INTERDISCIPLINARY RESEARCH AND DEVELOPMENT ON THE EFFECTS OF THE NATURE AND PROPERTIES OF CERAMIC MATERIALS IN THE DESIGN OF ADVANCED STRUCTURAL COMPONENTS

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ORIGINAL PAGE IS
OF POOR QUALITY

SUMMARY

A major redirection of research supported by the National Aeronautic and Space Administration on ceramic materials resulted in the establishment of an educational development and supportive research program at the University of Washington. The principal goal of the program is to advance design methodology, to improve materials and to develop engineers knowledgeable in design with and use of high performance ceramic materials.

Planning is continuing and full implementation of the academic development began during this report period. The organization of the supportive research effort has been completed and the results of the first full year is reported. Communications with appropriate industrial organizations and government agencies have been established and are continuing to assure future mutual support and assistance.

The previous IDL research was terminated during the report period.

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INTRODUCTION

The redirection of the interdisciplinary research program on ceramic materials last year resulted in a new program involving educational development and supportive research on structural design with ceramic materials in high technology. The IDL projects have been phased out this year. Both are supported by NASA grant number [redacted] 48-002-004. Aspects of each program will be discussed in this report. Planning and organization were the major features of the first year of the educational development and supportive research effort, and both areas became operational during the current year.

Administrative responsibility for this program remains vested to the Dean of the College of Engineering, who appoints a board of faculty member to establish policy and approve the general operations. At present, the Ceramic Structural Materials Board consists of the following:

- J. I. Mueller, Ceramic Engineering, Principal Investigator and Chairman
- B. W. Mar, Civil Engineering, Associate Dean of Engineering for Research
- J. G. Dash, Physics, representing the Dean of the College of Arts and Sciences
- D. G. Dow, Electrical Engineering
- A. S. Kobayashi, Mechanical Engineering
- W. D. Scott, Ceramic Engineering
- T. G. Stoebe, Metallurgical Engineering

Within the total program are two coordinating committees, one for research and the other for educational development. The former explores possible directions and the latter develops and presents the course work and design problems. The membership of each of these is given in the appropriate sections of this report.

Complete interdisciplinary cooperation was a necessity in the organization of the educational development portion of this program. Success in this area has been due to two factors, first a capable and enthusiastic faculty and, second, the experience previously gained through the IDL program.

Although this report discusses these projects which formulated during the reporting period, a final report on the total IDL effort will be prepared and distributed in the near future.

EDUCATIONAL DEVELOPMENT

Coordinating Committee

J. I. Mueller, Ceramic Engineering, Acting Chairman
R. J. H. Bollard, Aeronautics & Astronautics
B. J. Hartz, Civil Engineering
A. S. Kobayashi, Mechanical Engineering
W. J. Love, Mechanical Engineering
A. D. Miller, Ceramic Engineering
W. D. Scott, Ceramic Engineering
R. Taggart, Mechanical Engineering
O. J. Whittemore, Ceramic Engineering

Objectives for 1977-78

1. Initiate the academic development program, including the design problem sequences.

2. Prepare and distribute information on this and other campuses in order to secure additional student interest in the program.

3. Complete the first-phase processing and testing facility to support both the educational development and research areas.

4. Introduce appropriate subject matter regarding brittle (ceramic) materials in departmental third-year courses.

5. Develop a plan for industrial cooperation.

Progress

A series of experimental courses for the academic portion of this program were outlined and approval obtained at all levels of the University. A two-track plan was originally established, one for students at each of the undergraduate and graduate levels with both tracks to include academic and design courses. All courses have the designation of each participating department, viz, Ceramic Engineering, Mechanical Engineering, etc. The following diagram indicates the sequences of courses in each track with the number of credit hours listed parenthetically:

QUARTER	AUTUMN	WINTER	SPRING	SUMMER
Senior Track	→ 475(3)	→ 476(3)	→ 496(3)	
Graduate Track		→ 479(5)	→ 536(3)	→ 536(3)

The dotted line indicates that some exceptional seniors would have the option of enrolling in 479, with permission, then continuing with the Senior design problem.

Academic Courses

Approval of these courses was obtained in May 1977 and 479 was offered in the summer quarter on a trial basis to a select group of advanced M. S. students with an interdisciplinary mix. The results of this were discussed in our last report.

Based upon a review of this course several revisions were made and were implemented during the winter quarter 1978. The course utilized a scenario rather than strictly topical approach. The initial period were devoted to methods and requirement for the design of a gas turbine utilizing present technology. Subsequently the advantages and limitations of higher inlet temperatures were discussed and the nature of properties of ceramic materials was introduced. Period following this were devoted to failure mechanisms, structural evaluation of test data and the application of these in the design of a turbine rotor.

Design Projects

Plans for the senior design project were completed and the course (496) offered for the first time during the spring quarter 1978. A total of nine graduate students were enrolled, their baccalaureate degrees being ceramic engineering (6), civil engineering (2) and mechanical engineering (1). The assignment was to design a disc to be used as the hub of a rotor in a gas turbine engine, using the following basic design data -

Turbine inlet temperature - 1100° (2010°F)

Maximum hub temperature - 650°C (1200°F)

Mass Flow - 2.7 Kg/sec (6 lbs/sec)

Pressure ratio 8:1 from compressor inlet at one atmosphere and 20°C (70°F)

Minimum disc thickness for blade attachment - 13mm (0.5 in.)

Velocity at mid-point of blade height - 30 m/sec (98 ft/sec)

Specified Material - Al_2O_3

Maximum diameter for hub and blades - 200mm (8 inches)

Allow for 13mm (0.5 in.) central hole for shaft

Students were required to --

1. Calculate stresses as a function of disc radius and the angular velocity.

2. Design, process and test MOR specimens.

3. Determine Weibull statistics and from the stress field assess the probability of survival of disc.

4. Test commercial MOR specimens.

5. Repeat item 3, above.

6. Choose final design and justify. including economic considerations.

It was originally intended that discs based upon 6, above, would be fabricated and tested to failure but facilities for spin testing were not completed.

The seven participating faculty members were available as "consultants" to each group. As might be anticipated, several problems developed in this initial offering. The final critique was most helpful with the students, in general, showing enthusiasm for the course.

Plans were also completed for the graduate design problem (536) which is being offered for the first time during summer quarter. Thermal stresses will be introduced requiring a material change to silicon nitride. It is contemplated that the spin test pit will be available during the quarter.

Work Book

The extended notes used as a work book in all courses is currently under revision. This book is a compilation of 11 topical units written by participating faculty members. It is contemplated that revisions and editing will be completed this summer and available to new students during the offerings in 1978-79.

Iterative Design

Participating faculty members consider the educational development program as an exercise in iterative design and we are, therefore, just completing the initial testing of our preliminary design. Several changes have been suggested and are under study by the design group (Educational Development Committee). One major change in the original plan was made as the result of our analysis of the student response to the announcement regarding the general availability of this series during the 1978-79 academic year. It was generally agreed that a 5-credit course offers scheduling difficulties for graduate students. This, plus the inadvisability of offering two courses simultaneously in the same quarter due to faculty loads, suggested a redefinition of the academic "tracks" of this series. We now plan a single series with the first two courses (476 and 496) open to both seniors and graduate students. The design problem sequence (536) will be available to graduate students only. This will necessitate a rescheduling of material from the 5-credit 479 course with a single 3-credit course (476). This is deemed possible and the details are presently under final consideration. The course sequence would then be:

QUARTER		WINTER	SPRING	SUMMER	AUTUMN
Senior Track	→	476(3) →	496(3) ↘		
Graduate Track	↗			536(3) →	536(3)

Facilities and Equipment

Funding has been approved for renovation of a portion of Roberts Hall to be dedicated for use of this program. This space will be used for processing and test facilities. A delay in the preparation of plans has precluded these facilities being completed as anticipated in our last report.

Installation of the Brew nitriding furnace was completed and it is currently in use. The spin test unit and a simple thermal shock test rig are under construction and should be completed and available for use in the design problem course this summer, even though final installation of the former will be delayed pending completion of space renovation.

SUPPORTIVE RESEARCH

Coordinating Committee

A. E. Gorum, Ceramic Engineering, Chairman
A. F. Emery, Mechanical Engineering
J. L. Bjorkstam, Electrical Engineering
W. D. Scott, Ceramic Engineering
R. G. Stang, Metallurgical Engineering

Objectives

The goal of the research portion of this program is to keep abreast of the overall program in high performance structural ceramics in this country and elsewhere, and to do research in those areas that are deemed most appropriate whether it be related to materials or to structures. This should result in the University of Washington being a central focus for information and research in the area of high performance ceramics. The objective for the current year is to carry on for the most part those research programs initiated last year and to continue to develop additional interest among the University faculty in research areas deemed appropriate.

Organization

Emphasis at the present time is on the structures and related materials programs in a ceramic turbine engine or other heat engines that would benefit from this type of material.

The research program has been established so that all departments in the University that may contribute to the program are aware of the goals of the program and they participate in defining the individual areas of research that are to be undertaken. Those departments now involved are physics, aeronautics and astronautics, mechanical engineering, electrical engineering, civil engineering, metallurgical engineerng and, of course, ceramic engineering.

The general planning of the program is carried out by the ceramic engineering faculty and coordinated by the above named. The overall research program is derived by analysis of proposals submitted from various departments of the University of Washington with emphasis being on the relationship of the proposed work to the needs defined in the area of structural ceramics.

Progress

Ten programs were supported during the past year. These will be detailed in the following section.

Three new programs were initiated June 15, 1978 and are as follows:

The Reaction Bonding of Silicon Nitride: Physical Structure Relationships
O. J. Whittemore, Professor, Ceramic Engineering

Reliability Prediction of the Strength of Ceramic Materials Under Complex Stress States
B. J. Hartz, Professor, Civil Engineering

Characterization of Silicon Nitride by Auger Electron Spectroscopy and Low-Energy-Electron Diffraction
S. C. Fain, Jr., Associate Professor, Physics

Additional funding is being sought in the general area of high performance ceramics. When new funds are obtained, they will be used to support selective segments or extensions of the present program, and the present funds will be used to support additional programs. Several programs have been submitted and merit funding.

Failure Analysis in Ceramic Structure Materials
A. D. Miller, Associate Professor, Ceramic Engineering

Proof Loading of Ceramic Structures
Colin B. Brown, Professor, Civil Engineering

A Critical Evaluation of Thermal Shock Test Procedures and the Development of Methods to Predict Thermal Stress Fracture
Ashley F. Emery, Professor, Mechanical Engineering
Albert S. Kobayashi, Professor, Mechanical Engineering

Exploratory Research on Silicon Nitride Composites
David B. Fischbach, Research Professor, Ceramic Engineering

Thermal Depolarization Studies in Silicon Nitride
Thomas G. Stoebe, Professor, Metallurgical Engineering

RESEARCH ON HIGH PURITY MATERIALS (Si_3N_4 and SiC)

Faculty Supervisor: A. E. Gorum, Research Professor
Ceramic Engineering

Graduate Assistants: Charles Newquist, Ph.D. Student
Research Assistant

George Reini, M.S. Student
Research Assistant

Douglas Gilbert, M.S. Student
Research Assistant

Purpose:

The purpose of this part of the CSM program is

1. To learn how to make high purity Si_3N_4
2. To establish base line data (structure-property relationships) for high purity Si_3N_4
3. To understand the role of additives, if they are really necessary, in various kinds of processing

The program at present breaks down into specific areas

A. Whisker growth and evaluation:

1. Preparation of large whiskers (single crystals)
2. Definition of mechanical properties
3. Structure and orientation determinations (x-ray)
4. Dislocation studies by electron transmission and etching techniques

B. Determine the role of Si_3N_4 whiskers in reaction bonded Si_3N_4:

1. Using coarse grained metallurgical grade Si, observe the morphology of whisker growth, the structure of the whiskers, the effect of grain size and distribution on the amount and type of whiskers formed, and most important define the role of the whiskers in the final mechanical properties of reaction bonded Si_3N_4.
2. Compare the properties and whisker morphology of the metallurgical grade silicon with those of high purity silicon. This comparison should define to some extent the effect of additives. Of particular interest will be the high temperature properties if the whisker structure is similar.

C. Preparation and evaluation of high purity reaction bonded Si_3N_4 to obtain base line data for high purity material.

1. Nitriding of fine grained compacts of pure silicon.

a. Observe progress by ceramography as nitriding progresses.

b. Determine mechanical properties of high purity reaction bonded Si_3N_4.

c. Determine amount and character of porosity as well as unreacted silicon.

d. Postulate optimum grain size and distribution and make specimens to optimize structure and evaluate. This may well involve bi- or tri-modal distribution to optimize density as well as to control the reaction bonding characteristics.

D. Sintering Studies - Si_3N_4.

Very fine grained Si_3N_4 is now available from two sources. Work to date has all employed additives which degrade the properties. Investigation of methods to make a high purity material by this technique. The development of a fugative sintering aid is the primary goal.

E. Decomposition of inorganic polymers to produce high purity fine grained Si_3N_4 and SiC. Recently work has been initiated in two or three organizations, primarily in Japan, to produce SiC fibers for composites. We propose to go directly from the polymer by various processing techniques such as press forming, injection molding, and extrusion to a final shape. This technique has a great deal to offer in energy savings as compared to reaction bonding.

<u>Relevance</u>:

By understanding the behavior of high purity materials prepared by different techniques, and the role of structural components such as whiskers, grain size, grain boundaries, etc., it should be possible to make materials that relate to specific applications whether it might be a heat exchanger tube or a turbine rotating part. It also allows trade-offs as to properties, energy requirements for certain processes, etc.

<u>Objectives</u>:

The second year will see parts of the research finished and others well along. The third year should be characterized by the completion of other areas of interest and initiation of new lines of wor.. depending on the results of the research and the way the general field of structural ceramics in tne country is moving.

Progress:

The two furnaces, a small 1800°C vacuum atmosphere furnace, and the large Brew furnace for reaction bonding are operating and samples being prepared. Two high temperature testing furnaces are operable and ready for testing. Programs B, C, and D are underway, and A will start shortly. The thin section apparatus for optical characterization specimen is still to be procured.

EVALUATION OF TESTS FOR MECHANICAL PROPERTIES OF CERAMIC MATERIALS

Faculty Supervisor: R. J. H. Bollard, Professor
Aeronautics and Astronautics

Graduate Assistant: Majid Kani, M.S. Student
Research Assistant

Purpose:

To evaluate the accuracy of commonly employed material characterization and strength tests for brittle materials and from this critical study explore the feasibility of improved testing techniques.

Relevance:

Due to the brittle nature of ceramic materials it is inherently difficult to devise tests where the simple and uniform stress states desired for characterization and strength assessment can be accurately created. For example, the simple tension test is difficult to conduct due to possible grip misalignment producing high bending stresses and due to possible specimen failure in the grips. Furthermore, the random distribution of surface and interior flaws which greatly affect the strength, lead to a statistical prediction of the survivability of a designed structure under load. The interpretation of specimen failure data in the life or survivability prediction for a structure requires an accurately conducted test sequence using a large number of specimens in which the stress state at failure is well known. Economics, ease of test reproduction, among other factors, has led to the common usage of the 3-point or 4-point bend test for failure prediction within the structural ceramic industry. These relatively simple tests have been adopted widely also as quality control and acceptance tests. These are basically poor characterization tests for even the "perfect" specimen due to the stress and strain gradients; in the inability to separate surface and internal effects in the failure mechanism and in the error producing "imperfect" specimen geometries and support systems during the test.

The research program is initially directed to theoretically and experimentally determining the scale of these errors in the 3 and 4-point bending tests and then to apply the developed assessment techniques to promising alternative tests.

The development of characterization and strength tests of predictable accuracy will allow more accurate prediction of stresses in a loaded structure and, more importantly, a more accurate prediction of the related probability of failure of the structure. Furthermore, with the sources of error due to the specimen imperfections and testing technique understood and, hopefully, quantified, the influence of processing parameters can be more accurately identified. This knowledge is essential to the efficient design of structures made from brittle materials.

Objectives:

December - June 1978

The development of a finite element program to determine the mechanical state throughout a 4-point bend test specimen for various specimen geometry, loading system geometry, friction and specimen imperfection parameters. This program will be employed to assess the errors introduced relative to the commonly employed simple beam formula by each of the variable parameters

1978 - 1979

To apply the program to a comprehensive error analysis for the 4-point and 3-point MOR test for each of the parameters of importance contained in the general listing above for both mechanical state and failure. Carry out experimental comparisons where possible using Al_2O_3 specimens with careful control over the processing variables in the attempt to isolate these from the variables under study. If it proves desirable more suitable brittle material will be considered.

The development of similar programs to analyze the mechanical state for all loadings up to failure of candidate alternative tests.

Progress:

The program development was carried out and has been applied to the determination of the error introduced by loading system friction. The results compare favorably with published theoretical data.

STATISTICAL DISTRIBUTION OF MODULUS OF RUPTURE RESULTS FOR CERAMIC MATERIALS

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

Graduate Assistant: Tomio Hosokawa, M. S. Student
Research Assistant

Purpose:

To study the influence of certain test variables on the statistical distribution of the modulus of rupture in ceramic materials and to evaluate the adequacy of the Weibull distribution at the low end of the probability distribution curve.

Relevance:

Both aspects of this project have extremely important practical applications since the modulus of rupture is the most widely used measure of mechanical properties of ceramic materials. Also, the most important part of the probability distribution curve is in the range of low probabilities of failure where the Weibull distribution, although widely used, does not accurately fit the observed test data in many cases.

Objectives:

a) To develop a test fixture and instrumentation to rapidly break a large number of small ceramic specimens in a modulus of rupture type test and to obtain M.O.R. values for statistical analysis with variation of certain parameters in the M.O.R. test.

b) To try to determine something about the sensitivity of the statistical measures of M.O.R. strength to M.O.R. test variables for nominally the same specimens by analyzing a large amount of M.O.R. test data. The test variables varied were:
   i) the end supports under the specimens were changed from knife-edge line supports to spherical ball supports.
   ii) 3 point and 4 point M.O.R. tests with different spans were used (4 cases).
   iii) the specimens, which were alumina substrates with a "notch" on one side, were inverted for each of the above 4 cases to put either the "notched" or "smooth" side of the specimens in tension.

c) To try to obtain an improved Weibull distribution fit to the data at the low probability-of-failure end of the probability distribution curve.

Progress:

a) Accomplishments. The test device and peak reading indicator were assembled and used to break a total of several thousand specimens under the various loading and test conditions. An analysis of a significant portion of this data has been made and is the basis of an M. S. thesis to be submitted this summer.

b) Problem areas. It is not possible in the time available to slice a number of specimens to obtain M.O.R. data on the alumina substrate material without the notch, i.e., without the complex stress distribution associated with the notch. This was due to jigging problems and the loss of saw blades. Also there is some concern about the inability to control the rate of loading with the test fixture and possible influence of this on the results. There was not time to run control tests to study this variable - but it is believed that the tests were run consistently enough to be statistically meaningful. However, it is proposed to build a simple controlled rate loading device to go with this fixture and to add a printer for automatic data recording.

c) Progress toward current year objectives. All of the planned tests were completed except for the sliced bars for unnotched specimens and plan stress notched specimens. Most of the Weibull analysis and A plane stress Finite Element Analysis has been completed. The 3-dimensional Finite Element Analysis was not completed mainly because of difficulty with the SAP IV Finite Element program which was to be used for this analysis. A new SAP IV tape has been obtained and it should be possible to proceed with a 3-d analysis.

FRACTURE TOUGHNESS DETERMINATION OF CERAMIC MATERIALS

Faculty Supervisor: Ashley F. Emery, Professor
Mechanical Engineering

Albert S. Kobayashi, Professor
Mechanical Engineering

Graduate Assistant: Tusher Basu, Ph.D. Student
Research Assistant

Purpose:

The purpose of this exploratory investigation is to identify and to provide solutions to critical fracture mechanics problems in ceramic structures. The long range goal of this research effort is to define the fracture mechanics requirements necessary to maintain the integrity of a ceramic structure in a hostile environment.

Relevance:

(a) Possible solutions to identifiable problems in fracture of ceramics could lead to design procedure which reduces the fracture sensitivity of ceramic structures exposed to a hostile environment for a prolonged period of time.

(b) This basic research in fracture of ceramics will contribute to the state of art in predictive procedures for sustained stress crack growth and for statistical fracture strength and in the use of fracture mechanics to study erosion of ceramics.

Objectives:

(a) The first year objective was to identify critical areas where contributions to the state-of-art in the fracture of ceramics could be made. Development of a fracture specimen suitable for high temperature testing was identified as suitable area of investigation.

(b) The second year objective was to develop such fracture specimen. Simultaneously, an exploratory study for incorporating applied fracture mechanics to ceramic structural design was to be initiated.

(c) The third year objective is to determine the fracture toughness and stable crack growth characteristics of a typical structural ceramic using the developed fracture specimen.

Progress:

(a) Accomplishments: Most of the effort was expended in arriving at a specimen design with reduced sensitivity to the coefficient of friction between the loading pin and crack mouth opening. In the current design friction effect, although not negligible, has been reduced by a factor of four from the previous design. Stress intensity factor for the redesigned fracture specimen has been derived and experimental errors in stress intensity factor determination were assessed.

(b) Problem Area: An in-situ experimental procedure for measuring the coefficient of friction between the loading pin and the surfaces of crack mouth opening must be devised. Simultaneous measurements of the crack mouth opening, applied pin load and pin displacement in the actual test setup using a fracture specimen with a blunted crack tip should provide the necessary data for inferring a coefficient of friction if the elastic properties of the test specimen material at the test temperature are known.

(c) Progress Towards Current Year Objectives: Approximately twenty specimens machined from 1/4-inch thick hot pressed polycrystalline alumina disks (COORS AD-998) will be prepared. Also silicon carbide loading fixtures will be ordered shortly. The fracture specimen will be tested at about 1000°C in a vacuum furnace attached to an 30 Kips Instron Testing Machine.

(d) Degree Recipient: None

(e) Paper Presented: None

(f) Paper Published: "A Fracture Specimen for High Temperature Testing", by A.S. Kobayashi; L.I. Staley; A.F. Emery; and W.J. Love, <u>Fracture Mechanics of Ceramics</u>, Vol. 3, <u>Flaws and Testing</u>, edited by R.C. Bradt, D.P.H. Hasselman, and F.F. Lange, Plenum Press, 1978, pp. 451-461.

<u>Correlative Research</u>:

A.F. Emery, A.S. Kobayashi, and W.J. Love are completing their four year EPRI research contract on static three-dimensional fracture mechanics and dynamic fracture of pressurized pipes and pipe joints. The states of art in fracture mechanics and fracture dynamics advanced through this research effort are directly applicable to our research program in ceramic fracture. In addition, A.S. Kobayashi is in his fourteenth year of contract research with ONR involving dynamic fracture which again is directly applicable to our work in ceramic fracture.

MEASUREMENT OF CRACK VELOCITY IN CERAMICS

Faculty Supervisor: W. D. Scott, Professor
Ceramic Engineering

Graduate Assistants: Diane Martin, M.S. Student
Research Assistant

Kenneth Davido, M. S. Student

Purpose:

To develop new instrumentation techniques to measure and record crack growth velocities in opaque ceramic materials using a calibrated electrical grid applied directly to the specimen.

Relevance:

a. Ceramic materials are subject to static fatigue or slow crack growth under stress. In some materials, e. g., glass, this occurs at room temperature in the presence of water vapor. In other materials, such as Si_3N_4, it occurs at high temperature, probably by combined stress corrosion and plastic deformation mechanisms. Methods of predicting lifetimes of components in service, notably proof test techniques, require a detailed knowledge of crack growth behavior. The technique is to establish the size of the largest possible crack with a proof test and predict how long it will take this crack to grow to a critical size in the service stress. Thus, crack growth velocity data is essential.

b. Present techniques of obtaining crack growth data are tedious, require many specimens, and are open to some analytical criticism. The technique proposed here would provide a greater amount or more accurate information from a single specimen. It would enable researchers in materials development to evaluate rapidly new materials with regard to the important parameter of slow crack growth behavior.

Objectives:

The first year objective was to evaluate the feasibility of the technique at room temperature using microscope slides. We know from other workers the crack growth behavior of this material. We will be evaluating the following points:

a. What is the relationship of the metal grid fracture to the underlying ceramic fracture? Does the metal grid fracture follow the ceramic fracture exactly?

b. What level of electrical signals can be generated through the grid and how can these be measured and recorded?

c. How reproducible, reliable, and applicable are the photo-lithographic techniques used to form the metal grid system?

The second year objective is to secure better control of the experimental variables of ambient temperature and atmosphere to attempt to reduce the variability of experimental measurements.

Progress:

As stated in the previous progress report, the first year objectives have been attained. The voluminous data is now being analyzed by K. Davido who is now a part-time student. These results show a strong, systematic dependence of crack velocity with K_I when large changes in K_I are made by changing the applied load on the DCB specimen. For example, a change of K_I from 0.15 to 0.4 (MPa) $^{3/2}$ produces a crack velocity change from 2×10^{-9} to 10^{-7} M/S.

At low velocities ($<10^{-8}$ M/S) and constant load, although K_I increases as the crack length increases, the velocity does not increase as predicted by the load change experiments, and in fact, the velocity decreases slightly. This observation may indicate that the assumed conditions for calculating K_I may be changing possibly through changes in the crack tip configuration. At higher velocities, the agreement between experiments at constant load with crack length increasing and load increment experiments is good.

Analysis of the data involves velocity calculations for line breakage at 20 μm interials, and a typical constant load experiment may break over 100 lines. A computer program to do this analysis has been developed and tested, and the complete data set for five samples is being analyzed.

The experimental arrangement for holding and loading the DCB specimen has been redesigned to provide positive atmosphere and temperature control. If the tendencies toward crack arrest at low velocities is confirmed by data analysis of samples already tested, then this effect will be investigated further with relative humidity and temperature as controlled variables.

Another uncontrolled variable is damage at the base of the crack-guiding side groove introduced by the diamond slitting wheel. Experiments have been carried out to reduce residual damage by HF etching of the side groove, but results to date are inconclusive.

A STUDY OF HIGH TEMPERATURE CREEP IN CERAMIC MATERIALS FOR STRUCTURAL APPLICATIONS

Faculty Supervisor: Robert G. Stang, Assistant Professor
Metallurgical Engineering

Graduate Assistant: David Hata, M. S. Student
Research Assistant

Purpose:

The purpose of this project is to study high temperature deformation in hot pressed Si_3N_4. The research is focusing on the contribution of grain boundary sliding (GBS) to the creep strain.

Relevance:

The microscopic mechanisms which control high temperature plastic deformation must be determined to identify the best method for strengthening materials used in these applications. For materials used in creep applications this is often done by determining the stress and temperature dependencies. This data is used to formulate a model which describes the strain rate as a function of stress and temperature. The effects of impurity additions and microconstituents on the parameters in models of this type can be used to optimize and/or predict the deformation behavior of the material under study for different stress and temperature conditions.

Objectives:

First Year

1. Design of a compression creep machine capable of operating in air at temperature up to 1450°C and stresses up to 80,000 psi.

2. Selection of a suitable candidate material.

Progress:

This research program requires construction of a compression creep machine capable of testing materials at stresses up to 5.52×10^2 MPa (80,000 psi) and at temperatures up to 1450"C in air. This equipment is being designed so it could be used by other investigators to study materials which might be produced in the future. The experiments planned on hot pressed Si_3N_4 will serve two purposes; first the strain time data generated will be used to check the apparatus to make sure that the data produced is in agreement with that produced by other investigators working with the same material under similar conditions. In addition, a series of experiments is planned to investigate the contribution of grain boundary sliding to the total creep process. GBS will be monitored by studying the offset, at the grain boundaries, of lines scribed on the polished surface of hot pressed Si_3N_4. These results should lead to important conclusions concerning the importance of GBS on the rate controlling creep process as a function of stress and temperature.

The design and construction of a creep machine capable of operating at stresses up to 552 MPa (80,000 psi) and at temperatures up to 1450°C in air is essentially complete. The apparatus has been tested and modified so that the sample load is known and reproducible at room temperature. The furnace will soon be installed and the system tested at temperature. Techniques are currently being developed to fabricate samples from hot pressed Si_3N_4 (NC-132) supplied by AMMRC through A. E. Gorum. Sample preparation is difficult because the existing cutting, utilizing a diamond wheel is barely adequate and surface grinding equipment is non-existent. This may require the utilization of an outside shop. Unless unforeseen difficulties with the apparatus are encountered, we expect to run the first creep tests during summer quarter.

Data available in the (1,2) suggests that creep deformation in this material occurs by grain boundary sliding under certain conditions of stress and temperature. Use of this material in our experimental program will allow us to compare data obtained from our apparatus with data published in the literature. In addition, by scribing lines on the polished surfaces of undeformed samples, we can monitor the contribution of grain boundary sliding to the total creep strain by measuring the offsets in these lines at grain boundaries. This should provide a unique opportunity to check the theories based on grain boundary sliding with experiment.

1. R. Kossowsky, D. G. Miller and E. S. Diaz, Journal of Mat. Sci. 10 (1975) p. 983.

2. M. S. Seltzer, Cer. Bull. 56 (1977) p. 418.

Correlative Research Programs Supervised by R. G. Stang:

High-Temperature Low-Stress Creep of Copper 1% Al_2O_3 Composite Material, Myung-Shik Han, M. S. Candidate.

Orthopedic Internal Fixation, with Professors T. G. Stoebe and D. A. Spengler, M. D., S. M. Colella, M. S. Candidate.

An Investigation of the Effect of Stress Changes on the Subgrain Structure Developed During High Temperature Creep in High Purity Aluminum, Iris Ferreira, Ph.D. Candidate.

Effect of Heat Treatment on the Mechanical Properties of P/M 7075 Aluminum Alloys, Melvin Nilsen, M. S. Candidate, Sponsored by The Boeing Company.

OPTO-ACOUSTICAL TECHNIQUE FOR LOCATING DEFECTS IN CERAMICS

Faculty Supervisor: John L. Bjorkstam, Professor
Electrical Engineering

Graduate Assistant: Michael Morgan, M. S. Student
Research Assistant

Purpose:

This work has as its purpose the development of an opto-acoustical technique for use in detecting small ($\gtrsim$ 20 μm) internal (as well as surface) flaws in ceramics. Our method addresses two major problems: (1) the generation of high intensity acoustical pulses with sufficient high frequency content to give appreciable scattering from flaws in the range mentioned above, and (2) to detect the presence of such flaws without the necessity for fabricating receiving transducers on each part to be tested.

Relevance:

The technique which we are developing will be generally applicable to the detection of smaller flaws, and at greater depth, than has been possible heretofore in usual ceramic materials where scattering from normal grains leads to high acoustical attenuation. We believe that our method will allow generation of acoustical energy densities ≈ 4 orders of magnitude greater than conventional techniques in the necessary frequency range for detection of significant small flaws in both structural and electronic ceramic components.

Our investigations bring to bear instrumental tools and signal processing techniques which have been highly developed in electrical engineering, as well as in other disciplines. They also add further breadth to a strong bioengineering and ocean engineering acoustical program which exists in this electrical engineering department.

Objectives:

Year 1 (1977-78): To carry out a theoretical evaluation of the potential for success of the proposed methods and to demonstrate generation and detection of short acoustical pulses in some simple materials.

Year 2 (1978-79): To improve the efficiency of optical to acoustical energy conversion as well as detection methods, to investigate the effect of grain size upon acoustical pulse propagation in ceramics, and to demonstrate the detection of small inhomogeneities in typical ceramic materials.

Year 3 (1979-80): To demonstrate the detection of small surface flaws in ceramics and to develop beam scanning techniques.

Progress:

Probably the most significant progress during this period was recognizing that the detection, and determination of harmonic content of acoustical energy scattered to the surface could be greatly simplified by making use of a photographic film technique to detect the Brillouin scattering of the probing detection laser beam. The ability of the film to integrate over multiple shots of the high energy pulsed laser train which generates the acoustical energy should lead to a significant increase in sensitivity as well as simplicity. Such a system is nearly completed and is expected to be ready for initial tests within a month. If these tests are successful as we anticipate, our first year objective will have been met.

Surface Characterization of Silicon Nitride

Faculty Supervisors: E.A. Stern, Professor
Physics

J.G. Dash, Professor
Physics

S.M. Heald, Research Associate

Graduate Assistant: H. Meuth, M.S. Student
Research Assistant

Purpose:

We propose to identify and characterize the surface properties of Si_3N_4 that are important to sintering processes. This will be done by the coordinated application of two different probes of the surface properties: adsorption isotherm and extended x-ray absorption fine structure (EXAFS) measurements. The isotherms will be used to identify the population distribution of different types of adsorption sites characterized by their differing atom binding energies. The microscopic structure of these sites on an atomic scale can then be determined by the EXAFS measurements. Studies of samples produced under varying conditions should allow us to correlate the microscopic surface properties with sintering parameters.

Relevance:

At present, detailed knowledge on an atomic scale of the microscopic physical processes involved in sintering is not available. The present work is aimed at developing such knowledge for one particular material, Si_3N_4, but with the expectation that much of the same knowledge can be carried over to other materials. Once the physical basis of the sintering process is better understood, then this knowledge can be applied to optimizing the fabrication of other technologically important products. In the case of Si_3N_4, these include components exposed to high temperature environments such as high performance gas turbines and combustion chambers. In the past, much of this optimization has been on a trial and error basis.

Objective:

The first year of this project was devoted to construction and testing of necessary equipment, and the beginning of initial isotherm measurements. From the results of these initial measurements we will be able to refine our equipment and prepare for making EXAFS measurements. EXAFS measurements will begin during the second year which will be on the better characterized system of Kr on graphite in order to fully develop the use of adsorbed Kr as a surface probe. Also, initial EXAFS measurements will begin on Si_3N_4 at this time, which will continue in earnest during the third year. Any further isotherm measurements suggested by the EXAFS results will also be made during the third year.

Progress:

The isotherm equipment was completed during this period, and isotherms of Kr on Grafoil and Kr on Si_3N_4 were made. Figures 1 and 2 show the isotherm curves we obtained for the two cases. The Si_3N_4 sample was a hot press sample from a fractured turbine blade. It had very little surface area and therefore was a quite critical test of our apparatus. Encouragingly, the isotherms for Kr absorbed on Si_3N_4 showed some structure which indicates that the surface has some homogeneity. This means that we have a good possibility of being able to define well by EXAFS the different surface regimes exposed on the Si_3N_4 samples. We are awaiting the samples to be prepared here which are stopped at various stages of processing so as to continue our isotherm measurements on them.

For the Kr on Grafoil system, we are preparing samples for EXAFS measurements for which we have been scheduled the beginning of July at SSRL.

We have managed to maintain our first year objectives, and are confident that we will make successful progress for the following year.

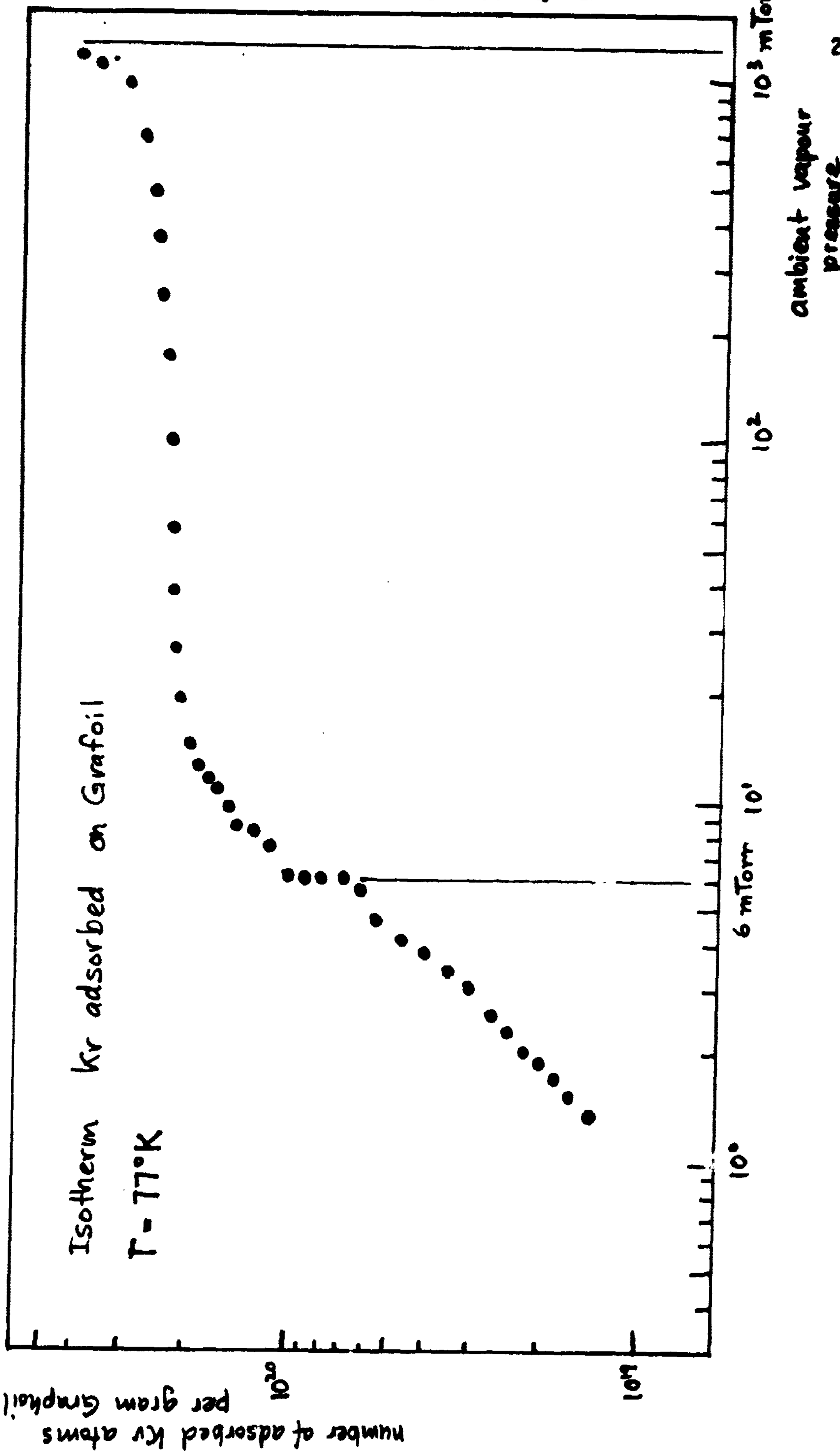
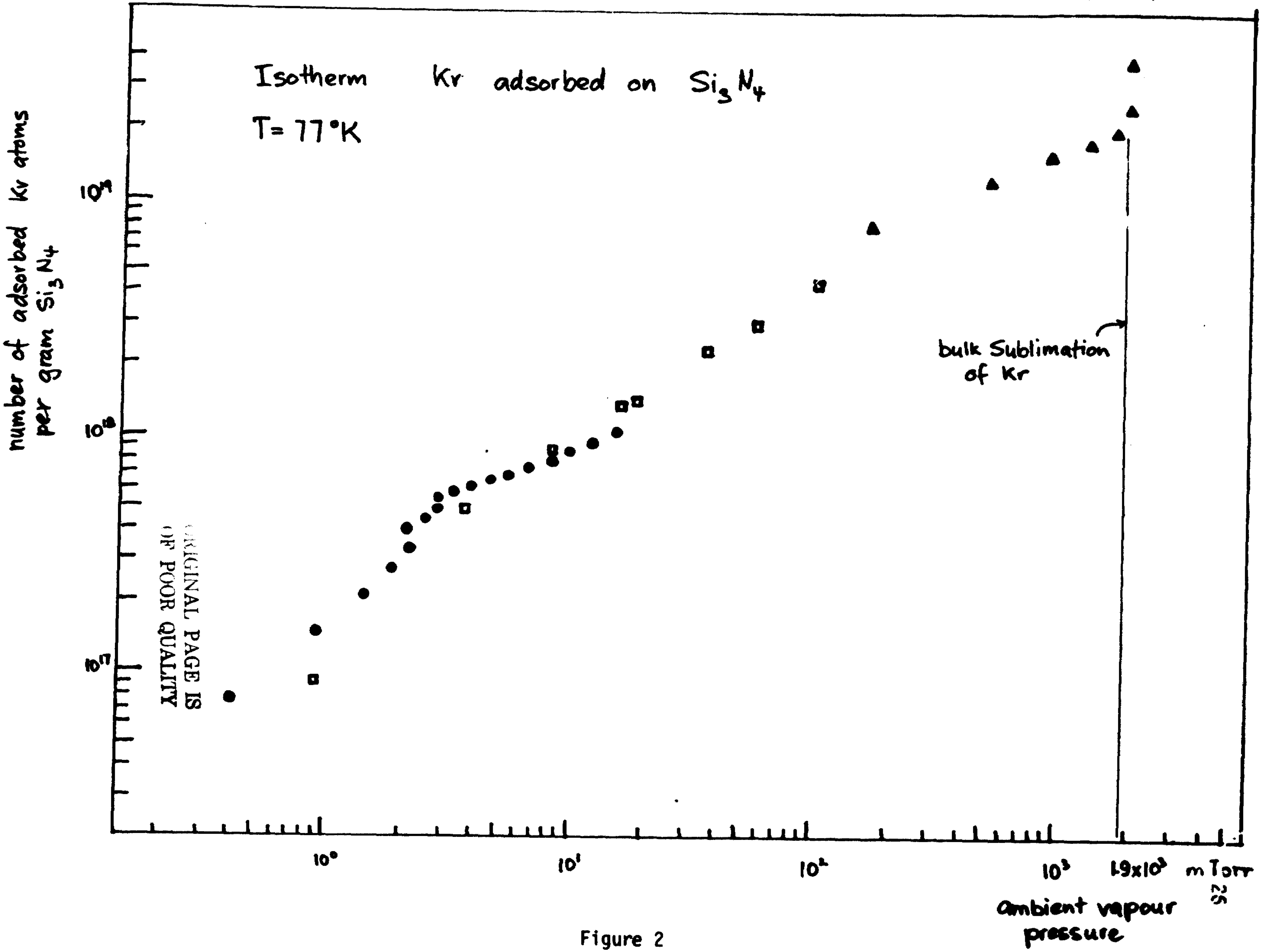

Isotherm Kr adsorbed on Grafoil
T = 77°K
10³ mTorr
10²
10¹
10⁰
6 mTorr
ambient vapour pressure
10²⁰
10¹⁹
number of adsorbed Kr atoms per gram Grafoil

Figure 1



Isotherm Kr adsorbed on Si3N4
T= 77°K
number of adsorbed Kr atoms per gram Si3N4
10^19
10^18
10^17
bulk Sublimation of Kr
10^0
10^1
10^2
10^3
1.9x10^3
m Torr
ambient vapour pressure

Figure 2

INTERDISCIPLINARY RESEARCH PROGRAM

Prior to 1976, the major thrust of the NASA-funded effort at this institution was towards interdisciplinary research on the nature and properties of ceramic materials. The program, in recent years, had been divided into three major research areas - solid electrolytes, ceramic fibers, and processing. Reorientation necessitated non-renewal of funds for these projects except for those manned by students to whom support commitments had been made.

As stated previously, the IDL portion of this program terminated during this report period. The following covers the research in this category. A final report on the total IDL program is being prepared and will be distributed in the future.

CARBON MATERIALS RESEARCH

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

Graduate Assistants: Seshadri Srinivasagopalan, Ph.D. Student Research Assistant (Army Research Grant)

Kunio Komaki, Ph.D. Student Research Assistant (Army Research Grant)

Purpose:

To investigate the dynamic and static stress-induced behavior of carbon fibers and its relationship to structure and processing. To investigate the structural characterization and the nature of thermally-induced structural evolution in carbons of various types.

Relevance:

The properties of carbon materials are very sensitive to microstructure, which varies widely dependent on both precursor and processing. Raman spectroscopy, diamagnetic susceptibility, electrical and dynamic mechanical behavior are being used in combination with x-ray diffraction and micrographic observations to develop improved material characterization techniques and determine structure/property relationships with primary emphasis on carbon fibers and difficult-to-graphitize bulk carbons. The electronic and mechanical property studies on carbon fibers are supported by a grant from the Army Research Office.

Objectives:

1. Complete the analysis, interpretation and publication of NASA-sponsored research on the structure, properties and characterization of carbon materials and terminate these projects.

2. Obtain a better fundamental understanding of the micromechanical characteristics of carbon fibers thru investigation of piezoresistance and dynamic torsional behavior.

Progress:

A paper reporting on the discovery and characteristics of additional lines in the Raman spectra of carbon materials was published. A method of plotting diamagnetic susceptibility data to display information on both structural perfection and texture was developed. The torsional behavior of carbon fibers from pitch mesophase (PM), rayon and PAN has been rationalized in terms of microstructural characteristics. Papers on the characterization of carbon materials by diamagnetic susceptibility measurements; and on the torsional behavior of fibers from PM were accepted for presentation at and publication in the proceedings of the 5th London International Conference on Industrial Carbon and Graphite.

For carbon materials with cylindrically symmetric textures, megnetic information on the state of structural development ("crystallinity") and degree of layer-plane preferred orientation may be indicated simultaneously on a single diagram by plotting X_z, the diamagnetic susceptibility measured parallel to the symmetry axis z, against X_{xy}, the susceptibility perpendicular to this axis. X_a, the crystallite susceptibility parallel to the layers, is assumed to be constant, insensitive to structure. Then constant values of $R_z \equiv < \sin^2 \phi >$, where ϕ is the angle between z and the crystallite c-axes, plot as radial lines emanating from the point $X_z = X_{xy} = X_a \approx 0.3 \times 10^{-6}$ emu/g, where the structure may be characterized as amorphous. Data points move outward from this origin with increasing layer size and perfection; and, when layers are well developed, move inward with increasing hexagonal (ABAB) layer stacking order. Ideal structure boundaries, defined by characteristic values of the trace susceptibility $X_T = X_z + 2 X_{xy}$, run diagonally across the diagram from upper left to lower right: For hexagonal graphite $X_T = 22$; and for turbostratic (random stacking) or rhombohedral (ABC) graphite $X_T \approx 37$ at room temperature. Ideal fiber texture ($R_z = 1.0$) lies parallel to the abscissa; ideal transversely isotropic texture (PC plate, $R_z = 0.0$) lies parallel to the ordinate; and isotropic textures ($R_z = 2/3$) lie along the line $X_z = X_{xy}$. Validity and usefulness of this diagram have been confirmed with data on pyrolytic carbons, electrographites and carbon fibers.

The high torsional moduli ($G \approx 35$ GPa) observed for some fibers from PAN are associated with strong circumferential layer textures near the fiber periphery whereas PM fibers with low G values (~10 GPa) have appreciable radial texture components. High tensile modulus fibers, regardless of precursor, exhibit behavior consistent with a fibrillar (stranded-cable) substructure; but the fibrillar size appears to be significantly larger in PM than in PAN or rayon fibers. Apparatus for controlled-temperature fiber piezoresistance measurements has been put into operation; apparatus for investigating the temperature-dependence of the dynamic torsional behavior is still in development.

Papers on the characterization of carbon materials by Raman spectroscopy, the diamagnetic characterization of carbon fibers, and the microhardness characteristics of carbon materials are in preparation.

Papers Published:

"Glassy Carbon Graphitization: Density Change", D.B. Fischbach and Michael E. Rorabaugh, High Temperatures-High Pressures, 1977, Vol. 9, pp. 199-205.

"New Lines in the Raman Spectra of Carbons and Graphite", R. Vidano and D.B. Fischbach, J. Amer. Ceramic Soc. 61 (1/2) 13-17, Jan/Feb. (1978).

Papers Submitted:

"Characterization of Carbon Materials by Diamagnetic Susceptibility", D.B. Fischbach, Proceedings of the 5th London International Carbon and Graphite Conference, Sept. 1978, 6 pages (accepted).

"Dynamic Torsional Behavior of Carbon Fibers from Pitch Mesophase", D.B. Fischbach and S. Srinivasagopalan, 9 pages ibid.

CERAMIC PROCESSING: SURFACE CHARACTERIZATION

Faculty Supervisors: J. G. Dash, Professor
Physics

O. E. Vilches, Associate Professor
Physics

Graduate Assistant: Manu Tejwani, Ph.D. Candidate
Research Assistant

Purpose:

The purpose of this project was to find a way of producing very large area ceramic powders (in particular MgO) with excellent surface homogeneity. Very refined Kr-adsorption measurements at liquid nitrogen temperatures were used to characterize the surface after various production steps.

Relevancy:

a. Well characterized ceramic powders are needed to gain a better understanding of ceramic processing, in particular sintering. Methods developed with MgO can be used to study other materials, like Si_3N_4. MgO itself is widely used.

b. The production of large amounts of well-characterized, surface uniform MgO has given physical adsorption research a surface very different to those materials currently available, mainly graphite. In our own laboratory, we are now measuring the heat capacity of Kr adsorbed on MgO. In addition, we have prepared samples for other laboratories to study light scattering with adsorbed ^3He films (MIT), NMR with ^3He films (Wesleyan University), neutron scattering with various films (UCLA), and tapped helium films (CALTECH).

Objectives:

We plan to continue research into producing different size particles by controlling the environment where the MgO smoke is produced. We also plan to study the effect of eliminating the presence of nitrogen when the smoke is produced. •This research will not be supported under this program.

Progress:

We have constructed an additional adsorption apparatus since our original set up is being used for Si_3N_4 studies.

We installed a compacted sample of MgO in a calorimeter, and have been producing Kr-adsorption isotherms at various temperatures and are starting heat capacity measurements of adsorbed Kr-films one atomic layer thick.

During one of the NASA program reviews work done under this grant was noted by Tektronix, Inc. who use MgO as secondary electron emitter in storage oscilloscopes. They have now awarded our group a six-month contract to continue this study of MgO powders produced an/or used at their plant.

Papers Submitted:

An article describing all the findings of this research project was submitted to the Journal of Physical Chemistry at the end of 1977, and after a minor revision was scheduled to be published in the June 15 issue.

GRAIN BOUNDARY EFFECTS IN BETA-ALUMINA

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

Graduate Assistant: Douglas O. Powell, Ph.D. Candidate

Purpose:

The purpose of this project is to improve the understanding of the role of grain boundaries in the conduction process in fast ionic conductors, particularly in beta-alumina. The effects of grain boundaries will be studied with respect to their purity, orientation and extent.

Relevance:

Since the application of solid electrolytes will almost certainly involve polycrystals rather than single crystals, it is important to understand the degree of influence of grain boundaries upon the conduction process. Any contribution as to the nature of the conduction process will help to provide a predictive capability in the development of improved systems. If the results can be generalized to describe the importance of boundary processes in low-activation energy electrolytes, a further predictive capability will be realized. This work, which utilizes a.c. conductivity measurements as an experimental tool, is complementary to the tracer diffusion studies on polycrystalline beta-alumina done previously under Professor Sarian's supervision.

Objectives:

Objectives for 1978 are:

1. To evaluate the a.c. conduction behavior of oriented polycrystalline specimens by comparison with the behavior of equivalent circuit models of possible processes occurring in the specimen.

2. To verify the applicability of chosen equivalent circuit models by varying sample geometry and by conducting four-probe measurements.

3. To correlate the results of the a.c. conduction experiments with results from tracer diffusion experiments on polycrystalline specimens.

4. To develop a physical model of the grain boundary region in beta-alumina which will improve our ability to predict microstructural effects upon d.c. conductivity.

Progress:

The data collection and reduction phase of the work is still in progress. The final results of this phase of the work are expected within the next six months. No additional funds will be expended in this area.

APPENDIX I

Papers Presented:

J. I. Mueller and A. E. Gorum, "Design of Structural Ceramics," presented at the 2nd Annual Conference on Composites and Advanced Materials, American Ceramic Society, Cocoa Beach, January 1978.

J. I. Mueller, "Structural Design with Ceramic Materials - A University Program," presented at the 80th Annual Meeting, American Ceramic Society, May 1978.

Papers Submitted:

D. B. Fischbach, "Characterization of Carbon Materials by Diamagnetic Susceptibility," Proceedings of the 5th London International Carbon and Graphite Conference, Sept 1978, 6 pages (accepted).

D. B. Fischbach and S. Srinivasagopalan, "Dynamic Torsional Behavior of Carbon Fibers from Pitch Mesophase," 9 pages ibid.

J. G. Dash, R. E. Ecke, J. J. Stoltenberg, O. E. Vilches and O. J. Whittemore, Jr., "Uniform MgO Adsorbents," to be published in the Journal of Physical Chemistry in the June 1978 issue.

J. I. Mueller, "An Educational Program on Structural Design with Brittle (Ceramic) Materials," to be published in Proceedings of the Third Army Materials Technology Conference - Ceramics for High Performance Applications.

Papers Published:

D. B. Fischbach and M. E. Rorabaugh, "Glassy Carbon Graphitization: Density Change," High Temperature/High Pressures, 1977, Vol. 9, pp 199-205.

R. Vidano and D. B. Fischbach, "New Lines in the Raman Spectra of Carbons and Graphite," Journal of the American Ceramic Society, 61 (1/2) 13-17, Jan/Feb (1978).

A. S. Kobayashi, L. I. Staley, A. F. Emery, and W. J. Love, "A Fracture Specimen for High Temperature Testing," Mechanics of Ceramics, Vol. 3, Flaws & Testing. Edited by R. C. Bradt, D. P. H. Hasselman and F. F. Lange, Plenum Press, 1978, pp. 451-461.