MASTER

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

Technical Progress Report
for December 19, 1976 --December 18, 1977

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Ronald S. Gordon
Principal Investigator

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

December 1977

Prepared For
DEPARTMENT OF ENERGY
UNDER CONTRACT NO. EY-76-S-02-1591.*002

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED *EB*

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

TECHNICAL PROGRESS REPORT

December 18, 1977

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

Principal Investigator

Dr. Ronald S. Gordon
Professor

Research Assistant Professor

Dr. Dinesh K. Shetty

Graduate Research Assistants

Mr. Yasuro Ikuma

Mr. J.P. Singh

TABLE OF CONTENTS

	page
Abstract	1
I. Introduction	2
II. Review of Research Activities	3
2.1 Creep of Polycrystalline Alumina Doped with Mixed Transition Metal (Mn-Ti) Impurities	3
2.1.1. Experimental Procedures	5
2.1.2. Stress Dependence	6
2.1.3. Grain Size Effects	8
2.1.4. Apparent Creep Activation Energies	10
2.1.5. Effects of Oxygen Partial Pressure	13
2.1.6. Transient Effects	13
2.2. Stress Relaxation Creep Studies	19
2.2.1. Description of an Improved Stress Relaxation Test System	22
2.2.2. Analyses of Stresses and Strains in Relaxation Tests in Four Point Bend Specimens	26
2.2.3. Preliminary Stress Relaxation Deformation Studies on Polycrystalline, Iron-Doped MgO	31
III. References	39
IV. Bibliography of Papers, Theses and Invited Presentations	41
V. Acknowledgements	45
VI. Principal Investigator	45

ABSTRACT

The research described in this report covers work on:

1. The effect of mixed transition metal (Mn-Ti) impurities on the creep of polycrystalline alumina
2. Stress relaxation deformation which includes work on (1) the design and construction of an improved experimental apparatus (2) the analysis of stresses and strains in stress relaxation tests and (3) preliminary stress relaxation tests at 1350-1450°C on iron-doped (0.53 cation %), polycrystalline MgO.

Some of the significant findings include:

1. Small amounts (0.2-0.4 cation %) of mixed transition metal impurities (Mn-Ti) enhance the creep rate of polycrystalline alumina by factors between two and four orders of magnitude.
2. The stress, grain size, and temperature dependencies indicate that diffusional creep is important in polycrystalline alumina doped simultaneously with Mn and Ti.
3. Transient creep, which can last for periods in excess of 250 hours at temperatures $< 1250^{\circ}\text{C}$ in polycrystalline alumina doped simultaneously with Mn and Ti, is probably due to a non-equilibrium distribution of lattice defects which leads to a time-variant diffusion coefficient.
4. Stress relaxation tests on polycrystalline MgO doped with iron can be used to clearly distinguish the transition between viscous and power law creep as the stress is increased.
5. Stress relaxation tests over a range of impurity concentrations, grain sizes, and temperatures will give important input data for the construction of creep deformation maps, particularly in transition regions between competing deformation mechanisms.

I. INTRODUCTION

The research activities under contract E(11-1)-1591 during the period December 19, 1976 to December 18, 1977 are described in this report.

Major effort was devoted to

- (1) Dead load creep tests on polycrystalline alumina doped simultaneously with Mn and Ti
- (2) The design and construction of an improved apparatus for conducting stress relaxation deformation studies at elevated temperatures
- (3) The analysis of stresses and strains in stress relaxation tests under conditions of four point bending
- (4) Preliminary stress relaxation tests on polycrystalline MgO doped with iron.

Four papers were published or accepted for publication this year. Reprints are attached to this report.

1. "Activation Energies in the Diffusional Creep of Polycrystalline Ceramics", J. D. Hodge, P.A. Lessing and R.S. Gordon, J. Amer. Ceram. Soc., 60(7) 318 (1977).
2. "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., 12 1598 (1977).
3. "Grain Growth and Creep in Polycrystalline Magnesium Oxide Fabricated with and without a LiF Additive", J. D. Hodge and R. S. Gordon, Ceramurgia International (in press)
4. "Creep of Polycrystalline Alumina, Pure and Doped with Transition Metal Impurities", P. A. Lessing and R. S. Gordon, J. Mater. Sci., 12 2291(1977).

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. This year research was conducted on (1) the effect of mixed dopants (e.g. Mn-Ti) on the creep of polycrystalline Al_2O_3 at elevated temperatures and (2) stress-relaxation deformation studies at elevated temperatures on polycrystalline MgO doped with iron.

2.1 Creep of Polycrystalline Alumina Doped With Mixed Transition Metal (Mn-Ti) Impurities

It has been established that both a quadrivalent (Ti^{4+}) and a divalent (Fe^{2+}) impurity cation in solid solution enhance the steady state creep rate of polycrystalline alumina significantly over that of undoped or chromium doped material.^(1,2) Similar effects have been reported on the effects of quadrivalent (Ti^{4+}) and divalent (Mn^{2+} , Fe^{2+}) cations in enhancing sintering rates.⁽³⁻⁶⁾

If both impurities (i.e. Ti^{4+} , Fe^{2+}) go into the lattice substitutionally for aluminum, then effectively negative (Fe'_{Al}) and positive (Ti^0_{Al}) impurity centers would be formed. One might expect that, if both of these impurities were present in solution simultaneously, a hindering effect would occur in the overall influence of these impurities on sintering and creep rates. That is, if the impurity defect centers were strongly associated, the corresponding charge compensating lattice defects (aluminum interstitials and vacancies) would annihilate each other leading to no net enhancement of lattice diffusion. Consequently, sintering and diffusional creep kinetics might be slower in

systems with mixed dopants than in those with single dopants of either Fe (Mn) or Ti.

However, some sintering data exist with double dopants that lead to an opposite conclusion. First, some data have been published by Rao and Cutler⁽⁶⁾ on the effects of Fe^{2+} and Ti^{4+} on the sintering of alumina. Over a range of temperatures 0.2% (cation) Ti enhanced the sintering rate by 3-4 orders of magnitude. When both dopants were present (i.e. 0.2% Ti and 3.2% Fe) the sintering rate was an order of magnitude higher than at an iron concentration of 3.2% and about a factor four lower than at titanium concentration of 0.2%. These results suggested that only a partial compensation existed between $\text{Ti}_{\text{Al}}^{\text{O}}$ and $\text{Fe}_{\text{Al}}^{\text{I}}$ and that defects associated with these impurity centers were annihilated only to a small extent. Furthermore, combinations of MnO and TiO_2 and CuO and TiO_2 have been shown to be more effective in lowering the sintering temperature of alumina than either oxide alone.⁽⁷⁾

Preliminary creep studies reported previously⁽⁸⁾ also confirm the effect of double dopants. Small amounts of Mn and Ti or Cu and Ti enhanced the creep rate of polycrystalline alumina significantly.

This past year extensive studies on the effects of stress, grain size, dopant concentration, temperature, and oxygen partial pressure have been conducted on the creep of polycrystalline alumina doped with both Mn and Ti at total impurity levels below 0.4 cation %. These studies along with transient creep effects, which are important in this system at low temperatures (1075-1300°C), will be discussed in this report.

2.1.1. Experimental Procedures

A high purity (99.999%) powder of α -alumina was prepared via an organometallic synthesis which is described elsewhere.^(2,8) The dopants, Mn (0.032-0.25 cation %) and Ti(0.12-0.24%) were introduced into the alumina by the following procedure: A solution was prepared by dissolving the appropriate amounts of TiCl_3 and $\text{MnCl}_3 \cdot 4\text{H}_2\text{O}$ in deionized water. α -alumina powder was added to this solution to form a slurry. The slurry was added to a concentrated and agitated solution of ammonium hydroxide leading to the precipitation of manganese and titanium hydroxides. After a thorough mixing, the precipitated slurry was filtered and dried at 100°C . Using isopropyl alcohol and high purity alumina grinding media the coprecipitated powder mixture was milled for about 6 hours. After evaporating the alcohol the resulting powder was calcined at 1000°C for 12-15 hours in air. The powder was milled again in isopropyl alcohol after calcination.

After evaporating the alcohol, the powder was vacuum hot-pressed (>98% of theoretical density) in graphite dies at 1200°C and 6000 psi for approximately 2 hours. The microstructure of the hot-pressed billets was uniformly equiaxed. Scanning electron microscope studies utilizing emitted x-rays revealed that the dopants were dispersed uniformly throughout the grains and not concentrated at grain boundaries.

In order to fabricate material with a range of grain sizes (13-43 μm) some specimens were annealed at temperatures up to 1450°C to promote grain growth.

It has become clear during the course of these studies that some of the impurities (e.g. Mn) were lost during the processes of hot-pressing

and high temperature annealing due to volatilization. Consequently, representative specimens after creep testing were analyzed for Ti and Mn by atomic absorption. All values reported for impurity contents are reliable to $\pm 10\%$.

Four-point dead load creep tests were conducted on beams $2 \times 5 \times 30\text{mm}$ in size. The creep testing apparatus and procedure have been described elsewhere⁽⁸⁾. Strain rates ($\dot{\epsilon}$) and stresses (σ) were calculated from standard relations assuming viscous behavior.⁽¹⁰⁾ Outer fiber strains were normally less than 1-2%. Creep experiments were conducted at temperatures ($\pm 0.5^\circ\text{C}$) between 1075 and 1300°C and at stresses up to 40 MN/m^2 . Most of the creep data were taken after 20-30 hours of creep testing. In some cases creep tests were extended up to 250 hours to examine the nature of the transient.

2.1.2. Stress Dependence

Stress change experiments were conducted on several specimens over a range of grain sizes ($25\text{-}30 \mu\text{m}$), stresses ($4\text{-}40 \text{ MN/m}^2$), and temperatures ($1179\text{-}1300^\circ\text{C}$). In all cases viscous (i.e. $\dot{\epsilon} \propto \sigma^N$; $N=1$) creep behavior was observed. In Figure 1 representative stress change experiments are shown. Here deflection rate versus load data plotted on a linear scale resulted in a straight line passing through the origin, a definitive test for viscous creep.

No evidence was found for slightly non-viscous creep (i.e. $\dot{\epsilon} \propto \sigma^N$ with N up to 1.7), which is typical for the creep of pure and doped polycrystalline alumina.^(1,2,11) Viscous creep has been reported previously⁽²⁾ in iron-doped (2 cation %) polycrystalline alumina for

Figure 1: Load - deflection rate behavior of creep in Mn-Ti doped polycrystalline Al_2O_3 .

creep tests conducted in a reducing atmosphere ($\sim 10^{-6}$ atm.) where more of the iron is divalent.

Due to the presence of a very long time transient in the creep of double-doped polycrystalline alumina, a stress-change experiment at short times (<20 hours) does not give reversible creep rates. A thorough discussion of this transient will be presented later in this report.

2.1.3. Grain Size Effects

Summarized in Figure 2 are all of the data taken to date at 1250°C on the effect of grain size on the creep of polycrystalline alumina doped with mixed transition metal impurities (Mn-Ti). The data represent 32 different tests, Mn/Ti ratios between 0.2 and 2.1, and total impurity levels between 0.18 and 0.37 cation %. Creep specimens with low Mn/Ti ratios (0.2-0.3) exhibited a very strong inverse grain size relationship (i.e. $\dot{\epsilon} \propto (GS)^{-m}$ with $m=4.2-4.8$). On the other hand, specimens with high Mn/Ti ratios (~ 2) also led to a significant, but somewhat weaker (i.e. $m=2.7$), grain size dependence.

All of the data in Figure 2 represent strain rates taken after 20-30 hours of creep testing. However, in view of transient effects which persist in some cases up to 250 hours, the creep rates shown in Figure 2 are overestimates (up to an order of magnitude) of steady state values and accordingly lead to uncertainties in the exact value of the grain size exponents. However, we can conclude at this time that increasing the grain size has a strong effect in inhibiting the

Figure 2: Grain-size dependence of creep of Mn-Ti doped polycrystalline Al_2O_3 at 1250°C .

creep of polycrystalline alumina doped with mixed transition metal impurities. Increases in the grain size by a factor of two led to decreases in the creep rate by up to a factor of almost twenty.

The significant enhancing effect of double doping on the creep rate can be seen from the data plotted in Figure 3. Here a comparison is made with undoped alumina and material doped with single impurities (Ti or Fe up to 2 cation %). All of the steady state creep data for the undoped and singly doped specimens were extrapolated from creep tests at higher temperatures ($>1350^{\circ}\text{C}$) using appropriate activation energies (120-130 kcal/mole)^(2,8). It is clear that double doping at low total impurity concentrations (0.2-0.4%) is far more effective in enhancing the creep of polycrystalline alumina than by doping with single impurities at concentrations between 0.5 and 2%. Even taking into account the long term transient nature of the creep process (i.e. slowly changing creep rates at 250 hours are up to an order of magnitude smaller than the creep rates after 20-30 hours of testing) in double-doped creep specimens, the creep rate of alumina is enhanced by 2-4 orders of magnitude by doping with small amounts (0.2-0.4%) of Mn and Ti or Fe and Ti.

2.1.4. Apparent Creep Activation Energies

Creep data taken from temperature change studies after 20-30 hours of creep testing indicate approximate apparent activation energies between 100 and 130 kcal/mole. Representative data are shown in Figure 4. In view of the transient effects which lead to uncertainties in the absolute value of the activation energy, we

Figure 3: Grain-size dependence of creep of "pure" iron, titanium, and Mn-Ti doped Al_2O_3 at 1250°C . ("Pure" iron and titanium doped Al_2O_3 are extrapolated from 1450°C).

Figure 4: Activation energies of creep of Mn-Ti Al_2O_3 .

can only conclude at this time that over the temperature range between 1075°C and 1300°C the creep of polycrystalline alumina doped with mixed dopants proceeds by some diffusional process. In view of the strong reciprocal grain size dependence, we suspect that oxygen grain boundary diffusion may be rate limiting, at least in part.

2.1.5. Effects of Oxygen Partial Pressure

The effect of oxygen partial pressure on the creep rate of polycrystalline alumina doped simultaneously with Mn and Ti is summarized in Table I. A typical experiment in which the oxygen partial pressure is repeatedly cycled between a high and low oxygen partial pressure is shown in Figure 5. A systematic interpretation of the effects summarized in Table I await the systematic collection of additional data. It is interesting to note, however, that the atmosphere effects in the double-doped specimens are opposite to what might have been expected from similar experiments with single dopants (Fe and Ti)⁽¹⁾. For example, in iron doped alumina a decrease in P_{O_2} leads to a decrease in the creep rate⁽¹⁾. However, for Mn-rich specimens double doped with Mn and Ti the opposite effect is seen. This apparent anomaly is also true for the Ti-rich double-doped specimens.

2.1.6. Transient Effects

In Figure 6 creep data are presented for long term tests (200-250 hours) which clearly illustrate the transient nature of the deformation process in double-doped specimens tested at low temperatures (1130-1175°C) at very low creep stresses ($\sim 2 \text{ MN/m}^2$). All of

Table I

<u>Impurity, concentration (cation %)</u>	<u>Mn/Ti</u>	<u>Increasing P_{O2}</u>
0.075% Mn	0.31	decreasing creep rate
0.24% Ti		
0.25% Mn	2.08	increasing creep rate
0.12% Ti		
1-2% Fe	---	decreasing creep rate
0.5% Ti	---	increasing creep rate

Figure 5: Effect of P_{O_2} on strain rate vs. time.

Figure 6: Transient creep of Mn-Ti doped Al_2O_3 .

the data presented earlier in this report were taken after 20-30 hours of creep testing. It is clear that these creep rates are almost an order of magnitude larger than creep rates after 200-250 hours of creep testing. Until we can determine the cause of this transient, definitive conclusions on the exact stress, grain size and temperature dependencies cannot be made.

Considerable time and effort have been expended this year in trying to understand the cause of this long term transient. Several mechanisms have been investigated and discarded for various reasons. These include: (1) simultaneous grain growth, (2) strain or work-hardening by dislocation motion, (3) diffusional creep transient caused by a redistribution of stresses, and (4) defect and/or impurity homogenization and/or equilibration.

The simultaneous occurrence of diffusional creep and grain growth will give rise to a long term transient because of the strong reciprocal grain size dependence. Extensive grain size measurements (1800 chord length measurements) before and after creep testing 200 hours at 1130°C revealed no grain growth to within $0.2\mu\text{m}$ for a $19.2\mu\text{m}$ grain size specimen. During the creep test the strain rate decayed by about an order of magnitude and thus eliminated grain growth as a viable explanation.

Dislocation slip and interaction can be eliminated as a viable mechanism, since the stress is about a factor of 50 below the yield stress of pure alumina at 1300°C ⁽¹²⁾. The lower temperatures (1075 - 1250°C) and impurity levels should make dislocation glide even more improbable.

Diffusional creep theory⁽¹³⁾ will predict a transient caused by a redistribution of normal stresses on the grain boundaries from the time of loading to the achievement of steady state conditions. In this theory transient strain rates will be described by a relation of the form, $\dot{\epsilon} \propto e^{-t/\tau}$ in which the transient relaxation time, τ , will be given by two limiting conditions depending on the value of a dimensionless parameter $\lambda (= \frac{\Omega C_V E}{kT})$. Ω is the vacancy volume, C_V is the concentration of lattice vacancies, E is the elastic modulus, and kT has its usual meaning. For $\lambda \gg 1$, $\tau \propto D_V / (GS)^2$ in which D_V is the defect diffusion coefficient and GS is the grain size. For $\lambda \ll 1$, $\tau \propto D_V C_V \Omega E / (GS)^2 kT$. The foregoing equations apply only to diffusional creep controlled by lattice diffusion. Similar equations, except for a cubic grain size dependence, apply for viscous creep rate limited by grain boundary diffusion⁽¹⁴⁾.

Using reasonable estimates for the diffusion constants, relaxation times on the order of minutes can be predicted. These are well below those which are seen (order of hours). Furthermore, the observed dependence of τ on stress and temperature are not consistent with the theory which predicts that τ is stress independent.

Having essentially eliminated grain growth, dislocation motion, and stress redistribution as the principal causes of the transient, we currently believe the phenomenon is caused either by a defect or impurity equilibrium process. While impurity segregation cannot be eliminated entirely, scanning electron microscopic (with the energy dispersive x-ray analyzer) examination on polished and fracture surfaces revealed no significant impurity inhomogeneity or segregation to the grain boundaries.

However due to the very low impurity concentrations, this technique is not very accurate.

We currently believe that defect equilibration via the diffusion of oxygen into or out of the sample as the valence states of Mn and Ti adjust to their equilibrium values is the most likely cause of the transient. Prior to creep testing all the specimens are annealed and equilibrated at higher temperatures (1300-1700°C) to promote a range of stable grain sizes. However at the creep test temperature ($\leq 1250^{\circ}\text{C}$), the time required for defect equilibration, which will involve some lattice or grain boundary diffusion over a distance at least on the order of a grain length, could easily exceed 100 hours (i.e. for grain sizes around 10-20 μm and lattice diffusion coefficients $\leq 10^{-12} \text{cm}^2/\text{sec}$). To check this possibility several experiments are under way. First the transient should be accompanied by an oxygen uptake on depletion from the sample giving rise to a weight change which could be measured as a function of time. Secondly by doping at lower impurity levels ($\sim 0.1\%$) and creep testing at higher temperatures ($\geq 1350^{\circ}\text{C}$) we should be able to promote steady state conditions and still observe the enhancing effect of the two impurities.

2.2 Stress Relaxation Creep Studies

The stress relaxation technique has been extensively used in plastic deformation studies of metallic materials^(15,16). The technique has been recently utilized in the studies of high temperature deformation behavior of ceramic materials^(8,17). In a stress relaxation test, a specimen of defined geometry is deformed at a constant deflection rate to a predetermined total load or total deflection and then the total deflection is held constant. The specimen continues to deform plastically while the

elastic deflection and hence the load drops with time. The rate of load drop, (dp/dt) , is related to the plastic deflection rate, $\dot{y}$, of the specimen through the total elastic compliance of the system, C , including specimen and the loading machine.

$$\dot{y} = -C \cdot \frac{(dp)}{dt} \quad (1)$$

The deflection rate, $\dot{y}$, and the instantaneous load, p , are then converted to plastic strain rate and stress, respectively, using the specimen geometry and loading arrangement.

The stress relaxation test has several advantages over the conventional constant deflection (or strain) rate tests or constant stress creep tests.

(1) The relationship between stress and plastic strain rate can be established over wide ranges of stress ($0.1-100 \text{ Mn/m}^2$) and strain rates ($10^{-3} - 10^{-9} \text{ second}^{-1}$) in a single test without fracturing the specimen.

(2) By using a "stiff machine" (i.e. contribution of machine compliance to C in Eq.(1) is negligible) one can minimize the total plastic strain accumulated in a relaxation test. Thus the stress-strain rate relationships are established at essentially constant plastic strain or at a state of constant "hardness"⁽¹⁷⁾ in the test material. Repetition of stress relaxation cycles then provides a means of studying the effects of strain hardening (or strain softening as the case may be) or plastic strain history on the stress-strain rate relationship.

(3) When the stress relaxation technique is adapted to the four point bending test geometry, it provides a means of determining outer fiber strain based only on specimen geometry. It eliminates a priori assumptions

regarding the plastic behavior of the material in the calculations of strains and stresses. This point will be elaborated further in the section on analysis of stress relaxation results in four point bend tests.

The unique advantages of the stress relaxation technique are particularly suited to achieving some of the objectives of the current research program on impurity effects on the creep of polycrystalline magnesium and aluminum oxides at elevated temperatures. These may be stated as follows:

- (1) To examine the transition in creep behavior from a low stress, viscous creep regime to one at high stresses which is nonviscous and has a power law stress dependence.
- (2) To study the effects of temperature (1350°C and 1450°C), grain size ($10\text{-}100\mu\text{m}$) and impurity additions (0.53 cation % Fe in MgO , 1 cation % Fe in Al_2O_3) on this transition in creep mechanism.
- (3) To establish the creep rate-grain size relationships in these systems both in the viscous and power law creep regimes.
- (4) To study the nature of solid solution hardening effects of impurity additions in the power law creep regime.

It is clear that dead load creep tests are inadequate to study creep behavior in the high stress regimes. The large strains that are accumulated under these conditions will either lead to tertiary creep and rapid fracture or the deflections involved will be too large and the analysis of the four point bend test geometry will no longer be applicable. A major effort was, therefore, devoted in the past year of this contract to developing an improved stress relaxation test procedure to complement the research activity in the dead load creep program. The progress made in this area is reported in the following three sections.

2.2.1. Description of an improved stress relaxation test system.

The success of the stress relaxation technique is critically dependent upon the temperature stability of the test system. Even small fluctuations in temperatures can affect the compliance, C , of the test system and can lead to load fluctuations during relaxation. To improve the thermal stability of the stress relaxation system and hence the range of strain rates that could be measured in a relaxation test, a new test system was designed and constructed during the past year. The new system is similar to the one that has been used in the past⁽⁸⁾. Modifications were, however, incorporated in the new system with the specific objectives of improving the performance. In Figure 7 a schematic cross sectional view of the stress relaxation system is shown. The whole system is designed to be interfaced with a floor model 'Instron' Universal Testing Machine. The major components of the system are:

(1) A new furnace with molybdenum metal wire as the heating element. This replaces the old furnace that featured MoSi_2 heating elements. While the new furnace is not as compact as the old one, it has greater thermal mass and thermal stability which is crucial in a relaxation test. The exterior of the furnace is constructed out of brass and is water cooled on all surfaces. This eliminates heat flow to the Instron drive screw and hence load fluctuations. A light, all-alumina, fibrous insulation makes the furnace light weight and facilitates easy mounting on the Instron frame. Four Thomson ball bushings are mounted on the furnace end plates. These enable the furnace to be moved up and down on guiding Thomson rods and facilitate access to the interior of the furnace for purposes of specimen

Figure 7: A schematic cross sectional view of the stress relaxation system.

mounting. A view of the furnace assembly as mounted on a stainless steel table in the Instron frame is shown in Figure 8A.

(2) A linear variable differential transformer (Shaevitz HR 050) and a nonrotating micrometer with a measurement sensitivity of 2×10^{-5} inch constitute the specimen center point deflection measurement system. As will be evident from the discussion in the following section, a continuous monitoring of the specimen deflection at the center point is essential for the determination of the strain in a four point bend specimen from purely geometric considerations. The body of the LVDT is mounted in an aluminum block fitted with ball bushings and movable along Thomson guide rods. The micrometer serves the dual purpose of calibrating the LVDT as well as positioning the LVDT body for desired initial output. The LVDT core is connected through a threaded core connector to an alumina rod that acts as a deflection measuring probe. The core assembly is spring loaded so as to ensure continuous contact of the probe tip with the lower face of the specimen. A ball bushing in the center of the stainless steel table maintains the axiality of the deflection measuring core assembly. The LVDT-micrometer system as mounted below the stainless steel table is seen in Figure 8A.

(3) Accurately machined dense alumina rods and sample pedestal constitute the loading assembly. Single crystal sapphire rods are used as support points and loading points on the specimen. A sapphire sphere is used to transmit the load from the loading rod to the specimen through a loading piece. The spherical contact point eliminates nonaxial forces being transmitted to the specimen. A closeup view of the specimen mounted on the pedestal with the associated loading assembly is shown in Figure 8B.

Figure 8 A: The stress relaxation test assembly as mounted on the Instron frame.

Figure 8 B: A close-up view of the specimen and loading assembly with sapphire loading points.

(4) A water cooled adaptor connects the loading rod to the load cell (Figure 8A, top).

(5) A new temperature controller-power supply unit (Research Incorporated Model No.: Thermac Model 625) allows temperatures to be controlled in the range 1000°C - 1750°C with a maximum fluctuation no greater than $\pm 0.25^{\circ}\text{C}$. Pt - Pt + 10% Rh thermocouples are used both to control and monitor the temperature. The temperature limitation of the relaxation system is primarily from room temperature fluctuations rather than the furnace temperature variations.

2.2.2. Analyses of Stresses and Strains in Relaxation Tests in Four Point Bend Specimens.

A rigorous analysis of stresses and strains in a four point bend test specimen under plastic deformation conditions is a complex problem. The complexity is due to the fact that a bend specimen is a composite of a large number of fibers subjected to different deformation conditions and the externally measured loads and deflections are complex resultants of the stresses and strains of the individual fibers. Varying numbers of assumptions are usually made to make the four point bend specimen amenable to analysis. Before we review some of the analyses that are commonly being used with four point bend tests, let us itemize the common assumptions inherent to all these analyses⁽¹⁸⁾.

(1) Plane cross sections before bending remain plane after bending; in other words, the strain at a point in a given cross section varies linearly with its distance from the neutral axis.

(2) The analysis is limited to beams that are long compared with their cross-sectional dimensions. In this case shear deflections can be neglected.

(3) Bending displacements are small compared with the length of the beam.

(4) The plastic behavior of the material in tension and compression are identical except for sign.

Under the constraints of the above four assumptions, the maximum strain in the outer fiber in a four-point bend test specimen should ideally be determined from only geometric considerations. Since the radius of curvature is constant between the inner load points, a simple relation exists between the maximum strain in the outer fiber and the deflection of the center of the beam relative to the load points. (10)

$$\epsilon_{\max} = \frac{4h}{a} (y_c - y_L) \quad (2)$$

where $\epsilon_{\max}$ = maximum strain in the outer fiber

y_c = deflection at the center of the beam

y_L = deflection at the load points

h = thickness of the beam

a = distance between load points.

A stress relaxation test system incorporating center point deflection measurement is ideally suited to making use of eqn (2) for strain and strain rate calculations. The center point deflection, y_c , is directly measured using the LVDT. The load point deflection, y_L , is obtained through equation (1). A measure of the compliance, C , of the loading system can be determined from the initial part of the loading curve.

When experimental difficulties preclude measurement of deflections at more than one point, which is usually the case in dead load creep tests, additional simplifying assumptions are needed to calculate strains based on deflection at a single point. Quite often plastic deflections are analyzed using elastic deflection formulae of the simple beam deflection theory. Then the outer fiber maximum strain is expressed

in terms of either the center point deflection or the load point deflection. The corresponding equations are^(8,10)

$$\epsilon_{\max} = \left[\frac{12h}{2L^2 + 2La - a^2} \right] y_c \quad (3)$$

and

$$\epsilon_{\max} = \left[\frac{6h}{(L-a)(L+2a)} \right] y_L \quad (4)$$

where L = distance between the outer support points.

Hollenberg et al⁽¹⁰⁾ have analyzed four point bending for the situation where the material is assumed to plastically deform according to a power law:

$$\dot{\epsilon} = A\sigma^N \quad (5)$$

The important assumptions made in their analysis are that

- (1) Stress power index N is independent of stress.
- (2) All the fibers in the specimen obey the same law with the same stress power index.

Under these assumptions the outer fiber strain is related to the load point deflection in the form

$$\epsilon_{\max} = \frac{2h(N+2)}{(L-a) [L+a(N+1)]} y_L \quad (6)$$

It should be noted that equation (6) reduces to the elastic strain equation (4) when $N = 1$, i.e. viscous creep.

In the stress relaxation program, plastic deflections were analyzed using both the ideal geometric relation (equation 2) as well as the simplified elastic analysis (equations (3) and (4)).

Simple elastic beam theory gives the simplest analysis for stress calculation which overestimates the actual stress in the outer fiber.

From simple theory the outer fiber stress can be written in terms of the

applied load as

$$\sigma_{\max} = \frac{1.5 (L-a) p}{bh^2} \quad (7)$$

where p = externally applied load.

As in the case of strain calculation the material is assumed to obey a power creep law (equation (5)) and it is implicitly assumed that N is independent of stress level as well as fiber position. Under these assumptions the maximum stress still occurs at the outer fiber but this stress is less than the elastic stress (eqn. 7). The equation relating stress to the applied load is shown to be^(10,18)

$$\sigma_{\max} = \frac{1.5 (L-a)p}{bh^2} \frac{(2N+1)}{3N} \quad (8)$$

Equation (8) reduces to equation (7) for $N = 1$ (viscous creep). It should also be noted that the overestimation of the stress by equation (7) increases as N increases above unity.

Bilde-Sorensen⁽¹⁹⁾ has recently presented an analysis which shows that equation (8) is still applicable when N is a function of stress. His analysis shows that there is no necessity for assuming a priori the value for stress power index N . Instead N can be interpreted as

$$N = (d \log \dot{\epsilon}_{\max} / d \log M) \quad (9)$$

Once N is determined at a particular load, the corresponding stress is calculated using equation (8). Sorensen's arguments are correct in a mathematical sense. But the physical implications of his analysis are not clear. This is particularly the case when a bend specimen undergoes a transformation in creep behavior, i.e., outer fibers obey say a power creep law ($N > 1$) and inner fibers obey the viscous law ($N = 1$). In such a situation it is not clear which of the two N values is determined by equation (9). Since the external moment M is a resultant of all the internal stresses it may

be interpreted that the N value determined by equation (9) is an intermediate value between the inner and outer fibers.

The calculation of the outer fiber stress is still possible in a situation when the stress sensitivity of the strain rate (i.e. stress exponent N) is a function of stress or equivalently a function of fiber position, i.e.

$$N = N(\sigma) = N(y) \quad (10)$$

where y is the distance from the neutral axis.

The basic assumption cited in the beginning of this section, viz. plane sections remain plane during bending, is still assumed. Then

$$\dot{\epsilon}(y) = \dot{\epsilon}_{\max} \frac{2y}{h} \quad (11)$$

Also the individual fibers follow the general creep equation:

$$\dot{\epsilon}(y) = A [\sigma(y)]^{N(y)} \quad (12)$$

and finally the externally applied moment must be balanced by the internal stresses

$$\therefore M = 2b \int_0^{h/2} \sigma(y) y dy \quad (13)$$

The problem posed here is redundant in nature. The function $N(y)$ can be determined only if $\sigma(y)$ is known and conversely $\sigma(y)$ can be determined only when $N(y)$ is assumed. Solution to this problem is possible only through an iterative numerical procedure. We assume a stress distribution, say initially linear; (elastic); then we linearize the strains and recalculate stresses. This procedure is repeated until stress distribution becomes independent of strain. A research program to examine the feasibility of such a calculation is part of the research proposal submitted with this report.

2.2.3. Preliminary Stress Relaxation Deformation Studies on Polycrystalline, Iron-Doped MgO.

Several preliminary experiments were carried out using the newly designed stress relaxation test system. Magnesium oxide with 0.53 cation % Fe was chosen as the material for initial study. Two temperatures (1350°C and 1450°C) and two grain sizes (25µm and 40µm) were selected for these tests. These conditions were chosen such that comparisons with dead load creep data⁽²⁰⁻²³⁾ could be facilitated.

Most of the stress relaxation data were analyzed using the elastic equations (equations (3) or (4) for strain and equation (7) for stress). Selected data were analyzed using a plastic analysis. This involved the use of equation (2) for strain which makes use of both load point and center point deflections. Thus the strains calculated are the ideal true strains based only on geometry. The corresponding maximum stresses were determined using the analysis of Bilde-Sorensen⁽¹⁹⁾. The calculated strain rates were used to evaluate the stress sensitivities of strain rates using equation (9). The stress exponents thus determined were used in calculating the stresses through equation (8). Of course, the limitations of this analysis discussed earlier are still valid here.

In Figure 9 a comparison is made of the two types of analyses (elastic and plastic) as applied to the same stress relaxation data. Both analyses show a clear transition in the creep behavior from a low stress, near viscous behavior ($N \approx 1.5$) to a high stress, nonviscous creep regime ($N \approx 4$). There is no significant difference in the stress exponents determined from the two plots. The stresses calculated by the elastic analysis are over-estimates of the plastic stresses by a factor

$$\frac{\text{Elastic stress}}{\text{Plastic stress}} = \frac{3N}{2N+1}$$

Figure 9: A comparison of two types of analyses of stress relaxation data obtained on four-point bend test specimens.

At higher values of the stress exponent, N , the elastic analysis increasingly overestimates the stress. This is clearly seen in the data of Figure 9. The strain rates calculated by the elastic analysis, on the other hand, are uniform underestimates of the actual geometric strain rates.

In Figure 10 a comparison is made of stress relaxation data with dead load creep data. In Figure 10 the experimental points correspond to the results of a stress relaxation test on iron-doped (0.53 cation % Fe) MgO at 1342°C with a 25 μ m grain size. The behavior predicted by dead load creep data under identical conditions is represented in the form of two straight lines⁽²³⁾ with slopes of 1 and 3 respectively. These represent the viscous and nonviscous regimes, respectively. The position of the transition point between these regimes was determined from the creep map that was generated for this material.⁽²³⁾ These maps were constructed using dead load creep test data.⁽²⁰⁻²²⁾ Two features are evident in Figure 10. In the low stress regime there is good agreement both in absolute creep rates as well as the slope, i.e. stress exponent N which is nearly 1. The stress relaxation data, however, indicate a transition to power law creep at a smaller stress level relative to the dead load transition. Two factors may contribute to this discrepancy. The total strain involved in the stress relaxation test is very much smaller than in the dead load test. In MgO+0.53 cation % Fe dislocation creep is expected to be dominant in the power law regime.⁽²¹⁾ So the increased plastic strain would lead to strain hardening and reduced strain rates with a shift in the transition point to higher stresses. Secondly creep maps were constructed on the basis of extrapolations from higher tempera-

Figure 10: A comparison of the stress relaxation results (experimental points) with the predicted behavior from dead-load creep study.

Figure 11: The stress dependence of creep rate in MgO-0.53 cation % Fe obtained by the stress relaxation technique at 1350°C at two grain-size levels - 25 μ m and 40 μ m.

ture dead load data.* Some error is therefore expected in the exact position of the lines in Figure 4 due to the uncertainties in the creep activation energy.

In Figure 11 a comparison is made of the stress dependence of the strain rates in $\text{MgO}+0.53$ cation % Fe at 1350°C for two grain sizes. As would be expected in the low stress regime, the finer grained specimen displays more viscous behavior than the coarse grained material. (Note: exponent 1.07 versus 1.49). The viscous-nonviscous transition is also shifted to a higher stress in finer grained material. The second interesting feature is the reversal of grain size dependence. In the viscous creep regime, of course, diffusional creep theories predict higher creep rates with smaller grain sizes. In the power law regime two features are different from the corresponding observations of dead load studies.⁽²¹⁾ We do see a grain size dependence here while in the dead load creep studies power law creep rates were independent of grain size.⁽²¹⁾ Secondly the stress exponents observed here are consistently larger than the value observed in the previous study (approximately 4.5 versus 3.5). The explanation for these differences may again lie in the basic difference in the two test conditions, viz. amount of total strain. The dead load studies were conducted in the large strain true steady state regime while stress relaxation experiments have been in the small strain or transient regime. Additional experiments investigating the strain hardening effects in the power law creep regime are necessary to resolve these differences.

In Figure 12 the temperature dependence of creep transitions is examined. Higher temperatures induce more viscous creep in the low stress

*Dead load creep tests in the power law creep regime were conducted at 1500°C ⁽²¹⁾.

Figure 12: The effect of temperature (1350°C and 1450°C) on creep rates and creep transition behavior in MgO-0.53 cation % Fe specimens.

regime as reflected by the lowering of the stress exponent. In the power law regime the stress exponent does not appear to change significantly. In the large grained material ($40\mu\text{m}$) it is likely that one sees a mixture of viscous and nonviscous creep processes at low stresses. This would explain the deviation of exponents from the ideal viscous stress exponent. Increasing the temperature enhances the viscous component and hence lowers the stress exponent.

III. References

1. G. W. Hollenberg and R. S. Gordon, "Effect of Oxygen Partial Pressure on the Creep of Polycrystalline Al_2O_3 Doped with Cr, Fe, or Ti," J. Amer. Ceram. Soc., 56(3) 140-147 (1973).
2. P. A. Lessing and R. S. Gordon, "Creep of Polycrystalline Alumina, Pure and Doped with Transition Metal Impurities," J. Mater. Sci. 12 2291-2302 (1977).
3. J. R. Keski and I. B. Cutler, "Effect of Manganese Oxide on Sintering of Alumina," J. Amer. Ceram. Soc., 48 (12) 653-54 (1965).
4. J. R. Keski and I. B. Cutler, "Initial Sintering of $Mn_xO-Al_2O_3$ ", J. Amer. Ceram. Soc., 51 (8) 440-44 (1968).
5. R. D. Bagley, I. B. Cutler, and D. L. Johnson, "Effect of TiO_2 on the Initial Sintering of Al_2O_3 ", J. Amer. Ceram. Soc., 53 (3) 136-141 (1970).
6. W. Raja Rao and Ivan B. Cutler, "Effect of Manganese Oxide on Sintering of Alumina," J. Amer. Ceram. Soc., 48(12) 653-54 (1965).
7. Ivan B. Cutler, Cyril Bradshaw, Carl J. Christensen and Edmond P. Hyatt, "Sintering of Alumina at Temperatures of $1400^{\circ}C$ and Below", J. Amer. Ceram. Soc., 40 (4) 134-39 (1957).
8. P. A. Lessing, "The Creep of Magnesia and Alumina Doped with Transition Element Impurities", Ph.D. Thesis, University of Utah, 1976.
9. G. W. Hollenberg, "The Effect of Oxygen Partial Pressure on the Creep of Polycrystalline Alumina Doped with Transition Metal Impurities", Ph.D. Thesis, University of Utah, 1972.
10. G. W. Hollenberg, G. R. Terwilliger, and R. S. Gordon, "Calculation of Stresses and Strains in Four-Point Bending Creep Tests", J. Amer. Ceram. Soc., 54 (4) 196-199 (1971).
11. A. H. Heuer, R. M. Cannon, and N. J. Tighe, pp. 339-65 in "Ultrafine-Grain Ceramics," Edited by J. J. Burke, N. L. Reed and Volker Weiss, Syracuse University Press, Syracuse, N. Y., 1970.
12. M. L. Kronberg, "Dynamical Flow Properties of Single Crystals of Sapphire, 1", J. Amer. Ceram. Soc., 45, (6), 274-79 (1962).
13. I. M. Lifshitz and V. B. Shikin, "Theory of Diffusional Viscous Flow of Polycrystalline Solids", Sov. Phys.-Solid State, 6 (9) 2211-18 (1965).

References

14. Ronald S. Gordon and G. R. Terwilliger, "Transient Creep in Iron-Doped Polycrystalline MgO," J. Amer. Ceram. Soc. **55** (9) 450-55 (1972).
15. F. Guiu and P. L. Pratt, "Stress Relaxation and the Plastic Deformation of Solids", Phys. Stat. Sol. **6**, 111 (1964).
16. E. W. Hart, "Theory of the Tensile Test", Acta Met **15**, 351, (1967).
17. J. T. A. Roberts, "Mechanical Equation of State and High-Temperature Deformation" ($\geq 0.5 T_m$) of Uranium Dioxide", Acta Met, **22**, 873, (1974).
18. I. Finnie and W. R. Heller, "Creep of Engineering Materials", pp. 135-137, McGraw-Hill Book Co., New York, (1959).
19. J. B. Bilde-Sørensen, "Maximum Fiber Stress in Bending Creep," J. Amer. Ceram. Soc., **58**, (1-2), 70, (1975).
20. R. T. Tremper, R. A. Giddings, J. D. Hodge, and Ronald S. Gordon, "Creep of Polycrystalline MgO-FeO-Fe₂O₃ Solid Solutions", J. Amer. Ceram. Soc., **57**, (10) 421-28 (1974).
21. P. A. Lessing and R. S. Gordon, "Impurity and Grain Size Effects on the Creep of Polycrystalline Magnesia and Alumina," in Deformation of Ceramic Materials, edited by R. C. Bradt and R. E. Tressler, pp. 271-296, Plenum Press, N. Y., 1975.
22. Ronald S. Gordon and J. D. Hodge, "Analysis of Mass Transport in the Diffusional Creep of Polycrystalline MgO-FeO-Fe₂O₃ Solid Solutions", J. Mater. Sci. **10**, 200-204 (1975).
23. J. D. Hodge, P. A. Lessing, R. S. Gordon, "Creep Mapping in a Polycrystalline Ceramic: Application to Magnesium Oxide and Magnesio-wüstite", J. Mater. Sci., **12**, 1593-1606 (1977).

IV. BIBLIOGRAPHY OF PAPERS, THESES AND
INVITED PRESENTATIONS

Papers

1. G. R. Terwilliger and Ronald S. Gordon, "Correlations Between Models for Time-Dependent Creep with Concurrent Grain Growth," J. Amer. Ceram. Soc. 54 (4) 218-219 (1969).
2. G. R. Terwilliger, H. K. Bowen, and Ronald S. Gordon, "Creep of Polycrystalline $\text{MgO-Fe}_2\text{O}_3$ Solid Solutions at High Temperatures," J. Amer. Ceram. Soc. 53 (5) 241-251 (1970).
3. Ronald S. Gordon, D. D. Marchant, and G. H. Hollenberg, "Effect of Small Amounts of Porosity on Grain Growth in Hot Pressed Magnesium Oxide and Magnesiowüstite," J. Amer. Ceram. Soc. 53 (7), 339-406 (1970).
4. G. W. Hollenberg, G. R. Terwilliger, and Ronald S. Gordon, "Calculations of Stresses and Strains in Four Point Bending Creep Tests," J. Amer. Ceram. Soc. 54 (4), 196-199 (1971).
5. D. D. Marchant and Ronald S. Gordon, "Grain Size Distributions and Grain Growth in MgO and $\text{MgO-Fe}_2\text{O}_3$ Solid Solutions," J. Amer. Ceram. Soc. 55 (1) 19-24 (1972).
6. Ronald S. Gordon and G. R. Terwilliger, "Transient Creep in Iron-Doped Polycrystalline MgO ," J. Amer. Ceram. Soc. 55 (9) 450-455 (1972).
7. Glenn W. Hollenberg and Ronald S. Gordon, "Effect of Oxygen Partial Pressure on the Creep of Polycrystalline Al_2O_3 Doped with Cr, Fe, or Ti," J. Amer. Ceram. Soc. 56 (3) 140-147 (1973).
8. Ronald S. Gordon, "Mass Transport in the Diffusional Creep of Ionic Solids," J. Amer. Ceram. Soc. 56 (3) 147-152 (1973).
9. G. W. Hollenberg and Ronald S. Gordon, "Origin of Anomalously High Activation Energies in the Sintering and Creep of Impure Refractory Oxides," J. Amer. Ceram. Soc. 56 (2) 109-110 (1973).
10. R. T. Tremper, R. A. Giddings, J. D. Hodge and Ronald S. Gordon, "Creep of Polycrystalline $\text{MgO-FeO-Fe}_2\text{O}_3$ Solid Solutions," J. Amer. Ceram. Soc. 57 (10) 421-428 (1974).
11. Ronald S. Gordon and J. D. Hodge, "Analysis of Mass Transport in the Diffusional Creep of Polycrystalline $\text{MgO-FeO-Fe}_2\text{O}_3$ Solid Solutions," J. Mater. Sci. 10 200-204 (1975).

12. R. S. Gordon, "Ambipolar Diffusion and its Application to Diffusion Creep," in Mass Transport Phenomena in Ceramics, edited by A. R. Cooper and A. H. Heuer, pp. 445-464, Plenum Press, N. Y. (1975)
13. P. A. Lessing and R. S. Gordon, "Impurity and Grain Size Effects on the Creep of Polycrystalline Magnesia and Alumina" in Deformation of Ceramic Materials, edited by R. C. Bradt and R. E. Tressler, pp.271-296, Plenum Press, N. Y. (1975).
14. P. A. Lessing, R. S. Gordon, and K. S. Mazdiasni, "Creep of Polycrystalline Mullite," J. Amer. Ceram. Soc. **58** (3-4) 149 (1975).
15. J. D. Hodge, P. A. Lessing and R. S. Gordon, "Creep Mapping in a Polycrystalline Ceramic: Application to Magnesium Oxide and Magnesiowüstite," J. Mater. Sci. **12** 1598 (1977).
16. J. D. Hodge, P. A. Lessing, and R. S. Gordon, "Activation Energies in the Diffusional Creep of Polycrystalline Ceramics," J. Amer. Ceram. Soc., **60**(7) 318 (1977).
17. J. D. Hodge and R. S. Gordon, "Grain Growth and Creep in Polycrystalline Magnesium Oxide Fabricated with and without a LiF Additive", Ceramurgia International. (in press)
18. Paul A. Lessing, James D. Hodge, and Ronald S. Gordon, "High Temperature Creep of Polycrystalline MgO and Al₂O₃", Proceedings of Second International Conference on Mechanical Behavior of Materials, August 1976, Boston (in press).
19. Paul A. Lessing and Ronald S. Gordon, "Creep of Polycrystalline Alumina, Pure and Doped with Transition Metal Impurities," J. Mater. Sci. **12** 2291 (1977)

Theses

1. H. K. Bowen, "Creep of Magnesia," Senior Thesis, University of Utah (1967).
2. G. R. Terwilliger, "Creep of Polycrystalline Magnesia," Ph.D. Thesis, University of Utah (1968).
3. D. C. Moore, "Hot Pressing Magnesia with an Iron Dopant," Senior Thesis, University of Utah (1970).
4. D. D. Marchant, "Grain Size Distributions and Grain Growth in MgO and MgO-Fe₂O₃ Solid Solutions," Senior Thesis, University of Utah (1968).
5. R. T. Tremper, "The Effect of Nonstoichiometry on the Creep of Iron-Doped Polycrystalline Magnesia," Ph.D. Thesis, University of Utah (1971).
6. G. W. Hollenberg, "The Effect of Oxygen Partial Pressure Changes on the Creep of Alumina Doped with Transition Metal Impurities (Cr, Ti, Fe)," Ph.D. Thesis, University of Utah (1972).
7. P. A. Lessing, "Thermogravimetric Analysis of Ti- and Fe-Doped Al₂O₃," Senior Thesis, University of Utah (1971).
8. P. A. Lessing, "The Creep of Magnesia and Alumina Doped with Transition Metal Impurities," Ph.D. Thesis, University of Utah (1976).
9. J. D. Hodge, "Diffusional Creep Phenomena in Polycrystalline MgO," Senior Thesis, University of Utah (1976).

Invited Presentations

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. "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

The support of this research by the Department of Energy under Contract No. EY-76-S-02-1591.*002 is acknowledged.

VI. PRINCIPAL INVESTIGATOR

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