIEDEL, "Dislocations" (Pergamon Press, London, 1964).

Received 8 October and accepted 8 November 1973.