UNIVERSITY OF WASHINGTON

College of Engineering

Ceramic Engineering Division

Interdisciplinary Research Concerning the Nature and Properties of Ceramic Materials

NASA Research Grant Number NGL 48-002-004

Semiannual Status Report Number 13

June 16, 1969 through December 15, 1969

James I. Mueller, Professor
Ceramic Engineering
Principal Investigator

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INTRODUCTION

The Ceramic Materials Research Program at the University of Washington was established June 1, 1963, under National Aeronautics and Space Administration Grant Number NGL 48-002-004. The principal purposes of the grant are to encourage interdisciplinary research upon the nature and properties of ceramic materials and to assist this institution in the development of an enduring research capability in ceramics and ceramic engineering. The funds are, therefore, used to make financial support available for research on ceramic materials conducted by members of the University faculty and to purchase such items of capital equipment as are deemed desirable for the implementation of the stated purposes.

The program, planned to study the effects of various energy environments upon ceramic materials, is divided into several broad research areas, namely: chemical, mechanical, atomic and molecular, and processing. The research program is planned and coordinated by the Ceramic Materials Research Committee, appointed by the Dean of the Graduate School. The current membership includes James I. Mueller, Ceramic Engineering, Chairman and Principal Investigator; T. F. Archbold, Metallurgical Engineering; J. Gregory Dash, Physics; Billy J. Hartz, Civil Engineering; Irene C. Peden, Electrical Engineering; and O. J. Whittemore, Jr., Ceramic Engineering. Administration of the program is coordinated by a board consisting of E. C. Lingafelter representing the Graduate School, Chairman; H. Myron Swarm, Associate Dean, College of Engineering; D. H. Polonis, Chairman of the Department of Mining, Metallurgical and Ceramic Engineering; and James I. Mueller, Principal Investigator.

GENERAL PROGRAM REPORT

This is the thirteenth semiannual status report and it covers the first half of the seventh year of operation under this grant. During the report period, a total of thirty-seven projects were supervised by twenty-three faculty members in eight academic disciplines of the University. Several new projects were initiated during the report period. Professor John Macklin, Department of Chemistry, initiated a project on Raman spectral studies of ZrC and other ceramic materials, and Professor Richard Zupp, Metallurgical Engineering Division, began work on crystal growing to supplement other projects in the program.

An additional full-time faculty position in ceramic engineering was established effective September, 1969. This was filled by Dr. Alan D. Miller. The part-time academic, part-time research position was filled by Dr. David B. Fischbach, formerly on the staff at the Jet Propulsion Laboratory. Dr. Fischbach has initiated both academic and research programs on carbon and graphite materials. This adds an entirely new dimension to our program and one which has definite orientation to space related research.

The Ceramic Materials Research Seminar, a period devoted to discussions of concepts and research of interest to the program, met for a total of six sessions during the report period. Speakers included two University of Washington faculty members, two visitors, and two graduate students. A complete list of seminar speakers and their topics is included as Appendix B.

The program supported the attendance of thirteen faculty members to a total of four technical meetings at which seven papers based upon work supported by the grant were presented. Papers published or presented resulting from work supported wholly or in part by the grant are listed with the individual status reports in Appendix C.

Funds from the grant were used to supplement University support of a visiting professor during the Summer Quarter. Dr. Wendell S. Williams, Professor of Physics and Ceramic Engineering at the University of Illinois, offered a four-week, three-credit course during the A term. The course, entitled "Physics of Strong Solids," had an enrollment of sixteen students.

The XVI Refractory Composite Working Group Meeting, co-hosted by the University of Washington and Battelle Northwest, was held at the Battelle Science Center in Seattle, October 13, 14, 15, 1969. Dr. David Fischbach was Arrangements Chairman and Conference Coordinator. Following this, the Pacific Coast Regional Meeting of the American Ceramic Society was held in Seattle with Dr. Alan D. Miller, of our staff, as General Chairman.

Due to the meetings mentioned above the annual fall technical review by Mr. James J. Gangler, NASA Technical Monitor, was more informal than in past years. Mr. Gangler discussed grant supported research with several of the research supervisors and the program in general with the Principal Investigator.

Graduate enrollment in Ceramic Engineering increased this fall, resulting in the total graduate enrollment being equivalent to previous years. Several of the graduate students have been working in industry and are returning for advanced degree work. Dr. Shiushichi Kimura completed a twelve-month appointment as a Senior Research Associate and has returned to the staff at Toyko Institute of Technology. Dr. Kimura contributed heavily to our program, working with Professor Alan D. Miller on the High Temperature Calorimeter Study.

The special study funded by a supplement to the grant to determine the impact of this program on various levels of the University of Washington has been completed. A report on this study was submitted to the Cffice of University Affairs on November 19, 1969.

RESEARCH STAFF

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CHEMICAL

(Zirconium-Oxygen-Carbon System)

This system was selected as the subject for study of the effects of chemical environment upon ceramic materials. Several faculty members from various disciplines are participating in a coordinated program to obtain knowledge regarding this system.

GAS-SOLID EQUILIBRIUM

James I. Mueller
Professor, Ceramic Engineering

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.

Influence of Oxygen Activity on the Structure of Zirconium Oxide

K. M. Nair
Predoctoral Associate, Ceramic Engineering (June 15 - November 10)
Ph.D. Thesis Research

The Zr - ZrO_2 system was investigated between the temperature 700°C and 1600°C using controlled oxygen partial pressures. The system was approached from two directions, (1) starting with pure metal powder and oxidizing it in argon and oxygen mixtures and (2) starting with pure ZrO_2 powder and reducing it in different mixtures of CO and CO_2.

Two lower valence oxides of zirconium were observed by x-ray powder diffraction analysis. One of them had a f.c.c. zinc blend (ZnS) type crystal structure with a_o = 3.8956 Å. The composition ZrO is proposed. The second oxide, Zr_3O, had a hexagonal-close-packed crystal structure similar to that of zirconium metal, with oxygen atoms occupying one-third of the interstitial positions in an ordered manner. The stability of this oxide was determined.

A series of reduced ZrO_2(black) samples were prepared at two temperatures 1200°C and 1470°C and varying oxygen partial pressures. Another set of ZrO_2(black) samples was prepared in the temperature range 1000°C and 1600°C and in hydrogen atmospheres. These reduced ZrO_2(black) samples were reoxidized to ZrO_2(white) at 1000°C in an air atmosphere. The d-spacings and relative intensities of ZrO_2(black) and ZrO_2(white) did not show any change with change in oxygen activity or temperature.

A mechanism for the formation of reduced ZrO_2(black) is proposed. The apparent activity of free Zr atoms in the monoclinic ZrO_2(black) lattice is caluclated.

Mr. Nair received his Ph.D. degree in Ceramic Engineering in December, 1969. Thesis title: "Unfamiliar Oxidation State of Zirconium and the Effect of Oxygen Activity on the Structure of ZrO_2".

Studies of the Zirconium Dioxide-Carbon Reaction

S. K. Sarkar
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

Heating zirconium carbide, various mixtures of zirconium oxide and carbon, and mixtures of zirconium oxide and zirconium carbide at 1800°C in a carbon monoxide atmosphere results in the formation of a f.c.c. phase (ZrC_xO_y) with lattice parameter between 4.673 and 4.689 Å. The determination of composition indicates that the lattice parameter decreases linearly with the oxygen content. Lattice parameter varies non-linearly with C and sum of C and O_2.

There is evidence of equilibrium being attained in some, if not in all cases. Assuming the activity of carbon to be unity in ZrC_xO_y of composition $ZrC_{0.856}O_{0.07}$, the oxygen activity has been calculated to be 1.36×10^{-15}.

The standard free energy of formation of this composition has been calculated to be -39.5 kcal/mole, assuming the activity of zirconium to be 10^{-3}.

The final composition of ZrC_xO_y depends on the temperature and the superincumbent CO pressure.

For the attainment of the equilibrium, the material transfer from the external environment is essentially through the CO phase.

Mr. Sarkar received his Ph.D. degree in Ceramic Engineering in August, 1969. Thesis title: "Solubility of Oxygen in Zirconium Carbide".

SOLID-SOLID EQUILIBRIUM

Norman W. Gregory
Professor, Department of Chemistry

A thermodynamic and kinetic study of chemical reactions in oxide-carbide graphite systems.

Interaction of Metal Oxides with Graphite and of Metal Carbides with Metal Oxides

Juey Hong Rai
Research Assistant, Chemistry (June 15 - June 30)
Ph.D. Thesis Research

The objective of this research is to study kinetic and thermodynamic properties of the reaction of CaO and graphite and of ZrC and CaO by torsion effusion measurement of steady state pressures of Ca(g) and CO(g) generated in effusion cells.

Mr. Rai has completed his Ph.D thesis and presented his research to his supervisory committee. His thesis describes fully his method for deriving thermodynamic data from steady state effusion pressures, measured at various temperatures as a function of time. His results on the CaO-graphite reaction are in very good agreement with those predicted from complementary thermodynamic studies. A direct study of this reaction has not been reported earlier. It also appears likely that the reaction of CaO with ZrC has given results which will be useful as an aid in characterizing the ZrC phases used as initial reactants.

A paper entitled "The Time Dependence of Effusion Cell Steady-State Pressures of Carbon Monoxide and Calcium Vapors Generated by the Interaction of Calcium Oxide and Graphite", by Juey Hong Rai and N. W. Gregory, has been accepted for publication in the Journal of Physical Chemistry (expected March 1970).

A paper entitled "An Effusion Study of the Reaction of Zirconium Carbide and Calcium Oxide", by Juey Hong Rai and N.W. Gregory, has been accepted for publication in the Journal of Physical Chemistry (expected April 1970).

Mr. Rai received his Ph.D. degree in Chemistry in August, 1969. Thesis title: "A High Temperature Torsion Effusion Study of the Interaction of Metal Oxides with Graphite and with metal Carbides".

BONDING IN INTERSTITIAL COMPOUNDS

Alan D. Miller
Assistant Professor, Ceramic Engineering

A better understanding of electronic bonding in interstitial compounds is sought by this study.

Ultrasoft X-ray Emission Studies

James W. Rue
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The purpose of this project is to: (1) study the ultrasoft x-ray emission of carbon, oxygen, and zirconium in Zr-C-O compounds and (2) relate the spectral data obtained to the electronic energies in Zr-C-O compositions whose band structures have been predicted.

Due to a discontinuous drop in x-ray tube current at low pressures and slow response in the vacuum system, preliminary operation of the vacuum spectrometer was hampered by an erratic behavior. This problem was solved by constructing a power supply that operates at low pressure and automatically switches over to the x-ray tube at a prescribed set point. In addition an adsorption pump was installed to increase pumping speed and thus increase the system's response.

Subsequent operation of the spectrometer has shown the necessity for improved resolution in the spectral emission. This problem will be solved by installing a curved analyzing crystal system. Currently ways of incorporating a curved crystal into the spectrometer are being investigated.

The investigation of instrument error on the recorded emission spectra is still being conducted.

Raman Studies of Ceramic Materials

J. W. Macklin
Assistant Professor, Chemistry

Jack Surendranath
Research Assistant, Chemistry
M.S. Chemical Engineering

The nature of the chemical bonding in Refractory Metal Carbides is not well understood and previous studies apply somewhat indirect methods, e.g., band structure studies. The vibration spectrum of a compound offers a direct measure of inner atomic and intermolecular forces and, to a large extent, the nature of the bonding involved. The purpose of this project will be to measure the Raman spectra of ceramic materials (single crystals and powders) to the end of understanding the character of their bonding. A Raman spectrometer utilizing a laser source will be used in this study. From the Raman spectrum one can determine: (a) symmetry of the bond structure from measured polarizabilities and group theoretical considerations; (b) the forces between the lattice constituents from the measured vibrational frequencies and a normal coordinate analysis considering the known local symmetry and symmetry of the vibration measured; and (c) the type of bonding involved from relative intensity measurements and possibly the bond order for cases in which the crystal structure is known.

ZIRCONIUM OXIDATION

Thomas F. Archbold
Associate Professor, Metallurgical Engineering

This research project is investigating the characteristics and mechanisms of the early states of oxidation of zirconium metal. The oxide crystal structure and metal-oxide orientation relationships are to be determined as a function of oxygen partial pressure and temperature.

Zirconium Oxidation

R. Darolia
Research Assistant, Metallurgical Engineering
M.S. Thesis Research

A detailed examination of the structure, grain size, and transformation characteristics of thin zirconia films is being made by the use of electron microscopy. The kinetic data obtained by the previous student on the project, Dr. L. P. Srivastava, are being used in an attempt to predict the oxide characteristics. During the report period, Mr. Darolia has directly observed the transition of zirconia to the molten state, followed by rapid cooling. The analysis of the photographs, to determine viscosity data, is in progress. We plan to investigate molten TiO_2 in an attempt to confirm the validity of the technique. The kinetics-structure correlation is near completion; this will be followed by a detailed study of the oxidation of ZrC.

A paper entitled "The Effect of Gas Flow Rate on the Oxidation Kinetics of Zirconium," by L. P. Srivastava and T. F. Archbold has been published in Scripta Metallurgica, vol. 3, p. 377, (1969).

CALORIMETRIC INVESTIGATION OF CERAMIC AND RELATED MATERIALS

Alan D. Miller
Assistant Professor, Ceramic Engineering

The objectives are the construction of a high-temperature diphenyl-ether drop calorimeter and the study of heat capacity and heats of transformation of ceramic and related materials.

High-Temperature Drop Calorimetry

Shiushichi Kimura
Research Associate, Ceramic Engineering

John Negrych (NSF Trainee)
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The enthalpy of three specimens of ZrC_x was measured over the temperature range 200°-1400°C. The initial compositions of the specimens were $ZrC_{1.0}$, $ZrC_{0.86}$ and $ZrC_{0.6}$. As expected the enthalpies showed a regular decrease as the carbon content decreased. At present carbon, oxygen and nitrogen analyses are being conducted on the specimens at Los Alamos Scientific Laboratory. Upon receipt of the chemical analysis results, more refined analysis of the data will be made. A second series of measurements on specimens of different carbon contents is in progress.

MECHANICAL PROPERTIES

Research upon the mechanical properties of ceramic materials is underway to develop a more thorough understanding of the brittle fracture mechanisms in single crystals and polycrystalline ceramics.

ALUMINUM OXIDE BICRYSTALS

William D. Scott
Assistant Professor, Ceramic Engineering

The purpose of this work is to study the properties of grain boundaries in aluminum oxide.

Mechanical Properties of Aluminum Oxide Bicrystals

Raymond L. Bertolotti
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The purpose of this project is to study grain boundary sliding in aluminum oxide by subjecting grain boundaries of selected, controlled misorientation to pure shear loading at elevated temperatures.

Experiments have continued on bicrystal grain boundary sliding. A small amount of apparent grain boundary shear offset has been produced with loads of 1000 to 2000 psi at 1500-1700°C for one to two hours. It is not clear if this offset is true sliding or is local deformation adjacent to the boundaries by normal basal slip mechanisms. This question is being investigated further through tests on suitably oriented bicrystals.

The relative interfacial energy of rhombohedral twins has been measured and a paper for publication has been completed.

Interfacial Energies of Aluminum Oxide Bicrystals

G. Achutaramayya
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The purpose of this project is to determine the relative interfacial energy of low angle dislocation tilt boundaries in aluminum oxide.

Low-angle dislocation tilt boundaries have been produced and initial measurements of relative interfacial energy indicate low energies for these low angles as expected. Further measurements over a range of angles will be required to test the functional dependence of the energy.

A computer program has been developed by the research supervisor and an undergraduate assistant to analytically evaluate the surface geometry indicated by interference photographs. This program involves digitizing the optical interferometer data and, through least squares curve fits, producing mathematical expressions for surface topography in the region of a thermally etched grain boundary groove.

Mobility of Grain Boundary in Alumina

Robert W. Burns
Predoctoral Associate, Ceramic Engineering (NDEA Fellow)
Ph.D. Thesis Research

The purpose of this project is to measure the mobility of low-angle tilt boundaries in aluminum oxide under the influence of an electric field and a surface energy driving force.

During the period covered by support on this grant, the student developed experimental techniques and apparatus. This included orientation slicing and polishing of alumina crystals and development of gas train and fabrication of heating elements for a Centorr high temperature furnace.

Surface Energy of Alumina

O. M. Bhandari
Research Assistant, Ceramic Engineering
M.S. Thesis Research

During his first three months on this project, the student investigated the feasibility of three different research projects on viscoelastic behavior, mechanical properties of alumina and surface energy. He selected surface energy measurements and will attempt to measure a "γ-plot" for alumina and to critically evaluate the relative energy measurement techniques used by several investigators in this field.

ZrC COATINGS

Colin J. Sandwith
Assistant Professor, Mechanical Engineering

James D. Danberg
Research Assistant, Mechanical Engineering
M.S. Thesis Research

Leonard Johnson
Research Assistant, Mechanical Engineering
M.S. Thesis Research

The purpose of this project is to determine bond strengths and microhardness and to design and apply a new test of mechanical properties of ZrC plasma flame-sprayed coatings.

Mr. Danberg's M.S. Thesis for the separation of ZrC coating has been approved. His master's degree will be dated Winter Quarter 1970.

The purpose of Mr. Johnson's project is to develop a test to evaluate the shear strength of the interface between flame sprayed ZrC and the metal substrate. The test under consideration has been designed to provide parameters for designing with plasma flame sprayed coating from an easily reproduced tensile specimen. The design parameter of interest is the maximum allowable strain parallel to the interface before separation of the coating. Stress analysis calculations indicate that this parameter can be determined by elongating to failure a sheet metal substrate coated with narrow strips, oriented perpendicular to the tensile axis, of ZrC. Interpreting test results should be relatively easy because the strain at which separation occurs can be calculated from the load at failure. Practical limitation of fabricating the test specimen and the optimum theoretical specimen configuration are being evaluated at this time. Specimens will be fabricated in January 1970 and hopefully tests will be completed by June 1970.

DEFECT PROPERTIES OF IONIC AND CERAMIC CRYSTALS

Thomas G. Stoebe
Associate Professor, Metallurgical Engineering

Richard R. Zupp
Assistant Professor, Metallurgical Engineering

This project concerns the growth of single crystals with controlled defect structures, the characterization of these defect structures, and the effects of different defect structures on the properties of crystals with the NaCl structures.

Mechanical Properties of LiF Single Crystals

Hira L. Fotedar
Predoctoral Associate, Metallurgical Engineering
Ph.D. Thesis Research

David Wilson
NSF Institute Participant

Work has been completed regarding the effects of stress relaxation experiments on the subsequent work hardening behavior in LiF. The effect of strain rate on dislocation density and activation parameters in deformation are now under active investigation.

Mechanical Properties of MgO Single Crystals

M. Srinivasan
Research Assistant, Metallurgical Engineering
M.S. and Ph.D. Thesis Research

The investigation of the room temperature mechanical properties of high purity MgO and MgO containing Fe^{+2}, Fe^{+3} and Ni^{+2} has been completed. Preliminary work on the investigation of dislocation configurations in such crystals using electron microscopy techniques is currently under way.

Thermodynamic Studies of MgO Single Crystal Growth

David A. Bucy (NDEA Fellow)
Research Assistant, Metallurgical Engineering
M.S. Thesis Research

Harshodrai Vora
Research Assistant, Metallurgical Engineering
M.S. Thesis Research

The purpose of this portion of the project is to determine the effect of thermodynamic variables on the defect structure of MgO single crystals. The planned approach is to first develop crystal growth techniques by which single crystals of MgO can be grown routinely, then to introduce controlled variations in magnesium activity, oxygen activity, and dopant activity during crystal growth, and to determine their effects on the product crystals. Two methods of MgO single crystal preparation have been explored during the report period: growth from a flux in a temperature gradient and growth by the plasma Verneuil technique.

A controlled atmosphere furnace was constructed so that Ni crucibles can be used for the flux growth runs under a protective atmosphere of purified argon. A one-day trial run at the crystal growth temperature of 1200° to 1300°C verified that the Ni crucible is fully protected by the argon atmosphere and that there is no reaction of the Ni crucible with the flux which is 17 w/o MgO - 70.5 w/o WO_3 - 8.63 w/o P_2O_5 - 3.87 w/o Na_2O. One five-day crystal growth run has been made to date. Although some difficulty was encountered in the protective atmosphere system, and this led to partial oxidation of the Ni crucible, twenty-four small MgO crystals were obtained. The dimensions of the largest crystals are approximately 6 mm x 3 mm x 3 mm, and portions of some of the crystals are green because of contamination with NiO from the partially oxidized Ni crucible. Subsequent growth runs will be continued for longer times when the protective atmosphere system is repaired.

Design and construction of equipment for MgO crystal growth by the plasma Verneuil technique was completed during the report period. Difficulties have been encountered with the ancillary RF generator, which will be rebuilt to provide RF plasmas of higher power.

David A. Bucy received his M.S. degree in Metallurgical Engineering in August, 1969. His thesis is entitled "Design and Construction of an Induction Plasma System for Single Crystal Growth in the Verneuil Geometry".

M. Srinivasan received his M.S. degree in Metallurgical Engineering in August, 1969. His thesis is entitled "Effect of Impurities on the Mechanical Behavior of MgO Single Crystals".

A paper entitled "Effect of Impurities on the Mechanical Behavior of MgO Single Crystals," by M. Srinivasan and T. G. Stoebe has been submitted for publication to the Journal of Applied Physics.

CONTINUUM STRESS ANALYSIS OF CRYSTALLINE CERAMICS

The purpose of this research is to apply modern computational methods, recently developed for complex aerospace problems, to the problem of evaluation of stresses in polycrystalline ceramics caused by anisotropic material properties. This should lead to a better understanding of the mechanical properties and behavior of these materials and of the influence of crystalline structure on strength.

Thermal Stresses in Crystalline Ceramics

B. J. Hartz
Professor, Civil Engineering

Colin B. Brown
Associate Professor, Civil Engineering

G. K. Das
Research Assistant, Civil Engineering
Ph.D. Thesis Research

Exhaustive studies are made on the anisotropic elastic and thermal properties of aluminum oxide crystals and the findings are documented in a detailed research report in preparation. An attempt is made to gather the latest information on these properties and a large amount of data has been collected, derived or reorganized which is of importance especially for three dimensional analysis.

The test results on bicrystals are reviewed and the strength characteristics of single crystal alumina is also compiled. The fracture of test specimens is discussed in the light of this knowledge. The different possible techniques of solution to the stress analysis have been documented are are included in the detailed paper.

As discussed in the earlier report, approximate techniques are used to solve this problem. Two dimensional (plane stress program) and three dimensional (Elas-4 program) finite element solutions are used to solve this problem. Finally, a "projective technique" using higher order polynomials and regular subdivisions is reviewed in detail and proposed to be the best technique for this problem.

"Plane stress" finite element results are obtained for each test specimen along transverse as well as longitudinal sections. Different mesh sizes are used to indicate the convergence characteristics for this problem. The effect of variation of elastic and thermal properties are also studied. Over 20 different analysis were made. The maximum stresses obtained varied from 37,000 psi to 54,000 psi. This is within psi lower range of strength of alumina crystals (43,000 psi to 155,000 psi).

As discussed earlier, the anisotropic properties make it desirable to use three dimensional analysis. At first, the Elas-4 program was used for this purpose. However, the program now is being converted to the CDC 6400 computer from DCS and once this is accomplished it is hoped that the larger storage will allow a finer mesh for analysis than was possible at the time.

Thermal Stresses in Crystalline Ceramics (continued)

Finally, the "projective technique" has been considered. Conversion to CDC and considerable rewriting was necessary to make the existing computer program workable. Although this looks most promising, no results are available as yet.

Grain Boundary Energy and Crystal Distortion Due to Surface Energy Changes

Michael Held (Unsupported)
Part-time Graduate Student, Civil Engineering
M.S. Thesis Research

This project is aimed directly at analytical calculations of the crystal distortions and of the associated internal stress fields caused by interfaced energies of aluminum oxide bicrystals. The experimental results are being obtained in the project on interfacial energies under the direction of Professor W. D. Scott.

Work is continuing on this with only limited results available.

Continuum Stress Analysis of Ceramic Materials

Max D. Coon
Assistant Professor, Civil Engineering

Maurice B. Cooper
Predoctoral Associate, Civil Engineering
Ph.D. Thesis Research

The object 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. A set of room temperature tests of Titanium Carbide beams, in three point loading, have been conducted. The carrying capacity of heat treated beams will be compared with the results of these tests to judge the value of the residual stresses. The furnace element and container have been constructed and are being assembled. The high temperature testing will begin shortly.

Strain Energy in Crack Propagation

Robert J. Campbell
Assistant Professor, Ceramic Engineering

Sang-Moo Park
Research Assistant, Ceramic Engineering
M.S. Thesis Research

An attempt will be made to correlate the energy involved in propagation of cracks in single crystals. The approach will be to use a single crystal of $BaTiO_3$ with two areas of polarization established to provide strain input. The amount of strain will be related to the electrical field applied.

Literature surveys are in progress and materials procurement has started.

This project was stimulated by knowledge of failure of polycrystalline under water transducers as a result of high power service. It is anticipated that this research may permit improved design of such equipment.

Nondestructive Determination of Residual Stresses

Robert J. Campbell
Assistant Professor, Ceramic Engineering

Derrille K. Thayer (Edward Orton Ceramic Foundation)
Research Fellow, Ceramic Engineering
M.S. Thesis Research

This research will attempt to measure stresses in fired bodies by nondestructive tests. These determinations will permit estimation of the effect of such stresses on properties such as thermal shock and ultimate strength.

Literature surveys are completed and sample preparation has begun.

This investigation should provide methods of prediction of long-term strengths, crazing and thermal shock resistance. The principal approach visualized at present involves determination of sonic modulus of elasticity and rate of propagation of sonic energy through pieces in stressed or relaxed configurations.

ATOMIC AND MOLECULAR

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.

EFFECTS OF RADIATION UPON CERAMIC MATERIALS

James I. Mueller
Professor, Ceramic Engineering

William M. Ziniker
Senior Research Associate, Ceramic Engineering

Jack K. Merrow
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

Coimbatore S. Krishnan
Research Assistant, Ceramic Engineering
Ph.D. Thesis Research

John M. Rusin
Research Assistant, Ceramic Engineering
Ph.D. Thesis Research

Thermoluminescence of Oxide Single Crystals

The technique of thermoluminescence is being used to study the effect of ultraviolet radiation upon MgO and CaO single crystals. The mechanism of coloration of these crystals is being sought in terms of trace impurities and lattice defects. Thermoluminescence data is being collected for MgO crystals of various origins. The completion of a very sensitive single photon counting system has made possible the measurement of the spectral composition of the various glow peaks. Most MgO samples exhibit a red emission only. The structure of this emission identifies it as that of Cr^{3+} which is a common substitutional impurity in MgO. An exceptional crystal shows no detectable red emission, but rather a broad emission peak centered at 6000A. No similar luminescence has been reported and we are tentatively assigning it to Mn^{2+} on the basis of similarities to other luminescent materials activated by Mn^{2+}. Initial excitation measurements show that an absorption peak at about 2800A is responsible for this luminescence.

Surface Defects on Oxide Powders

In order to study the optical properties of electronic centers existing on the surfaces of powders, one must make all measurements under ultra high vacuum conditions. We are presently constructing a small glass vacuum system which will enable us to degass our samples at high temperature in a very clean vacuum. Subsequent irradiation will form electronic centers at the surface which can then be studied with much of our present apparatus.

DOMAIN DYNAMICS IN ISOMORPHOUS FERROELECTRICS

John L. Bjorkstam
Professor, Electrical Engineering

This research has been concerned with the optical and dynamical properties of domains in ferroelectric KH_2PO_4 and its isomorphs.

Ferroelectric Domains and Domain Motion in KH_2PO_4 and KD_2PO_4

James A. Aikins
Research Assistant, Electrical Engineering
M.S. Thesis Research

Due to unexpected results in the As^{75} Nuclear-Magnetic-Resonance (NMR) spectrum of KH_2AsO_4 and RbD_2AsO_4 just above the ferroelectric phase transition, emphasis on this project has changed*. In particular, we interpret the appearance of additional resonances in the spectrum as resulting from long-range, long-lived ($\tau \gtrsim 10^{-4}$ sec.) correlations in the hydrogen (or deuteron) ordering. If this is in fact the case, one should observe a critical slowing down of polar fluctuations into the kilocycle range. Such critical slowing should be evident as a decrease in the dielectric constant as the measuring frequency is increased through the kilocycle range. The results should contribute appreciably to our understanding of the ferroelectric phase transition in KDP type ferroelectrics.

*The NMR work mentioned above is funded by the National Science Foundation.

STUDIES ON GASH

Edward C. Lingafelter
Professor, Department of Chemistry

Louis P. Torre
Research Assistant, Department of Chemistry
Ph.D. Thesis Research

The objective of this study is to investigate the structural basis for the ferroelectricity of Guanidinium Chromium Sulfate Hexahydrate (GCrSH) and its aluminum isomorph (GASH).

An apparatus was designed and constructed to enable systematic study of hysteresis loops at various frequencies (including quasi-static conditions). Behavior of a small single crystal of GASH indicated that the crystal could be adjusted to a nearly fully poled state under zero applied voltage. Two complete sets of x-ray diffraction intensities were collected under these conditions. However, analysis of the x-ray data indicate that the crystal was not completely poled. We are now continuing the study of the electrical behavior of the crystal in an attempt to specify conditions which will maintain polarization of the crystal during collection of x-ray data.

STRUCTURE AND PROPERTIES OF CARBON MATERIALS

David B. Fischbach
Research Associate Professor, Ceramic Engineering

The properties and behavior of carbons and graphites are especially dependent on microstructure, because of the strong intrinsic anisotropy; and a wide range of structural variation occurs in these materials. A better fundamental understanding of the capabilities and limitations of carbon materials is being sought through investigation of the inter-relationships of properties and structure in selected model carbons.

Effect of Plastic Deformation on Graphitization

The purpose of this study is to investigate the influence of high temperature basal-plane shear deformation on the graphitization of pyrolytic carbon.

Pyrolytic carbon specimens cut at an angle to the average basal-plane orientation and then deformed in tension at 2500 - 3000°C are available from previous work of the principal investigator while employed at the Jet Propulsion Laboratory. Specimens with well-defined narrow zones of shear deformation have been selected for further study. It is planned to compare the x-ray diffraction structure in the sheared zones with that in adjacent unsheared portions of the same sample, and to correlate the results with the amount of shear, deformation temperature, etc. Techniques for preparing samples of the sheared material (bands $\leq$ 0.03" wide) are now being investigated.

Previous studies showed that plastic deformation in either tensile or compression modes substantially enhanced the rate of graphitization of pyrolytic carbon. However, the influence of basal shear was ambiguous. The layer flattening and mobility produced by shear should facilitate graphitization. On the other hand, the stacking disorder characteristic of ungraphitized carbons can be represented in terms of random layer shear using the turbostratic model.

The Anisotropy of Carbon Fibers

C. B. Scott
Research Assistant, Ceramic Engineering
M.S. Thesis Research

The objective is to determine the diamagnetic behavior of carbon fibers and relate it to the structure and to other structure-sensitive properties, with special attention to anisotropy, to gain further insight into the structure of these unique carbons.

A Faraday susceptibility balance has been designed and is being constructed using existing capital equipment (a 4" electromagnet and a recording semi-micro automatic balance). The height and orientation of the magnet relative to the suspended sample will be adjustable to facilitate calibration and anisotropy measurements. It is planned to correlate the susceptibility anisotropy with x-ray diffraction preferred orientation texture, Young's modulus and electrical resistance. Samples of carbon fibers with a wide range of preferred orientation texture, prepared from both rayon and polyacrylonitrile precursors, have been obtained or ordered. A continuous literature survey is being made because of the intense activity in carbon fiber research and development.

The structure of carbon fibers is unusual and interesting. It combines a strong layer plane orientation texture with a high degree of crystallographic disorder. The importance of the preferred orientation for high elastic modulus is well established, but the significance of crystallographic disorder is just beginning to be appreciated. No information on magnetic behavior of carbon fibers is available, but magnetic studies on other carbon materials have been fruitful.

A paper entitled "On the Relationship of Mechanical Properties to Structure in Carbons and Graphite," by D. B. Fischbach, was presented at the XVI Refractory Composite Working Group Meeting in Seattle, Washington, October, 1969.

A paper entitled "High Temperature Isotropic Plasticity of Graphite," by D. B. Fischbach has been accepted for publication by the Philosophical Magazine. (Paper based on experimental work performed at Jet Propulsion Laboratory.)

MÖSSBAUER STUDIES

J. G. Dash
Professor, Department of Physics

Edward A. Stern
Professor, Department of Physics

Robert L. Ingalls
Assistant Professor, Department of Physics

Hanan Shechter
Visiting Assistant Professor, Department of Physics (Returned to Technion, Israel Institute of Technology, August 15, 1969)

Gerald A. Erickson
Predoctoral Associate, Department of Physics
Ph.D. Thesis Research

John R. Nett
Research Assistant, Department of Physics
Ph.D. Thesis Research

C. D. West
Predoctoral Associate, Department of Physics
Ph.D. Thesis Research

Mössbauer Studies

This program is based upon the application of the Mössbauer effect to study properties of various materials with emphasis on atomic force constants, interatomic potentials, electronic valence states and distributions. The proposed program consists of three individual projects, charging in alloys, anharmonic force constants and study of insulators as a function of temperature and pressure.

A paper entitled "Anharmonicity in Thorium Oxide," by H. Shechter was presented at the Gordon Research Conference, Meriden, New Hampshire on August 4, 1969.

A paper entitled "Extreme Low Temperature Anharmonicity of Fe^{57} in ThO_2," by G. A. Erickson was presented at the Meeting of the American Ceramic Society, Seattle, Washington on October 17, 1969.

A paper entitled "Comments on Divalent Fe^{57m} Quadrupolar Coupling Constants," by R. Ingalls, has been published in Physical Review, December 1969.

A paper entitled "Mössbauer Studies of Lattice Dynamics, Fine and Hyperfine Structure of Divalent Fe^{57} in FeF_2," by D. P. Johnson and R. Ingalls, has been accepted for publication by Physical Review.

A paper entitled "Mössbauer Study of Low Temperature Anharmonicity in $ThO_2:Co^{57}$," by H. Shechter, J. G. Dash, G. A. Erickson and R. Ingalls, has been submitted for publication in Physical Review.

IMPURITY DIFFUSION IN MgO UNDER THE INFLUENCE OF AN ELECTRIC FIELD

William D. Scott
Assistant Professor, Ceramic Engineering

Chester A. Hinman (on leave)
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The purpose of this project was to measure the diffusion of Ni in MgO at high temperature 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 effects in MgO.

High-purity MgO single crystals have been subjected to D.C. fields at various temperatures for long periods of time. Several two-probe and four-probe measurement techniques have been used. The D.C. conductivity increases with time and temperature. The current increases with applied field at a rate greater than expected from ohmic behavior. The results of these experiments are being analyzed to identify and separate the various conductivity mechanisms.

The graduate student working on this project has been on leave for the period covered by this report, and is working on the final presentation of his dissertation.

PROCESSING

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

CERAMIC PROCESSING

O. J. Whittemore, Jr.
Associate Professor, Ceramic Engineering

Characterization and Forming

Douglas J. Calkins (NSF Trainee)
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

Daniel B. Leiser
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The objectives of this project are (1) to study ceramic forming methods and (2) to correlate characteristics of particles and agglomerates with forming and subsequent product properties. Present activities are being devoted to the study of compaction.

In a study of small void filling as related to brittle fracture of glass spheres, carefully sorted to 250 μ size and uniform sphericity have been vibrated to minimum volume (64% relative density) in a 1/2 inch diameter mold. Compaction has been conducted in an "Instron" tester at from 5000 to 40,000 psi to obtain compaction curves. The maximum pressure gives 72% relative density. Before removal, pore distribution has been determined by mercury intrusion. A breakthrough at 90 microns pore size is noted on the initial packing (this agrees with simple cubic packing) and the distribution curves show 90 micron pores with increasing amounts of finer pores after increasing compaction pressures. Particle sizing after compaction does not indicate bimodal distributions but a spectrum of sizes from the original sphere size down.

Compaction of closely sized particles of fused magnesia and alumina and of two shape varieties of mullite has been studied. Relative compaction was found to decrease with both particle size and eccentricity. On most materials, loading rate (varied from 0.05 to 5.0 cm/min.) was not found to be significant. Fracture after compaction at various pressures has been determined on magnesia and angular mullite and was found to vary with particle eccentricity.

Initial Stages of Sintering

J. Joseph Sipe
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The objective of this project is to study the initial stages of sintering where pore growth occurs. Pore growth has been shown to occur during the initial sintering of several ceramic materials, and it also has been shown to occur simultaneously with shrinkage.

Work is being concentrated on Fe_2O_3. Scanning electron microphotographs of lightly sintered Fe_2O_3 indicate a complex structure despite the simple original spherical particle shape. Particles appear to be organized in groups and different pairs of spheres appear to sinter at large differences in rates. The currently accepted two-sphere model of sintering seems not to be useful in interpreting this sintering. However, the varying rates of sintering may explain pore growth in initial stages.

A paper entitled "Variables in the Compaction Behavior of Ceramic Particles," by D. B. Leiser and O. J. Whittemore was presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Seattle, on October 17, 1969.

MISCELLANEOUS

SURFACE DIFFUSION

Alan D. Miller
Assistant Professor, Ceramic Engineering

Edward H. Randklev
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The primary objective of this study is the determination of the effect of temperature on the diffusion coefficient of chromium on aluminum oxide.

Diffusion anneals performed in vacuum have confirmed the feasibility of using a point source diffusion couple geometry for an oxide system. However, at the annealing temperatures required, the vapor loss of chromia was found to be prohibitively high for anneals run in vacuum, which eliminated their usefulness in determining diffusivity values. Diffusion anneals performed in purified argon atmosphere are presently in progress. Attention has also been directed toward the phenomena of linear thermal faceting of select surfaces of single crystal aluminum oxide.

DEVELOPMENT AND TESTING OF CALCIA REFRACTORIES

O. J. Whittemore, Jr.
Associate Professor, Ceramic Engineering

James T. Benson
Research Assistant, Ceramic Engineering
M.S. Thesis Research

Calcia is an inexpensive, very refractory material largely neglected because of hydration.

Preliminary work has indicated that good refractories can be made by sintering calcium hydroxide at relatively low temperatures. The objective of this work is to develop useful, feasible calcia refractories and to determine their properties.

Four commercial hydrated limes of varying analyses have been studied for their sinterability, purity and microstructure and one selected for development. Compacts of this material can be sintered to 97% relative density when heated to 1450°C for 12 hours. Brick compositions have been developed from granular material prepared by crushing these sintered compacts.

PORE STRUCTURE OF BONE

O. J. Whittemore, Jr.
Professor, Ceramic Engineering

Mrs. Anita Fagan
Junior, Ceramic Engineering

The pore size distribution of rat tibia and femurs has been studied by mercury porosimetry and procedures worked out. This work is being done in cooperation with Dr. David Baylink of the Veteran's Administration Hospital and the University Medical School to support his work on osteoporosis (or decrease of bone mass) of aging. Useful data on the size and volume of canalicular pores (0.2 and smaller) have been obtained on 0.15 g. samples. A brief paper was presented on this work at the Third Annual Bioengineering Symposium.

MATERIAL CHARACTERIZATION

James I. Mueller
Professor, Ceramic Engineering

David A. Boyd
Research Assistant, Ceramic Engineering
M.S. Thesis Research

The purpose of this project is to develop new methods of characterizing ceramic materials and to increase our abilities in utilizing existing tehcniques.

This work will involve use of x-ray defraction, electron microscopy, electron microprobe analysis to assist others in the process of characterization of materials of interest.

CERAMIC MATERIALS RESEARCH PROGRAM IMPACT STUDY

James E. Rosenzweig
Professor, Management and Organization

Fremont E. Kast
Professor, Management and Organization

John W. Stockman
Predoctoral Associate, Management and Organization
Ph.D. Thesis Research

The objective of this study was to determine the impact of the Ceramic Materials Research Program upon the University of Washington. The study investigated the impact of the CMRP at three levels: (1) the Ceramic Engineering Division, (2) the College of Engineering, and (3) the University.

This study has been completed and the final report was submitted to the Office of University Affairs, National Aeronautics and Space Administration on November 19, 1969. In the first phase of a three-phase project we identified the administrative, organizational, and structural relationships of the CMRP from the beginning of the program in 1963 to the present time. We investigated the changes in the program activity--such as number of graduate students, number of seminars, and extent of faculty involvement.

The second phase of the study: the interview-questionnaire program involved a social-psychological analysis of the attitudes, opinions, and reactions of the various participants in the CMRP. We also interviewed a number of people within the NASA organization to obtain information concerning NASA-sponsored university programs.

The final stage of the study: the interpretation and evaluation of data and the writing of the final report, was completed during the Summer and Autumn, 1969. We reported our findings concerning the impact of the CMRP and also provided information concerning the organization and administration of CMRP, the interface between this program and NASA, and a general evaluation of progress toward program objectives. The draft of the final report on this research was reviewed with NASA officials in Washington D.C. on August 5, 1969.

Mr. John W. Stockman completed his Doctorate in Business Administration in August, 1969. He received his support from the grant funds during the 1968-69 academic year for research on this program. His thesis is entitled "An Appraisal of the Impact of an Interdisciplinary Research Program: The Ceramic Materials Research Program at the University of Washington".

APPENDIX A-1

Distribution of Projects Within the University According to Research Areas

Academic Department	Number of Projects	Chemical	Mechanical	Atomic & Molecular	Process	Miscl.
Ceramic Engineering	21	5	6	4	2	4
Chemistry	3	2	-	1	-	-
Civil Engineering	3	-	3	-	-	-
Electrical Engineering	1	-	-	1	-	-
Management & Organization	1	-	-	-	-	1
Mechanical Engineering	1	-	1	-	-	-
Metallurgical Engineering	4	1	3	-	-	-
Physics	3	-	-	3	-	-
TOTAL	37	8	13	9	2	5

APPENDIX A-2

Number of Students and Faculty Involved in Research Supported by Grant Funds

Academic Department	Number of Projects	Faculty	Research Faculty	Under Grads	MS	Ph.D.	Total Grads
Ceramic Engineering	21	6	2	1	6	15	21
Chemistry	3	3	-	-	-	3	3
Civil Engineering	3	3	-	-	1	2	3
Electrical Engineering	1	1	-	-	1	-	1
Management & Organization	1	2	-	-	-	-	-
Mechanical Engineering	1	1	-	-	2	-	2
Metallurgical Engineering	4	3	-	-	4	1	5
Physics	3	4	-	-	-	3	3
TOTAL	37	23	2	1	14	24	38

APPENDIX B

Ceramic Materials Research Seminars

"Phonon Scattering by Conduction Electrons and Lattice Vacancies in Transition Metal Carbides", Dr. Wendel S. Williams, Visiting Professor, Ceramic Engineering

"Grain Boundary Diffusion", P. L. Pratt, Professor of Crystal Physics Imperial College, London

"Solubility of Oxygen in Zirconium Carbide", Satya K. Sarkar, Predoctoral Research Associate, Ceramic Engineering

"Heat Content of Defect Zirconium Oxide at High Temperature", Dr. S. Kimura, Research Associate, Ceramic Engineering

"Raman Spectroscopy", Dr. John W. Macklin, Assistant Professor, Chemistry

"Unfamiliar Oxidation State of Zirconium and the Effect of Oxygen Activity on the Structure of ZrO_2", Mr. K. Manikatan Nair, Predoctoral Assistant, Ceramic Engineering

"Research in Ferroelectric Ceramics at the University of New South Wales", Dr. Fennimore N. Bradley, Associate Professor of Ceramic Engineering, Michigan Technological Institute, Houghton, Michigan

APPENDIX C

Theses Published:

"Design and Construction of an Induction Plasma System for Single Crystal Growth in the Verneuil Geometry"
David A. Bucy, M.S., Metallurgical Engineering

"Effect of Impurities on the Mechanical Behavior of MgO Single Crystals"
M. Srinivasan, M.S., Metallurgical Engineering

"Unfamiliar Oxidation State of Zirconium and the Effect of Oxygen Activity on the Structure of ZrO_2"
K. M. Nair, Ph.D., Ceramic Engineering

"Solubility of Oxygen in Zirconium Carbide"
S. K. Sarkar, Ph.D., Ceramic Engineering

"A High Temperature Torsion Effusion Study of the Interaction of Metal Oxides with Graphite and with Metal Carbides"
Juey Hong Rai, Ph.D., Chemistry

"An Appraisal of the Impact of an Interdisciplinary Research Program: The Ceramic Materials Research Program at the University of Washington"
John W. Stockman, Ph.D., Business Administration

Papers Accepted for Publication:

"The Time Dependence of Effusion Cell Steady-State Pressures of Carbon Monoxide and Calcium Vapors Generated by the Interaction of Calcium Oxide and Graphite," Juey Hong Rai and N. W. Gregory has been accepted by the Journal of Physical Chemistry.

"An Effusion Study of the Reaction of Zirconium Carbide and Calcium Oxide," Juey Hong Rai and N. W. Gregory, has been accepted by the Journal of Physical Chemistry.

"High Temperature Isotropic Plasicity of Graphite," D. B. Fischbach, has been accepted by the Philosophical Magazine.

"Mössbauer Studies of Lattice Dynamics, Fine and Hyperfine Structure of Divalent Fe^{57} in FeF_2," D. P. Johnson and R. Ingalls, has been accepted by Physical Review.

Papers Submitted for Publication:

"Effect of Impurities on the Mechanical Behavior of MgO Single Crystals," M. Srinivasan and T. G. Stoebe, has been submitted to the Journal of Applied Physics.

"Mössbauer Study of Low Temperature Anharmonicity in ThO_2:Co^{57}," H. Shechter, J.G. Dash, G. A. Erickson and R. Ingalls, has been submitted to Physical Review.

Papers Published:

"The Effect of Gas Flow Rate on the Oxidation Kinetics of Zirconium," L. P. Srivastava and T. F. Archbold, Scripta Metallurgica, Vol. 3, p. 377, (1969).

"Comments on Divalent Fe^{57m} Quadrupolar Coupline Constants," R. Ingalls, Physical Review, December 1969.

Papers Presented:

"On the Relationship of Mechanical Properties to Structure in Carbons and Graphite," D. B. Fischbach. Presented at the 16th Refractory Composites Working Group Meeting, Seattle, October 13-15, 1969.

"Extreme Low Temperature Anharmonicity of Fe^{57} in ThO_2," G. A. Erickson, J. G. Dash, R. L. Ingalls and H. Shechter. Presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Seattle, October 17, 1969.

"Anharmonicity in Thorium Oxide," H. Shechter. Presented at the Gordon Research Conference, Meriden, New Hampshire, August 4, 1969.

"Variables in the Compaction Behavior of Ceramic Particles," D. B. Leiser and O. J. Whittemore, Jr. Presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Seattle, October 17, 1969.

"Heat Content of Defect Zirconium Carbide at High Temperature," A. D. Miller, S. Kimura, H. W. Schimmelbusch and J. I. Mueller. Presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Seattle, October 17, 1969.

"Oxygen Solubility in Zirconium Carbide," S. K. Sarkar and J. I. Mueller. Presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Seattle, October 17, 1969.

"Current Ceramic Research at the Toyko Institute of Technology," Shiushichi Kimura. Presented at the Eastern Section of the American Ceramic Society, Richland, Washington, September 18, 1969.

APPENDIX D

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