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Transient and steady state creep behaviour of polycrystalline MgO

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Stress change experiments during compressive creep tests at high stresses on polycrystalline MgO at 1596 K have shown that the creep rate at any instant during transient and steady state creep is predicted by the ratio, r/h, where r is the rate of recovery ($= -\partial\sigma/\partial t$) and h is the coefficient of strain hardening ($= \partial\sigma/\partial\epsilon$). Over most of transient and steady state creep, when h is constant and the decrease in creep rate, $\dot{\epsilon}$, is a direct result of a decrease in r, the variation of the creep strain, ϵ, with time, t, is accurately described as

$$\epsilon = \epsilon_0 + \epsilon_T(1 - e^{-mt}) + \dot{\epsilon}_S t$$

where ϵ_0 is the instantaneous strain on loading, ϵ_T the transient creep strain, m relates to the rate of exhaustion of transient creep and $\dot{\epsilon}_S$ is the steady creep rate. Deviations from this equation occur during the initial 10 to 15% of the transient creep life, when h increases rapidly.

The variations in $\dot{\epsilon}$, r and h during transient and steady state creep are explained in terms of a model for creep in which the rate-determining process is the diffusion controlled growth of the three-dimensional dislocation network within subgrains to form dislocation sources allowing slip to occur.

1. Introduction

In many of the investigations which have been carried out on the high temperature creep behaviour of magnesia, it has been observed [1-7] that the initial specimen strain on loading is followed by a transient stage during which the creep rate decreases with time until a steady state is achieved when the creep rate appears to remain constant. Most of these investigations have concentrated on the steady state condition, whilst comparatively few studies have been devoted to the transient creep stage even though it has been recognized [8] that information on the rate controlling processes during creep of ceramics can be obtained from a consideration of the shape of the transient creep curve.

It is widely accepted that, during high temperature creep of fine grained, polycrystalline MgO under low stresses, the linear dependence of the steady creep rate on the applied stress, σ, is associated with stress-directed diffusion through the lattice [9, 10] or the grain boundaries [11]. Under these conditions, a transient creep stage is observed [3, 4, 6] although on a diffusion creep model, a steady creep rate would be expected immediately after loading [8]. This occurence of a transient stage has been attributed [3, 4] to simultaneous grain growth during creep. However, transient creep has been reported [6] when grain growth has been retarded in Fe-doped MgO and in MgO tested at low temperatures. Expressing the total strain-rate, $\dot{\epsilon}$, as the sum of the transient, $\dot{\epsilon}_{tr}$, and the steady state, $\dot{\epsilon}_s$, creep rates as

$$\dot{\epsilon} = \dot{\epsilon}_{tr} + \dot{\epsilon}_s \quad (1)$$

it has been demonstrated [6] that the variation of the transient creep rate with time, t, can be described as

$$\dot{\epsilon}_{tr} = \dot{\epsilon}_0 e^{-t/\tau}. \quad (2)$$

The initial transient creep rate, $\dot{\epsilon}_0$ (i.e. $\dot{\epsilon}_{tr}$ when t is zero) was found to be linearly dependent on applied stress, and the relaxation time, τ, was shown to be thermally activated. This behaviour was considered to be consistent with a diffusion creep mechanism [6].

Although studies have been made of the

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transient creep behaviour of magnesia at low stresses, little work has been carried out on the transient stage under high stress conditions. At high stresses, when the steady creep rate varies approximately as σ^3, creep is generally considered to occur by dislocation movement [5, 7, 8]. The present work was, therefore, undertaken to study the deformation processes occurring and the strain/time relationships during transient and steady state creep of magnesia in the high stress regime.

2. Experimental procedure

The detailed analysis (wt%) of the magnesia was as follows: 99.85% MgO, 0.1% SiO_2, <0.02% CaO, Al_2O_3 and Fe_2O_3 and <0.012% B_2O_3. The material was produced from specially prepared $Mg(OH)_2$, which was calcined at 1570 K, pressed into bars and sintered at 1920 K. Cylindrical specimens of magnesia (4.25 mm diameter and 6.4 mm long) were prepared from the bars having 93 to 95% theoretical density and having an average grain diameter in the range 10 to 14 μm [12].

A series of compression creep tests were carried out at 1596 (±1) K over a range of stresses from 70 to 85 MN m^{-2}, using a constant stress machine [7] capable of determining specimen strains to 10 nm. The creep tests were carried out at a temperature considerably below the sintering temperature employed to prepare the samples, in order to avoid grain growth during creep. Grain size measurements before and after creep showed that no detectable growth had occurred during the tests.

3. Experimental results

3.1. Strain/time relationship during creep

For each creep test, the high initial creep rate, observed immediately following the initial specimen extension on loading, decreased continuously to an apparently constant rate. In all cases, about 60 strain/time readings were taken at approximately constant time intervals during the creep test. All strain readings were then converted to true strain values.

A least-squares analysis was carried out over the period of apparently constant creep rate, during which about 30 strain/time readings had been recorded. For each test, a linear strain/time relationship was found with a correlation coefficient of better than 0.9999. Furthermore, the average difference between the measured strain/time readings and the calculated best straight line was <0.000 02 over this period when the total specimen strain was ~0.01. The strain/time readings taken during this period of apparently constant creep rate were then divided into three consecutive groups, whose gradients were determined separately. Applying student's t test to the differences between these gradients gave values of t in the range 0.2 to 0.9 (for 16 degrees of freedom) showing that the gradients were not significantly different. Thus, the creep rate is constant rather than continuously decreasing or associated with an inflection in the creep curve.

The observation that a steady state exists, indicates that the time dependence of the creep strain cannot be adequately described by an equation of the form [13]

$$\epsilon = \epsilon_0 + at^{1/3} + \dot{\epsilon}_s t \qquad (3)$$

where ϵ_0 is the instantaneous strain on loading and a is a transient creep parameter. This equation predicts an infinite initial creep rate and thereafter a continuously decreasing creep rate. Similarly, since an inflection in the creep rate does not occur, the strain/time relationship cannot be represented as [14]

$$\epsilon = \epsilon_0 + bt^{\alpha} + ct^{\beta} \qquad (4)$$

where α is <1 and β is >1. The material parameters, b and c, depend on the stress and temperature.

An equation which does not predict an infinite initial creep rate but which does lead to a steady creep rate has been proposed by McVetty [15] and Garofalo [16] as

$$\epsilon = \epsilon_0 + \epsilon_T (1 - e^{-mt}) + \dot{\epsilon}_s t \qquad (5)$$

where ϵ_T is the total transient strain and m is a parameter relating to the rate of exhaustion of transient creep. For each creep test, the best value of the parameters ϵ_0, ϵ_T, m and $\dot{\epsilon}_s$ was determined by a least squares analysis using a digital computer [17]. The accuracy with which Equation 5 represents the creep curve was then established by computing the average difference between each experimental reading and the best fitting curve, using the derived constants. Over most of the creep curve, the average difference between the measured strain, ϵ, and the calculated value, ϵ_c, applying the derived constants was <0.000 05. Equation 5, therefore, accurately describes the variation of creep strain with time except during the initial 10-15% of the transient creep life, when the difference ($\epsilon_c - \epsilon$) increased

progressively as t approached zero (Fig. 1). This behaviour is exactly the same as that reported for a wide range of metals and alloys [17-19].

It is noteworthy that, with $m = 1/\tau$, differentiating Equation 5 gives Equation 2. Thus, Equation 5 appears to describe the time dependence of the creep strain when creep occurs either by stress directed diffusion or by dislocation movement.

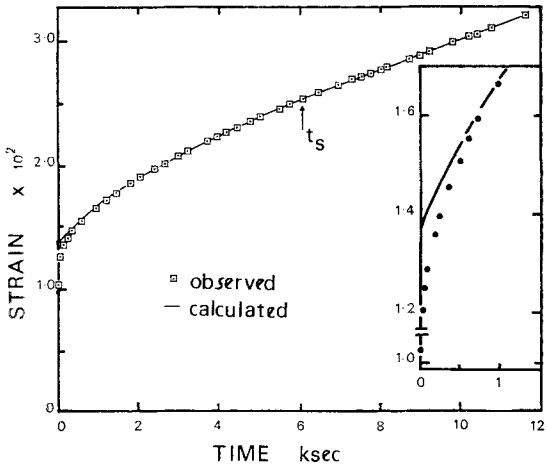

Figure 1 The variation of the true creep strain with time during transient and steady state creep of polycrystalline MgO at 84.6 MN m^{-2} and 1596K. The time to the onset of steady state creep, t_s, was ~6 ksec under these conditions.

3.2. The effect of small stress changes during transient and steady state creep

It has been established previously [7] that when the applied stress, σ, is reduced by a small amount $\Delta\sigma$ (~0.05σ) during steady state creep of magnesia at high stresses, an incubation period of zero rate is observed before the creep rate accelerates to the value expected at the reduced stress level. The present work shows that a small stress reduction (~0.05σ) at any instant during the transient stage also results in an incubation period (Fig. 2). The occurrence of an incubation period of zero creep rate suggests that the flow stress must change by recovery processes before deformation can commence at the reduced stress level. Thus, the deformation process is recovery-controlled during both transient and steady state creep.

Recovery theories of creep at high temperatures are based on the concept that strain

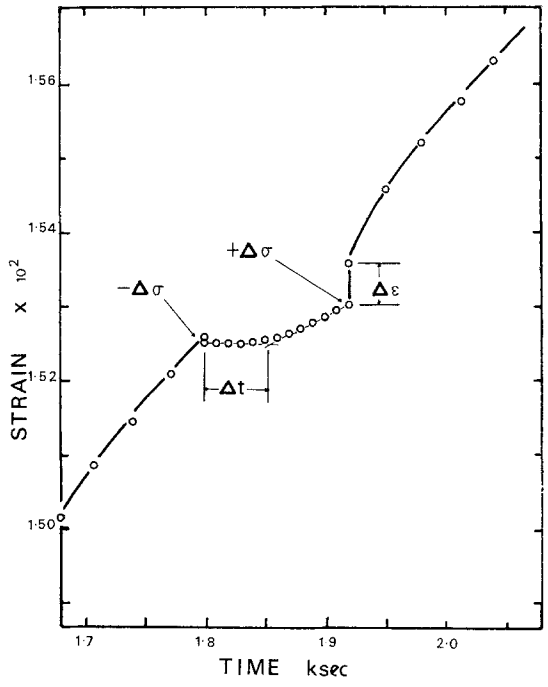

Figure 2 The strain/time behaviour following small stress changes during transient creep of polycrystalline MgO at 84.6 MN m^{-2} and 1596K.

hardening resulting from deformation is continuously annealed out by recovery processes to maintain a constant flow stress, σ. The creep rate, $\dot{\epsilon}$, can then be expressed as [20, 21]

$$\dot{\epsilon} = d\epsilon/dt = r/h \qquad (6)$$

where r ($= -\partial\sigma/\partial t$) is the rate of recovery and h ($= \partial\sigma/\partial\epsilon$) is the coefficient of strain hardening. From the duration of the incubation period, Δt, following a stress reduction, $\Delta\sigma$ (up to ~0.15σ) for magnesia [7], a measure of the rate of recovery r, can be obtained as $\Delta\sigma/\Delta t$. Similarly the coefficient of strain hardening, h, is measured as $\Delta\sigma/\Delta\epsilon$, where $\Delta\epsilon$ is the strain* obtained on increasing the stress by $\Delta\sigma$. As reported previously [7] for steady state creep, the creep rate at any instant during the transient stage can be predicted by the ratio, r/h (Fig. 3a).

The variation of $\dot{\epsilon}$, r and h during transient and steady state creep is shown in Fig. 3. Clearly from Fig. 3b, it can be seen that r decreased continuously throughout the transient stage becoming constant at the start of steady state creep. The value of h increased rapidly during the early part of the transient creep life

*It must be noted that the strain, $\Delta\epsilon$, includes a small elastic contribution (~ 15%) which is comparable to the experimental error and thus has little effect on the value of h.

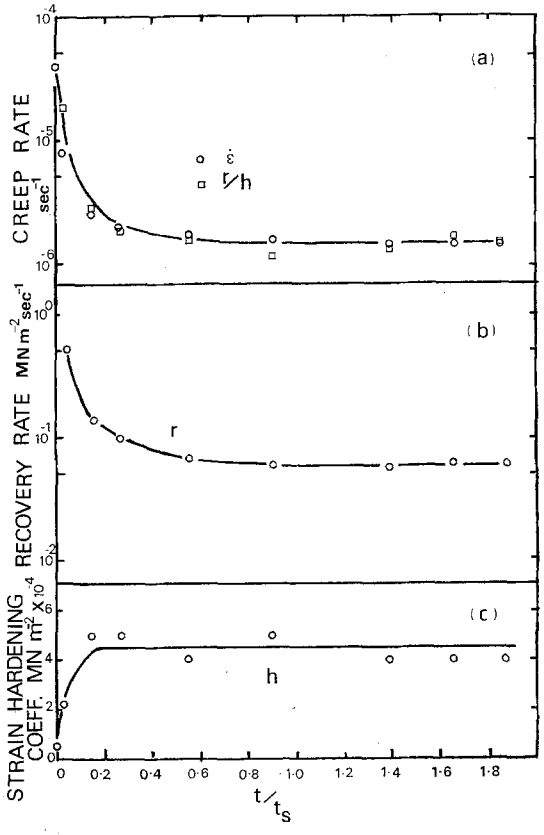

Figure 3 The variation of (a) the creep rate, $\dot{\epsilon}$, and the ratio, r/h, (b) the rate of recovery, r and (c) the coefficient of strain hardening, h, during transient and steady state creep at 84.6 MN m^{-2} and 1596K.

before attaining a constant value (Fig. 3c). Measurement of the creep rate, the rate of recovery and the coefficient of strain hardening is difficult during the early stages of the creep curve, when the creep rate is high and decreasing rapidly. However, it appears that the deviations from Equation 5 (Fig. 1) occur during the period when h increases markedly (Fig. 3b). When h is constant and the creep rate, $\dot{\epsilon}$, is directly proportional to the rate of recovery, r, Equation 5 accurately describes the creep curve. Similar behaviour has been reported for a number of pure metals and alloys [17-19].

4. Discussion

The dislocation substructure developed during compressive creep of single and polycrystalline MgO at high stresses, has been investigated using both etch-pit and transmission electron microscopy techniques [22-24]. During steady state creep it has been established that the grains are divided into more or less well-defined subgrains, with a three-dimensional dislocation network within the subgrains. However, this subgrain boundary structure is not developed immediately on loading [23]. Early in the transient stage, the sub-structure is characterized by slip bands and diffuse dislocation walls which develop into nearly parallel cell walls. These gradually transform to the equiaxed cell arrangement found in the steady state. The dislocation density within the subgrains decreases throughout the transient stage, becoming constant during steady state creep. Similar observations on the changes in dislocation arrangement during transient and steady state creep have been reported for sodium chloride [25].

The dislocation structure developed during creep has been interpreted [22] in terms of a model [26] for creep based on the growth by recovery processes of the three-dimensional dislocation network within the subgrains until links* are formed which are sufficiently long to act as dislocation sources allowing slip to occur. On this model, creep deformation occurs by dislocation glide even though the rate determining process is the diffusion-controlled growth of the dislocation network. The existence of an incubation period on decreasing the stress would then 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 processes.

This model can be developed to account for the observed variations in $\dot{\epsilon}$, r and h during transient and steady state creep (Fig. 3). It is suggested that immediately after the initial specimen strain on loading, all of the dislocation links of length, l_s, ($\geqslant 2\alpha\mu\boldsymbol{b}/\tau$, where α is approximately unity, μ the shear modulus, $\boldsymbol{b}$ the Burgers vector and τ is the local resolved shear stress) will have operated as dislocation sources. Subsequent creep deformation then occurs by growth of the dislocation network providing new sources. The creep rate at any instant will therefore depend on the rate of production of sources and the average strain per source. From the reported changes in dislocation structure during creep, it would appear that, early in the creep life, the dislocations generated at a source move considerable distances across the slip

*The term "link" is defined as an idealized straight length of dislocation connecting two nodes in the three-dimensional dislocation network.

planes forming distinct slip bands. However, the distances moved and, hence, the average strain per source, would decrease considerably as a subgrain structure develops early in the transient stage. This decrease in the average strain per source could account for the rapid increase in h (i.e. a marked decrease in the strain, $\Delta\epsilon$, for a given stress increment, $\Delta\sigma$) at the start of a creep test (Fig. 3c). Generation of dislocations at a source results in local strain hardening so that the next slip event is likely to occur elsewhere The rate of production of new sources will be governed by the rate of growth of the three-dimensional dislocation network which is related to the rate of recovery r. Using Friedel's theory of network growth [27], the recovery rate varies as $\rho^{3/2}$, where ρ is the density of dislocations within the subgrains. On this basis, the observed decrease in ρ during the transient stage [23] accounts for the decrease in r, and hence $\dot{\epsilon}$, throughout transient creep (Figs. 3a and b).

On this model, although the rate of generation of sources and the average strain per source varies as the dislocation arrangement changes during the transient stage, the rate determining process, namely the diffusion controlled growth of the three-dimensional dislocation network, is the same throughout transient and steady state creep.

5. Conclusions

Creep tests carried out at high stresses on polycrystalline MgO at 1596 K have shown that:

1. the time dependence of the true creep strain during most of transient and steady state creep can be described accurately by the equation

$$\epsilon = \epsilon_0 + \epsilon_T (1 - e^{-mt}) + \dot{\epsilon}_s t .$$

Deviations from this equation occur during the initial 10 to 15% of the transient creep life. As with metallic materials, this behaviour can be explained in terms of variations in the rate of recovery and the coefficient of strain hardening during creep;

2. the changes in the creep rate, the rate of recovery and the coefficient of strain hardening during transient and steady state creep can be interpreted on the basis of a model for creep in which the rate-determining process is the diffusion-controlled growth of the three- dimensional dislocation network within subgrains, to form dislocation sources which allow slip to occur.

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References

1. E. M. PASSMORE, R. H. DUFF and T. VASILOS, *J. Amer. Ceram. Soc.* **49** (1966) 594.
2. J. H. HENSLER and G. V. CULLEN, *ibid* **51** (1968) 179.
3. H. TAGAI and T. ZISNER, *ibid* **51** (1968) 303.
4. G. R. TERWILLIGER, H. K. BOWEN and R. S. GORDON, *ibid* **53** (1970) 241.
5. T. G. LANGDON and J. A. PASK, *Acta Met.* **18** (1970) 505.
6. R. S. GORDON and G. R. TERWILLIGER, *J. Amer. Ceram. Soc.* **55** (1972) 450.
7. J. M. BIRCH and B. WILSHIRE, *J. Mater. Sci.* **9**, (1974) 794.
8. T. G. LANGDON, D. R. CROPPER and J. A. PASK, "Ceramics in Severe Environments" (Plenum, New York, 1971) p. 297.
9. F. R. N. NABARRO, "Report on Conference on Strength of Solids", University of Bristol, 1947. (The Physical Society, London, 1948) p. 75.
10. C. HERRING, *J. Appl. Phys.* **21** (1950) 437.
11. R. L. COBLE, *ibid* **34** (1963) 1679.
12. D. R. F. SPENCER, *Trans. Brit. Ceram. Soc.* **11** (1972) 123.
13. A. H. COTTRELL and V. AYTEKIN, *Nature* **160** (1947) 328.
14. J. DE LACOMBE, *Rev. Met.* **36** (1939) 178.
15. P. G. MCVETTY, *Mech. Eng.* **56** (1934) 149.
16. F. GAROFALO, "Fundamentals of Creep and Creep-Rupture in Metals" (Macmillan, New York, 1965).
17. W. J. EVANS and B. WILSHIRE, *Met. Sci. J.* **4** (1970) 89.
18. D. SIDEY and B. WILSHIRE, *Met. Sci. J.* **3** (1969) 56.
19. W. J. EVANS and B. WILSHIRE, *Trans. Met. Soc. AIME* **242** (1968) 1303.
20. E. OROWAN, *J. West. Scot. Iron Steel Inst.* **54** (1946-47) 45.
21. R. W. BAILEY, *J. Inst. Metals* **35** (1926) 27.
22. J. B. BILDE-SORENSEN, *J. Amer. Ceram. Soc.* **55** (1972) 606.
23. W. HUTHER and B. REPPICH, *Phil. Mag.* **28** (1973) 363.
24. J. B. BILDE-SORENSEN, *Acta Met.* **21** (1973) 1495.
25. J. P. POIRIER, *Phil. Mag.* **26** (1972) 713.
26. P. W. DAVIES and B. WILSHIRE, *Scripta Met.* **5** (1971) 475.
27. J. FRIEDEL, "Dislocations" (Pergamon, Oxford, 1964).

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