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IMPURITY EFFECTS ON THE CREEP OF POLYCRYSTALLINE MAGNESIUM AND ALUMINUM OXIDES AT ELEVATED TEMPERATURES

MASTER

Technical Progress Report
for December 19, 1978 - December 18, 1979

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DISCLAIMER

TECHNICAL PROGRESS REPORT

December 18, 1979

PROJECT TITLE	Impurity Effects on the Creep of Polycrystalline Magnesium and Aluminum Oxides at Elevated Temperatures
CONTRACT	EY-76-S-02-1591.*002
CONTRACTOR	University of Utah Department of Materials Science and Engineering
PROJECT PERSONNEL	<u>Principal Investigator</u> Dr. Ronald S. Gordon Professor <u>Graduate Research Assistant</u> Mr. Yasuro Ikuma

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I ABSTRACT

The research described in this report covers work on:

1. The effect of mixed transition metal impurities (Fe-Ti, Mn-Ti, and Mg-Ti) on the steady state creep of polycrystalline aluminum oxide

2. Stress relaxation deformation studies on polycrystalline MgO and Al_2O_3, pure and doped with iron

3. Theoretical analysis of ambipolar diffusion taking into account interfacial defect creation and/or annihilation processes at grain boundaries.

Some of the significant findings include:

1. Diffusional creep controlled by an interfacial defect creation-annihilation process at grain boundaries has been experimentally established for polycrystalline alumina doped either with titanium or a mixed Mg-Ti dopant. Interfacial kinetics correspond to a reciprocal relationship between the steady state creep rate and the grain size.

2. Interfacial effects become important in two limiting situations:
(a) at small grain sizes when cation grain boundary diffusion is slow
(b) at intermediate grain sizes providing cation lattice diffusion is very high and cation grain boundary diffusion is slow.

3. Divalent transition metal impurities such as Fe^{2+} apparently enhance aluminum grain boundary diffusion to a larger degree than aluminum lattice diffusion.

4. Diffusional creep in Fe-doped Al_2O_3 and in the Fe-Ti and Mn-Ti double doped systems is controlled by various combinations of aluminum lattice and grain boundary diffusion.

II. INTRODUCTION

The research activities under contract E(11-1) - 1591 during the period December 19, 1978 to December 18, 1979 are described in this report.

Major effort was devoted to:

1) Dead load creep tests on polycrystalline alumina doped with several cation impurity pairs: Fe-Ti, Mn-Ti, and Mg-Ti

2) Theoretical analysis of ambipolar diffusion taking into account the possibility of kinetics dominated by interfacial defect creation and/or annihilation processes at the grain boundaries

3) Stress relaxation tests on polycrystalline MgO doped with iron and polycrystalline Al_2O_3, pure and doped with iron

One paper was published this year. A reprint is submitted with this report.

"Stress Relaxation Technique for Deformation Studies in Four-Point-Bend Tests - Application to Polycrystalline Ceramics at Elevated Temperatures" D. K. Shetty and R. S. Gordon, J. Mater. Sci. 14 (9) 2163-2171 (1979).

A complete listing of the bibliography of papers and theses prepared under this contract is given at the end of the report in Section IV.

III REVIEW OF RESEARCH ACTIVITIES

In this section the research tasks which have been conducted under the contract for the current period will be described. This year research was conducted on (1) the effect of mixed cation dopants on the diffusional creep of polycrystalline Al_2O_3, (2) an analysis of interfacial grain boundary processes in ambipolar diffusional creep and (3) stress relaxation deformation studies on polycrystalline MgO and Al_2O_3, pure and doped with iron

3.1 Creep of Polycrystalline Alumina Doped with Mixed Cation Impurities

This year steady state creep experiments were conducted on high density (>99%) polycrystalline alumina doped with cation impurities at the following compositions (cation percent):

1. 0.2% Ti
2. 0.15 % Ti + 0.05% Fe
3. 0.1% Ti + 0.1% Fe
4. 0.05% Ti + 0.15% Fe
5. 0.2% Fe
6. 0.05% Ti
7. 0.05% Ti + 0.05% Mg
8. 0.05% Ti + 1.0% Fe
9. 0.24% Ti + 0.075% Mn
10. 0.12% Ti + 0.25% Mn

Four point bend dead load creep tests were conducted over a range of grain sizes (5-100 μM temperatures (1100-1500^{0}C), stresses (2-17 MN/m^2) and oxygen partial pressures (10^{-11} - 0.86 atm).

3.1.1 Fe-Ti System

The vast majority of experiments were conducted in this system particularly at the 0.2% total impurity concentration ranging from pure Ti, various Ti/Fe ratios, and pure Fe.

The most significant results were obtained from the studies on the effect of grain size. Perhaps the most important finding was the discovery of a nearly reciprocal grain size dependence of the steady state creep rate ($\dot{\varepsilon}$) at the 0.05% Ti, and 0.05% Ti + 0.05% Mg, and 0.2% Ti compositions (ie $\dot{\varepsilon}\ \alpha\ (GS)^{-m}$ with m = 1.0-1.3). A creep grain size exponent (m)of unity is the first experimental verification of diffusional creep rate-limited by an interfacial (grain boundary) defect creation (or annihilation) process in polycrystalline alumina. At the 0.2% Fe and the mixed Fe-Ti compositions, the creep grain size exponent was nearly 2 (i.e. a reciprocal square grain size dependence) which was indicative of Nabarro-Herring diffusional creep rate-limited by aluminum lattice diffusion. The effects of oxygen partial (i.e. decreasing creep rates with decreasing oxygen partial pressure) and the creep activation energies (∿ 130 kcal/mole) at these compositions were also consistent with kinetics governed by aluminum lattice diffusion.

Experiments at the 1% Fe + 0.05% Ti composition revealed (1) a reciprocal grain size dependence indicative of kinetics rate-limited by aluminum lattice diffusion (2) significantly enhanced creep rates (factor ∿5) over the 1.0% Fe composition and (3) oxygen partial pressure effects consistent with the dominance of the defect structure by divalent iron (i.e. increasing creep rates with decreasing oxygen partial pressure).

3.2.2 Mg-Ti System: Nearly reciprocal grain size dependencies (i.e. m=1.2-1.3) and identical creep rates were observed for the 0.05% Ti and 0.05% Ti + 0.05% Mg compositions indicating the dominance of the interfacial grain boundary reaction in diffusional creep of alumina at these compositions. Apparently very little compensation occurs between divalent magnesium and quadrivalent titanium.

3.1.3 Mn-Ti System: Steady state creep experiments at the compositions studied revealed creep grain size exponents between 2 and 3 and hence kinetics controlled by a combination of aluminum lattice and grain boundary diffusion.

3.1.4 General Conclusions from Creep Studies with Mixed Cation Dopants

The nearly reciprocal grain size dependence found at the 0.05% Ti, 0.2% Ti, and 0.05% Ti + 0.05 Mg% compositions is apparently the key to understanding (1) the effect of mixed valence cation dopants on the diffusional creep of polycrystalline alumina, and(2) the dominant defect structure in Al_2O_3 (i.e. aluminum cation Frenkel versus Schottky defects). In titanium-doped material, the Ti^{4+} ions in substitutional solid solution and the compensating aluminum lattice vacancies enhance aluminum lattice diffusion to such a degree that diffusional creep becomes rate limited by vacancy creation and/or annihilation at the grain boundaries (i.e. interface control). Apparently Ti^{4+} dominates the defect structure via the creation of aluminum ion vacancies in the mixed Fe-Ti compositions at the 0.2% concentration of total impurities. As the Ti/Fe cation ratio increases at a fixed concentration of total impurities (0.2%), the diffusional creep rate (and the aluminum lattice diffusion coefficient) increases to a maximum before the grain boundary interfacial reaction becomes dominant at the pure Ti composition. At this limit the creep rate either decreases or remains constant (independent of the Ti^{4+} concentration) depending on the grain size.

Ti^{4+} apparently also dominates the defect structure in the mixed Mg-Ti compositions. Here, as in the Ti-doped material, interfacial defect creation (or annihilation) at grain boundaries becomes rate limiting due to high aluminum ion mobilities in the lattice.

Interfacial effects become dominant in diffusional creep when the concentration of defects controlling lattice diffusion become too large for rapid equilibration at grain boundaries. In this case, grain boundaries are not perfect sinks and sources of lattice defects, as is commonly assumed in diffusional creep theories.

The Mn-Ti impurity couple is the most effective in enhancing the diffusional creep rate of polycrystalline alumina ($\sim$3-4 orders of magnitude enhancement in creep rate). In this mixed impurity system, the concentrations of divalent and quadrivalent impurities are apparently enhanced significantly over levels present in alumina doped singly either with Mn or Ti.

In both the Fe-Ti and Mn-Ti systems, the variable valent ions, Fe and Mn, apparently enhance aluminum grain boundary diffusion and also assist in equilibration at grain boundaries since interfacial creep kinetics are never observed.

In view of the results on the effects of mixed cation dopants, the intrinsic defect structure in aluminum oxide has been reassessed. It is apparent that the the dominance of Schottky defects can explain effectively the diffusional creep characteristics of polycrystalline $A\ell_2O_3$, pure and doped with single impurities such as Mg, Fe (Fe^{2+}, Fe^{3+}), Cr and Ti (Ti^{3+}, Ti^{4+}) as well as impurity pairs such as Fe-Ti, Mn-Ti, and Mg-Ti. Several significant findings can be explained in terms of a Schottky defect model for alumina which cannot be readily interpreted if aluminum Frenkel defects are the dominant intrinsic defects. These include

(1) the absence of any minimum creep rates due to the simultaneous doping of divalent and quadrivalent substitutional cation impurities. If aluminum ion Frenkel defects were dominant, then one would expect that the compensating defects (i.e. aluminum vacancies and interstitials) for the quadrivalent and divalent impurities would annihilate each other and lead to a substantial reduction in the aluminum ion diffusivity. The opposite result is observed.

(2) The occurence of a rate limiting interfacial (grain boundary) process for the creation and annihilation of cation vacancies when the concentration of dominant lattice defects (aluminum ion vacancies compensating for substitutional quadrivalent titanium) becomes very large. Interfacial reactions to not become important in the case of divalent impurities because the dominant compensating defect is an oxygen vacancy and creep is controlled by the much smaller concentration of a minority defect (i.e. aluminum ion interstitials) which does not create problems associated with defect creation or annihilation at grain boundaries. If Frenkel defects were dominant, one would expect grain boundary

saturation and interfacial kinetics as the concentration of divalent impurity increased. Instead one finds no evidence of interface control but a competition between aluminum lattice and grain boundary diffusion in the control of diffusional creep rates as is evident from creep grain size exponents ranging between 2 and 3.

3.2 Generalized Ambipolar Diffusional Creep Theory Including Grain Boundary Interfacial Effects

In order to take into account interfacial defect reactions at grain boundaries, the ambipolar diffusional creep equation developed earlier on this contract for the compound $A_\alpha B_\beta$ must be modified. In the general situation, three different processes can control creep ((1) lattice diffusion, (2) grain boundary diffusion and (3) an interfacial defect reaction) and two diffusing species must be considered. Assuming the rate of the interfacial defect reaction for species A and B to vary linearly with stress, the following generalized creep equation was derived:

$$\dot{\varepsilon} = \frac{44\Omega_V \sigma}{kT\,(GS)^3} D_{\text{Complex}} \qquad (1)$$

$$D_{\text{Complex}} = \frac{1}{\dfrac{\alpha}{\dfrac{\dfrac{(GS)^3\Omega_V D_A^{\ell} K_A}{11\,\Pi}}{\dfrac{(GS)D_A^{\ell}}{\Pi} + \dfrac{(GS)^2 K_A \Omega_V}{11}} + \delta_A D_A^{b}} + \dfrac{\beta}{\dfrac{\dfrac{(GS)^3\Omega_V D_B^{\ell} K_B}{11\,\Pi}}{\dfrac{(GS)D_B^{\ell}}{\Pi} + \dfrac{(GS)^2 K_\beta \Omega_V}{11}} + \delta_B D_B^{b}}} \qquad (2)$$

Here, D_A^{ℓ} and D_A^{b} are the $A^{\beta+}$ ion lattice and grain boundary diffusivities, D_B^{ℓ} and D_B^{b} are the $B^{\alpha-}$ ion lattice and grain boundary diffusivities, δ_A and δ_B are the effective widiths of the regions of enhanced diffusion near the grain boundaries for $A^{\beta+}$ and $B^{\alpha-}$ ions, and K_A and K_B are the interfacial rate constants for anion and cation vacancy (or interstitial) creation on annihilation, respectively, at the grain boundaries. (No interfacial processes are assumed to be required for boundary diffusion processes)

Let us examine several limiting cases for this theory. First in the limit of very rapid anion grain boundary diffusion (good assumptions for both $A\ell_2O_3$ and MgO), we can write the following expression for D_{complex} . In this case

$$\alpha D_{\text{complex}} = \frac{\dfrac{(GS)^3 \Omega_V D_A^{\ell} K_A}{11\,\Pi}}{\dfrac{(GS) D_A^{\ell}}{\pi} + \dfrac{(GS)^2 K_A \Omega_V}{11}} + \delta_A D_A^{b} \qquad (3)$$

LIMIT I: Rapid Cation Interface Reaction

$$\frac{(GS)}{\pi} D_A^{\ell} <<< \frac{(GS)^2 K_A \Omega_V}{11} \qquad (4)$$

In this case

$$D_{\text{complex}} = \frac{1}{\alpha}\left[\frac{(GS)\ D_A^{\ell}}{\pi} + \delta_A\ D_A^{\ b}\right] \qquad (5)$$

and diffusional creep is just a competition between cation lattice and cation grain boundary diffusion.

LIMIT II : Slow Cation Interface Reaction

$$\frac{(GS)}{\Pi} D_A^{\ell} >> \frac{(GS)^2 K_A\ \Omega_V}{11} \qquad (6)$$

In this case,

$$D_{\text{complex}} = \frac{1}{\alpha}\left[\frac{(GS)^2 \Omega_V K_A}{11} + \delta_A D_A^{b}\right] \qquad (7)$$

and

$$\dot{\varepsilon} = \frac{44\Omega_V\sigma}{kT\alpha}\left[\frac{\Omega_V K_A}{(GS)11} + \frac{\delta_A D_A^{b}}{(GS)^3}\right] \tag{8}$$

LIMIT II A: Rapid Cation Grain Boundary Diffusion

In this case,

$$\dot{\varepsilon} = \frac{44\Omega_V\sigma\delta_A D_A^{b}}{kT\,(GS)^3} \tag{9}$$

and diffusional creep is controlled by cation grain boundary diffusion

LIMIT II B: Slow Cation Grain Boundary Diffusion

In this case

$$\dot{\varepsilon} = \frac{4\Omega_V^2\,K_A\,\sigma}{\alpha\,kT\,(GS)} \tag{10}$$

and diffusional creep is controlled by the cation interfacial defect reaction in the limit of small grain sizes.

LIMIT III: Slow Cation Grain Boundary Diffusion

In this case,

$$\varepsilon = \frac{44\Omega_V\sigma}{\alpha kT}\;\frac{\left[\dfrac{D_A^{\ell}}{\pi}\;\dfrac{K_A\,\Omega_V}{11}\right]}{\left[\dfrac{(GS)D_A^{\ell}}{\pi} + \dfrac{(\sigma S)^2\,K_A\,\Omega_V}{11}\right]} \tag{11}$$

LIMIT III A: Small Grain Size and/or High D_A^{ℓ}

In this case,

$$\frac{(GS)\,D_A^{\ell}}{\pi} >> \frac{(GS)^2 K_A\Omega_V}{11} \tag{12}$$

$$\dot{\varepsilon} = \frac{4\Omega_V^2\,\sigma\,K_A}{\alpha kT\,(GS)} \tag{13}$$

and diffusional creep is again interface reaction controlled. The important point to be made here is that interface controlled creep kinetics are not restricted to the small grain size limit. They can be controlling in intermediate to large grain size material providing cation lattice diffusion is very large and cation grain boundary diffusion is slow. Referring to section 2.1, the creep of Ti-doped (and Mg-Ti compositons) $A\ell_2O_3$ apparently satisfies these conditions (i.e. intermediate grain size, very high $D^{\ell}_{A\ell}$ and apparently low $\delta_{A\ell} D^{b}_{A\ell}$).

3.3 Stress Relaxation Deformation of Polycrystalline MgO and $A\ell_2O_3$, "Pure" and Doped with Iron

This year numerous four point bend stress relaxation tests were conducted at 1350-1450°C over a range of grain sizes (6-60 μm). Tests were conducted on polycrystalline MgO and $A\ell_2O_3$ at the following compositions, temperatures, and grain sizes:

Material	Dopant Cation %	Temperature (°C)	Grain Sizes (μm)
MgO	2.65% Fe	1350	11,54,57
$A\ell_2O_3$	undoped	1450	23
$A\ell_2O_3$	1.0% Fe	1450	6

3.3.1 Iron-Doped MgO. Four point bend stress relaxation tests were performed at 1350°C on polycrystalline MgO doped with 2.65 cation % Fe which was fabricated at essentially two grain sizes: 11 and 54-57 μm. The composition and grain sizes were selected to maximize the contribution to deformation by diffusional creep so that the transition stress between power law creep ($\dot{\varepsilon} \propto \sigma^N$; $N \sim 3$-5) and diffusional creep could be clearly seen. The experiments were successful in this sense in that transitions between viscous and non-viscous deformation were detected, particularly in the fine grain size material. However as reported earlier, the actual correspondence in creep rates at given stress levels between strain rates derived from the stress relaxation tests and the dead load creep values was poor. At high stresses (short times in stress relaxation), the strain rates calculated from load relaxation data were significantly higher than the dead load values. On the other hand at low stresses (long times in stress relaxation), the inferred strain rates from stress relaxation were significantly lower than the dead load creep rates. This was even true when stress relaxation and creep experiments were performed in succession on the same specimen at the test temperature.

3.3.2 $A\ell_2O_3$, "Pure" and Doped with Iron.

Stress relaxation tests at 1450°C on polycrystalline $A\ell_2O_3$, pure and doped with iron also revealed reasonably sharp transitions between slightly non-viscous creep ($\dot{\varepsilon} \alpha \sigma^N$, $N \sim 1.3$-2) at low stresses (long times) and power law creep ($\dot{\varepsilon} \alpha \sigma^N$; $N \sim 5$) at high stresses (short times). However as in the case with stress relaxation tests on MgO the correspondance with dead load creep rates was poor. Strain rates inferred from stress relaxation at high stresses (short times) exceed dead load creep rates, while those inferred from low stress (long time) measurements were significantly below dead load creep rates.

3.3.3 Analysis of stress Relaxation and Correspondance with Dead Load Creep

At long times in the stress relaxation test, one might expect stress relaxation to follow an equation of the form

$$\sigma = \sigma_0 \exp(-E_1 t/\eta_1) \qquad (14)$$

in which E_1 is the elastic modulus and η_1 is the viscous (diffusional) creep viscosity ($= \dfrac{kT(GS)^3}{44\Omega_V D_{complex}}$). Thus it should be possible to measure E_1/η_1 from stress relaxation, compute η_1 knowing E_1, and compare the experimental viscosity with that computed from dead load diffusional creep reasurements (i.e. $\dot{\varepsilon} = \sigma / \eta_1$).When this comparison was made for stress relaxation and creep of polycrystalline $A\ell_2O_3$ doped with 1% Fe, reasonably good agreement was achieved between η_1 measured from stress relaxation (at long times) and η_1 calculated from diffusional creep theory (i.e. $D_{complex} = f\,(D_{A\ell}^{\ell}$ and $\delta_{A\ell}\ D_{A\ell}^{b}$)). Even with this agreement, the correspondance between creep rates calculated from stress relaxation data and dead load creep measurements was poor.

Consequently the assumptions made in calculating strain rates from stress relaxation data were reexamined. When this is done, it is clear that in the stress relaxation analysis the following assumptions for total strain have been assumed:

$$\varepsilon_{Total} = \varepsilon_{Elastic} + \varepsilon_{Plastic} \qquad (15)$$

For stress relaxation at constant total strain

$$\dot{\varepsilon}_{Total} = 0 = \dot{\varepsilon}_{Elastic} + \dot{\varepsilon}_{Plastic} \qquad (16)$$

and

$$\dot{\varepsilon}_{Plastic} = - \dot{\varepsilon}_{Elastic} = - \frac{\dot{\sigma}}{\dot{E}} \qquad (17)$$

That is, in stress relaxation, the elastic (instantaneous recoverable) strain is converted into plastic strain. If diffusional (viscous) creep represents the plastic strain, then we have the classical Maxwell body corresponding to a spring and dashpot in series. A generalized Maxwell body including viscous and non-viscous elements should have the following creep characteristics:

1) on the application of the load, elastic deformation takes place instantly followed by steady state creep. No transient creep should be observed

2) When the load is removed after the creep test, the elastic strain will be recovered instantly. However there will be no time delayed strain recovery.

Careful examination of the dead load creep characteristics of polycrystalline MgO and $A\ell_2O_3$ reveal, however, the presence of significant transient creep at the beginning of the test and, more importantly, the occurence of significant recovery (time delayed recoverable strain) when the stress is removed. These observations are consistent with the presence of an anelastic contribution to the strain rate, i.e.

$$\varepsilon_{\mathrm{Total}} = \varepsilon_{\mathrm{Elastic}} + \varepsilon_{\mathrm{Plastic}} + \varepsilon_{\mathrm{Anelastic}} \tag{18}$$

For stress relaxation

$$\dot{\varepsilon}_{\mathrm{Total}} = 0 = \dot{\varepsilon}_{\mathrm{Elastic}} + \dot{\varepsilon}_{\mathrm{Plastic}} + \dot{\varepsilon}_{\mathrm{Anelastic}} \tag{19}$$

and

$$\dot{\varepsilon}_{\mathrm{Plastic}} = -\frac{\sigma}{E} - \dot{\varepsilon}_{\mathrm{Anelastic}} \tag{26}$$

In our stress relaxation analysis we have implicitly assumed $\dot{\varepsilon}_{\mathrm{Anelastic}}$ to be small or near zero. This can only be true in the absence of transient creep and recovery effects.

In equation (2), the first term is positive since $\dot{\sigma} < 0$. At short times in a stress relaxation test, $\dot{\varepsilon}_{\mathrm{anelastic}}$ $(=\sigma/\eta_2) > 0$ (assuming a Kelvin element, $\varepsilon = \frac{\sigma}{E_2}$ $(1-\exp -E_2t/\eta_2)$ and $\dot{\varepsilon}_{\mathrm{plastic}}$ will be overestimated, when $\dot{\varepsilon}_{\mathrm{Anelastic}}$ is assumed to be zero. At long times, $\dot{\varepsilon}_{\mathrm{Anelastic}}$ $(= \dot{\sigma}/E_2) < 0$ and $\dot{\varepsilon}_{\mathrm{Plastic}}$ will be underestimated when $\dot{\varepsilon}_{\mathrm{Anelastic}}$ is ignored. These effects are in accord with the general experimental observations.

Thus it appears that anelastic deformation effects are responsible for the lack of direct correspondance between dead load creep rates and those calculated from stress relaxation measurements assuming "Maxwellian" behavior. Experiments are currently under way to measure the anelastic contributions to the strain rate in these materials.

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3. "Mass Transport Processes in the High Temperature Creep of Polycrystalline Ceramics," Gordon Research Conference on Corrosion, July (1973).
4. "Impurity and Grain Size Effects on the Creep of Polycrystalline Magnesia and Alumina" Conference on Plastic Deformation of Ceramic Materials, Pennsylvania State University, July (1974).

5. "Ambipolar Diffusion and its Application to Diffusion Creep," University Conference on Ceramic Science, Case Western Reserve University, Cleveland, June (1974).

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V ACKNOWLEDGEMENTS

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VI PRINCIPAL INVESTIGATOR

Dr. Gordon spends time throughout the year in supervisory activities and preparing talks and papers on work related to this contract.