Contract #C00-2408-1

PRESSURE SINTERING AND CREEP DEFORMATION

-- A JOINT MODELING APPROACH

Progress Report

Michael R. Notis

Lehigh University

Bethlehem, Pennsylvania 18015

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ABSTRACT

Studies have been initiated to compare the densification during later stages of pressure sintering and the creep deformation behavior for two oxide materials, CoO and MgAl_2O_4 , as a function of stress, temperature, grain size and porosity. To date, hot pressed specimens have been fabricated, densification kinetics have been observed and creep specimens have been made. A high temperature dead-load creep facility has been constructed and is now operational.

Studies of the densification kinetics during final stage pressure sintering of MgAl_2O_4 have been interpreted in terms of both phenomenological and atomistic constitutive equations. Densification kinetics indicate non-viscous deformation mechanisms to be operative and the rate controlling process appears to be extrinsic oxygen-lattice diffusion.

Three new and unique deformation (densification)-mechanism maps were developed in order to demonstrate deformation effects. A map in log grain size-temperature space was used to illustrate the effects of microstructure and temperature at constant stress. A map in log stress - log grain size space was developed to demonstrate stress and grain size effects on densification behavior at constant temperature. Finally, maps have been generated in log stress-temperature space that display contours of constant stress exponent across dominant mechanism fields. These maps provide the background necessary for the generation of quantitative three-dimensional deformation maps.

I. Project Scope and Significant Results

The purpose of this research is to compare the densification during the later stages of pressure sintering and the creep deformation behavior for two oxide materials, CoO and MgAl_2O_4 , as a function of stress level, temperature, grain size, porosity and stoichiometry. During the course of the past eight months, emphasis has been placed on three major areas of concern: experimental developments, developments related to interpretation of pressure-sintering kinetics and developments related to the technique of deformation (or densification) mapping.

A. Experimental Developments

The first months of this project were concerned with the fabrication of hot pressed specimens of cobalt oxide and magnesium aluminate spinel. Specimens of both materials have now been produced in a wide variety of final grain size and porosity; densification kinetics have been monitored and evaluated during all of these experiments and the densification rates will ultimately be compared with the creep rates to be measured. Additional specimens of cobalt oxide have been provided to Professor Coble at M.I.T. for studies on diffusion rates in this system. These measurements will ultimately be used in conjunction with the present research.

A high temperature dead-load creep test facility has been constructed, proved-in and calibrated. Hot pressed specimens have been stabilized-annealed and cut into creep specimens; initial creep measurements on cobalt oxide will be made during the next few months.

Studies have been made concerning grain growth effects in hot pressed cobalt oxide. At temperatures less than 1100°C in air grain growth

appears to follow a grain size-squared time law and has a process activation energy of approximately 44,500 kcal/mole. At higher temperatures the grain size exponent was observed to increase but it was not possible to calculate an activation energy because of scatter in the data. In all cases, the grain growth rate constants were quite small, indicative of growth limited by residual porosity.

B. Developments Related to Pressure-Sintering Kinetics of Ceramic Oxides

A study of the pressure-sintering densification kinetics of MgAl_2O_4 has been performed within the temperature range, 1400° - 1525°C and the pressure range, 5000 - 10,000 psi. The results were analyzed in terms of the phenomenological models of Murray et al. and of Wolfe. A "multi-mechanism" analysis procedure was employed to model the densification behavior and the results were also analyzed in terms of atomistic densification mechanisms.

The densification rate equation of Murray, et al., derived from a consideration of Newtonian-viscous densification behavior, was found to be inadequate as a model for the densification behavior of MgAl_2O_4 . The Wolfe rate equation, based on non-viscous densification behavior, provided good agreement between predicted and experimentally observed densification rates. An iterative computer solution technique was devised to determine the effective stress exponent which best fit the experimental behavior. These stress exponents were in the range 1 to 3 depending upon the temperature and applied pressure of the experiment and upon the particular stress correction term used in the analysis.

The multi-mechanism analysis was based on the simultaneous, independent operation of 4 deformation mechanisms: (1) Nabarro-Herring bulk viscous flow, (2) Coble grain boundary viscous flow, (3) grain boundary sliding,

and (4) dislocation climb-controlled deformation. These relationships, originally derived to model creep deformation behavior, were modified for application to pressure-sintering deformation (densification) studies. An analysis technique was developed whereby a value for the lattice diffusion coefficient of the ionic species controlling the densification rate could be calculated from experimental data. The experimental lattice diffusion coefficient was found to be

$$D_L = 4.01 \exp(-103100/RT)$$

and was considered to be a measure of extrinsic lattice diffusion of the oxygen ion.

C. Developments Related to the Technique of Deformation (Densification)

Mapping

Early in the research, emphasis was placed on the inclusion in the maps of deformation mechanisms usually observed in experimental practice but for which dependable constitutive equations have not been available; this would at least provide upper bound limits for the creep rate contributions for processes such as grain boundary sliding. Similarly, we have investigated the incorporation of SEQUENTIAL-DEPENDENT creep mechanisms into deformation maps in contrast to the SIMULTANEOUS-INDEPENDENT mechanisms initially studied by Ashby. A discussion of the importance of these effects is included in the manuscript attached in Appendix I.

It has previously been shown by the present investigator that densification-mechanism maps displaying the dominant densification mechanisms in log stress-temperature space at constant grain size (Figure 1) allow a

comparison of the effects of stress and temperature on the densification and creep deformation behavior of a material. Densification-mechanism mapping techniques have now been developed to illustrate the pertinent relationships on two dimensional planes in log stress-temperature-log grain size space.

Densification-mechanism maps have been developed to display the dominant densification mechanisms in log stress - log grain size space at constant temperature (Figure 2). This type of map illustrates areas of dominance for mechanisms which might not appear on maps in log stress - temperature space. This was the case in the study of MgAl_2O_4 where at $0.3 \mu\text{m}$ the grain boundary sliding mechanism did not appear as a dominant field. However at 1500°C , GBS is shown to have a field of dominance above $1 \mu\text{m}$.

Densification-mechanism maps displaying the dominant densification mechanisms in log grain size - temperature space at constant stress (Figure 3) have been developed to facilitate a comparison of the effects of grain size and temperature on the mode of densification. This type of map forms a complimentary series with maps in log grain size - log stress space and in log stress-temperature space which can be used to illustrate relationships between dominant mechanisms under any combination of stress, temperature, and grain size.

The work performed in conjunction with the generation of three different types of deformation maps described above provides the foundation necessary to produce three-dimensional deformation maps. This will be one of our major areas of emphasis during the following year.

Deformation (densification)-mechanism maps have also been developed to display contours of constant stress exponent across dominant mechanism fields in log stress-temperature space (Figure 4). These maps have proven useful in the present research by their illustration of the changes in stress exponents expected from changes in levels of stress. Investigators studying densification or creep behavior of materials should find this type of map valuable in their correlation of the stress exponents for the deformation process with the operation of diffusional flow or dislocation mechanisms.

Figure 1
Deformation Map in Stress-Temperature Space

Figure 2

Deformation Map in Stress-Grain Size Space

Figure 3
Deformation Map in Grain Size - Temperature Space

Figure 4
Deformation Map in Stress-Temperature Space
with Contours of Constant Stress Exponent

II. Scientific Personnel Performing Research on this Current Project

1. M. R. Notis, Principal Investigator, Assistant Professor in the Department of Metallurgy and Materials Science and Senior Staff Member of the Advanced Materials Laboratory of the Materials Research Center -- 2.0 man-months as of August 31, 1974; estimated 2.8 man-months total at the end of the contract year.
2. V. Krishnamachari, Postdoctoral Research Assistant -- 6.5 man-months as of August 31, 1974; estimated 10.5 man-months total at the end of the contract year. Dr. Krishnamachari has concentrated on construction of a high temperature dead load creep testing facility during the past few months and has guided the production of hot pressed specimens to be used in creep testing and has spent considerable time on computer analysis of hot pressing data.
3. P. Urick, Research Engineer -- 3 man-months. Mr. Urick was responsible for the preparation of powders and for the fabrication of the hot-pressed specimens used in this study.
4. C. Vinante, Laboratory Assistant -- 0.75 man-months. Mr. Vinante is a member of the Center for Applied Mathematics at Lehigh and has helped with the programming related to two-dimensional deformation maps.
5. R. Green, Laboratory Assistant -- 0.5 man-months. Mr. Green is a member of the Department of Chemical Engineering and has helped with the computer programming of three-dimensional views.

6. R. H. Smoak, Research Assistant -- unpaid. Mr. Smoak is completing his Ph.D. requirements and has applied the mapping procedures developed by Mr. Vinante to his work on spinel.
7. D. Knorr, Undergraduate Project Assistant -- unpaid. Mr. Knorr has worked with the Principal Investigator to study the inclusion of grain boundary sliding mechanisms and sequential deformation mechanisms into the deformation maps.
8. C. Gasdaska, Undergraduate Summer Assistant -- unpaid. Mr. Gasdaska performed experimental work related to grain growth effects in cobalt oxide.