# INTERDISCIPLINARY RESEARCH CONCERNING THE NATURE AND PROPERTIES OF CERAMIC MATERIALS

NASA RESEARCH GRANT NUMBER NGL 48-002-004

Semiannual Status Report Number 16

December 16, 1970 - June 15, 1971

UNIVERSITY OF WASHINGTON

College of Engineering

Ceramic Engineering Division

James I. Mueller

Professor, Ceramic Engineering

Principal Investigator

UNIVERSITY OF WASHINGTON
SEATTLE, WASHINGTON 98105

*Department of Mining,*
*Metallurgical,*
*and Ceramic Engineering*

June 30, 1971

To All Concerned:

This is the sixteenth semi-annual status report submitted under the National Aeronautics and Space Administration Grant NGL 48-002-004. In our last report, dated January, 1971, we used a new format which, generally, was well received. Now, as part of a continuing effort to improve our methods of communication, we are modifying the content and format of our June report.

In the past, we have attempted to indicate the status of all of our research projects in every semi-annual report. A review of these efforts, however, shows that the most significant research accomplishments seem to come during the middle of each calendar year. Thus, although we recognize our responsibility to submit semi-annual reports, we suggest that we may serve our readers better by emphasizing research in the December report and by focusing on the operational details of our program in the June report. We plan to make our normal distribution of the former but to send the June report only to NASA personnel and to members of our technical monitoring committee.

I wish to express my appreciation to Professor Myron L. White of our Humanistic-Social Studies Department for his suggestions and assistance in the development of our new reporting methods. We hope that, with these changes, we can continue to report effectively on our program and yet reduce unnecessary repetition. If the response to the present report is as favorable as that we received last January, we shall feel that we have moved another step toward our goal of improved communication.

Sincerely yours,

James I. Mueller
Principal Investigator

JIM/cm

Enc.

TABLE OF CONTENTS

LIST OF TABLES

# OPERATIONAL REPORT

## Introduction

This report deals with the operation in 1970-71 of the Ceramic Materials Research Program at the University of Washington. The program is supported by Grant NGL 48-002-004 from the National Aeronautics and Space Administration for "Interdisciplinary Research on the Nature and Properties of Ceramic Materials."

As in the past, the planning and coordination of the program has been supervised by the Ceramic Materials Research Committee consisting of James I. Mueller, Ceramic Engineering, Chairman and Principal Investigator; Thomas F. Archbold, Metallurgical Engineering; J. Gregory Dash, Physics; Billy J. Hartz, Civil Engineering; Irene C. Peden, Electrical Engineering; and William D. Scott, Ceramic Engineering. Administration of the program has been supervised by a board consisting of H. Myron Swarm, Associate Dean of the College of Engineering, Chairman; Edward C. Lingafelter, representing the Dean of the Graduate School; Douglas H. Polonis, Chairman of the Department of Mining, Metallurgical and Ceramic Engineering; and James I. Mueller, Principal Investigator. Members of both groups are appointed by the Dean of the Graduate School.

During 1970-71, the program operated for the first time at a reduced level of funding. Nevertheless, research supported by the grant actively continued in all areas. Actually, the total monies which were expended exceeded the $250,000 allocated, but the additional funds were those carried over from 1969-70 so that a large capital expenditure might be made

for a scanning electron microscope. Acquisition of this equipment increased the program's research capability, and arrangements for its operation have extended interdisciplinary relationships. The educational benefits of the grant at this institution continued to be and have been extended to NASA personnel at the centers. In addition, the content and format of the December report were changed; the favorable response to the change has resulted in a decision to continue the new style of this report.

## Research

### General Program

The Research Committee attempted to give maximum support to the research program by supporting graduate students. Consequently administrative and summer faculty funds were reduced, and the full-time post-doctoral position in ceramic engineering was eliminated. The result was that, in 1970-71, research projects were continued at nearly the same level as in 1969-70. Table I compares the level of activity for the two years.

TABLE I. COMPARISON OF RESEARCH ACTIVITY, 1969-70 and 1970-71

| | 1969-70 | 1970-71 |
|---|---|---|
| Number of Projects | 39 | 33 |
| Number of Academic Disciplines | 7 | 7 |
| Number of Faculty Supervisors | 21 | 17 |
| Number of Research Faculty | 2 | 1 |
| Number of Graduate Students | 33 | 38 |
| (MS) | (9) | (15) |
| (Ph.D.) | (24) | (23) |

Although, as Table I shows, the number of projects dropped somewhat in 1970-71, the number of academic disciplines involved in the program remained the same. Moreover, faculty and graduate students of these disciplines carried on projects which were well distributed amoung the program's major research areas: chemical environment, mechanical properties, surface phenomena, atomic and molecular effects, and processing. Details of this distribution appear in Table II. Appendix A (p. 11) provides a list of the research projects which also identifies the faculty and graduate students participating and includes the objectives of each project.

TABLE II. DISTRIBUTION OF PROJECTS BY DISCIPLINE AND RESEARCH AREA, 1970-71

| | Number of Projects | Chem. | Mech. | Surface Phenomena | Atomic & Molecular | Process. | Miscl. |
|---|---|---|---|---|---|---|---|
| Ceramic Engineering | 22 | 3 | 3 | 4 | 5 | 4 | 3 |
| Chemistry | 3 | 2 | - | - | 1 | - | - |
| Civil Engineering | 2 | - | 2 | - | - | - | - |
| Electrical Engineering | 1 | - | - | - | 1 | - | - |
| Mechanical Engineering | 1 | - | 1 | - | - | - | - |
| Metallurgical Engineering | 2 | 1 | 1 | - | - | - | - |
| Physics | 2 | - | - | - | 2 | - | - |
| TOTAL | 33 | 6 | 7 | 4 | 9 | 4 | 3 |

Table I also indicates for 1970-71 a decrease in the number faculty involved. As noted above, however, this decrease was largely the result of planning, and its effect was partially offset by the larger number of graduate students who were engaged in research supported by the grant. A breakdown of faculty and graduate student participation is shown in Table III.

TABLE III. NUMBER OF STUDENTS AND FACULTY IN GRANT-SUPPORTED RESEARCH

| | Number of Projects | Faculty | Research Faculty | Under-grads | MS | Ph.D. | Total Grads |
|---|---|---|---|---|---|---|---|
| Ceramic Engineering | 22 | 5 | 1 | 1 | 9 | 13 | 22 |
| Chemistry | 3 | 3 | - | - | - | 3 | 3 |
| Civil Engineering | 2 | 2 | - | - | 1 | 1 | 2 |
| Electrical Engineering | 1 | 1 | - | - | 1 | 1 | 2 |
| Mechanical Engineering | 1 | 1 | - | - | 1 | - | 1 |
| Metallurgical Engineering | 2 | 3 | - | - | 3 | 1 | 4 |
| Physics | 2 | 2 | - | - | - | 4 | 4 |
| TOTAL | 33 | 17 | 1 | 1 | 15 | 23 | 38 |

Perhaps the momentum of research activity which was maintained in the past year under reduced funding is a mark of the program's maturity. In any event, evidence for this maturity is to be found in the number of papers which resulted during 1970-71 from many of the research projects. Table IV indicates by academic discipline the number of these papers which were published, and it shows, as well, the increase of publication in the professional literature which has taken place over the last three years. In addition,

Appendix B (p. 24) lists the authors, titles, and other details not only of the papers published in 1970-71 but also of the papers which were presented at professional meetings, the papers which have been accepted for publication, and those which have been submitted.

TABLE IV. NUMBER OF PAPERS PUBLISHED IN 1970-71 AND THE TWO PRECEDING YEARS

| | 1968-69 | 1969-70 | 1970-71 |
|---|---|---|---|
| Ceramic Engineering | 2 | 1 | 9 |
| Civil Engineering | - | 2 | - |
| Electrical Engineering | - | 1 | 1 |
| Metallurgical Engineering | 1 | 1 | 4 |
| Physics | 5 | 2 | 1 |
| TOTAL | 8 | 7 | 15 |

## Refractory Surface Insulation (RSI) Program

Over and above the effort devoted to the general research program, special attention was given to ceramic surface insulation when it became apparent early in the last year that such insulation might possibly be used for space shuttle vehicles. It was evident that a lack of fundamental knowledge existed regarding the nature and properties of the materials involved. Thus, Professor Mueller visited several of the NASA research centers and contractors concerned with the development of these materials and was asked to accompany an *ad hoc* NASA task committee in order to determine the "state of the art" in this area.

In the Fall of 1970, a small project was begun at the University of Washington in order to study the effects of time and temperature upon the recrystallization and morphology of quartz and mullite fibers. This project was carried on principally by ceramic engineering faculty members and graduate students as an "extra-effort," although one student was supported for a period of twelve weeks.

The increased interest in ceramic insulation resulted in additional contract funding to NASA research centers and contractors for developing and optimizing these materials. In March, 1971, the University submitted a single proposal to all NASA research centers, requesting support for an accelerated effort to obtain the fundamental information believed to be necessary. Recently, the NASA-Ames Research Center approved a contract for this work, which will support a total of one postdoctoral research associate and three graduate students for a period of approximately 15 months. OART also approved a supplement of $3,000 to the basic grant in order to cover the additional travel which Professor Mueller performed in connection with the RSI program. A list of the facilities which he visited is given in Appendix C (p. 28).

## Research Capability

During the past year, grant funds for 1970-71 and funds carried over from 1969-70 were used to purchase a Cambridge Scanning Electron Microscope. This, together with the x-ray diffraction and fluorescent spectrographic equipment, electron microscope, and electron microprobe, provides a wide capability for analyzing all types of materials.

It was believed that all of this equipment would be used more efficiently if a full-time operator were employed. However, decreases in grant and university funds have precluded their use for this purpose. Therefore, a Materials Analysis Cost Center was established in April, 1971. Under this arrangement, any University program may use the equipment on an approved rate schedule, and everyone is charged the same rate, regardless of source of funds. The income thus obtained will be used for the operator's salary and fringe benefits, equipment maintenance and up-dating, and standard supplies required for operation.

The initial results of the new arrangement have been very encouraging, for additonal interdisciplinary relationships are developing with faculty members and graduate students from the life and physical sciences and from the other engineering departments. These results underline the importance of using grant funds to purchase capital equipment which not only increases research capability but also enhances interdisciplinary dialogue.

## Education

If the communication of research results reached a high point in 1970-71, so also did the benefits which the program provides in the education of graduate students. During the past year, 8 Master's degrees and 5 Doctor's degrees were awarded to students who have been supported by the NASA grant; both figures give further evidence of the program's maturity. Table V indicates by academic discipline the numbers of students who earned either the MS or PhD last year; in addition, it indicates the growth in these numbers over the last three years.

TABLE V. NUMBER OF GRADUATE DEGREES AWARDED

(Figures reflect awards to only those students supported by the NASA grant)

| | 1968-69 | | 1969-70 | | 1970-71 | |
|---|---|---|---|---|---|---|
| | MS | PhD | MS | PhD | MS | PhD |
| Aeronautics & Astronautics | - | - | - | 1 | - | - |
| Business Administration | - | - | - | 1 | - | - |
| Ceramic Engineering | - | - | 1 | 4 | 5 | 3 |
| Chemistry | - | - | - | 1 | - | 1 |
| Electrical Engineering | 1 | - | - | - | 1 | - |
| Mechanical Engineering | - | - | 1 | - | - | - |
| Metallurgical Engineering | 2 | 1 | 2 | - | 2 | - |
| Physics | - | - | - | - | - | 1 |
| TOTAL | 3 | 1 | 4 | 7 | 8 | 5 |

A list of students receiving degrees in 1970-71 and the degrees they earned is in Appendix D (p. 29); details concerning their theses are to be found in the final section of Appendix B (p. 27).

As in past years, the activities of 1970-71 also included special educational efforts. The Ceramic Materials Research Seminar continued throughout the year, covering a wide variety of topics. Speakers included 2 visitors and 11 students. A complete list of speakers and their topics will be found in Appendix E (p. 30).

Funds from the grant were used to supplement University support of a visiting professor during the summer quarter of 1970. Dr. D. L. Johnson, Professor of Materials Science, Northwestern University, offered a four-week, three-credit course entitled "Sintering and Other Capillarity Phenomena in Materials." Twenty students were enrolled.

At the request of the NASA-Langley Research Center, Professor David B. Fischbach offered a 1-week course entitled "Carbon Materials" in December, 1970. A total of 20 Langley personnel participated.

## Reporting Methods

Finally, there has been a determined effort this last year to study and improve the reporting of the research program. As the letter of transmittal has already indicated, the format and content of this report and its predecessor have been modified experimentally in an attempt to achieve better communication. The basic content of the December report consisted only of research project titles, the names of research personnel, and statements of project objectives. Accompanying this material was a return questionnaire by means of which recipients could request an individual detailed report on any or all of the projects listed. This questionnaire, a copy of which is included as Appendix F (p. 31), also requested from the respondents some additional information including the number of individuals to which each report was circulated, a statement as to whether or not the respondents wished to continue receiving future reports, and any comments which they might wish to volunteer. A summary of the responses to these questionnaires may be found in Appendix F (pp. 32-34).

A large majority of the persons receiving the December report reacted very favorably to its attempt at individualized reporting, which eliminates the need to wade through a bulky document in order to obtain the information a reader really wants. Consequently, the next December's report will have the same content and format. As before, however, all copies of the report sent to NASA Headquarters and to the program's technical monitors

will include the detailed reports, as well as the basic document.

Although this report does not contain a questionnaire, comments on and reactions to its new designs will be very welcome.

Appendix A

## LIST OF PROJECTS, PERSONNEL AND OBJECTIVES

### CHEMICAL ENVIRONMENT

The zirconium-oxygen-carbon system was selected as the subject for study of the effects of the chemical environment upon ceramic materials in order that a problem area could be defined for interested faculty members.

#### Ultrasoft X-ray Emission Studies

Faculty Supervisor: Alan D. Miller
Assistant Professor, Ceramic Engineering

Graduate Assistant: James W. Rue, Ph.D. Candidate
Predoctoral Research Associate II

Objective: The purpose of the study is to develop an ultra-soft X-ray spectrometer capable of measuring the X-ray spectrum of the carbides and oxycarbides of zirconium in the soft region (10-100Å). Of particular interest are the spectra resulting from transitions involving electrons in bonding states. The information contained in these spectra is of interest in helping to describe the state of chemical bonding in the compounds.

#### Raman Studies of Ceramic Materials

Faculty Supervisor: John W. Macklin
Assistant Professor, Chemistry

Graduate Assistants: Jack G. Surendranath, Ph.D. Candidate
Research Assistant

Dagmar Cronn, Ph.D. Candidate
Research Assistant

Objective: The purpose of this project is to measure the Raman spectra of ceramic materials to the end of understanding the character of their bonding.

## Zirconium Oxidation

Faculty Supervisor: Thomas F. Archbold
Associate Professor, Metallurgical Engineering

Graduate Assistant: Ramgopal Darolia, Ph.D. Candidate
Research Assistant

Objective: This research project has been involved with the determination of the characteristics and mechanisms of the early stages of oxidation of zirconium metal.

## High Temperature Calorimetry

Faculty Supervisor: Alan D. Miller
Assistant Professor, Ceramic Engineering

Graduate Assistant: John A. Negrych, Ph.D. Candidate
National Science Foundation Trainee

Objective: The purpose of this study is to develop a high-temperature isothermal drop calorimeter for use in measuring the heat contents of ceramics and related compounds. The current objective is the measurement of heat capacities of zirconium carbide as a function of temperature and carbon content.

## Gas-Solid Equilibria

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Objective: The composition and pressure of the gaseous phase(s) associated with the solid phases at various temperatures materially affect the equilibrium of a system. It is the purpose of this research to study the effects of these variables upon the Zr-O-C system. (Currently inactive.)

## Solid-Solid Equilibrium

Faculty Supervisor: Norman W. Gregory
Professor, Department of Chemistry

Objective: The objective of this research is to study kinetic and thermodynamic properties of the reactions in oxide, carbide and graphite systems by torsion effusion measurement of steady state pressures of gases generated in effusion cells. (Currently inactive.)

# MECHANICAL PROPERTIES

All ceramic materials are affected by severe environments. Research in these categories is directed toward fundamental studies on the fracture and deformation characteristics.

## ZrC Coatings

Faculty Supervisor: Colin J. Sandwith
Assistant Professor, Mechanical Engineering

Graduate Assistant: Leonard Johnson, M. S. Candidate
Research Assistant

Objective: The coating-metal interface shear strength and substrate strain that produces permanent deformation in the coating are being evaluated by tensile tests. The long term objective of these tests is to provide parameters for designing with plasma flame sprayed coatings from an easily reproduced specimen.

## Defect Properties of Ionic and Ceramic Crystals

Faculty Supervisors: Thomas G. Stoebe
Associate Professor, Metallurgical Engineering

Richard R. Zupp
Assistant Professor, Metallurgical Engineering

Graduate Assistants: H. L. Fotedar, Ph.D. Candidate
Predoctoral Research Associate II

M. Srinivasan, Ph.D. Candidate
Predoctoral Research Associate I

H. Vora, M.S. Candidate
Research Assistant

Objective: This project concerns the growth and characterization of single crystals of the NaCl structure. Current work includes studies of mechanical deformation parameters in high purity LiF, and crystal growth and mechanical property studies in MgO.

## Mechanical Behavior of Carbon Fibers

Faculty Supervisor: David B. Fischbach
Research Associate Professor, Ceramic Engineering

Graduate Assistant: Roy E. Henrichsen, Ph.D. Candidate
Predoctoral Research Associate I

Objective: Investigate the dynamic modulus and internal friction behavior of carbon fibers.

## Continuum Stress Analysis of Ceramic Materials

Faculty Supervisor: B. J. Hartz
Professor, Civil Engineering

Graduate Assistant: Michael Held (Unsupported), M.S. Candidate
Part-time Graduate Student, Civil Engineering

Objective: To couple the computer oriented numerical "Finite Element Method" of stress analysis with current experimental work in crystalline ceramic materials for a better understanding of materials behavior and properties and to subsequently invert this process to bring the analytical tools and materials research to bear on the Engineering design problem.

## Thermal Stresses in Crystal and Ceramics

Faculty Supervisor: Max D. Coon
Assistant Professor, Civil Engineering

Graduate Assistant: Maurice B. Cooper, Ph.D. Candidate
Predoctoral Research Associate I

Objective: The objective of this study is to make use of the ductility of ceramics at high temperatures to introduce residual stresses in such a way as to improve the load carrying capacity of structural elements.

## Strain Energy in Crack Propagation

Faculty Supervisor: Robert J. Campbell
Assistant Professor, Ceramic Engineering

Graduate Assistant: Sang Moo Park, M.S. Candidate
Research Assistant

Objective: An attempt is being made to correlate energy involved in propagation of cracks through polycrystalline bodies. $BaTiO_3$ bodies were chosen for models because previous work had suggested limited plastic behavior.

## Nondestructive Determination of Residual Stresses

Faculty Supervisor: Robert J. Campbell
Assistant Professor, Ceramic Engineering

Graduate Assistant: Derrile K. Thayer, M.S. Candidate
Research Assistant
Orton Ceramic Foundation Fellow

Objective: This research is aimed at proving the feasibility of determining porosity in bodies by non-destructive techniques. The use of ultrasonic pulse transmission related to elastic modulus at ambient and high pressures is being pursued.

# SURFACE PHENOMENA

The importance of Surface Phenomena on the nature and properties of ceramic materials justifies our research on surface energy. Although previously included in other areas in previous reports these projects are separately identified in this report.

## Interfacial Energy of Low Angle Tilt Boundaries

Faculty Supervisor: William D. Scott
Associate Professor, Ceramic Engineering

Graduate Assistant: G. Achutaramayya, Ph.D. Candidate
Predoctoral Associate II

Objective: The purpose of this project is to determine the relative interfacial energy of low angle dislocation wall tilt boundaries formed by polygonization of deformed aluminum oxide crystals.

## Mobility of Grain Boundaries in Alumina

Faculty Supervisor: William D. Scott
Associate Professor, Ceramic Engineering

Graduate Assistant: Robert W. Burns, Ph.D. Candidate
Predoctoral Associate II

Objective: To measure the mobility of well-characterized low angle grain boundaries under the influence of surface energy and electric field driving forces.

## Relative Energy of Deformation Twin Boundaries in Alumina

Faculty Supervisor: William D. Scott
Associate Professor, Ceramic Engineering

Graduate Assistant: O. M. Bhandari, M.S. Candidate
Research Assistant

Objective: To evaluate technique of thermal grooving as a method of measuring interfacial energy and to measure twin boundary energy in alumina.

## Surface Diffusion Studies

Faculty Supervisor: Alan D. Miller
Assistant Professor, Ceramic Engineering

Graduate Assistant: Edward H. Randklev, Ph.D. Candidate
Predoctoral Research Associate II

Objective: This study is concerned with the phenomenon of linear thermal faceting on surfaces of single crystal aluminum oxide. The study of the growth and morphology of these facets is expected to provide information relative to surface diffusion rates in specific crystallographic directions.

# ATOMIC AND MOLECULAR EFFECTS

Research in this area consists of studying the electronic properties of ceramic materials, principally metal carbides or related structures, in an attempt to gain further understanding of atomic bonding and charge transfer. Also, research on radiation effects upon ceramic materials is included.

## U-V Photolysis-Radiation Ceramic Studies

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Graduate Assistant: Coimbatore S. Krishnan, Ph.D. Candidate
Predoctoral Research Associate I

Objective: To understand the luminescence mechanism of impurity ions in ultra violet irradiated MgO single crystals using thermoluminescence and electron spin resonance techniques with compensating ions.

## Irradiation Damage of Sapphire & Ruby

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Graduate Assistant: John M. Rusin, Ph.D. Candidate
Predoctoral Research Associate I

Objective: The purpose of this research is to study the pre-existing defects in sapphire and the effect of U-V colors on optical degradation in ruby.

Domain Dynamics In Isomorphous Ferroelectrics

Faculty Supervisor: John L. Bjorkstam
Professor, Electrical Engineering

Graduate Assistants: Guy J. Adriaenssens, Ph.D. Candidate
Predoctoral Research Associate II

James A. Aikins (Unsupported), M.S. Candidate
Research Assistant, Electrical Engineering

Objective: To relate cooperative atomic effects near the phase transition in $KH_2PO$ and (KDP) type ferroelectrics to macroscopic dielectric phenomena; particularly domain formation and propagation.

Studies on GASH

Faculty Supervisor: E. C. Lingafelter
Professor, Chemistry

Graduate Assistant: Louis P. Torre, Ph.D. Candidate
Predoctoral Research Assistant

Objective: To use x-ray crystallography to determine the structural changes which occur when the ferroelectric material Guanidinium Chromium Sulfate Hexahydrate is subjected to an electric field, and to infer the mechanism for the rearrangements. Hysteresis loops of the crystal will be monitored to indicate change in the dielectric properties

White Allotropic Graphite

Faculty Supervisor: David B. Fischbach
Research Associate Professor
Ceramic Engineering

Graduate Assistant: Robert M. Haugen, M. S. Candidate
Union Carbide Grant-in-Aid

Objective: Attempt to verify the existence of a reported new allotrope of crystalline carbon and define conditions under which it forms.

The Magnetic Behavior and Structure of Carbon Fibers

Faculty Supervisor: David B. Fischbach
Research Associate Professor, Ceramic Engineering

Graduate Assistant: C. Bruce Scott, M.S. Candidate
Research Assistant

Objective: To measure the diamagnetic susceptibility of carbon fibers and relate it to the structure to obtain further insight into the nature of these unique materials.

Mössbauer Studies

Faculty Supervisor: Robert L. Ingalls
Associate Professor, Department of Physics

Graduate Assistants: Gerald A. Erickson, Ph.D. Candidate
Predoctoral Research Associate II

John R. Nett, Ph.D. Candidate
Research Assistant

C. D. West, Ph.D. Candidate
Predoctoral Research Associate II

Objective: One object of this program is to apply the Mössbauer effect to study properties of various materials with emphasis on atomic force constants interatomic potentials, electron valence states and distributions.

Liquid Glass Phase Transition Study

Faculty Supervisor: Edward A. Stern
Professor, Department of Physics

Graduate Assistant: Dale Sayers, Ph.D. Candidate (Unsupported)
Predoctoral Research Associate

Objective: A program of specific heat and extended x-ray absorption fine structure measurements of amorphous solids is also in progress. Its purpose is to examine the nature of the liquid to glassy state transition.

Impurity Diffusion in MgO Under the Influence of an Electric Field

Faculty Supervisor: William D. Scott
Associate Professor, Ceramic Engineering

Graduate Assistant: Chester A. Hinman (on leave) Ph.D. Candidate
Ceramic Engineering

Objective: The purpose of this project was to measure the diffusion of Ni in MgO at high temperatures in an electric field. Initial experiments disclosed anomalous reduction of the nickel impurity and apparent electrolysis of the MgO under the applied fields. The project was then modified to investigate D.C. conductivity mechanisms.

# PROCESSING

Research in this area is intended to gain basic information on processes used for fabricating ceramics.

## Compaction

Faculty Supervisor: O. J. Whittemore, Jr.
Professor, Ceramic Engineering

Graduate Assistants: Daniel B. Leiser, Ph.D. Candidate
Predoctoral Research Associate II

Arun K. Chattopadhyay, M.S. Candidate
Research Assistant

Objective: To study compaction of ceramic particles. Statistical analyses are being made of the interrelationships of variables in compaction including; material, particle size, particle shape, compaction rate.

## Sintering

Faculty Supervisor: O. J. Whittemore, Jr.
Professor, Ceramic Engineering

Graduate Assistants: J. Joseph Sipe, Ph.D. Candidate
Predoctoral Research Associate II

K. Aihara, Ph.D. Candidate
Research Assistant

Objective: To study the initial stage of sintering where pore growth occurs. Pore growth has been shown to occur during the initial sintering of $Fe_2O_3$ and MgO, and under certain conditions, $Al_2O_3$.

## The Graphitization Process

Faculty Supervisor: David B. Fischbach
Research Associate Professor
Ceramic Engineering

Objective: The influence of various microstructural and processing parameters (initial microstructure, internal stress, hot deformation, etc.) on the development of crystalline order in initially disordered carbons is being investigated to gain a better understanding of graphitization process.

## The Structure and Graphitization Behavior of Pyrolytic Carbons Deposited in a Fluidized Bed

Faculty Supervisor: David B. Fischbach
Research Associate Professor
Ceramic Engineering

Graduate Assistant: Ronald L. Beatty, Ph.D. Candidate
Union Carbide Predoctoral Fellow

Objective: To investigate the influence of initial as-deposited microstructure on the graphitization behavior of pyrolytic carbons with a wide range of structures.

## MISCELLANEOUS

Research listed in this category does not necessarily fall under one of the energy-environment areas. Certain research projects included herein are used as pilot studies for possible future expansion.

### Carbon as a Biomedical Material

Faculty Supervisor: David B. Fischbach
Research Associate Professor, Ceramic Engineering

Graduate Assistants: J. Nilles
Research Associate, Biomedical Engineering

M. Lapitsky, M.S. Candidate
Research Assistant, Office of Engineering Research

Objective: The suitability of carbons and graphites as bone implant materials is being investigated with special attention to factors affecting bone ingrowth and adherence.

### Devitrification of Glass to Ceramic Seals

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Graduate Assistant: Alan T. Dull, M.S. Candidate
Tektronix, Research Fellow

Objective: The purpose of this research is to determine the mechanism of devitrification of glasses and for glass to ceramic seals.

### Materials Characterization

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Graduate Assistant: Alan T. Dull, M.S. Candidate
Research Assistant

Objective: This was a pilot study on the nature and properties of ceramic fiber materials and the effects of time and temperature upon their devitrification. (Project participants included four members of the ceramic engineering faculty and 6 otherwise supported graduate students.)

# REFRACTORY SURFACE INSULATION

This research, initiated as a project with grant support, is currently being funded by a contract with NASA Ames Research Center. Its objective is to obtain fundamental information which will lead to a proper understanding of materials being considered for space shuttle whose characteristics and properties may be affected by the severe environment to which they may be exposed in use.

## Effect of Thermal Environments on Fibers and Uncoated Tiles

Faculty Supervisor: James I. Mueller
Professor, Ceramic Engineering

Research Associate: Daniel B. Leiser
Research Associate, Ceramic Engineering

Graduate Assistants: Alan D. Dull, M.S. Candidate
Research Assistant

Jerry Jermann, M.S. Candidate
Research Assistant

Objective: To determine the effect of time and temperature on devitrification reaction properties and morphology of "as-received" fibers and to study the effects of processing on these materials.

# Appendix B

## PAPERS AND PUBLICATIONS

### PAPERS PUBLISHED

"Fracture Induced in $Al_2O_3$ Bicrystals by Anisotropic Thermal Expansion," Henry Y. B. Mar and W. D. Scott, Journal of the American Ceramic Society 53 (10) 555-558 (1970).

"Effect of Impurities on the Mechanical Behavior of MgO," M. Srinivasan and T. G. Stoebe, J.A.P. 41 3726, August 1970.

"Effects of Lattice Defects on Thermoluminescence in Lithium Fluoride Crystals," T. G. Stoebe, G. M. Guilmet and J. K. Lee, Radiation Effects 4 189 (1970).

"Single-Crystal Growth of Magnesium Oxide by the Flux Method," H. Vora and R. R. Zupp, Materials Research Bulletin 5, 977-982 (1970).

"Dynamics In Isomorphous Ferroelectrics," J. L. Bjorkstam, J. Phys. Soc. Japan 28, Supplement, 101 (1970).

"Mössbauer Study of Low Temperature Anharmonicity in $ThO_2$: $Co^{57}$," H. Shechter, J. G. Dash, G. A. Erickson and R. Ingalls, Physical Review 28 613 (1970).

"Preparation of Samples for Sintering of submicron Particles by Transmission Electron Microscopy," J. J. Sipe and O. J. Whittemore, Journal of the American Ceramic Society 53, 9, 525 (1970).

"Compaction Behavior of Ceramic Particles," D. B. Leiser and O. J. Whittemore, Jr., American Ceramic Society Bulletin, 49, 8, 714-7 (1970).

"Effect of Work Hardening, Strain Rate and Impurity Content on the Work Hardening Characteristics of LiF Single Crystals," H. L. Fotedar, T. G. Stoebe, Philosophical Magazine, 23, 859-867 (1971).

"The Kinetics and Mechanism of Graphitization," D. B. Fischbach, Chemistry and Physics of Carbon, P. L. Walker, Jr., editor (Dekker, New York) vol. 7, pp 1-105, February, 1971.

"The Graphitization Process," D. B. Fischbach, Tauso (Japanese Carbon Journal) late 1970 and early 1971, pp. 115-120 (exact reference not yet available).

"Tensile Creep Behavior of Glassy Carbon," D. B. Fischbach, Carbon 9, 193-203 (1971).

"An Economical and Demountable Diffusion-Pump Cold Trap," W. M. Ziniker, The Journal of Vacuum Science and Technology, Vol 7, No. 6, Nov./Dec. 1970.

"Appropriate Ceramic Materials Research at the University of Washington," James I. Mueller, Proceedings of the 17th Annual Composite Working Group Meeting, 1970.

"A Lime Refractory for High Temperature Use," J. T. Benson, O. J. Whittemore, Jr., American Ceramic Society Bulletin, 50, 6 545 (1971).

# PAPERS PRESENTED

"Current Problems in Interpretation of Ultra-soft X-ray Spectra," J.W. Rue and A.D. Miller, presented at the 23rd Pacific Coast Regional Meeting of the American Ceramic Society, San Francisco, October 30, 1970.

"Chemical Bonding in Refractory Metal Compounds," A.D. Miller presented at the 23rd Pacific Coast Regional Meeting of the American Ceramic Society, San Francisco, October 30, 1970.

"The Mechanism of Graphitization," presented at the joint NSF-JSPS Summer Seminar on Carbon Materials, Wakayam, Japan, 3-5 September, 1970.

"Pore Growth During the Initial Stages of Sintering, " J.J. Sipe, O.J. Whittemore, Jr. presented at the American Ceramic Society Meeting, Basic Science Division, Chicago, Illinois, April, 1970.

"Ceramic Powder Compaction by Particle Fracture Using a Glass Sphere Model," D.J. Calkins, O.J. Whittemore, Jr., presented at the American Ceramic Society Annual Meeting, Refractories Division, Chicago, Illinois, April, 1971.

"Specimen Geometry Effects and Deformation Characteristics in Ionic Crystals," H.L. Fotedar, T.G. Stoebe, presented at the Spring Meeting of the Metallurgical Society, Atlanta, Georgia, May, 1971.

"Deformation Characteristics of LiF Single Crystals, H.L. Fotedar, T.G. Stoebe, presented at the Spring Meeting of the Metallurgical Society, Atlanta, Georgia, May, 1971.

"Ceramic Surface Insulation for Space Shuttle," J.I. Mueller, presented at the 25th Tri-Section Meeting of the Pacific Northwest sections of the American Ceramic Society in Portland, May 1971.

## PAPERS ACCEPTED FOR PUBLICATION

"Work Hardening Characteristics of LiF Single Crystals," H. L. Fotedar and T. G. Stoebe, Phil. Magazine in press.

"The Graphitization Process," D.B. Fischbach, Tauso, ( Japanese Carbon Journal).

"Compressive Creep of Aluminum Oxide Single Crystals," W.D. Scott, R.L. Bertolotti, Journal of the American Ceramic Society.

## PAPERS SUBMITTED FOR PUBLICATION

"Ceramic Powder Compaction Studies Using a Glass Sphere Model," D.J. Calkins and O.J. Whittemore, Jr., submitted for presentation at the annual meeting of the American Ceramic Society, May 1971.

"Specimen Geometry Effects and Deformation Characteristics in Ionic Crystals," H.L. Fotedar, M. Srinivasan, D.A. Wilson and T.G. Stoebe.

# THESES PUBLISHED

"Low Frequency Dielectric Relaxation in Potassium Dihydrogen Arsenate,"
James A. Aikins, M.S., Electrical Engineering

"Interfacial Energy of Twin Boundaries in Aluminum Oxide,"
Om Bhandari, M. S., Ceramic Engineering

"Melting of Zirconia by Electron Beam Heating and Calculation of Surface Tension of Liquid Zirconia,"
Ramgopal Darolia, M. S., Metallurgical Engineering

"Devitrification of Lead Zinc Borosilicate Solder Glass,"
Alan Dull, M.S., Ceramic Engineering

"The Mössbauer Effect of $Fe^{57}$ in Thorium Oxide,"
Gerald Erickson, Ph.D., Physics

"Variables in Ceramic Powder Compaction,"
Daniel B. Leiser, Ph.D., Ceramic Engineering

"Thermoluminescence in Ultraviolet Irradiated Magnesium Oxide Single Crystals,"
Jack K. Merrow, Ph.D., Ceramic Engineering

"Fracture Behavior of Polycrystalline $BaTiO_3$,"
Sang Moo Park, M.S., Ceramic Engineering

"The Magnetic Behavior and Structure of Carbon Fibers,"
C. Bruce Scott, M.S., Ceramic Engineering

"Pore Growth During the Initial Stages of Sintering,"
John J. Sipe, Ph.D., Ceramic Engineering

"Determination of Ultrasonic Compression and Shear Ware Velocities in Protoenstatite as a Function of Density,"
Derrille K. Thayer, M.S., Ceramic Engineering

"X-ray Studies of Three Coordination Compounds,"
Louis P. Torre, Ph. D., Chemistry

"Single Crystal Growth of Magnesium Oxide by the Flux Method,"
Harshadri Vora, M.S., Metallurgical Engineering

## Appendix C

## REFRACTORY SURFACE INSULATION VISITATIONS

| | | |
|---|---|---|
| October | 26, 1970 | Lockheed Missile and Space Corporation, Burbank, California |
| | 27 | Lockheed Missile and Space Corporation, Palo Alto, California |
| | 30 | NASA-Ames Research Center, Sunnyvale, California |
| Nov. | 16 | McDonnell-Douglas Aerospace Corp., St. Louis, Mo. |
| Dec. | 10 | NASA-Langley Research Center, Newport News, Va. |
| Jan. | 25, 1971 | Johns-Manville Fiberglass Research, Waterville, Ohio |
| | 26 | Grumman Aerospace Corp., Beth Page, N. Y. |
| | 27 | North American Rockwell Corp., Washington, D. C. |
| | 29 | Babcock-Wilcox, Corp., Augusta, Ga. |
| Feb. | 1* | McDonnell-Douglas Aerospace Corp., St. Louis, Mo. |
| | 2* | NASA-Manned Spaceflight Center, Houston, Texas |
| | 3* | General Electric Space Science Laboratory, Valley Forge, Pa. |
| | 3* | NASA Headquarters, Washington, D. C. |
| | 23 | Lockheed Missile and Space Corporation, Sunnyvale, Calif. |
| | 23 | NASA-Ames Research Center, Moffett Field, California |
| March | 16-18 | Marshall Space Flight Center, Huntsville, Alabama |
| | 19 | NASA-Langley Research Center, Newport News, Va. |
| April | 30 | Battelle Memorial Institute, Columbus, Ohio |

*These visits made as ex officio member of NASA ad hoc committee consisting of:

Mr. Louis Vosteen, NASA-LaRC
Mr. James J. Gangler, NASA-OART
Mr. Norman Peil, NASA-OMSF

Appendix D

## GRADUATE DEGREES AWARDED

### MASTER OF SCIENCE

James A. Aikins, M.S. in Electrical Engineering, March, 1970.

Om Bhandari, M.S. in Ceramic Engineering, March, 1971.

Ramgopal Darolia, M.S. in Metallurgical Engineering, December, 1970.

Alan Dull, M.S. in Ceramic Engineering, March, 1971.

Sang Moo Park, M.S. in Ceramic Engineering, March, 1971.

C. Bruce Scott, M.S. in Ceramic Engineering, March, 1971.

Derrille K. Thayer, M.S. in Ceramic Engineering, March, 1971.

Harshadari Vora, M.S. in Metallurgical Engineering, August, 1970.

### DOCTOR OF PHILOSOPHY

Gerald Erickson, Ph.D. in Physics, March, 1971.

Daniel B. Leiser, Ph.D. in Ceramic Engineering, March, 1971.

Jack K. Merrow, Ph.D. in Ceramic Engineering, August, 1970.

John J. Sipe, Ph.D. in Ceramic Engineering, December, 1970.

Louis P. Torre, Ph.D. in Chemistry, June, 1970.

Appendix E

## CERAMIC MATERIALS RESEARCH SEMINARS

"Composites Materials," Mr. S. W. Bradstreet, Consultant, Dayton, Ohio.

"Pore Growth During the Initial Stages of Sintering," Mr. John J. Sipe, Predoctoral Research Associate, Ceramic Engineering.

"High Temperature Materials for Space Shuttle," Mr. Ronald Torgorson, Boeing Company, Seattle, Washington.

"Study of Valence Band Structure for Soft Emission," Mr. James Rue, Predoctoral Research Associate, Ceramic Engineering.

"Variables in Ceramic Powder Compaction," Mr. Daniel B. Leiser, Predoctoral Research Associate, Ceramic Engineering.

"The Magnetic Behavior and Structure of Carbon Fibers," Mr. C. Bruce Scott, Research Assistant, Ceramic Engineering.

"Vibrational Spectra of ZrC Carbide," Mr. Jack Surendranath, Research Assistant, Chemistry.

"Radiation Damage in Sapphire and Ruby," Mr. John M. Rusin, Predoctoral Research Associate, Ceramic Engineering.

"Interfacial Energies of Tilt Boundaries in Alumina," Mr. Gaddipati Achutaramayya, Predoctoral Research Associate, Ceramic Engineering.

"Interfacial Energy of Twin Boundaries in Alumina," Mr. O. M. Bhandari, Predoctoral Research Associate, Ceramic Engineering.

"Fracture Processes in Ceramic $BaTiO_3$," Mr. Sang Moo Park, Research Assistant, Ceramic Engineering.

"Devitrification of Lead Zinc Borosilicate Solder Glass," Mr. Alan Dull, Research Assistant, Ceramic Engineering

"Powder Growth Characteristics of Decomposed MgO," Mr. K. Aihara, Research Assistant, Ceramic Engineering.

Appendix F

## RESULTS OF QUESTIONNAIRE, DECEMBER REPORT

### COPY OF QUESTIONNAIRE

Please send me the complete report on the projects whose numbers are circled.

| Report No. | Title (Supervisor) | Report No. | Title (Supervisor) |
|---|---|---|---|
| 1 | X-ray Emission (ADM) | 17 | Surface Diffusion Studies (ADM) |
| 2 | Raman Studies (JWM) | 18 | UV Photolysis (JIM) |
| 3 | Zirconium Oxidation (TFA) | 19 | Irradiation Damage (JIM) |
| 4 | High Temp. Calorimetry (ADM) | 20 | Domain Dynamics (JLB) |
| 5 | Gas-Solid Equilibrium (JIM) | 21 | Studies on GASH (ECL) |
| 6 | Solid-Solid Equilibrium (NWG) | 22 | Compaction (OJW) |
| 7 | ZrC Coatings (CJS) | 23 | Sintering (OJW) |
| 8 | Defect Properties (TGS) | 24 | Graphitization (DBF) |
| 9 | Mechanical Behavior of Carbon Fibers(DBF) | 25 | Graphitization of Pyrolytic Carbons(DBF) |
| 10 | Continuum Stress Analysis (BJH) | 26 | White Allotropic Graphite (DBF) |
| 11 | Thermal Stresses in Crystal Ceramics(MDC) | 27 | Magnetic Behavior of Carbon Fibers (DBF) |
| 12 | Strain Energy (RJC) | 28 | Carbon, Biomedical (DBF) |
| 13 | Residual Stresses (RJC) | 29 | Mössbauer Studies (RLI) |
| 14 | Interfacial Energy (WDS) | 30 | Glass Phase Transition Study (EAS) |
| 15 | Mobility of Grain Boundaries (WDS) | 31 | Impurity Diffusion in MgO (WDS) |
| 16 | Deformation Twin Boundaries (WDS) | 32 | Devitrification of Glass to Ceramic Seals (JIM) |

Your status report is circulated to_____members of our staff.

We wish to be (retained on) (deleted from) your mailing list.

Please (retain) (correct) the addressee on your mailing list as follows:

Name_____

Title_____

Address_____

# RESULTS FROM RESPONDENTS' CARDS

| | |
|---|---|
| Number of Reports Issued | 60 |
| Number of Responses Returned | 17 |
| Total Number of Individuals Reading Report | 117 |
| Number of Individual Detailed Reports Requested | 152 |
| Maximum Number Individuals Reading Single Copy of Request | 22 |
| Minimum Number of Individuals Reading Single Copy of Request | 1 |

A detailed breakdown of responses will be found on the next page.

# INDIVIDUAL RESPONDENTS

| Name of Respondent | Organization | No. Reading Report | No. of Complete Reports Requested |
|---|---|---|---|
| A. G. Eubanks | NASA-Goddard Space Flight Center | 4 | 3 |
| T. Heslin | NASA-GSFC | 3 | 4 |
| Eldon Mathauser | NASA-Langley Research Center | 6 | 5 |
| Alan D. Franklin | National Bureau of Standards | 1 | 10 |
| Earl T. Hayes | Bureau of Mines | 1 | 5 |
| R. Nathan Katz | Army Matl. & Mech. Res. Center | 10 | 12 |
| W. G. Ramke | Air Force Mtls. Lab. | 15 | 14 |
| J. L. Daniel | Battelle-Northwest | 12 | 6 |
| P. L. Farnsworth | Battelle-Northwest | 22 | 5 |
| George W. Farwell | University of Washington | 5 | 0 |
| F. V. Lenel | Rensselaer Polytechnic Inst. | 1 | 2 |
| Thomas D. McGee | Iowa State University | 7 | 15 |
| R. J. Diefendorf | Rensselaer Polytechnic Inst. | 1 | 5 |
| William B. Crandall | IIT Research Institute | 14 | 20 |
| Research Inst. Library | University of Dayton | library copy | 32 |
| Louis R. McCreight | GE-Valley Forge Space Center | 4 | 7 |
| Gene Wakefield | Texas Instruments | 5 | 7 |

# REQUESTS FOR INDIVIDUAL PROJECT REPORTS

| Index No. | Project | Supervisor | Number of Requests |
|---|---|---|---|
| 1 | X-ray Emission | Alan D. Miller | 1 |
| 2 | Raman Studies | John W. Macklin | - |
| 3 | Zirconium Oxidation | Thomas F. Archbold | 3 |
| 4 | High Temperature Calorimetry | Alan D. Miller | 2 |
| 5 | Gas-Solid Equilibria | James I. Mueller | 1 |
| 6 | Solid-Solid Equilibrium | Norman W. Gregory | 1 |
| 7 | ZrC Coatings | Colin J. Sandwith | 4 |
| 8 | Defect Properties | Thomas G. Stoebe | 5 |
| 9 | Mechanical Behavior of Carbon Fibers | David B. Fischbach | 7 |
| 10 | Continuum Stress Analysis | Billy J. Hartz | 2 |
| 11 | Thermal Stresses in Crystal Ceramics | Max D. Coon | 6 |
| 12 | Strain Energy | Robert J. Campbell | 4 |
| 13 | Residual Stresses | Robert J. Campbell | 6 |
| 14 | Interfacial Energy | William D. Scott | 4 |
| 15 | Mobility of Grain Boundaries | William D. Scott | 4 |
| 16 | Deformation Twin Boundaries | William D. Scott | 3 |
| 17 | Surface Diffusion Studies | Alan D. Miller | 5 |
| 18 | UV Photolysis | James I. Mueller | 2 |
| 19 | Irradiation Damage | James I. Mueller | 4 |
| 20 | Domain Dynamics | John L. Bjorkstam | 2 |
| 21 | Studies on GASH | Edward C. Lingafelter | - |
| 22 | Compaction | O. J. Whittemore, Jr. | 6 |
| 23 | Sintering | O. J. Whittemore, Jr. | 6 |
| 24 | Graphitization | David B. Fischbach | 5 |
| 25 | Graphitization of Pyrolytic Carbons | David B. Fischbach | 7 |
| 26 | White Allotropic Graphite | David B. Fischbach | 7 |
| 27 | Magnetic Behavior of Carbon Fibers | David B. Fischbach | 5 |
| 28 | Carbon, Biomedical | David B. Fischbach | 8 |
| 29 | Mössbauer Studies | Robert L. Ingalls | 1 |
| 30 | Glass Phase Transition Study | E. A. Stern | 1 |
| 31 | Impurity Diffusion in MgO | William D. Scott | 2 |
| 32 | Devitrification of Glass to Ceramic Seals | James I. Mueller | 5 |

DISTRIBUTION LIST:

Mr. Charles T. Bersch
Naval Research Center

Dr. John Buckley
NASA-Langley Research Center

Mr. Gene Cataldo
NASA-Marshall Space Flight Center

Mr. G.C. Deutsch
NASA Headquarters

Dr. Alfred G. Eubanks
Goddard Space Flight Center

Dr. Allen Franklin
National Bureau of Standards

Mr. James J. Gangler
NASA - OART Headquarters

Mr. Charles A. Hermach
NASA-Ames Research Center

Mr. Robert E. Johnson
NASA-Manned Space Flight Center

Mr. Hal J. Kelly
United States Bureau of Mines

Dr. H.H. Kurzweg
NASA - OART Headquarters

Dr. Robert A. Lad
NASA-Lewis Research Center

Mr. Howard Larson
NASA-Ames Research Center

Dr. Martin Leipold
University of Kentucky

Mr. L.R. McCreight
General Electric Company

Mr. Andree Meyer
NASA-Manned Space Center

Mr. Eldon E. Mathauser
NASA-Langley Research Center

Mr. R.R. Nash
NASA Headquarters

Dr. H.B. Probst
NASA-Lewis Flight Center

Dr. H.E. Rorschach, Jr.
Rice University

Mr. Robert Schwinghamer
NASA-Marshall Space Flight Center

Dr. Stephen E. Weiberley
Rensselaer Polytechnic Institute

Mr. Louis Vosteen
NASA-Langley Research Center

University Personnel:

Ceramic Materials Research Administrative Board
Ceramic Materials Research Committee
Dean C.H. Norris, College of Engineering
Dean G. Beckman, College of Arts & Sciences
Mr. George W. Farwell, Vice President of Research
Project Supervisors