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College of Engineering

Ceramic Engineering Division

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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 NsG-484. The principal purposes of the grant are to encourage multidisciplinary 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. A. Pifer, Chairman of the Department of Mining, Metallurgical and Ceramic Engineering; and James I. Mueller, Principal Investigator.

GENERAL PROGRAM REPORT

This is the eleventh semiannual status report and it covers the first half of the sixth year of operation under this grant. During the report period, a total of thirty-two projects were supervised by nineteen faculty members in eight academic disciplines of the University. Several new projects were initiated during the report period. Professors B. J. Hartz and M. D. Coon, Department of Civil Engineering, began projects on the mechanical properties of ceramic materials. Professors James Rosenzweig and Fremont Kast, Department of Management and Organization, School of Business Administration, initiated a study to determine the socio-academic effects of the grant upon the University.

The Ceramic Materials Research Seminar, a period devoted to discussions of concepts and research of interest to the program, met for a total of twelve sessions during the report period. Speakers included two University of Washington faculty members and ten visitors. The exchange seminar programs with Battelle-Northwest Laboratories and with Tektronix, Inc. have continued and an exchange program was initiated during the report period with the U.S. Bureau of Mines in Albany, Oregon. A complete list of seminar speakers and their topics is included as Appendix B.

The program supported the attendance of twelve faculty members to a total of nine technical meetings at which fourteen papers based upon work supported by the grant were presented. Of particular significance is the role of our supervisors in the program of the Pacific Coast Regional Meeting of the American Ceramic Society. The twelve papers based upon work supported by this grant presented by ten faculty members represented about 17% of the technical papers scheduled for this meeting. Papers published or presented resulting from work supported wholly or in part by the grant are listed with the individual status reports and in Appendix C.

Funds from the grant were used to supplement University support of a visiting professor during Summer Quarter. Dr. Edmund K. Storms, Staff Scientist, Los Alamos Scientific Laboratory, offered a four-week, three-credit course during the "b" term of Summer Quarter. The course, entitled "Refractory Carbide Compounds," had an enrollment of eighteen students.

In light of Dr. Storm's visitation, the first Working Group Meeting on Basic Properties of Actinide and Transition Metal Refractory Compounds was held August 8-9 at the Battelle Science Center in Seattle. This conference, co-sponsored by Battelle-Northwest and the Ceramic Materials Research Program, was very successful and a second meeting is being planned for the summer of 1969. Details of the conference will be found in Appendix D.

An exchange program was initiated with Rensselaer Polytechnic Institute wherein a faculty member from each school involved with the NASA-IDL programs spent one half month at the other institution. Professor Heribert Wiedemeier, of RPI, spent the period July 22 to August 5 on our campus and Professor A. D. Miller visited RPI from August 16 to August 30. Each spent the time discussing common research interests with faculty and students of the other institution.

The new selective service laws relative to draft deferments for graduate students had a definite effect upon the program. Only two new graduate students began their studies in the ceramic engineering division this fall. These were from a total of ten students who had been accepted and offered assistantships. Of the eight declining, one is attending another institution, one entered the military service, and the remaining six accepted employment in hopes of getting occupational deferments. In order to maintain an active research level, Dr. Shiuschi Kimura, a member of the ceramic staff at Tokyo Institute of Technology, was given a nine-month appointment as Senior Research Associate. He is currently working with Prof. A. D. Miller on the high temperature calorimeter study.

Invited visitors again attended the annual fall technical review October 28-29, 1968. In addition to Mr. James J. Gangler, NASA technical monitor, a total of seventeen visitors from other federal agencies, industry, universities and non-profit research organizations attended. A summary of this program will be found in Appendix E.

Professor T. G. Stoebe, Metallurgical Engineering Division, received an NSF grant for a High School Teachers' Research Participation Program during the summer of 1968. This supported a total of five high school science teachers for a period of two months, during which time they worked with faculty members and graduate students on current research. Two of these were associated with projects of this program. They included Mr. David Wilson, who was associated with Professor Stoebe working on "Mechanical Properties of LiF Single Crystals" and Mr. James R. Ezell, working with Professor A. D. Miller on "Bonding in Interstitial Compounds."

RESEARCH STAFF

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CHEMICAL

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.

Studies of the Zirconium Dioxide-Carbon Reaction

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

A ternary material (zirconium oxycarbide) of 98% or more purity has been produced from a mixture of ZrO_2 and carbon heated to 1800°C in a graphite-tube furnace with a CO atmosphere. The composition of this compound has not yet been established, but the lowest lattice parameter observed was 4.671Å.

Information has been obtained regarding the kinetics of oxycarbide formation at a given temperature as a function of time and CO pressures. Variation of CO pressure has not shown any effect on the kinetics of oxycarbide formation from a mixture of ZrO_2 and carbon. Higher CO pressures, however, result in the formation of oxycarbide which shows better x-ray diffraction peak resolution. Initial oxidation studies of the oxycarbide indicate that it may have a slightly better oxidation resistance than pure ZrC.

Zirconium oxycarbide has been formed from mixtures of ZrO_2 and ZrC at 1800 and 1900°C. Depending upon the temperature and CO pressure, one or two f.c.c. phases are formed. The intensity of x-ray diffraction peaks indicate the rate of oxycarbide from these mixtures is much slower than from the ZrO_2-graphite mixtures.

Apparatus has been set up for the determination of nitrogen by Dumas method, and this system is in the process of calibration. Oxygen and carbon determinations on the oxycarbides are being made using the Leco Analyzers.

A paper entitled "The Zirconium-Oxygen-Carbon System: II, Identification of Zr_2CO," by S. K. Sarkar and J. I. Mueller, was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

Influence of Oxygen Activity on the Structure of Zirconium Oxide

K. M. Nair
Predoctoral Associate, Ceramic Engineering
Ph.D. Thesis Research

The purpose of this study is the determination of the effects of low oxygen partial pressure at high temperatures upon the stability of ZrO_2. It is planned to study the possible existence of lower oxides of zirconium, their dependence upon oxygen activity and temperature, and the influence of the formation of such an oxide upon the formation of a ternary compound in the Zr-O-C system.

No stable oxide compounds of zirconium lower than ZrO_2 have been observed in the equilibrium study of Zr-O systems. Study has been concentrated on two typical temperatures (1) just above the monoclinic-tetragonal transformation (1200°C) and (2) below the tetragonal-cubic transformation of ZrO_2 and between log pO_2 = -9.8 to log pO_2 = -13.5. No observable changes both in the relative intensities and in the d-spacings are noticed, but the color of the sample varies from pure white to grey. Change of temperature in hydrogen atmosphere does not produce any change in the d-spacings. These 'black oxides' change to an almost white color when heated in air or oxygen without any change in the x-ray diffraction pattern.

Non-equilibrium study of Zr-O systems has resulted in a material which has a lattice parameter (a_o) of 3.8956Å with the ZnS structure. Further study on the oxidation of this material under extremely oxygen-free argon atmosphere is underway, hoping to explain the "blackening" property of ZrO_2 and the mechanism of formation of ternary compounds.

A paper entitled "The Zirconium-Oxygen-Carbon System: I, Introduction," by J. I. Mueller and A. D. Miller, was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

SOLID-SOLID EQUILIBRIUM

N. 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
Ph.D. Thesis Research

The objective of this research is to study kinetic and thermodynamic characteristics of chemical reactions between graphite and metal oxides and between metal oxides and metal carbides by measuring the gas pressures (of CO, or, where formed, of CO + volatile metal) developed by the interacting solids in torsion effusion cells.

During this period a considerable amount of experimental work has been done on reactions involving calcium oxide. Steady-state pressures of Ca(g) and CO(g), generated by reaction of CaO with graphite and by CaO with ZrC, respectively, in effusion cells, have been followed as a function of time by the torsion effusion method. Pressures decrease in a manner generally consistent with the assumption that the rate of production of vapor is controlled by the interfacial contact area and, in the second case, by diffusion of the reactants through the product layer. Such a model was used to derive a suitable form for extrapolation of steady-state pressures back to time zero to derive predicted equilibrium constants for the reactions. The results, and associated thermodynamic properties, are in good agreement with expected values. A treatment of this kind appears necessary for the study of oxide and carbide solid phases by the effusion method.

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.

The soft x-ray spectrometer was assembled and is being checked out. Considerable time and effort have been spent optimizing the thin window assembly on the detector to give well-controlled pressures in the vacuum chamber. Once this problem is adequately solved, the data collection can begin.

Structural Ordering of Zr(C,O)

John N. Hale,
Research Assistant, Ceramic Engineering
M.S. Thesis Research

This project will investigate the degree of ordering, if any, in the nonmetal sublattice of the phase $ZrC_{0.5}O_{0.5}$. It is felt that if this composition is distinct from the ZrC phase there must be strong ordering among the carbon and oxygen atoms.

Various Zr(C,O) compositions have been synthesized in the amounts necessary for the fabrication program. The carbon, oxygen and nitrogen contents are being determined using combustion techniques. A modified Damas nitrogen apparatus was built to evaluate the nitrogen contents.

Arrangements have been made with Oak Ridge National Laboratory to perform neutron diffraction experiments on these materials when the compositions are well known.

A graphite die hot-press is being assembled for the fabrication of high density specimens.

A paper entitled "The Zirconium-Oxygen-Carbon System: III, Preliminary Study of Electronic Band Structure" by A. D. Miller and J. I. Mueller, was presented at the American Chemical Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

ZIRCONIUM OXIDATION

Thomas F. Archbold
Associate Professor, Metallurgical Engineering

This research project is investigating the characteristics and mechanisms of the early stages 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

L. P. Srivastava
Predoctoral Associate
Ph.D. Thesis Research

This project is investigating the short-time oxidation kinetics of zirconium and morphology of the oxide films. Both electropolished and abraded specimens have been oxidized at temperatures in the range 440-856°C. The gas flow rate was found to influence the oxidation kinetics up to a certain point, beyond which there is no effect. The partial pressures of oxygen were 0.01, 0.2, and 1 atm at the various temperatures, while the maximum oxidation time was 3 minutes. The analysis of the weight change vs time of oxidation data indicates that the kinetics can be expressed as a combination of an adsorption term (linear) and a diffusion term (parabolic). A consideration of the pressure dependency indicates that the oxide films are n-type below approximately 700°C and p-type above this temperature. The metal's thermal and mechanical history, as well as the surface preparation, are reflected in the kinetic data and the electron microscope observations of the oxides.

L. P. Srivastava received a Ph.D. in Metallurgical Engineering December 19, 1968. Thesis title: "The Early Stages of the Oxidation of Zirconium Metal."

A paper entitled "Oxidation of Zirconium" by T. Archbold and L. P. Srivastava was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

R. Darolia
Research Assistant, Metallurgical Engineering (September 15 to December 15)
M.S. Thesis Research

Dr. Srivastava's preliminary electron microscope studies of the stripped zirconia films will be completed. Efforts will be made to extend the research to the oxidation of ZrC and to examine the films produced by the reaction of zirconium with air.

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

Shiuschi Kimura
Research Associate, Ceramic Engineering (December 1 to December 15)

Hugo W. Schimmelbusch
Research Assistant, Metallurgical Engineering (June 15 to June 30)
M.S. Thesis Research

The construction of the calorimeter has been completed. The calorimeter consists of the following: drop mechanism, vertical high temperature tube furnace, upper and lower gates, calorimeter vessel-fin assembly, vacuum-inert gas valving system, sample weigh equipment, constant temperature bath, furnace control and sample temperature measuring equipment, and inert gas purifying train.

Calorimetric investigations of non-stoichiometric ZrC have been initiated. One of the results will be to obtain the heat capacity as a function of composition at various temperatures and to obtain information on its defect structure.

Hugo W. Schimmelbusch received an M.S. degree in Metallurgical Engineering December 19, 1968. Thesis title: "Construction of a High Temperature Diphenyl-Ether Calorimeter."

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
Research Assistant, 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.

Flat surfaces of exceptional microscopic smoothness have been produced on alumina and several bicrystals have been fabricated. A device for applying constant load during high temperature deformation has been constructed and tested along with associated strain measuring instrumentation. A bicrystal has been sheared and it is believed to be the first observation of grain boundary sliding in this type of specimen. A complex pattern of basal slip and cracking was also present. Further experiments will be conducted to isolate and further investigate the sliding phenomenon.

One of the primary mechanisms of polycrystalline deformation at high temperature is thought to be grain boundary sliding. The kinetics of this process and the relationship of boundary sliding to dislocation motion in the crystals adjacent to the boundary will be investigated in pressure sintered bicrystals of controlled misorientation.

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.

Small single crystal specimens for deformation and polyginization have been oriented and cut from boule material. A device for bending these crystals was constructed, and, although made of Mo, was not strong enough to deform the alumina at 1400°C. Redesign of the specimen dimensions and loading arrangement is being carried out.

A paper entitled "Ceramic Materials for Structural Components" by W. D. Scott was presented at a meeting of the Pacific Northwest Ceramic Association at Harrison Hot Springs, British Columbia, November 9, 1968.

ZrC COATINGS

Colin J. Sandwith
Assistant Professor, Mechanical Engineering

James D. Danberg
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.

Tensile bond strength tests show that a change in the type of exit nozzle (angle of impingement between plasma and powder, and location of impingement) used on the plasma gun can alter the bond strength by as much as 40%. Nozzle B (83° is angle between the pure plasma gas and the pure powder, and impingement takes place just prior to exit from the enclosed nozzle) gives the best results - 5,500 psi.

A computer program (CINDA-Crystler Improved Differential Analyser) has been applied to predicting the surface temperature during heating by a moving heat source as a function of coating type, coating thickness, source output, source velocity, sensor location, and, most important, size and type of unbound. This method of predicting the output from nondestructive infra-red test yields in a few minutes (8 minutes computer time) approximate test results for any reasonable combination of the parameters and eliminates the near impossible task of fabricating specimens with known types of unbonds or separations. Results from this program indicate convention sensors and heat sources (10 Btu/min. over circular region of approximately 1/2" diameter) are not suitable for detecting an unbound which exhibits from 60 to 100% of the surrounding heat transfer properties.

A paper entitled "Nondestructive Thermal Test for Separation of ZrC Coatings" by James Danberg and C. J. Sandwith was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

DEFECT PROPERTIES OF IONIC AND CERAMIC CRYSTALS

Thomas G. Stoebe
Assistant Professor, Metallurgical Engineering

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

Mechanical Properties of LiF Single Crystals

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

An investigation of the effects of strain rate, temperature and impurity content on yielding and work hardening has been performed.

A literature review on latent hardening has been completed and work in this area is commencing.

A paper entitled "Effect of Impurities on Mechanical Properties of LiF Single Crystals" by H. L. Fotedar and T. G. Stoebe was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 23, 1968.

A paper entitled "Factors Affecting the Mechanical Behavior of LiF Single Crystals" by H. L. Fotedar and T. G. Stoebe has been published in Scripta Metallurgica 2, 443 (1968).

Mechanical Properties of MgO Single Crystals

M. Srinivasan
Research Assistant, Metallurgical Engineering
M.S. Thesis Research

Deformation studies in highly pure and Ni-doped MgO single crystals are being performed as a function of temperature to determine the effects of purity and temperature on yielding behavior.

Ageing Behavior of LiF Crystals

J. K. Lee
Research Assistant, Metallurgical Engineering
M.S. Thesis Research

The lattice defect structure of LiF single crystals is being studied after quenching and during low temperature ageing using ionic conductivity techniques.

CONTINUUM STRESS ANALYSIS OF CRYSTALLINE CERAMICS

B. J. Hartz
Professor, Civil Engineering

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.

Stresses in Crystalline Ceramics due to Anisotropic Mismatch

Girindra Das
Predoctoral Research Associate, Civil Engineering (September 16 to December 15)
Ph.D. Thesis Research

Initially this work is aimed at a better theoretical solution for the stresses in the aluminum oxide bicrystals investigated experimentally by Mr. Henry Y. B. Mar under the direction of Professor W. D. Scott.

Due to the boundary conditions the solution of a realistic approximation to this problem by classical methods in the theory of elasticity does not seem possible. Consequently the most promising numerical techniques, that of "finite element representation" and closely related numerical methods suitable for automatic calculation on high speed digital computers, are being pursued.

In order to evaluate the approximation involved this problem is first being solved using two-dimensional elasticity models for the bicrystal with induced stresses due to anisotropic thermal expansion, with the possibility of superposition of plate bending solutions for the warping of the crystals. Following this, a three-dimensional finite element model will be used. However, since available three-dimensional finite element programs are considerably restricted in size, it is not expected that this will give adequate results and a three-dimensional technique, closely related to "finite elements" but using high order polynomials and regular subdivisions to make possible automation of the computation, is being adapted to this problem. This appears to be the best possibility at present for a realistic assessment of the stresses in the bicrystal. Using this method, it is expected that a realistic evaluation can also be made of the mechanical strength of the bicrystals and of the stresses caused by anisotropic material properties in stressed polycrystalline ceramics.

Residual Stresses in Ceramic Beams

Max D. Coon
Assistant Professor of Civil Engineering

A series of tests is proposed based on the hypothesis that it is possible to induce favorable residual stress distributions in ceramic structural components by utilizing a program of heating - loading - unloading and cooling, making use of the considerable ductility of the ceramic material at elevated temperature. If this is possible for certain ceramic materials it should be possible to obtain considerably improved strengths for these materials by this device.

Preliminary investigation has been made to identify the material characteristics necessary for this hypothesis. A sequence of tests has been devised capable of establishing whether favorable residual stresses can be induced in a particular material by a simple heating - loading - unloading - cooling cycle.

The material characteristics needed appear to be within the realm of certain ceramic materials and the tests can be conducted without extensive modification to available equipment.

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

L. Annapoorni
Research Assistant, Ceramic Engineering (June 16 to September 15)
M.S. Thesis Research

Interest in the effects of ultra-violet radiation upon the properties of certain ceramic materials continues to be strong in many laboratories. Efforts here are centered on understanding the thermoluminescence induced in crystals by ultra-violet radiation in terms of trace impurities and lattice defects. The thermoluminescent glow curves from several MgO crystals containing various impurities have been measured and the difficult work of correlating these emissions with specific impurities and defects is in progress. This work involves the study of the glow curves as a function of irradiation time and temperature and irradiation wavelength. Monitoring the strength of impurity absorption bands during heating will also give hints as to the role of certain impurities in the thermoluminescent process. Measurement of the wavelength spectra of the glow peaks continues to be a difficulty due to the very low intensities of these emissions.

DOMAIN DYNAMICS IN ISOMORPHOUS FERROELECTRICS

John L. Bjorkstam
Professor, Electrical Engineering

The objective of this investigation is to develop a microscopic model which explains essential features of the polarization reversal process in hydrogen bonded ferroelectrics of the KH_2PO_4

Ferroelectric Domains and Domain Motion in KH_2PO_4 and KD_2PO_4

Richard E. Oettel
Predoctoral Associate, Electrical Engineering
Ph.D. Thesis Research

The domain velocity for partially deuterated KH_2PO_4 samples was found to have an exponential temperature dependence of the form log v = K/T over the temperature interval 160°K - 205°K. (The Curie temperature for these samples is 209°K.) In addition to this activity some attention has been given to the optical properties of domains. Under certain conditions domains can be made to act as tunable optical waveguide, a property which has potentially useful applications.

In addition to contributions in understanding the basic microscopic crystal properties which limit the switching rate of ferroelectrics, our research is concerned with the general problem of domain formation and polarization reversal in ferroelectrics. Many of the proposed uses for ferroelectrics depend upon these properties.

A paper entitled "Optical Observation of Domain Nucleation, Interaction and Motion in KH_2PO_4 and KD_2PO_4 During Polarization Changes" by Richard E. Oettel and John L. Bjorkstam, was presented at the American Ceramic Society Pacific Coast Regional Meeting in Pasadena, California, October 24, 1968.

STUDIES ON GASH

Edward C. Lingafelter
Professor, Department of Chemistry

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

The objectives of this study are to investigate the mechanism of the ferroelectricity of Guanidinium Chromium Sulfate Hexahydrate (GCrSH) and its aluminum isomorph (GASH).

The electrical circuitry used to display the hysteresis loops of both large and small (x-ray size) crystals is now fully operative.

X-ray diffractometer data have been collected on a GCrSH crystal while it was poled in the direction of the c axis with a d.c. voltage large enough to ensure that the condition of the crystal corresponded to the saturation region of its hysteresis loop. To prevent the crystal from arcing while in the presence of the x-ray beam, the crystal was enclosed in a coat of glue. During the x-ray data collection, the crystal was changing its structure in some unknown fashion. Also, the hysteresis loops obtained at intervals during the x-ray data collection had different characteristics and indicated that the poling voltage should be increased to maintain the crystal in a saturated condition when exposed to x-rays. Eventually the crystal arced and decomposed.

In order to devise some technique for determining the structure of a poled crystal of GCrSH, we are currently systematically studying the effects of a.c. and d.c. fields and x-rays on the hysteresis loops of both large and small GCrSH crystals. To determine if the hysteresis loops depend upon the frequency of the applied a.c. fields, the circuitry is being adapted to include use of a variable d.c. voltage source. It is hoped that these studies also will provide some additional information for understanding the ferroelectric mechanism in GCrSH.

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

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

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

C. D. West
Research Assistant, Department of Physics
Ph.D. Thesis Research

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, each of which is separately described below.

Part A. Charging in alloys

The silver-tin system is being studied via the isomer shifts observed in the Mössbauer spectra. A theoretical analysis of this experimental shift provides relations that can be used for the study of the electron band structure.

Part B. Anharmonic force constants

A study of ThO_2 containing Fe impurities is nearing completion. Designed to test a simple theory of anharmonic force constants developed last year in connection with the work of $FeCl_2$, the present study appears to confirm our prediction that low temperature anharmonicity occurs when the nearest-neighbor ionic cages provide a "rattle space" larger than the caged ion. In ThO_2 the rattle space provided is about 0.5 Angstroms: measured f-factors and the known crystal structure of the pure material yield approximately the same value. A detailed analysis of f and the line shift indicate that the potential acting on the Fe ions has a central maximum. The measured quadrupole splittings and line widths indicate that the relaxation times of the electronic levels are comparable with the level splitting, and a detailed comparison has been made by means of an extension of the theory of relaxation effects of nmr lines. On the basis of our results, we believe that ThO_2:Fe will display ferroelectric ordering. A sample containing a high concentration of non-radioactive Fe has been prepared for testing by Professor Bjorkstam.

Pact C. Study insulators as a function of temperature and pressure

1) Three experiments in progress are outlined as follows: The Mössbauer effect is used to find the valence state and its symmetry in the case of Fe-57 impurities in insulators. A Mössbauer pressure cell was built for pressures up to 300,000 atm. This system will be first used on ThO_2 as a complementary study to the anharmonicity.

2) The effect of electric polarization on the relative intensity of the quadrupole-split lines of some insulators is being studied. The experimental results for the first material ($FeSiF_6 \cdot 6H_2O$) are being computer-analyzed according to existing theory.

3) The $KFeF_3 \cdot XH_2O$ has shown interesting results, apparently strongly affected by the amount of water in the crystal. Different degrees of drying cause changes from completely divalent spectrum for the Fe-57 ions to entire trivalent spectrum. Although it is not yet understood how this effect can be interpreted, the results may explain the source of impurities in this material.

A paper entitled "Low-Temperature Anharmonicity in $FeCl_2$," by D. P. Johnson and J. G. Dash, has been published in the Phys. Rev. 172, 3 (August 15, 1968).

A paper entitled "Low-Temperature Anharmonicity and the Debye-Waller Factor," by J. G. Dash, D. P. Johnson and W. M. Visscher, has been published in the Phys. Rev. 168, 3 (April 15, 1968).

A paper entitled "Quadrupole Hyperfine Anisotropy in $Fe(NH_4SO_4)_2 \cdot 6H_2O$ and its Comparison with the Magnetic Susceptibility," by R. Ingalls, K. Ono and Louis Chandler, has been published in the Phys. Rev. 172, 2 (August 10, 1968).

A paper entitled "Mössbauer Studies of Low Temperature Anharmonicity," by J. G. Dash, was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

IMPURITY DIFFUSION IN MgO UNDER THE INFLUENCE OF AN ELECTRIC FIELD

William D. Scott
Assistant Professor, Ceramic Engineering

Chester A. Hinman
Predoctoral Associate, Ceramic Engineering (June 16 to September 20)
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.

A paper entitled "Effect of Magnesium Oxide on the Interfacial Energy of Alumina Bicrystals" by M. W. Matson and W. D. Scott was presented at the Pacific Coast Regional Meeting of the American Ceramic Society in Pasadena, California, October 23, 1968.

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

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. This phenomenon has been shown to occur during the sintering of several ceramic materials, and it has also been shown that it may occur simultaneously with shrinkage. Auxiliary objectives are to determine whether pore growth occurs generally during sintering and the controlling mechanisms.

A study of the compaction behavior of Linde A alumina versus degree of aggregation was conducted so that a range of initial porosities (and, to some extent, pore sizes) can be prepared for sintering studies. Linde A, compacted as received, has a bimodal pore distribution due to aggregation which can be eliminated by dispersion before compaction. Then, the pore size is reduced but not to the one-seventh ratio to particle size found in other compacted aluminas.

Surface areas were determined several times on several powders of sintering interest to also establish the precision and deviation of the "Sorbet" apparatus.

Characterization and Forming

Douglas J. Calkins
Predoctoral Associate, Ceramic Engineering **(June 16 to September 15)**
Ph.D. Thesis Research

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

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

To study small void filling as related to brittle fracture, glass spheres of uniform size and sphericity are required. Another particle discriminator has been designed and constructed together with a special feeder, which will sort spheres precisely and can be operated without constant attention. Other equipment constructed include a "felvation" unit in which particle sieving is achieved by carrying particles upward in a stream of water through sieves. To determine the pore distribution after compaction, mercury porosimetry will be employed. The present equipment has been improved by adding a fan to control the pressure chamber temperature and more precise pressure gauges are being added for additional accuracy.

Compaction studies to 25000 psi in carbide dies have been conducted on fused magnesia (cube-shaped particles), and on two different shapes of fused nullite and the data fitted to the two-part equation of Cooper and Eaton. Studies to 50,000 psi are being planned, using smaller dies in the Instron machine.

A study was completed of pore size distributions in compacts of various fine aluminas with varying additions of wax. The addition of wax did not affect the pore distribution between the alumina particles (determined by burning out the wax) until over 30 volume % had been added. The range of pore sizes was found to be narrow and the pore size was found to be one-seventh the particle size of two aluminas.

A paper entitled "Compaction as a Ceramic Forming Process," by O. J. Whittemore, Jr., was presented at the American Ceramic Society, Pacific Coast Regional Meeting, Pasadena, California, October 24, 1968.

A paper entitled "Compaction as a Ceramic Forming Process," by O. J. Whittemore, Jr. was presented at a meeting of the Pacific Northwest Ceramic Association, Harrison Hot Springs, British Columbia, November 9, 1968.

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 the investigation is the determination of the effect of temperature on the surface diffusion coefficient of chromium on aluminum oxide.

During this report period construction and assembly of furnacing was the primary effort. In addition, the specimen assembly necessary to achieve the experiment configuration was fabricated and assembled.

FORSTERITE DIELECTRICS

James I. Mueller
Professor, Ceramic Engineering

Roger W. Hanson
Research Assistant, Ceramic Engineering (July 16 to August 22)
M.S. Thesis Research

Various raw materials and firing temperatures are being studied as to their individual and combined effects upon the crystalline composition and microstructure. Reaction studies are underway using qualitative and quantitative x-ray diffraction analyses of specimens fired in a gradient furnace. Reflected and transmitted optical microscopy and pore size are also being used.

FERROELECTRIC MATERIALS STUDY

Robert J. Campbell, Jr.
Assistant Professor, Ceramic Engineering

The objective of this study is the development of an explanation of the mechanism of energy storage in ferroelectric materials, resulting from displacement of atoms in the crystal lattice.

USE OF CERAMICS IN CONTROLLING ANTENNA SYSTEM PARAMETERS

Irene C. Peden
Associate Professor, Electrical Engineering

The purpose of this research is to study the characteristics of ceramics as modelling materials for antenna systems.

Ceramic Models of the Lunar Surface

Richard L. Hendrickson
Research Assistant, Electrical Engineering
M.S. Thesis Research

A conceptual model of the lunar surface is developed and presented, in terms of a stratified, lossy dielectric medium. Only those properties of the lunar material are treated which are pertinent to the electromagnetic characteristics of concern in the design and implementation of a future radio communication system on the moon. The results of specific laboratory measurements on an apparently suitable terrestrial substitute for the lunar soil are presented. Optical measurements of the brightness curves of naturally occurring olivine basalt rock were made, utilizing photometric equipment constructed in the laboratory. These measurements were made to determine the validity of the selection of basalt as a suitable lunar substitute material, following methods used in similar optical investigations reported in the literature. The complex dielectric constants of solid and powdered samples of basalt were measured, for the frequency range expected to be practical for lunar radio communications, 100 khz to 30 mhz.

A modeling facility, utilizing ceramic dielectric materials in the microwave frequency range, is available for experimental analysis of certain antenna characteristics. The properties of this model are sufficiently close to those of the desired lunar model to justify its use without modification. The theoretical antenna pattern of an electrically short, vertical radiator over a circular ground plane was computed, using the dielectric properties of basalt for the ground constants of the surrounding medium. This pattern is compared with the pattern computed for the same antenna using the ground constants of the existing model. The theoretical analysis was made using a computer program previously written for an analytical solution to the problem, as reported in the literature. Model measurements of the antenna patterns of a vertical monopole over a ground plane are presented in a study of the effects of burial in the lunar surface. The results are correlated with empirical and theoretical information, abstracted from the published work of others, in order to provide some of the basic information necessary to the meaningful study of lunar communications. In addition, a brief analysis of wave propagation over the lunar surface is given, including curves for surface wave propagation losses, utilizing analytical expressions available in the literature.

Richard L. Hendrickson received an M.S. degree in Electrical Engineering December 19, 1968. Thesis title: "A Ceramic Dielectric Model of the Lunar Surface, with Application to Lunar Antenna and Propagation Studies."

SINTERING OF CALCIA

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

Dennis Hotaling
Research Assistant, Ceramic Engineering
M.S. Thesis Research

The objective of this study is to determine the mechanisms of sintering of calcium oxide, especially from calcium hydroxide as the precursor.

Mr. Hotaling has asked leave for a year in which to obtain industrial experience.

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

We have identified the administrative, organizational, and structural relationships of the CMRP from the beginning of the program in 1963 to the present time. We have investigated the changes in the program and the factors causing these changes. Various measure of the level of program activity--such as number of graduate students, number of seminars, and extent of faculty involvement--have been utilized. This historical perspective of the CMRP provides the basis for the interview-questionnaire phase of the study.

The next phase of the study will involve a social-psychological analysis of the attitutes, opinions, and reactions of the various participants in the CMRP. Initially, we will utilize semi-structured interviews to obtain information from participants--graduate students, faculty, and administrative staff. We will also develop more structured questionnnaires in order to obtain explicit information. This interview-survey phase will be completed during the first six months of 1969. We will then move to the interpretation and evaluation of data and the writing of the final report.

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	17	6	2	3	3	3
Chemistry	2	1	-	1	-	-
Civil Engineering	2	-	2	-	-	-
Electrical Engineering	2	-	-	1	-	1
Management & Organization	1	-	-	-	-	1
Mechanical Engineering	1	-	1	-	-	-
Metallurgical Engineering	4	1	3	-	-	-
Physics	3	-	-	3	-	-
TOTAL	32	8	8	8	3	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	17	5	2	3	4	11	15
Chemistry	2	2	-	-	-	2	2
Civil Engineering	2	2	-	-	-	1	1
Electrical Engineering	2	2	-	-	1	1	2
Management & Organization	1	2	-	-	-	1	1
Mechanical Engineering	1	1	-	-	1	-	1
Metallurgical Engineering	4	2	-	1	4	2	6
Physics	3	3	-	-	-	3	3
TOTAL	32	19	2	4	10	21	31

APPENDIX B

Ceramic Materials Research Seminars

"High Temperature Electrical and Thermoelectric Properties of Actinide Oxides"
Dr. G. Lambert Bates, Battelle-Northwest

"Single Crystal Growth of Transition Metal Disulfides"
Dr. Sidney R. Butler, National Science Foundation, Washington, D.C.

"Proposed Theory for the Static Fatigue Behavior of Brittle Ceramics"
Dr. D. P. H. Hasselman, Berkeley, California

"U-C-O-N Phase Diagram"
Dr. J. L. Henry, U.S. Bureau of Mines Research Laboratories, Albany, Oregon

"NASA Sponsored Research at RPI"
Dr. Alan D. Miller, Assistant Professor, Ceramic Engineering, University of Washington

"A Student's View of Graduate Study in England"
Mr. John Rusin, Penberthy Electromelt, Seattle

"Electronic Structure of Disordered Systems"
Dr. Edward A. Stern, Professor of Physics, University of Washington

"A Review of High-Temperature Chemistry Research at Los Alamos"
Mr. Edmund K. Storms, Visiting Lecturer, Los Alamos Research Laboratories

"Composite Materials Technology"
Dr. A. D. Thomas, Jr., Materials Section Head, Tracor, Inc., Austin, Texas

"Evaporation Studies on the Chalcogenides"
Dr. Heribert Wiedemeier, Associate Professor, Chemistry, Rensselaer Polytechnic Institute, Troy, New York

"Phase Transformations in Titanium Alloys"
Dr. James Williams, North American Rockwell Science Center

"Microstructures of Graphites in Relation to Processing and Applications"
Mr. Eugene M. Woodruff, Battelle-Northwest, Richland, Washington

APPENDIX C

Theses Published:

"The Early Stages of the Oxidation of Zirconium Metal"
L. P. Srivastava, Ph.D., Metallurgical Engineering

"A Ceramic Dielectric Model of the Lunar Surface, with Application to Lunar Antenna and Propagation Studies"
R. L. Hendrickson, M.S., Electrical Engineering

"Construction of a High Temperature Diphenyl-Ether Calorimeter"
H. W. Schimmelbusch, M.S., Metallurgical Engineering

Papers Published:

"Factors Affecting the Mechanical Behavior of LiF Single Crystals," H. L. Fotedar and T. G. Stoebe, Scripta Metallurgica 2, 443 (1968).

"Low Temperature Anharmonicity in $FeCl_2$," D. P. Johnson and J. G. Dash, Phys. Rev. 172, 3 (August 15, 1968).

"Low-Temperature Anharmonicity and the Debye-Waller Factor," J. G. Dash, D. P. Johnson and W. M. Visscher, Phys. Rev. 168, 3 (April 15, 1968).

"Quadrupole Hyperfine Anisotropy in $Fe(NH_4SO_4)_2 \cdot 6H_2O$ and its Comparison with the Magnetic Susceptibility," R. Ingalls, K. Ono and Louis Chandler, Phys. Rev. 172, 2 (August 10, 1968).

"Structural Changes on Reheating Plasma-Sprayed Alumina," Vere S. Thompson and O. J. Whittemore, Jr., Bulletin of the American Ceramic Society, 47, 7, p. 637 (1968).

Papers Presented:

The following papers were presented at the Pacific Coast Regional Meeting of the American Ceramic Society held in Pasadena, California, October 23-24, 1968:

"Effect of Impurities on Mechanical Properties of LiF Single Crystals," H. L. Fotedar and T. G. Stoebe

"Effect of Magnesium Oxide on the Interfacial Energy of Alumina Bicrystals," M. W. Matson and W. D. Scott

"The Zirconium-Oxygen-Carbon System: I, Introduction," J. I. Mueller and A. D. Miller

"The Zirconium-Oxygen-Carbon System: II, Identification of Zr_2CO," S. K. Sarkar and J. I. Mueller

"The Zirconium-Oxygen-Carbon System: III, Preliminary Study of Electronic Band Structure," A. D. Miller and J. I. Mueller

"Oxidation of Zirconium," T. Archbold and L. P. Srivastava

"Nondestructive Thermal Test for Separation of ZrC Coatings," James Danberg and C. J. Sandwith

"Optical Observation of Domain Nucleation, Interaction and Motion in KH_2PO_4 and KD_2PO_4 During Polarization Changes," Richard E. Oettel and John L. Bjorkstam

"Mossbauer Studies of Low Temperature Anharmonicity," J. G. Dash

"Compaction as a Ceramic Forming Process," O. J. Whittemore, Jr.

The following papers were presented based upon previous work not being reported in this status report:

"Mechanically Assisted Polarization of Ferroelectrics," R. E. Deno and R. J. Campbell, Jr.

"Effect of Lanthanum Doping on the Microstructure of $BaTiO_3$," R. P. Burley and J. I. Mueller, October

"Ceramic Materials for Structural Components," W. D. Scott. Presented at a meeting of the Pacific Northwest Ceramic Association at Harrison Hot Springs, British Columbia, November 9, 1968

"Compaction as a Ceramic Forming Process," O. J. Whittemore, Jr. Presented at a meeting of the Pacific Northwest Ceramic Association, Harrison Hot Springs, British Columbia, November 9, 1968.

APPENDIX D

Working Group Meeting on Basic Properties of Actinide and Transition Metal Refractory Compounds

August 8-9, 1968

A two-day working group meeting, co-sponsored by Battelle-Northwest Laboratories and the University's Ceramic Materials Research Program, was held to discuss progress and problems in the field of basic properties of actinide and transition metal refractory compounds. The discussion was limited to the fields of thermodynamics, electronic structure and properties and mechanical properties.

Each of the above areas was introduced by a distinguished researcher in the particular field: Dr. E. K. Storms of Los Alamos Scientific Laboratory on thermodynamics; Dr. R. G. Lye of the Research Institute for Advanced Studies, Martin-Marietta Corp. and Dr. W. S. Williams of the University of Illinois on mechanical properties. The balance of the time in each subject area was spent on reports from the various laboratories represented on current research and a general discussion.

The participants included seventeen people all actively engaged in research in the field representing fifteen laboratories, five of which were government laboratories, seven were industrial laboratories or research institutes and three were university laboratories.

The meeting was felt by all concerned to be extremely valuable in providing information as to what work is currently in progress and in providing fresh insight into individual problems. The consensus was that the meeting should be continued on an annual basis. The second meeting will be held at Los Alamos in the summer of 1969.

APPENDIX E

Ceramic Materials Research Program Review
October 28-29, 1968

As part of the programmed effort to develop continuing communication with others of similar research interests, representatives of government and industrial research organizations were invited to the autumn program review. Invitations were sent to appropriate individuals in all federal agencies and to officials of the Pacific Coast ceramic industry, aerospace industry and non-profit research organizations.

The morning program each day was devoted to 10-15 minute briefings by the faculty research supervisors. During the afternoons, the attendees visited the various faculty members in their laboratories and discussed their research in detail.

Favorable reaction was received relative to this type of presentation with most of the attendees indicating their pleasure in being able to observe and discuss on-going research rather than having to experience the normal delays involved in presentation and publication.

SUMMARY OF ATTENDEES

University Personnel	
Project Supervisors	16
Other Faculty & Administrators	5

Visitors:

	Distribution by State						
	Wash.	Oregon	Calif.	Ohio	New York	D.C.	Total
Industry							
Ceramic	1	-	1	2	-	-	4
Aerospace	3	-	-	-	-	-	3
Other	-	3	-	-	-	-	3
Federal Agencies							
NASA	-	-	-	-	-	1	1
NBS	-	-	-	-	-	2	2
USBM	-	2	-	-	-	-	2
Non-Profit Research	1	-	-	1	-	-	2
University	-	-	-	-	1	-	1
TOTAL	5	5	1	3	1	3	18

APPENDIX F

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