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

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Technical Progress Report

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TECHNICAL PROGRESS REPORT

December 18, 1976

PROJECT TITLE

Impurity Effects on the Creep of
Polycrystalline Magnesium and Aluminum
Oxides at Elevated Temperatures

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ABSTRACT

The research described in this report and the attached preprints covers work on:

1. Viscous creep of fine-grained MgO doped with iron
2. The effects of residual fluorine on diffusional creep and grain growth kinetics in polycrystalline MgO
3. The effects of transition metal impurities and grain size on the creep of polycrystalline Al_2O_3
4. The construction of creep deformation maps for polycrystalline MgO and Al_2O_3 , pure and doped with iron

Some of the significant findings include:

1. Three well defined regimes have been identified for the diffusional creep of polycrystalline MgO and Al_2O_3 , pure and doped with transition metal impurities: (1) cation grain boundary diffusion (2) cation lattice diffusion, (3) anion grain boundary diffusion
2. Creep deformation maps have been developed for ambipolar diffusional creep and applied to pure and doped MgO and Al_2O_3
3. Iron in solid solution enhances grain boundary diffusivities (cation and anion) in polycrystalline MgO in addition to magnesium lattice diffusion via the creation of cation lattice vacancies
4. Residual fluorine in polycrystalline MgO leads to enhanced magnesium lattice and grain boundary diffusivities and hence to increased diffusional creep and grain growth rates
5. Viscous Coble (diffusional) creep, which is rate-limited by oxygen grain boundary diffusion, has been identified in reduced iron-doped (2 cation %) and double-doped (1/4% Mn and 1/4% Ti) polycrystalline Al_2O_3

I. INTRODUCTION

The research activities under Contract E(11-1)-1591 during the period December 19, 1975 to December 18, 1976 are described in this report and in the attached preprints. (1-6)

Major effort was devoted to the following:

1. Additional data on grain size effects on the creep of polycrystalline MgO have been obtained for material with 0.05 cation % iron. These data in combination with previous measurements made during the course of the contract were used to study the effect of trivalent iron in solid solution on lattice and grain boundary diffusion processes in polycrystalline MgO.
2. Dead-load creep and grain growth experiments were conducted on polycrystalline MgO, fabricated with and without a LiF additive. (4) Residual fluorine was found to enhance the magnesium lattice and grain boundary diffusivities leading to enhanced creep and grain growth kinetics.
3. Further analysis was conducted on the development of creep deformation maps in polycrystalline MgO and Al_2O_3 , pure and doped with iron in solid solution. (1,3,5) These maps illustrate the important deformation mechanisms which operate in these systems and the range of variables (stress, temperature, and grain size) over which they are dominant.
4. Additional studies (6) have been conducted on the creep of polycrystalline Al_2O_3 doped simultaneously with 1/4 cation % Mn and 1/4 cation % Ti over a range of stresses (4-40 MN/m²), grain sizes (18-40 μm) and temperatures (1100-1300°C).

Six papers have been prepared for publication this year. Two have been approved for publication (1,2) and four are under review. (3-6)

1. "High Temperature Creep of Polycrystalline MgO and Al_2O_3 ", Paul A. Lessing, James D. Hodge, and Ronald S. Gordon, Proceedings of Second International Conference on Mechanical Behavior of Materials, August 1976, Boston (in press).
2. "On Activation Energies in the Diffusional Creep of Polycrystalline Ceramics", J. D. Hodge, P. A. Lessing and R. S. Gordon, J. Amer. Ceram. Soc., (in press).

3. "Creep Mapping in a Polycrystalline Ceramic: Application to Magnesium Oxide and Magnesio-wüstite", J. D. Hodge, P. A. Lessing, and R. S. Gordon, J. Mater. Sci., (submitted for publication).
4. "Grain Growth and Creep in Polycrystalline Magnesium Oxide Fabricated with and without a LiF Additive", J. D. Hodge and R. S. Gordon, Ceramurgia International (submitted for publication).
5. "Creep of Polycrystalline Alumina, Pure and Doped with Transition Metal Impurities", P. A. Lessing and R. S. Gordon, J. Mater. Sci., (submitted for publication).
6. "Creep of Polycrystalline Alumina Doped with Mixed Transition Metal (Mn-Ti) Impurities", P. A. Lessing, Y. Ikuma, and R. S. Gordon, J. Amer. Ceram. Soc., (submitted for publication).

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.

II. REVIEW OF RESEARCH ACTIVITIES

In this section the research activities which have been conducted under the contract for the current period will be described exclusive of that which is presented in the attached preprints.

2.1 Viscous Creep of MgO-FeO-Fe₂O₃ Solid Solutions

This year additional data on grain size effects in the creep of polycrystalline MgO have been obtained at one dopant level (0.05 cation % Fe) in creep tests at 1350°C. These data in combination with previous measurements made during the course of the contract permit some conclusions to be drawn concerning the effects of doping on the important transport parameters in polycrystalline MgO.

Using a technique developed by Terwilliger, et. al.,⁽⁷⁾ additional experiments were conducted during the current period at the 0.05 cation % dopant level at 1350°C. In this method, previously obtained grain growth data are used to assign grain sizes to the creep specimen at various times during the deformation test. By plotting the assigned grain sizes against the corresponding creep rates, the grain size dependence can be determined from the decay in strain rate due to the occurrence of simultaneous grain growth.

In Figure 1 grain growth data at 1350°C are shown for polycrystalline MgO doped with 0.05% Fe. Included in the plot are grain sizes of specimens which have been creep tested for various periods of time at 1350°C. These data indicate that grain growth in the control specimens is comparable to that observed in specimens which were creep

Figure 1. Grain Growth in Polycrystalline MgO Doped with 0.05 Cation % Iron

tested at the same temperature. All grain sizes were determined on polished surfaces using the linear intercept method of counting 400-500 grains. Grain sizes in Figure 1 are reported as linear intercepts.

The grain growth data in Figure 1 approximately follow cubic kinetics (i.e. $D^3 - D_0^3 = kt$ with $k = 690 \mu\text{m}^3/\text{h}$), a result in good agreement with previously reported data by Gordon, et. al.⁽⁸⁾ at 1400°C and 1500°C at the same dopant level. The grain growth rates are also consistent with the earlier work. All specimens for the grain growth and creep studies were over 99% dense.

Using the grain growth data in Figure 1 in combination with dead-load creep data at the same temperature, the effect of grain size on the viscous creep of polycrystalline MgO doped with 0.05 cation % Fe was determined at 1350°C in the regime of low stress ($< 10 \text{ MN/m}^2$). These results reveal grain size exponents m (i.e. $\dot{\epsilon} \propto (\text{GS})^{-m}$) ranging between 2.4 and 2.8 which are indicative of a viscous creep process in which transport by both magnesium grain boundary ($\delta_{\text{Mg}} D_{\text{Mg}}^b$) and magnesium lattice diffusion (D_{Mg}^l) is comparable. Terwilliger, et. al.⁽⁷⁾ reported a grain size exponent of ~ 3 in creep tests at 1300°C in material at the same dopant concentration. Values of D_{Mg}^l and $\delta_{\text{Mg}} D_{\text{Mg}}^b$ equal to $(7.7 \pm 4.4) \times 10^{-13} \text{ cm}^2/\text{sec}$ and $(6.0 \pm 2.1) \times 10^{-16} \text{ cm}^3/\text{sec}$, respectively, were calculated by a procedure described elsewhere⁽⁴⁾.

Enough diffusion values at various dopant levels have now been accumulated that we can begin to look at the effects of impurities on the relevant mass transport parameters for these systems. Values of D_{Mg}^l , $\delta_{\text{O}} D_{\text{O}}^b$, and $\delta_{\text{Mg}} D_{\text{Mg}}^b$ at 1350°C are tabulated in Table I. Included also

TABLE I

Mass Transport Parameters in Polycrystalline
MgO and MgO-FeO-Fe₂O₃ Solid Solutions

1350°C

Dopant Level (cation %)	m	D_{Mg}^l (cm ² /sec)	$\delta_{O_3}^{D_O^b}$ (cm ³ /sec)	$\delta_{Mg_3}^{D_{Mg}^b}$ (cm ³ /sec)
Undoped	2.0*	$3.6 \times 10^{-13}^{**}$	---	$> 10^{-17}^{***}$
0.05	2.6	7.7×10^{-13}	---	6.0×10^{-16}
0.53	1.94	2.3×10^{-12}	---	---
2.65	2.38	9.0×10^{-12}	1.6×10^{-14}	---
5.3	2.38	15.1×10^{-12}	3.2×10^{-14}	---
Magnesium Tracer****		1.15×10^{-12}	---	---

* 2.6 when residual fluorine is present

** 1.3×10^{-12} cm²/sec when residual fluorine is present⁽⁴⁾

*** 2.0×10^{-15} cm³/sec when residual fluorine is present⁽⁴⁾

For "pure" MgO the magnesium boundary diffusivity must be
in excess of 10^{-17} cm³/sec since the kinetics are controlled
by magnesium lattice diffusion

**** Extrapolated from data at higher temperatures

are the grain size exponents m ($\dot{\epsilon} \propto (GS)^{-m}$) observed in creep for the various compositions. The effect of dopant concentration on D_{Mg}^l is shown in Figure 2. It is clear that the magnesium lattice diffusion coefficient is a strong function of the iron concentration.

Using standard chemical procedures^(9,10) the concentration of divalent (Fe^{2+}) and trivalent (Fe^{3+}) iron were determined at three dopant levels (0.53, 2.65, and 5.30 cation %) at 1350°C. These results are tabulated in Table II. At high iron concentration most of the iron is trivalent. In dilute solutions (≤ 0.5 cation %) more of the iron is in the divalent state. Assuming the concentration of cation vacancies to be one-half the concentration of trivalent iron, a plot was made (Figure 3) of the magnesium lattice diffusion coefficient versus the concentration of lattice vacancies. While the vacancy concentration increases between the 0.05 and 5.3 dopant concentrations by a factor of approximately 200, the magnesium lattice diffusivity inferred from creep measurements increases by only a factor of twenty (20). This result implies that many of the vacancies introduced by trivalent iron are not able to move freely in the lattice to contribute to diffusional creep. Defect complexing at the relatively high defect concentrations (1-2%) is probably responsible for the observation.⁽¹⁰⁾

Last year⁽¹²⁾ a contradiction existed between the mass transport parameters for undoped MgO and the 0.05% dopant level. D_{Mg}^l for the undoped material was a factor of 3 larger than the 0.05% dopant level. This discrepancy was found to be due to the presence of residual fluorine present in the lattice.⁽⁴⁾ When MgO was fabricated without the LiF additive smaller creep and grain growth rates were measured (Table I).

Figure 2. Effect of Iron in Solid Solution on Magnesium Lattice Diffusion in Polycrystalline MgO

Figure 3. Effect of Cation Vacancy Concentration on Magnesium Lattice Diffusion in Iron-Doped Polycrystalline MgO

TABLE II

Correlation Between Cation Vacancy Concentration and
Cation Lattice Diffusivity at 1350°C

Dopant Level (cation %)	$\frac{\text{Fe}^{+3}}{\text{Fe}^{+2}}$	Cation Vacancy* %	D_{Mg}^{c} (cm ² /sec)
0	---	---	0.36×10^{-12}
0.05	$\sim 0.7^{**}$	$\sim .01^{**}$	0.77×10^{-12}
0.53	0.76	0.11	2.3×10^{-12}
2.65	5.91	1.13	9.0×10^{-12}
5.30	8.77	2.38	15.1×10^{-12}

* % = $\left(\frac{\text{number vacancies}}{\text{number cation sites}} \right) 100$

** Estimated from data of Brynestad and Flood⁽¹¹⁾

Finally the data in Table I also indicate that iron in solid solution while enhancing magnesium lattice diffusion also enhances to some degree the magnesium and oxygen grain boundary diffusivities. Magnesium grain boundary diffusion in polycrystalline MgO apparently is also enhanced by residual fluorine which remains after hot-pressing with a LiF additive.⁽⁴⁾

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2. "The Effects of Impurities and Atmosphere on the Creep of Polycrystalline MgO and Al₂O₃," M.I.T., Lehigh University, Cornell University (1972).
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).
6. "Impurity and Grain Size Effects on the High Temperature Creep of Polycrystalline Al_2O_3 and MgO " Special Meeting Japanese Ceramic Society, Tokyo, Japan, June (1975).

V. ACKNOWLEDGEMENTS

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VI. PRINCIPAL INVESTIGATOR

In addition to full time research on the contract for a portion of the summer, Dr. Gordon spends time throughout the year in supervisory activities and preparing talks and papers on work related to this contract.