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

Ronald S. Gordon
Principal Investigator

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Technical Progress Report
December 19, 1972 to December 18, 1973

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Division of Materials Science and Engineering
University of Utah
Salt Lake City, Utah 84112

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PROJECT
PERSONNEL Principal Investigator
 Dr. Ronald S. Gordon
 Associate Professor

 Graduate Research Assistant
 Mr. Paul A. Lessing

 Undergraduate Assistant
 Mr. James D. Hodge

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VI. PRINCIPAL INVESTIGATOR

ABSTRACT

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

- 1) Creep rate - grain size effects for polycrystalline, iron-doped MgO (0.53 and 2.65 cation % Fe) at low stresses and grain sizes $\leq 65 \mu\text{m}$.
- 2) A theoretical analysis of mass transport in the creep of iron-doped, polycrystalline MgO
- 3) Non-viscous creep behavior of pure and iron-doped, polycrystalline MgO over a range of grain sizes (91-321 μm)
- 4) Creep rate - grain size effects for pure and iron-doped, polycrystalline Al_2O_3 over a range of grain sizes (5-71 μm) and stresses (100-400 Kg/cm^2)

Some of the significant findings include the following:

- 1) Nabarro-Herring creep has been conclusively identified as the mechanism for the creep in oxygen of polycrystalline MgO doped with 0.53 cation % iron or less. At higher dopant levels both Nabarro-Herring creep and Coble creep contribute to creep deformation in air
- 2) An analytical procedure has been developed to estimate from the creep data of polycrystalline, iron-doped MgO diffusion constants for magnesium and oxygen grain boundary diffusion and magnesium lattice diffusion
- 3) The creep in air at 1500°C of coarse grained iron-doped, (0.53 cation %) polycrystalline MgO consists of
 - a) viscous, Nabarro-Herring creep and
 - b) a non-viscous dislocation process which is independent of grain size

4) Nabarro-Herring creep has been identified in pure polycrystalline Al_2O_3 . In iron-doped, (1 cation %) polycrystalline Al_2O_3 both Nabarro-Herring and Coble diffusional creep processes contribute to creep deformation.

I. INTRODUCTION

The research activities under Contract AT(11-1)-1591 during the period December 19, 1972 to December 18, 1973 are described in this report and in the attached preprints. (1, 2)

Major effort was devoted to the following:

1. Additional creep rate - grain size data were collected to establish the grain size dependencies for the creep in oxygen of iron-doped, polycrystalline MgO at the 0.53 and 2.65 cation % dopant levels. Nabarro-Herring creep was identified ($\dot{\epsilon} \propto (GS)^{-2}$) at the 0.53% composition. (1) However at the higher composition of 2.65% a mixture of Nabarro-Herring and Coble creep behavior was observed (i.e. $\dot{\epsilon} \propto (GS)^{-2.38}$). (1)
2. A theoretical analysis of mass transport in the creep of iron-doped, polycrystalline MgO was conducted. (2) Using actual grain size data a method was devised to evaluate quantitatively the relative amounts of magnesium lattice and oxygen grain boundary diffusion in the creep of MgO doped at the 2.65% level.
3. Studies on the creep of polycrystalline MgO, pure and doped with iron (0.53 cation %), were extended to larger grain sizes (90-321 μm). At 1400-1500°C significant non-viscous creep behavior was observed ($\dot{\epsilon} \propto \sigma^N$; $1.7 < N < 3.6$). The transition between viscous and non-viscous creep behavior was determined.
4. High purity Al_2O_3 , which was synthesized by an organometallic procedure, was used as the base material in creep studies on polycrystalline Al_2O_3 , pure and doped with iron, over a range of grain sizes (5-71 μm). Nabarro-Herring creep was identified in pure Al_2O_3 while the creep properties

of iron-doped Al_2O_3 were intermediate between Nabarro-Herring and Coble creep (i.e. $\dot{\epsilon} \propto (\text{GS})^{-2.38}$). By using the organometallic Al_2O_3 complications of discontinuous grain growth during annealing have been successfully avoided.

Three papers have been published (3, 4, 5) this year and are attached to this report.

1) "Mass Transport in the Diffusional Creep of Ionic Solids," Ronald S. Gordon, J. Am. Ceram. Soc., 56 (3) 147-152 (1973).

2) "The Effect of Oxygen Partial Pressure on the Creep of Polycrystalline Al_2O_3 Doped with Cr, Fe or Ti," G. W. Hollenberg and R. S. Gordon, J. Am. Ceram Soc., 56 (3) 140-147 (1973).

3) "Origin of Anomalous High Activation Energies in the Sintering and Creep of Impure Refractory Oxides," G. W. Hollenberg and Ronald S. Gordon, J. Am. Ceram Soc., 56 (2) 109-110 (1973).

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

Finally the principal investigator attended the Gordon Research Conference on Corrosion (July, 1973) and presented an invited talk on "Mass Transport Processes in the Diffusional Creep of Polycrystalline Ceramics."

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 briefly. This discussion will focus only on research not covered in the preprints attached to this report.^(1, 2) In the latter work related to grain size effects in iron-doped polycrystalline MgO and a mass transport analysis in this system is presented.

2.1 Non-Viscous Creep of Large Grain Size MgO, Pure and Doped With Iron

Preliminary studies have been conducted on both pure and iron-doped (0.53 cation %), large grained, polycrystalline MgO. Grain sizes were varied between 91 and 321 μm . Initial experiments were first attempted at approximately 1350°C; however, creep rates continually decreasing with time were observed. Since no grain growth occurred in these specimens, this time decay of the strain rate was attributed to a "dislocation hardening process." Subsequent tests, conducted at higher temperatures 1400-1500°C, resulted in steady state creep behavior. Recovery processes resulting in steady state creep behavior should become predominant as the temperature is increased.

At temperatures between 1400 and 1500°C several stress change experiments were conducted. In general, non-viscous (i.e. $\dot{\epsilon} \propto \sigma^N$; $1.7 \leq N \leq 4.1$) creep behavior was observed. Data from typical stress change experiments are given in Figure 1 for creep tests at 1425 and 1500°C. Data at 1500°C include tests conducted in both oxidizing (air) and reducing atmospheres (CO). It is interesting to note that in the non-viscous regime the creep rate is insensitive to changes in the oxygen partial pressure. This behavior is in sharp contrast to viscous creep in this system which is predominant at

Figure 1. Typical Non-viscous Creep in Iron-Doped, Polycrystalline MgO

smaller grain sizes. In the latter creep rates creep rates decrease as the oxygen partial pressure is lowered.⁽¹⁾

A summary of all the stress-strain rate experiments is given in Table 1. Inspection of these data reveals that the apparent stress exponents (N) continually decrease with decreasing grain size. A particularly interesting result of this trend is shown in Figure 2 where the effect of grain size on the creep rate is shown at two stress levels. At the lower stress (100 Kg/cm^2) a definite reciprocal grain size dependence is noted (i.e. $\dot{\epsilon} \propto (\text{GS})^{-m}$; $m \approx 2$). As the grain size increases ($> 100 \mu\text{m}$) its effect in decreasing the creep rate appears to diminish rapidly. At the higher stress level (300 Kg/cm^2) a sharp transition exists ($\sim 40 \mu\text{m}$) between grain size dependent and independent regimes. The two smallest grain size datum points (14 and $30 \mu\text{m}$) in Figure 2 (filled-in datum points) correspond to viscous creep data extrapolated from the 1300 - 1400°C temperature range. In this grain size regime viscous, Nabarro-Herring creep with an activation energy of 117 Kcal/mole has been well documented this past year.⁽¹⁾

The data in Figure 2 clearly demonstrate that viscous creep gives way to non-viscous creep at lower grain sizes as the stress level is increased. Secondly the strong dependence between the grain size and the stress exponent (Table 1) indicates significant contributions of viscous creep as the grain size is decreased below $100 \mu\text{m}$.

To check if the reason for the strong dependence of the grain size on the stress exponent is due, in fact, to a strong contribution of viscous creep let us assume that the two processes (i.e. viscous and non-viscous) are additive, i.e.

TABLE 1

Strain Rate-Stress Data
Pure and Iron Doped, Polycrystalline MgO

<u>Specimen</u>	<u>Temperature (°C)</u>	<u>Stress Range (kg/cm²)</u>	<u>Grain Size (μm)</u>	<u>Apparent Stress Stress Exponent (N)</u>	<u>Actual Non-Viscous Stress Exponent (N')</u>
<u>0.53 cation %</u>					
A	1500	90-350	321	3.6	3.9
B	1500	100-300	172	3.6	4.8
C	1500	120-270	119	3.1	4.5
D	1425	30-236	120	2.9	4.9
E	1500	100-300	91	1.7	3.0
<u>Pure MgO</u>					
A	1500	30-260	145	2.6	-

Figure 2. Grain Size Effect on Non-viscous Creep of Iron-doped, Polycrystalline MgO

$$\dot{\epsilon} = K\sigma^N = K\sigma^N + K''\sigma^{N'} \quad (1)$$

K , K' , and K'' are constants independent of stress. N is the apparent stress exponent and N' is the actual non-viscous stress exponent. To determine the non-viscous stress exponent equation (1) must be rearranged into the following form

$$\dot{\epsilon} - K'\sigma = K''\sigma^{N'} \quad (2)$$

Thus a plot of $\log [\dot{\epsilon} - K'\sigma]$ versus σ should result in a straight line with slope equal to N' . Evaluation of the term $[\dot{\epsilon} - K'\sigma]$ requires a knowledge of the viscous component $[K'\sigma]$.

Viscous Nabarro-Herring creep has been well documented at temperatures between 1300 and 1400°C.⁽¹⁾ Using a diffusion coefficient of 2.54×10^{-12} cm²/sec at 1350°C,⁽¹⁾ a linear stress dependence, and a reciprocal square grain size dependence, values of $K'\sigma$ can be evaluated at any grain size and stress at 1425 and 1500°C. Using this procedure values of $K'\sigma$ have been subtracted from all non-viscous creep data to determine estimates of the true non-viscous creep rate $[\dot{\epsilon} - K'\sigma]$. A typical log-log plot of $[\dot{\epsilon} - K'\sigma]$ versus σ is shown in Figure 3 for a creep test at 1500°C on a 119 μ m specimen. Included on the plot for comparison are the uncorrected creep data. It is clear that the viscous contribution is significant particularly at the low stress levels (< 200 kg/cm²). The corrected plot of only non-viscous creep data results in a stress exponent which is significantly higher (4.5 versus 3.1). In Table 1 a comparison is given between values of N and N' . The values of N' are significantly higher than the N values particularly as the grain size decreases. Most of the non-viscous

Figure 3. Comparison Between Apparent and Actual Non-viscous Creep Behavior

stress exponents (N') lie between 3.9 and 4.9. They are also independent of grain size as might be expected.

Thus it is clear that the creep of coarse grained, polycrystalline, iron-doped MgO consists of two processes.

1) A viscous process which predominates at small stresses ($\leq 200 \text{ kg/cm}^2$) and grain sizes ($\lesssim 100 \text{ }\mu\text{m}$).

2) A grain size independent non-viscous process with a stress exponent around 4.2 ± 0.4 .

One temperature change experiment was conducted in the non-viscous regime between 1350 and 1500°C on a specimen with a grain size of 91 μm . An activation energy of $\sim 102 \text{ Kcal/mole}$ resulted, somewhat lower than the 117 Kcal/mole reported for the viscous regime.⁽¹⁾ However, since the creep mechanism is mixed in that both viscous and non-viscous modes are contributing, additional experiments of this type need to be conducted over a range of grain sizes.

For reference some experiments were conducted on pure, polycrystalline MgO at 1500°C. In Figure 4 strain rate-stress data for a specimen with a grain size of 145 μm reveal a stress exponent of 2.6 somewhat lower than that observed in iron doped material at a comparable grain size. The relatively low stress exponent may be indicative of some viscous contribution to the overall creep rate. Included on Figure 4 for comparison are compressive creep data ($350\text{--}1140 \text{ kg/cm}^2$) of Langdon and Pask⁽⁶⁾ which have been extrapolated from 1200°C using their measured activation energy of $51 \pm 5 \text{ Kcal/mole}$. Considering the magnitude of the extrapolation reasonable agreement exists between the two sets of measurements.

Figure 4. Non-viscous Creep of Pure Polycrystalline MgO

Finally creep data at 300 and 100 kg/cm² are plotted on Figure 2 as a comparison with the doped material. These preliminary data indicate that the creep rates in the non-viscous regime are not very sensitive to small amounts of iron in solid solution.

In summary the following tentative conclusions can be drawn from the foregoing:

- 1) For grain sizes over $\sim 90 \mu\text{m}$ stress exponents between 1.7 and 3.6 indicated significant non-viscous contributions to the creep rate. In iron doped material viscous ($\dot{\epsilon} \propto \sigma$) and non-viscous ($\dot{\epsilon} \propto \sigma^{4.2}$) processes were occurring simultaneously. The non-viscous component is consistent with dislocation mechanisms such as those proposed by Nabarro and Weertman. (7, 8)
- 2) In the non-viscous regime the creep rate of iron-doped, polycrystalline MgO is independent of the grain size at high stresses ($\sim 300 \text{ Kg/cm}^2$). However at lower stresses ($\sim 100 \text{ Kg/cm}^2$) an inverse relation between the creep rate and grain size was observed which is consistent with a significant contribution of viscous creep, particularly as the grain size approaches $100 \mu\text{m}$.
- 3) In the non-viscous regime the creep rate of polycrystalline, iron-doped MgO was insensitive to changes in oxygen partial pressure and maybe even to the presence of iron in solid solution. Additional experiments over the entire range of grain sizes are required since, as the grain size decreases, viscous creep should become important and hence P_{O_2} and impurity effects should be observed.

2.2 Effect of Grain Size on Creep of Polycrystalline Al_2O_3 , Pure and Doped With Iron

High purity (99.999%) alumina powder, which was synthesized by the organometallic method described in last year's report,⁽⁹⁾ was used as the starting material to hot press dense (> 98%), pure and doped (Cr and Fe), polycrystalline specimens. At the present time results have been obtained for two Fe dopant levels (0.2 and 1.0 cation %) and pure Al_2O_3 . Studies on the chromium doped material are underway.

As reported previously^(4, 9) slightly non-viscous stress exponents (1.05-1.43) were consistently found in stress change creep experiments up to 400 kg/cm^2 as can be seen from the data in Table 2. No systematic variation in stress exponents were observed with either changes in grain size or dopant level. The large grain size specimens were obtained by high temperature annealing (> 1700°C). The microstructures were uniform and no evidence for discontinuous grain growth was observed.

Creep rate-grain size data at 1450°C and at a low stress of 50 kg/cm^2 are plotted in Figure 5 over an extensive range of grain sizes (6 - $71 \mu\text{m}$). In the case of pure Al_2O_3 the data give an excellent fit to the Nabarro-Herring Creep equation (i.e. $\dot{\epsilon} \propto (\text{GS})^m$; $m = 1.99$) for grain sizes between 6 and $48 \mu\text{m}$. These data are perhaps the best to date in establishing conclusively the presence of Nabarro-Herring creep in high purity, polycrystalline Al_2O_3 .

For specimens (20 - $70 \mu\text{m}$) doped with 1 cation % iron the grain size effect on the creep rate appears to be more pronounced than in the case of undoped material ($m = 2.38$ as compared to 1.99). Although more data are required, the higher grain size exponent suggests a mixed diffusional

TABLE 2
Stress Exponents at 1450°C in Polycrystalline
Al₂O₃, Pure and Doped with Iron

<u>Specimen</u>	<u>Cation % Fe</u>	<u>Grain Size (microns)</u>	<u>Stress Exponent N</u>
A	pure	6	1.37
B	pure	11	1.43
C	pure	12	1.39
D	pure	21	1.05
E	pure	48	1.28
F	0.2	18	1.13
G	0.2	25	1.15
H	1.0	17	1.23
I	1.0	25	1.18
J	1.0	71	1.33
K	1.0 (P _{O₂} ~ 10 ⁻⁸ atm)	25	1.29

Figure 5. Effect of Grain Size on Creep of Pure and Iron-doped, Polycrystalline Al_2O_3

creep mechanism in which cation lattice and oxygen grain boundary diffusion processes may be comparable in magnitude. Similar effects have been reported in iron-doped, polycrystalline MgO .^(1, 2) The grain size effects for the 0.2 cation % dopant level, although limited data are available at present, are consistent with the Nabarro-Herring relation (i.e. $n = 2$). The grain size exponents shown in Figure 5 are approximately the same up to stresses $\sim 400 \text{ kg/cm}^2$ since no systematic variation in the stress exponents with grain size exists (refer to Table 2).

Creep activation energies, determined from temperature change experiments, were 130-134 Kcal/mole for the pure and 0.2 % Fe specimens. An activation energy of 117 Kcal/mole was found for the 71 μm specimen at the 1 cation % level; whereas Hollenberg and Gordon⁽⁴⁾ reported a value of 148 Kcal/mole for a much smaller grain size (10 μm). It is clear that a systematic study of the effect of grain size on the activation energy will be required particularly if both lattice and boundary diffusion processes are contributing to the creep process.

Finally lattice diffusion coefficients have been calculated from the creep data at 1450°C using the Nabarro-Herring Creep equation (refer to Table 3). Reasonable agreement exists between the value computed from the creep of pure Al_2O_3 and the aluminum process value indicating cation diffusion controlled kinetics. Diffusion coefficients computed from the creep of iron-doped specimens are, as expected, higher. It should be noted that if both lattice and grain boundary diffusion contribute to the creep of iron-doped Al_2O_3 , the diffusion coefficients in Table 3 for the lattice contribution are underestimated.⁽²⁾

TABLE 3
Cation Lattice Diffusion Coefficients at 1450°C

<u>Description</u>	<u>D (cm²/sec)</u>	<u>Reference</u>
Pure Al ₂ O ₃ (Alkaxyd)	6.92 x 10 ⁻¹³	this study
0.2 cation % Fe	1.69 x 10 ⁻¹²	this study
1.0 cation % Fe	4.06 x 10 ⁻¹²	this study
1.0 cation % Fe	7.5 x 10 ⁻¹³	(4)
Aluminum Tracer	1.02 x 10 ⁻¹³	(10)

In summary the following tentative conclusions can be drawn from the foregoing:

- 1) Slightly non-viscous creep (i.e. $\dot{\epsilon} \propto \sigma^N$; $1.05 \leq N \leq 1.4$) is characteristic of polycrystalline Al_2O_3 , pure and doped, over a range of grain sizes (up to 70 μm), temperatures (up to 1500°C) and stresses (up to 550 kg/cm^2).
- 2) Creep rate-grain size data for pure polycrystalline Al_2O_3 (6-48 μm) give excellent agreement with Nabarro-Herring creep (i.e. $\dot{\epsilon} \propto (GS)^{-2}$) and kinetics controlled by cation lattice diffusion at 1450°C.
- 3) Preliminary creep rate-grain size data in air for iron-doped (1 cation %) polycrystalline Al_2O_3 (20-70 μm) are consistent with a mixed diffusional creep mechanism (i.e. significant contributions of both aluminum lattice and oxygen grain boundary diffusion).

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1. "The Effects of Impurities and Atmosphere on the Creep of Polycrystalline MgO," Materials Science Division, Argonne National Laboratory, April 1971.
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. "Extrinsic Diffusional Creep Phenomena in Polycrystalline Oxide Ceramics," Conference on Plastic Deformation of Ceramic Materials, Pennsylvania State University, July (1974).
5. "Relationship of Transport to Processes in Ceramics," Ninth Annual University Conference on Ceramic Science, Case Western Reserve University, Cleveland, June (1974).

V. ACKNOWLEDGMENTS

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VI. PRINCIPAL INVESTIGATOR

The principal investigator, Dr. Ronald S. Gordon, returned to the University of Utah on May 1, 1973 from his leave of absence at the General Electric Co. 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.