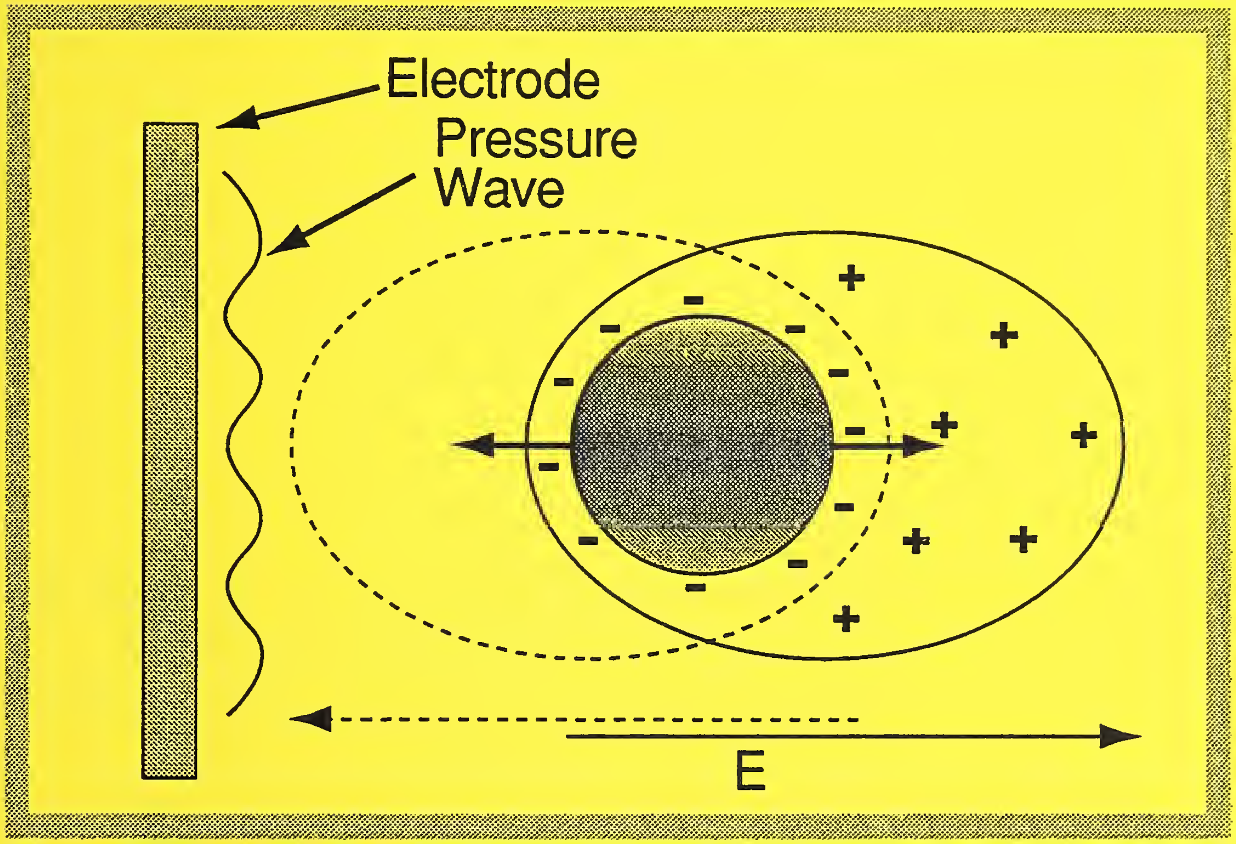
MSEL

Materials Science and Engineering Laboratory

CERAMICS



NISTIR 5747
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities
1995

Ceramics Division

Formation of advanced ceramic components by media-based powder processing requires stringent control of particle dispersion properties. To accomplish this task, new measurement techniques must be developed to characterize particle charge and agglomeration in concentrated suspensions. The cover schematic drawing depicts the electroacoustic response of a charged particle in a high-frequency electric field. Vibratory motion of the particle generates an ultrasonic pressure wave at the electrode surface. The phase and amplitude of the wave are directly related to particle charge and mass. The Ceramics Division program in powder characterization addresses the development of this technique for processing and research applications. This effort is conducted through a NIST-Industry consortium on Intelligent Processing of Powders. The consortium activities are expected to have a direct impact on the processing industry by providing methodology and material/process property data.

MSEL

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CERAMICS

S. W. Freiman, Chief
S. J. Dapkunas, Deputy

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Technical Activities 1995

U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

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Certain trade names and company products are mentioned in the text or identified in illustrations in order to adequately specify the experimental procedure and equipment used.

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OVERVIEW

The format of the 1995 NIST Ceramics Division Report is similar to that begun last year, emphasizing the programmatic nature of our activities rather than those of individual groups. Scientific and technical descriptions of research are organized by programs which are comprised of individual projects. Additional sections describe staff and skills, and important outputs of our research. This format reflects increasing intergroup activities and the desire to focus resources on the most critical technical issues. The participation of U.S. industry in all phases of the research has been strongly encouraged, e.g., the consortia on Machining of Ceramics, and Powder Processing from Slurries. The output of the Ceramics Division program is made available to U.S. industry and the ceramics community through a number of avenues including presentations, publications, and direct collaborations with companies and universities. During this past year the research program produced 147 publications, about 80 presentations, and 3 invention disclosures. Four new Cooperative Research and Development Agreements (CRADAs) were signed with companies and universities.

We are proud of the fact that a number of individuals in the Ceramics Division were the recipients of special awards during the year: Craig Carter was the first recipient of the Coble Award from the American Ceramic Society based upon his work on the modeling of the sintering process; Debra Kaiser received the Sigma Xi Outstanding Young Scientist Award for her work on the magnetooptical imaging of flux pinning in high T_c superconductors; Ron Munro, Ed Begley, and Joyce Harris were given Measurement Service Awards by the Standards Reference Data Office for their development of the first comprehensive database of the properties of high T_c superconductors; Tze-jer Chuang was made a Fellow of ASME, and Said Jahanmir was the recipient of an ASME Service Award.

One new employee, Patricia McGuiggan, was hired into the Division in 1995. Her expertise in measurements of surface phenomena at the atomic level is expected to contribute significantly to a new effort on microtribology.

Sandy Dapkunas completed a six-month term as an "NIST Industry Fellow." The general purpose of the Industry Fellow Program is to allow NIST personnel to spend time in U.S. Industry to obtain first-hand knowledge of how NIST can best serve industrial needs. Sandy worked at the Pratt and Whitney Division of United Technologies Corp. in West Palm Beach Florida with a group heavily involved in the development of coatings for aircraft engines. His goal during his stay there was to learn how materials transition from the laboratory to a product, and how NIST can impact this process. His experience there has already enhanced the NIST coatings program.

In 1995, the Ceramics Division continued a significant involvement with the Advanced Technology Program (ATP), through funding by ATP to work directly with several ATP awardees, through review and program monitoring, and through the efforts of Division personnel who served on ATP Source Evaluation Boards and in the development of ATP focused programs. In addition Subhas Malghan has served in the NIST Program Office for much of 1995.

The Ceramics Division research program has had the following significant acccomplishments in 1995:

- A microstructural model of strain effects in superconducting tapes has been developed. This model is based on synchrotron radiation microradiography, optical microscopy, and electrical resistance measurements.

- The utility of magneto-optical imaging for routine nondestructive analysis of high T_c superconductors, even when the specimen surfaces are not polished, has been demonstrated.

- NIST Standard Reference Database 62: High Temperature Superconductors, Version 1, has been completed. This database provides evaluated thermal, mechanical, and superconducting property data for these materials commonly called high temperature superconductors.

- An international workshop, jointly sponsored by NIST and the Institute for Mechanics and Materials, was held on April 22-23, 1995, in San Diego to discuss the scientific and standardization issues associated with instrumented indentation. Over ninety representatives from industry, academia and national labs around the world met to discuss the indentation process, from both an experimental and a theoretical point of view, and to address the problems underlying the lack of standardization in the field.

- Small angle neutron scattering studies indicate that the prevailing orientation of the cracks in plasma spray deposited coatings depends directly on the spray angle. Interlamellar pores, however, which are mostly parallel to the substrate surface, are unlike the cracks in that their orientation is not a strong function of spray angle. The results offer the first proof that the quantity and the character of the porosity in these materials can each be controlled by selecting the appropriate processing protocols.

- Thirteen new procedures for characterization of powders for secondary properties were developed as part of the International Energy Agency program on powder characteriztion. The procedures and 2000 powder samples were sent to the thirty-three participants from six countries.

- Two procedures for primary characterization of ceramic powders have already passed the ASTM C-28 (American Society of Testing and Materials) main committee ballot and adopted as standards. Three more procedures are in various committee levels. Six additional procedures will be drafted for ASTM adoption. Two standard reference materials (SRMs) have been produced using the procedures developed in this project.

- The results of studies conducted as part of the Ceramic Machining Consortium clearly show the effects on fracture strength of material batch-to-batch variations, repeatability, interlaboratory reproducibility, and machining conditions for three different silicon nitrides being considered for commercial applications.

- A thermal MOCVD system, designed and built at NIST for ferroelectric oxide film deposition, has been modified to include metalorganic bubblers with an oxygen preheater and improved temperature control. These modifications have improved the reproducibility of the deposition experiments. A set of processing conditions has been established for the deposition of epitaxial $BaTiO_3$ films on single crystal MgO and $KTaO_3$ substrates in the modified system.

- A non-parametric bootstrap statistical method has been applied to lifetime prediction of InP components exposed to water and to argon gas at 50% relative humidity. Use of nonparametric bootstrap method permitted lifetime predictions to be made and resulted in a proof-test level prediction which would provide a desired lifetime of 27 years at a 95% confidence level.

- The strain and bond distortions in epitaxial InAs, InGaAs and thicker InAs films have been studied via x-ray standing-wave and x-ray-absorption fine structure measurements. The results indicate that, for both InAs and InGaAs monolayers, strain and bond distortions are accurately described by macroscopic elastic theory. Thicker InAs films are found to collapse beyond a critical thickness of about 2 monolayers, accompanied by a large degree of structural disorder and bond-length relaxation.

- Standard Reference Materials (SRM's) were developed for ceramic Knoop and Vickers hardness which are compatible with ASTM and ISO standard test methods. SRM #2830, the Knoop SRM became available in January, 1996. SRM #2831, the Vickers standard, will be available in the summer of 1996. Two standard test methods that rely in part on the SRM's have been adopted by ASTM.

- CRADAs were signed with Digital Equipment Corp. (now Quantum) and Censtor to jointly study the tribochemistry of the head-disk interface and to develop new instrumentation required to measure head-disk durability. Several testers were donated by our industrial partners to NIST to help to accelerate the research effort.

Stephen W. Freiman
Chief, Ceramics Division

TECHNICAL ACTIVITIES

CERAMIC COATINGS

The Coatings program is a measurement and characterization effort which addresses the processing reproducibility and performance prediction issues associated with, primarily, thermal-spray deposited ceramic coatings. The program focuses on plasma-spray-deposited ceramic thermal barrier coatings used in aircraft gas turbines and expected to be used in land-based turbines and diesel engines. Sales in the thermal-spray industry are currently valued at over one billion dollars annually, a significant portion of which is ceramic thermal-barrier coatings. Collaborations have been established with industrial organizations including Pratt and Whitney, General Electric, Caterpillar, METCO, MetTech and Zircoa as well as the Thermal Spray Laboratory at the State University of New York at Stoney Brook and the Thermal Spray Laboratory at Sandia National Laboratory. The program includes collaboration with the National Aerospace Laboratory and the National Mechanical Engineering Laboratory, both in Japan, to examine functionally gradient materials. Research is also conducted on the processing and properties of chemical-vapor-deposited (CVD) diamond films in collaboration with Westinghouse, an Advanced Technology Program (ATP) awardee, and on Physical Vapor Deposited (PVD) ceramic coatings in collaboration with Praxair, another ATP awardee.

Participants in the NIST program are located in the Ceramics, Materials Reliability and Reactor Radiation Divisions of the Materials Science and Engineering Laboratory as well as the Chemical Science and Technology Laboratory.

The approach taken in the plasma-spray (PS) research has been to build on the analytical capabilities at NIST and the material processing capabilities of collaborators. The program has the following elements:

- development of techniques for characterization of physical and chemical properties of stabilized zirconia feedstock to provide data for increased processing reproducibility as well as data required for production of a Standard Reference Material suitable for calibration of light-scattering size distribution instruments used in industry for analysis of PS powder;

- development of scattering techniques to determine the quantity, size and orientation of porosity and microcracks in PS ceramic coatings suitable for use in modeling the thermomechanical behavior of these materials;

- development of methods to measure chemical, elastic modulus, and thermal properties on a scale suitable for use in microstructural models of behavior;

- development of techniques to model thermomechanical behavior of thermal-barrier coatings to enable more reliable performance prediction; and

- development of techniques for accurate measurement of the thermal conductivity of PS coatings by use of the guarded hot-plate technique suitable for incorporation in ASTM standards and to provide a method for comparison with routine industrial techniques.

Research on chemical mapping of powders and microstructures is conducted in the Microanalysis Division of the Chemical Science and Technology Laboratory. Thermal property research is conducted in the Materials Reliability Division and the Reactor Division participates in both the powder analysis and scattering projects. A strong attribute of the PS coatings research is the use of common materials for which complementary data can provide a more complete understanding of processing-microstructure-property relationships.

Research is also conducted on the formation of chemically-vapor-deposited (CVD) diamond films. This research is focused on the hot-filament method utilizing graphite as a carbon source. This research has allowed the determination of basic system parameters which control nucleation and growth of the diamond film in a relatively easily controlled, inexpensive process.

For additional information about the Coatings Program, please contact S. J. Dapkunas at 301-975-6119 or e-mail to Dapkunas@micf.nist.gov.

PROJECT TITLE: Micro-Mechanical Modeling and Computer Simulations

PROGRAM TITLE: Ceramic Coatings

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Technical Description:

This project is designed to elucidate the mechanics and physics of heterogeneous materials at the mesoscopic level and to develop computationally efficient algorithms and computational codes for simulating the micro-mechanical behavior of advanced ceramic microstructures.

Technical Objectives:

This research is designed to assist industry in developing new paradigms for elucidating micro-mechanical behavior, fracture, deformation, damage, and other nonlinear phenomena, in real and simulated microstructures. A technique for obtaining average linear response from a particular microstructure is envisioned. Predictions of response for simulated and digital representations of actual microstructures are a primary goal. Efficient storage and microstructural representation techniques are to be developed. The goal is the development of a set of tools which all materials scientists can and will want to use.

Outcomes:

A new paradigm for materials calculations is foreseen through the development of the computational tools which will forge this paradigm shift. The tools could be further developed by third parties and potentially lead to new commercial ventures associated with software repackaging. Finally, inexpensive means by which industry may test and design new microstructures may be produced.

Accomplishments:

Averaged elastic behavior of thermal barrier coatings was calculated on a microstructural basis from digitized images and compared with experimental measurements. The computational simulations were performed on random regions from a micrograph of polished sections of a plasma sprayed zirconia coating. Both plan and section views were considered. Elastic properties were treated as orthotopic in the plane. Experimental measurements were performed via Hertzian indentation with a spherical indenter on micro-hardness machine. The specimen area sampled for both the simulations and the experiments was approximately 0.01 mm^2.

Localized microstructural strains in an heterogeneous microstructure were calculated from a digitized micrograph of a polycrystalline alumina. Texture information, which was input into these calculations, was determined experimentally from backscattered Kikuchi patterns, electron backscattered patterns (EBSP), in a scanning electron microscope (SEM). Correlation of the microstructural simulations with a micro-Raman techniques for measuring localized strains in an heterogeneous microstructure was performed.

Initial studies of the elastic, thermal shock, and fracture behavior of silicon nitride have produced exciting results.

Outputs:

Several important outputs were derived from this project. A letter of appreciation was received from the Ford Research Division signifying their appreciation for our initial efforts. A computer code, called OOF (Object-Oriented Finite Elements), is currently available for alpha-testing.

Technical papers:

J. Xu, B.N. Cox, M.A. McGlockton, W.C. Carter, *"A Binary Model of Textile Composites II: Elastic Regime,"* Acta Met and Metall.. 43 9 3511-3524 (1995)

Impact:

Many industrial researchers have asked that we model their particular problems and have asked for use of our developed codes.

PROJECT TITLE: Instrumented Indentation

PROGRAM TITLE: Ceramic Coatings

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Technical Description:

In this program, the ability of instrumented (or continuously recording) indentation to determine various mechanical properties of ceramic coatings and bulk materials is studied. Work focuses primarily on standardization issues and on methods for determining such material properties as hardness, elastic modulus, yield strength, and resistance to microfracture.

Technical Objectives:

The program has four primary objectives:

1. To organize workshops and symposia that will bring the indentation community towards consensus on recommended test and analysis procedures, and to participate in indentation round robins.

2. To collect experimental data (load-displacement curves) and correlate them with analytical and numerical modeling of the indentation process, in order to better understand deformation processes beneath indenters, particularly in film-substrate and multilayer systems.

3. To use instrumented indentation to quantify hardness, Young's modulus, porosity and microcrack density in ceramic coatings, both to characterize as-prepared coatings and to quantify damage evolution.

4. To study the microstructural damage done in the indentation of brittle coatings and bulk material, and correlate with other material properties such as machinability and wear resistance that depend on microfracture processes.

Outcomes:

Recommended guidelines for test and calibration procedures will help users with different machines and at different labs compare results effectively.

Reliable mechanical property data at small length scales will aid designers of thin film and multilayer structures.

The use of indentation to introduce and quantify microcrack damage may lead to use of the technique to predict wear resistance and fracture toughness easily.

Accomplishments:

An international workshop on instrumented indentation, sponsored jointly by NIST and the Institute for Mechanics and Materials, was held in San Diego in April, 1995. The workshop is described under "Highlights," below. Follow-up symposia are planned for 1996.

A higher-load instrumented indenter system was designed and constructed to extend measurements to loads of 4 kg. The previous maximum load had been 0.1 kg.

Instrumented indentation was used to study the machinability of dental ceramics. Correlations were observed between the energy absorbed in an indentation loading-unloading cycle and the energy required to grind away a unit volume of material.

The technique was also used to quantify a) microcrack density in damaged silicon nitride and b) porosity in thermal spray coatings, both through shifts in the Young's moduli of the materials.

Output:

A detailed report on the Indentation Workshop is being prepared. In addition, several technical papers have published:

Technical papers:

"Quantifying Local Microcrack Density in Ceramics: A Comparison of Instrumented Indentation and Thermal Wave Techniques," D.T. Smith and L. Wei, *J. Amer. Ceram. Soc.* **78**, 1301 (1995).

K.G. Kreider, M.J. Tarlov, G.J. Gillen, G.E. Poirier, L.H. Robins, L.K. Ives, W.D. Bowers, R.B. Marinenko and D.T. Smith, "Sputtered Amorphous Carbon Nitride Films." *Journal of Materials Research*, in press.

L.M. Hsiung, R.L. Lankey, H.N.G. Wadley, D.T. Smith, J.Z. Zhang, J.W. Golz, B.L. Halpern and J.J. Schmitt, "Jet Vapor Deposited Aluminum-Aluminum Oxide Nanolaminates." For *"Novel Techniques in Synthesis and Processing of Advanced Materials," TMS/ASM Materials Week 1994,* in press.

Impact:

The instrumented indentation program in the Ceramics Division is still relatively new, and as yet has had only modest demonstrated impact in the industrial sector. One clear benefit, however, came from the workshop that is described below. At that workshop, many attendees from industry (25 from U.S. companies, 6 from foreign companies) had the opportunity to listen to, and talk to, many of the leading researchers in the field. (Some of the leading researchers are, in fact in the industrial sector, and several gave presentations at the workshop.) The interaction gave the industry participants an up-to-date look at how the technique can help them characterize the mechanical properties of the materials with which they work.

PROJECT TITLE: Characterization of Thermal Spray Zirconia Powders

PROGRAM TITLE: Ceramic Coatings

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Technical Objectives:

The objective of this project, conducted in collaboration with Pratt and Whitney (P&W) under the auspices of the National Center for Manufacturing Science (NCMS), was to characterize mass fraction (7 to 8%) yttria containing zirconia powders by physical and surface chemical techniques to identify differences between powders manufactured by different methods and producers and to develop correlations among powder properties and deposition efficiency.

Outcomes:

Two major outcomes are expected: (1) development of correlations among powder properties and deposition efficiency, (2) development of experimental procedures to characterize powders.

Accomplishments:

Through extensive interactions with thermal spray industry, thirteen powders produced by different types of manufacturing processes were selected for this study. These powders were manufactured according to the current P&W powder specifications but no correlations could be developed among the powder properties, coating quality and deposition efficiency (DE). This lack of correlation and powder variability was the motivation for conducting the present study.

The powder properties studied were: chemical composition, density (true, apparent and tap), particle size distribution, powder morphology, agglomerate integrity, interface chemistry, relative heat uptake by differential thermal analyses, phase composition, and specific surface area. The above characterization procedures were developed and tested. The powder properties that affected DE were tap density, relative heat uptake and selective PSD data. Data from characterization methods such as density, agglomerate integrity and optical and SEM micrographs were able to differentiate the powders by various manufacturing processes.

Impact:

Both the NIST developed characterization methods and the correlation established between the powder properties and the performance of the thermal barrier coatings have enabled P&W and other engine manufacturers to tighten powder specifications. This in turn would improve the quality of the thermal barrier coated parts.

Outputs:

A SRM zirconia powder is in preparation to help the thermal spray industry to standardize their experimental procedures such as PSD.

Technical Paper:

"Characterization and Processing of Spray Dried Zirconia Powders for Plasma Spray Application" P.Pei, S.G.Malghan, S.J. Dapkunas and P.H.Zajchowski, Journal of Thermal Spray Technology, In print

PROJECT TITLE: Processing / Microstructure Relationships in Plasma-Sprayed Coatings

PROGRAM TITLE: Ceramic Coatings

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Technical Description:

This project is designed to characterize the pore and crack microstructure of plasma-sprayed ceramic coatings as a function of processing parameters, and relate these to the properties of the deposit product.

Technical Objectives:

The technical objective of this project is a systematic investigation of processing-microstructure-property relationships leading to improved process models that can ultimately offer quantitative predictability of product plasma-sprayed ceramic microstructures.

Outcomes:

These investigations are expected to result in a better understanding of the processing-microstructure-property relationships in plasma-spray deposition of ceramics. Improved processing models will enable the prediction and control of the deposit properties as a function of the spray system and the spray parameters, optimization and reduction of development costs of deposits in engineering practice, and broadening of the field of applications due to increased reliability and predictability of the deposits.

Accomplishments:

Plasma-sprayed deposits are composed of splats created by the rapid solidification of molten or semi-molten feedstock particles. Rapid cooling of the particles results in the formation of metastable phases and complex microstructures. The main features in the microstructure of plasma-sprayed coatings are the splats, separated by interlamellar pores, voids around unmelted particles, and cracks that are most likely formed upon cooling.
Small-angle scattering was applied to the quantitative characterization of the crack and pore microstructure of plasma-sprayed deposits. The anisotropic character of the microstructure has been observed and the methods and techniques for separating the intralamellar crack population from the interlamellar pore population have been developed, in which the surface areas and volume of each are derived.

Outputs:

J. Ilavsky, H. Herman, C. C. Berndt, A. N. Goland, G. G. Long, S. Krueger, and A. J. Allen,"Porosity in Plasma-Sprayed Alumina Deposits" in Thermal Spray _ Industrial Applications, C. C. Berndt and S. Sampath, Eds. (ASM International, 1994) 709-714.

J. Ilavsky, A. J. Allen, G. G. Long, S. Krueger, H. Herman, C. C. Berndt, and A. N. Goland, "Anisotropy of the Surfaces of Pores in Plasma Sprayed Alumina Deposits" in Thermal Spraying _ Current Status and Future Trends, A. Ohmori, Ed. (High Temperature Society of Japan, 1995) 483-488.

J. Ilavsky, A. J. Allen, G. G. Long, S. Krueger, C. C. Berndt, and H. Herman, "Influence of Spray Angle on the Pore and Crack Microstructure of Plasma-Sprayed Deposits" J. Amer. Ceram. Soc., in press (1996).

A. J. Allen, N. F. Berk, S. Krueger, G. G. Long, H. Kerch, and J. Ilavsky, "New Developments in Multiple Small-Angle Scattering Studies of Advanced Ceramics," in Neutron Scattering in Materials Science _II, D. Neumann, T. Russell and B. Wuensch, Eds. (Materials Research Society, Pittsburgh, 1995) 347-352.

CERAMIC MACHINING

Recent studies have estimated that of the total production cost, machining can account for (30 to 60)% and sometimes even up to 90% for high-precision ceramic components. The high cost and sometimes uncertain reliability at times associated with machining damage in ceramic components in comparison with their metallic counterparts are often cited as impediments to the wide spread use of these materials. The goal of the NIST Ceramic Machining Program is to assist the U. S. industry, through a joint research program, in the development of precision machining technology for the manufacture of reliable and cost-effective products made from advanced ceramics. In addition to research within MSEL, joint research is conducted with the Precision Engineering Division, Automated Production Research Division, and Statistical Research Division. The specific projects address Grinding Optimization, Machinability Database, Nano-Precision Grinding, Mechanisms of Material Removal, Characterization of Machining, Damage, and Chemically Assisted Machining.

The research in this program is conducted in cooperation with ceramic producers and users as well as the academic research community and government laboratories. Much of this research is conducted as part of the NIST Ceramic Machining Consortium, consisting of 20 members from industry and academia, and which provides guidance on manufacturing issues and near term implementation of program results.

Current research materials include those intended for structural applications, such as silicon nitride, and dental materials, such as machinable glass.

For additional information about the Machining Program, contact Said Jahanmir at 301-975-3671 or e-mail requests to Said@enh.nist.gov

PROJECT TITLE: Ceramic Machining Consortium

PROGRAM TITLE: Ceramic Machining

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Technical Description:

This project provides measurement methods, data, and mechanistic information needed by industry for their effort in developing innovative, cost-effective methods for machining of advanced ceramics. The specific projects during the reporting period were as follows: (1) Grinding Optimization, (2) Machinability Database, (3) Nano-Precision Grinding, (4) Mechanisms of Material Removal, and (5) Characterization of Machining Damage. Industrial and academic organizations participate in this program by either joining the Ceramic Machining Consortium or by signing a CRADA for joint research on specific research tasks. The following is a list of organizations who were members of the Consortium during the last year: Ceradyne, Inc.; Cercome, Inc.; Cincinnati Milacron, Inc.; Corning, Inc.; Dow Chemical Company; Eaton Corporation; Eonic, Inc.; Ferro Corporation; Ford Motor Company; General Electric Company; General Motors Corporation; Georgia Institute of Technology; Norton Company; Stevens Institute of Technology; Texas A&M University; Torrington Company; Tower Oil and Technology Company; University of Connecticut; University of Maryland; University of Massachusetts; University of Rochester; and West Advanced Ceramics, Inc. In addition, the Norton Company contributed to the machining efforts through an individual CRADA.

Technical Objectives:

The objective of this program is to assist the U. S. industry, through a joint research program, in the development of precision machining technology for the manufacture of reliable and cost-effective products made from advanced ceramics.

Outcomes:

Five major outcomes are expected from this program: (1) recommendations for optimum sets of grinding parameters to be used on specific silicon nitride ceramics, (2) a PC-based database containing data and information on machinability of advanced ceramics, (3) guidelines for obtaining damage-free nano-precision surfaces made from bearing grade silicon nitride ceramics, (4) descriptive models for the process of material removal in grinding including the role of microstructure and the effect of mechanical properties on grinding, and (5) characterization of grinding-induced damage in different ceramics and evaluation of several non-destructive techniques for damage detection.

Accomplishments:

The effect of grinding parameters on strength and surface finish of three commercial silicon nitrides were evaluated in an industrial setting. The results showed no distinguishable change in fracture strength that could be associated with the different grinding conditions for the samples ground in the longitudinal direction of the flexure test bars. These tests also provided information on the repeatability and reproducibility of results obtained in grinding as reflected in surface roughness and strength of machined test samples.

The database structure and the search strategy for the Ceramic Machinability Database were completed. A prototype version was developed and evaluated internally.

Research on the microstructural aspects of the material removal process in grinding showed that intergranular fracture leading to grain dislodgement is the primary material removal process in materials with weak grain boundaries and interfaces. Analysis of ground surfaces showed that the debris generated by grinding contain extremely fine particles that are much smaller than the grain size of the material being ground. Suggesting that the grains are shattered just prior to separation from the surface.

The results of bi-axial strength tests on ceramics with a rising R-curve behavior showed that such materials have a greater tolerance toward machining-induced damage in comparison with ceramics possessing a flat R-curve. The results of this study suggest that in addition to the enhanced damage tolerance, these materials also posses an improved machinability through easy intergranular microfracture.

The feasibility of a thermal wave measurement method and an ultrasonic method were evaluated for the detection of machining-induced damage in ceramics. Both methods were found to be capable of detecting microcracks (on the order of 20 micrometers) that could reduce the strength of ceramic components.

Outputs:

The results of this research program were discussed with the industrial and academic partners on a semi-annual basis. In addition to these interactions, the NIST staff were invited to participate in more than a dozen conferences and technical workshops devoted to ceramic machining.

Technical Papers:

Ahn, H. S., Wei, L. and Jahanmir, S., "Non-destructive Detection of Damage Produced by A Sharp Indenter in Ceramics," *Manufacturing Science and Engineering*, PED-Vol. 68, ASME Press, New York, NY, (1994) 923-933.

Xu, H. H. K. and Jahanmir, S., "Microfracture and Material Removal in Scratching of Alumina," *J. Materials Sci.*, 30 (1995) 2235-2247.

Xu, H. H. K., Jahanmir, S. and Wang, Y., "Effect of Grain Size on Scratch Interactions and Material Removal in Alumina," *J. Am. Ceram. Soc.*, 78 (1995) 881-891.

Xu, H. H. K. and Jahanmir, S., "Effect of Grain Size on Scratch Damage and Hardness of Alumina," *J. Mat. Sci. Lett.*, 14 (1995) 736-739.

Xu, H. H. K. and Jahanmir, S., "Scratching and Grinding of a Machinable Glass-Ceramic with Weak Interfaces and Rising T-Curve," *J. Am. Ceram. Soc.*, 78 (1995) 497-500.

Xu, H. H. K. and Jahanmir, S., "Effect of Microstructure on Abrasive Machining of Advanced Ceramics," *Ceramic Eng. and Sci. Proc.*, 16 (1995) 295-314.

Liang, H. and Jahanmir, S., "Boric Acid as an Additive for Core-Drilling of Alumina," *J. of Tribology*, 117 (1995) 65-73.

Jahanmir, S., Hwang, T., Whitenton, E., Job, L. and Evans, C., "Measurement and Analysis of Forces in Grinding of Silicon Nitride," *Proc. of ASME Tribology Symposium*, H. Masudi (ed.), PD-Vol. 72, ASME Press, New York, NY, (1995) 15-24.

Strakna, T. J., Jahanmir, S., Allor, R. and Kumar, K., "Effect of Grinding Direction on Fracture Strength of Silicon Nitride," *Machining of Advanced Materials*, S. Jain and D. Yang (eds.), AMD-Vol. 208, ASME Press, New York, NY, (1995) 53-64.

Wei, H. H. H. Xu, and S. Jahanmir, "Evaluation of Thermal Wave Imaging Technique for Detection of Machining Damage in Ceramics," NIST IR 5645, March 1995.

Hsu, C. J., Wang, J. C., and Hsu, S. M., "Chemically Assisted Machining of Si_3N_4," Advances in Science and Technology 9, High Performance Materials in Engine Technology, P. Vincenzini, Editor, Techna, 1995.

Patents:

1. "Method of Fabricating Articles," U.S. Patent Application Serial No. 08/062,534.

Impact:

The seventeen companies that have signed CRADA's to either join the Ceramic Machining Consortium or to participate in a specific joint research project, have a direct access to the information generated in this program. The results of the NIST research program on ceramic machining is being used by these companies to develop new ceramic materials and grinding fluids, and to optimize their manufacturing operations to develop cost-effective methods. In addition, participation of the six academic institutions provides a unique opportunity both for the industry members and for the university faculty and their students. This program provides a unique educational opportunity for the students to participate in an industry-lead research program.

PROJECT TITLE: Chemically Assisted Machining of Ceramics

PROGRAM TITLE: Ceramic Machining

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Technical Description:

The US ceramic industry has identified the current machining technology with its low material removal rate as a major cost barrier to the introduction of advanced ceramics into mass markets.

Rapid machining of tough ceramics such as Si_3N_4 often is not feasible because of the rapid heat build up at the interface due to high friction, and the possible introduction of residual surface cracks which often render the component unusable. Machining research in Germany and Japan have emphasized ultra-stiff machine design, heat control, and continuous electrochemical wheel-dressing. Both countries have had national programs on machining of ceramics for the past five years. Many of the advanced machining tools are now designed and built in these countries. The machining industry in the U.S. is mostly small machine shops where expensive machinery often poses an unacceptable economic burden. Within these constraints, chemically assisted machining technology represents an attractive technological option, as well as a major competitive edge for the U.S. machining industry. Chemically assisted machining (CAM) technology, if it is brought into fruition will be low cost, will not require expensive machining tools, and will significantly lower the ceramic components manufacturing cost.

This project is to survey various chemical environments and to assess their effects on the rate of materials removal, surface finish, amount of diamond wear during ceramic machining processes; develop basic measurement methodology and standards to assess the effects of coolants on machining; conduct basic mechanism studies on promising chemical compositions, and based on the results, develop new chemistries to increase the machining rate, improve the surface quality at lower cost.

Technical Objectives:

The project objective is to increase the rate of ceramic machining while controlling the surface finish through modification of contact stresses and material properties by controlled surface chemical reactions. The technology should be capable of providing cost-effective machining techniques using existing machining tools.

Accomplishments:

A simple bench-top cutting device with a diamond wheel was used for screening chemistries. Tests were performed at various speeds and loads, and material removal rates and cutting forces were measured. Results have shown that coolant chemistry can influence the material removal rate by several order of magnitudes without changing the surface quality. There is no current coolant that is specifically designed for ceramic machining.

Toxicity and waste disposal are important criteria in the selection of chemistries as the machining industry is under constant pressure to avoid the use of halogens and other traditional chemistries used in the industry. Several chemistries have been identified and they significantly increase the machining rate.

We are working closely with Kennemetal Corp. who has an existing production line producing silicon nitride and Sialon wear inserts. Their input and guidance have proved to be invaluable. Samples of coolant and materials were supplied to us. These are materials currently being used in their production facility. This provides a sound baseline of current technology and practice. They have also provided test procedures for corrosion so that we can screen the chemistries more effectively.

A surface grinder was set up and instrumented. Precise grinding test procedures (constant feed rate and constant pressure procedures) were developed to assess chemical effects on machining. Results confirmed findings from the cutting tests. The G ratio, the ratio of diamond wheel wear to the amount of material removed, was measured using a wheel print method. A four fold increase in the G ratio was achieved using an experimental chemistry. Analysis of the results suggested that the wear of the diamond was crucial in controlling the materials removal rate. For grinding silicon nitride, a minimum grinding (tangential) force was necessary to start the material removal process and this minimum force was different for different fluids. A maximum grinding force beyond which further increase in grinding force would not increase the material removal rate but would increase the surface damage also existed. The maximum force is also different for different fluids. This suggested optimum grinding conditions existed for a particular wheel-fluid-workpiece combination that would have the fastest grinding rate and minimum surface damage. These findings have also been validated on a vertical grinder which was donated by Kennemetal.

Outputs:

Technical Publications:

Wang, J. And Hsu, S. M., "Chemically Assisted Machining of Ceramics," J. of Tribology, 116, 423-429, 1994.

Hsu, C. J., Wang, J. C., and Hsu, S. M., "Chemically Assisted Machining of Si_3N_4, "Advances in Science and Technology 9, High Performance Materials in Engine Technology, P. Vincenzini, Editor, Techna, 1995.

Patents:

A process to chemically assisted machining of ceramics, filed in 1994 with the US Patent Office.

A process to assist machining of ceramics using chemicals, patent disclosure filed in NIST in April 1995.

Impact:

The feasibility of chemically assisted machining of ceramics has been demonstrated. This opens up a new way to lower the cost of components made from advanced materials without the heavy investment requirement of expensive machinery to machine advanced materials. This technology allows the US industries to regain some competitive edge in the fabrication and manufacturing sector while providing a major advantage for the U.S. ceramic producers.

CERAMIC PROCESSING

Processing of ceramic powders to the green and sintered states is a major component in the production of ceramics with high degrees of reproducibility and cost-effectiveness. Several programs have been initiated in a number of countries involved in advanced ceramics to address these issues. Technical elements affecting the cost-effective manufacturing are synthesis of novel powders that have tailored properties, repeatability and reproducibility in the measurement of powder properties, procedures for characterization of secondary properties of powders, powder-aqueous environment interactions, and sintering behavior of green ceramics.

The program on ceramic processing is designed to address some of these issues with a focus on improvement of measurement quality in ceramic powders processing. This is accomplished by providing the U.S. ceramics community the ability to control ceramic powder properties so that cost-effective manufacturing can be achieved. The current program continues to emphasize structural ceramics with application of silicon nitride to automotive engines. The specific elements of the program are as follows:

- ceramic powder characterization techniques, measurement science, sensors and models for intelligent processing, standard methods and standard reference materials;
- powder processing science to understand interrelationships between powder characteristics and their processing environment; and
- novel processing and synthesis methods as applied to nanosize and ultra-pure powders.

Ceramic processing projects address the development of standard procedures, standard reference materials, improving the scientific basis of powder dispersion measurements in slurries, overall improvement in measurement accuracy, and leadership in national and international standardization activities including the ISO and ASTM. The powder characteristics of interest include physical and surface chemical properties, and phase composition. In addition, grain-boundary characterization, and crystallographic texture investigations are carried out using advanced characterization tools. In powder processing, our efforts have been focused around high-energy agitation milling of powders, textured Al_2O_3 microstructure, and hot-isostatic-pressing of silicon nitride.

For further information on the Ceramic Processing Program, contact S. J. Dapkunas at 301-975-6119 or e-mail to Dapkunas@micf.nist.gov.

PROJECT TITLE: Powder Characterization

PROGRAM TITLE: Ceramic Processing

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Technical Description:

This project is aimed at developing and providing a set of commonly agreed procedures to be used for characterization of ceramic powders used in the ceramic heat engine applications as well as development of the understanding of the role of interfacial energy anistropy on the microstructural evolution of ceramics.

Technical Objectives:

The overall objective of this project is to develop pre-standardization procedures for characterization of secondary properties of powders such as those for the measurement of slurry, pH, green density and porosity of green ceramics.

Outcomes:

Major measurement outcomes are expected to occur: (1) collection of the procedures for characterization of secondary properties, (2) ruggedness testing of the procedures by the participants through the method of a interlaboratory comparison study to determine the major variables and their impact on the resulting data, (3) refined of the procedures based on the results, (4) development of specific and detailed procedures for characterization of secondary properties to enhance the quality, repeatability and reproducibility of the measurements.

Impacts:

Many standards organizations such as ASTM, CEN (European Standards Committee) and JISE (Japan Standards Association) are using the round-robin data and procedures for developing their standards. This will lead to the development of international standards. Industrial laboratories are using the SRMs developed from this project.

Outputs:

L.H. Lum, S.G. Malghan, and S.B. Schiller, "Standard Reference Materials for Particle Size Analysis of Ceramic Powders by Gravity Sedimentation," Powder Technology, in press

Standard Reference Material 1978, Particle Size Distribution Standard for Sedigraph Calibration (October 1993).

Standard Reference Material 659, Particle Size Distribution Standard for Sedigraph Calibration

PROJECT TITLE: Sintering and Microstructure Development

PROGRAM TITLE: Ceramic Processing

Principal Investigator(s):

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Technical Description:

The driving force for the consolidation and densification of ceramic powders during sintering is the difference between the energy of the free surfaces which are eliminated and the energy of the grain boundaries which form. While the modelling of microstructure development during sintering of ceramics has generally assumed that all interfacial energies are isotropic, measurements of the interfacial energies have shown that most materials are anisotropic. Anisotropy of these interfacial energies will lead to differences in the local rate of densification, shape of pores and the location of pores (either attached to or separated from grain boundaries). When pore separation occurs, shrinkage of the pores effectively stops and a limiting density is reached. large differences in mobility of specific grain boundaries can result in a highly textured microstructure consisting of elongate grains. If a liquid phase is present, not all grain boundaries will be wetted depending on the local energy balance.

Technical Objectives:

The understanding of the effect of interfacial energy anisotrophy on the microstructure evolution of ceramics. The microstructural features of concern are the pore location, the distribution of liquid phases and the crystallographic texture.

Outcomes:

We have determinated the equilibrium (Wulff) shape of Al_2O_3 in air. The resultant shape was compared with the shapes predicted from theoretical calculations of the surface energy, and large discrepancies were found.

The conditions for wetting of low angle grain boundaries in Al_2O_3 by anorthrite glass have been measured. It was shown that not only is the degree of grain boundary misorientation important, but the normal to the boundary plane also controls the wetting.

A technique for measuring individual grain orientations from surface facetting using AFM measurements has been developed. This technique uses the observed faceting and the Wulff shape to determine the orientation and is being calibrated using orientations determined by electron backscatter patterns of the same grains.

Outputs:

Technical papers:

"Stability and Surface Energy of Wetted Grain Boundaries in Aluminum Oxide", D-Y. Kim, S.M. Wiederhorn, B.J. Hockey, C.A. Handwerker and J.E. Blendell, J. Amer. Ceram. Soc. 77(2)444-53(1994).

"Texture Measurement of Sintered Alumina using the March-Dollase Function", J.P. Cline, M.D. Vaudin, J.E. Blendell, C.A. Handwerker, R. Jiggetts, K.J. Bowman and N. Mendendorp, Advances in X-ray Analysis, 37, J.V. Gilfrich, ed., Plenum Press, New York, 1994, 473.

"The Equilibrium Shape of Internal Cavities in Sapphire" J-H. Choi, D-Y. Kim, B.J. Hockey, S.M. Wiederhorn, C.A. Handwerker, J.E. Blendell, W.C. Carter and A.R. Roosen, submitted to J. Amer. Ceram. Soc., Jan 1996.

Impact:

Allow materials development to use the anisotropy of materials to enhance/improve the properties in specific orientations, and to use changes in anisotropy (via doping) to control microstructure. Development of measurement techniques for easily determining the surface energy anisotropy.

PROJECT TITLE: Broad Based Manufacturing

PROGRAM TITLE: Ceramic Processing

Principal investigator(s):

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Technical Description:

Silicon nitride components for heat engines are under development in industry to take advantage of their superior wear resistance and high temperature strength. Shaping of these parts in a rapid manner with reproducible properties is necessary to allow production at a competitive cost which allow broad commercialization. This project addresses fundamental measurements required for the development of aqueous injection molding, an ATP funded program.

Technical Objectives:

This project is focused on the development of methods of characterizing the green body structure of parts made by aqueous injection molding and development of means of measuring the dispersion and other properties of dense suspensions of silicon nitride.

Outcomes:

This research is expected to enhance industry's capability to fabricate cost effect silicon nitride automotive components as well as industrial gas turbine hardware. This will be accomplished by the development of measurement methods such as electro-acoustic sonic amplitude measurement techniques which can be implemented by industry to control process materials.

ELECTRONIC AND PHOTONIC CERAMICS

The objectives of the Electronic and Photonic Ceramics program are to provide data, measurement methods, standards and reference materials, models, and understanding of the fundamental aspects of processing, structure, properties, and performance of electronic and photonic materials. The program supports generic technologies in bulk and thin-film ceramics in order to foster their efficient and economical use as electronic and/or photonic materials. The research addresses the science base underlying advanced electronic/photonic materials technologies together with associated measurement methodology.

The principal activities of the program are directed toward materials for electronic and photonic technology related to data processing, storage, display, and transmission. Four aspects of this technology being addressed are modulator materials, storage-media materials, materials for microwave transmission, and material for compact, short-wavelength radiation sources to increase storage density. In the area of modulator materials, MSEL scientists are evaluating film processing methods and their relationship to properties such as electro-optic modulator characteristics. In the area of storage-media materials, scientists are addressing issues in thin-film and bulk ferroelectric and photorefractive materials, in which the storage method involves either modification of the refractive and absorptive properties upon exposure to optical radiation or changes in material polarization by application of an electric field. Ferroelectric oxides are being studied because they possess large spontaneous polarizations, large electro-optic coefficients and large photorefractive effects. In the area of data transmission, phase diagrams important for the production of low-loss, thermally stable ceramics for fabrication of components to be used in microwave communication devices are being developed..

In the area of materials for short-wavelength sources, our studies are directed toward crystalline materials with large bandgaps,e.g. gallium nitride, aluminum nitride, and zinc selenide, because a large bandgap is required if a material is to emit radiation at short wavelengths. A key issue is the characterization of defects at very low concentration levels. Collaborations have been established for the purpose of providing data that will allow manufacturers to improve the quality of the materials.

For further information on the Electronic and Photonic Ceramics Program, please contact Grady White at 301-975-5752 or e-mail requests to Spruce@enh.nist.gov.

PROJECT TITLE: Molecular Chemical Synthesis of a Microwave Ferrite

PROGRAM TITLE: Electronic and Photonic Ceramics

Principal investigator(s):

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Project Description:

This project focusses on the use of aqueous solution chemistry to synthesize a model microwave ferrite material, nickel iron oxide, in fine-powder form.

Technical Objective:

The research is directed towards assisting industry to generate microwave ferrite materials economically from commonly available chemicals.

Outcomes:

The model synthesis system developed in this work is expected to have substantial generality for applications in the production of a variety of other, relevant microwave ferrites.

Accomplishments:

Two model systems for the synthesis of precursors to the technologically important ferrite, nickel iron oxide, $NiFe_2O_4$, were explored. One used tartaric acid to form complexes with iron and nickel ions, while the second used tartaric acid admixed with ethylene glycol. Spectrophotometric studies of the metal-organo complexes in solution suggested an optimum molar ratio of metal ion-to-complexing agent of 1 : 1.

The evolution of $NiFe_2O_4$ from the precursors as a function of temperature up to 1000 °C was explored. During this part of the study it was noted that the addition of ethylene glycol to the metal ion-tartaric acid complexes provided certain advantages. These included a lower $NiFe_2O_4$ crystallization temperature, substantially smaller particles, and minimal amounts of unreacted hematite remaining in the final product.

In both systems, typical yields of the product were (93 to 95)% of theoretical. Particle sizes were typically 1μm for the tartaric acid process, and 0.5μm for the tartaric acid-glycol process.

Output:

Technical Paper:

"Synthesis of $NiFe_2O_4$ by a Metal-Organo Complex Method", Joseph J. Ritter and Pichai Maruthamuthu, accepted for publication by the Journal of Materials Synthesis and Processing.

Impact:

The work supplies guidelines for a simple, economically viable, chemical approach to the production of microwave ferrites.

PROJECT TITLE: Phase Equilibria Studies of Dielectric Oxides for Wireless Communications

PROGRAM TITLE: Electronic and Photonic Ceramics

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Project Description:

Dielectric ceramics are used to fabricate a variety of components in cellular communications circuits that store, filter, and/or transfer electromagnetic energy with minimal loss (e.g., resonators (bandpass filters), circulators). The required properties for the ceramic materials include high dielectric constant, minimal dielectric loss (which precludes the use of ferroelectric oxides), and essentially zero temperature dependence of dielectric properties. Knowledge of phase equilibria relations is important owing to the additive nature of dielectric properties; e.g. practically all ceramic components are processed as mixtures to achieve compensation and a net overall zero temperature coefficient. Thus, identification and characterization of phases in equilibrium with the dielectric ceramic are essential in order to process ceramic components that meet current standards of tightly controlled dielectric constant ($\pm0.25\%$), low dielectric loss, and temperature coefficients near zero (±4 ppm/°C). Furthermore, in carrying out the determination of phase relations in a system, the existence of previously unknown compounds with potentially useful properties may be elucidated. The approach taken by the phase equilibria group focusses on the experimental determination of ternary (or higher) oxide systems containing one or more components that are known to give rise to materials with useful properties as dielectric ceramics for microwave communications. Efforts are directed to synthesis, structural characterization, and characterization of dielectric properties.

Technical Objectives:

To determine the phase relationships in ceramic materials used in Wireless communication devices.

Accomplishments:

The $BaO-Fe_2O_3-TiO_2$ system was chosen for determination to elucidate the phase relations between a magnetic component and the existing Ba-Ti-O microwave ceramics. High-dielectric-constant ceramic magnets are fundamental to a wide variety of communication applications as circulators and isolators. The $BaO-Fe_2O_3-TiO_2$ ternary phase diagram has been investigated at (1250 to 1270) °C in air. X-ray diffraction studies of approximately 150 polycrystalline specimens at room temperature confirmed the existence of sixteen ternary compounds. Two of these compounds, $BaFe_4Ti_2O_{11}$ and $Ba_{12}Fe_{28}Ti_{15}O_{84}$, have been previously reported, four were found to be isostructural with known chemically similar compounds, and ten apparently adopt new structure-types. The crystal structures of five of the new ternary phases are briefly described. The oxidation state of iron found in this study is similar to that reported for a study of the $BaO-Fe_2O_3-SnO_2$ system in air at 1200°C. No indications of reduction of Fe^{3+} to Fe^{2+} were observed; however, oxidation to Fe^{4+} was clearly indicated in an extensive solid solution $BaTi_{1-x}Fe_xO_{3-z}$, $x=0.06$ to 0.84, with the hexagonal $BaTiO_3$ structure. Substantial solid solution regions were also found for the hollandite-type structure ($Ba_xFe_{2x}Ti_{8-2x}O_{16}$, $x=1.07$ to 1.33), and for TiO_2 dissolved in the $BaFe_{12}O_{19}$ structure (end member $BaFe_{10.8}Ti_{0.9}O_{19}$). The complexity of the $BaO-Fe_2O_3-TiO_2$ system is attributed to the coordinative versatility of Fe^{3+} in a close-packed O/Ba-O matrix and the resulting opportunity to form a wide variety of layered structures with different stacking sequences and distortions from ideal packing.

The $SrO-TiO_2-Nb_2O_5$ system is of possible importance in the formation of useful compositions with high dielectric constant. $SrTiO_3$, with the cubic perovskite structure, has a very high dielectric constant, but unfortunately it has a very negative temperature coefficient of resonant frequency (T_f). It has been found that cubic $SrTiO_3$ is in equilibrium with $Sr_6Ti_2Nb_8O_{30}$ or some solid solution of similar composition with the tetragonal tungsten bronze (K_xWO_3) type structure. The T_f of the phases in this ternary system are not known. In addition, there are several possible phases of hexagonal structure related to $Sr_5Nb_4O_{15}$ which may exist on the join between $SrTiO_3$ and $Sr_5Nb_4O_{15}$. The phase $Sr_6TiNb_4O_{18}$ has been synthesized but "$Sr_8Ti_3Nb_4O_{24}$" has not been isolated, although the phase $Ba_8Ti_3Nb_4O_{24}$ is known. Instead, the new phase $Sr_7Ti_2Nb_4O_{21}$ was found to exist. Further studies will be continued in the ternary system to understand the compatibility relations, and the dielectric constants and T_f data will be evaluated.

Outputs:

Technical Papers:

"Magnetic Dielectric Oxides: Subsolidus Phase Relations in the $BaO\text{-}Fe_2O_3\text{-}TiO_2$ System", T.A. Vanderah, J.M. Loezos, and R.S. Roth.

"Preparation, Crystal Structure, Dielectric Properties, and Magnetic Behavior of $Ba_2Fe_2Ti_4O_{13}$", T.A. Vanderah, Q. Huang, W. Wong-Ng, B.C. Chakoumakos, R.B. Goldfarb, R.G. Geyer, J. Baker-Jarvis, R.S. Roth, and A. Santoro.

"Preparation and Crystal Structure of Sr_5TiNbO_{17}", A.R. Drews, W. Wong-Ng, R.S. Roth, and T.A. Vanderah.

PROJECT TITLE: Ferroelectric Oxide Thin Films for Photonics

PROGRAM TITLE: Electronic and Photonic Ceramics

Principal Investigator:

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Technical Description:

This project involves a study of the complex relationships between thin film deposition, microstructural features and electro-optical properties of barium titanate thin films deposited by by metalorganic chemical vapor deposition (MOCVD). Novel electro-optical measurement methodology is under development to determine the crystallographic orientation and polarization state of the thin films.

Technical Objective:

The objectives are to address key scientific issues vital to the advancement of ferroelectric oxide-based thin film photonic devices for information technology and communications industries and to develop appropriate measurement methodology as needed to study these issues.

Outcome:

The outcome will be the establishment of microstructural characterization and electro-optical measurement techniques needed to develop the processing/structure/property relationships of importance for ferroelectric oxide thin films for photonic devices.

Accomplishments:

A thermal MOCVD system, designed and built at NIST for ferroelectric oxide film deposition, has been modified to include an oxygen preheater and metalorganic bubblers with improved temperature control. These modifications have improved the reproducibility of the deposition experiments. A set of processing conditions has been established for the deposition of epitaxial $BaTiO_3$ films on single crystal MgO and $KTaO_3$ substrates in the modified system.

X-ray diffraction studies showed that the $BaTiO_3$ films could contain either epitaxial, polycrystalline or amorphous material, or a combination of epitaxial and amorphous or polycrystalline and amorphous phases. Second harmonic generation (SHG) in the films was measured as a function of the incidence angle of the fundamental beam as well as the direction of polarization of the fundamental beam. The SHG data on the as-grown films were not relatable to either the microstructure or the crystallographic orientation of the $BaTiO_3$ grains. Thus, SHG was shown not to be a viable technique for determining the orientation of $BaTiO_3$ thin films, despite contrary reports in the literature.

Outputs:

Technical Papers:

"Epitaxial growth of $BaTiO_3$ thin films at 600 °C by metalorganic chemical vapor deposition", D. L. Kaiser, M. D. Vaudin, L. D. Rotter, Z. L. Wang, J. P. Cline, C. -S Hwang, R. B. Marinenko, and J. G. Gillen, Appl. Phys. Lett **66**, 2801 (1995).

PROJECT TITLE: Defect Studies in Photonic Materials

PROGRAM TITLE: Electronic and Photonic Materials

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Technical Description:

This project provides: 1) observation, analysis, and evaluation of residual disorder in high quality optoelectronic/photonic single crystals; 2) determination of the effects of the various types of disorder on optoelectronic/photonic crystal properties and performance; and, 3) through collaboration with crystal growers and optoelectronic/photonic device fabricators, improved control of those particular types of crystalline disorder that most affect optoelectronic/photonic device performance.

Technical Objectives:

The objective of this project is the development and production of materials suitable for optical computing, optical communications, and optical information processing. This objective is achieved through the exploitation of 1) special high sensitivity measurement facilities and 2) photonic material structural interpretive expertise, neither of which is available in the private sector.

Outcomes:

Four outcomes are anticipated: 1) improved scientific and practical understanding of optical interactions in photonic crystals, 2) reliable structural requirements for high quality crystals capable of producing world-class devices for optical computing, other optical information processing, optical communications, and optical information storage; 3) improved growth of these crystals in the private sector, following establishment of the relevant structural requirements; and 4) quantitative verification that the structural goals have been met.

Accomplishments:

Through collaboration with Deltronic Crystal Industries and Tufts University we facilitated the commercial production of superior barium titanate and strontium barium niobate single crystals designed for use in holographic devices for high capacity information processing and storage. This accomplishment required, in turn the development of the two new capabilities: 1) the first direct observation of the storage of photorefractive information in these crystals and 2) complete mathematical models, used successfully in the detailed interpretation of the novel x-ray diffraction images obtained.

Through collaboration with NZ Technologies and Tufts University we demonstrated attainment of the first high quality epitaxy of a photorefractive oxide layers on an oxide substrate, a process with great technological potential for new forms of optical information processing. In doing so, we showed that the substrates used at this point will severely limit the performance of devices made from these layered devices in a manner that is in addition to any further limitations that are introduced through subsequent processing.

Through collaboration with Deltronic Crystal Industries and the Naval Research Laboratory we have shown that heavy doping of lithium niobate for improved photorefractive sensitivity and the periodic poling of undoped material both are strongly correlated with the prevalent defect structure in this material.

In collaboration with other NIST scientists we are developing an understanding of the generation of defects as III-V layers are deposited on high quality III-V substrates. Devices fabricated in this manner form the basis for novel optoelectronic/photonic approaches to information processing.

Outputs:

Some of the most important outputs of this project are the improved optoelectronic/photonic crystals that are likely to form the basis for novel information processing and storage technology. Other outputs are the information that will be used by others in the scientific and technical community to improve the performance of these and related materials and the understanding on which still further enhancement of performance can be based.

Technical Papers:

L.D. Zhu, J. Zhao, F. Wang, Peter E. Norris, G. D. Fogarty, B. Steiner, P. Lu, B. Kear, S.B. Kang, B. Gallois, M. Sinclair, D. Dimos, and M. Cronin-Golomb, *Epitaxial electro-optical* $Sr_xBa_{1-x}Nb_2O_6$ *films by single-source plasma-enhanced metalorganic chemical vapor deposition*, Appl. Phys. Lett. **67**, 1836-1838 (1995).

Bruce Steiner, L.E. Levine, Margaret Brown, and David Larson, *Residual disorder in low pressure, low thermal gradient liquid encapsulated Czochralski gallium arsenide observed in high resolution synchrotron diffraction imaging,* J. Crystal Growth, in press (1996)

G. Fogarty, B.Steiner, M. Cronin-Golomb, U. Laor, M. H. Garrett, J. Martin, and R. Uhrin, *Anti-Parallel Ferroelectric Domains in Photorefractive Barium Titanate and Strontium Barium Niobate Observed by High-Resolution X-Ray Diffraction Imaging*", JOSA **B,** in press (1996).

G. Fogarty, B. Steiner, M. Cronin-Golomb, R Uhrin, and J. Martin, *High Resolution X-Ray Diffraction Imaging of Anti-Parallel Ferroelectric Domains in Barium Titanate and Strontium Barium Niobate*", Proc. Photorefractive Materials, Effects, and Devices Topical Meeting, Estes Park, 9 (1995).

PROJECT TITLE: Growth and characterization of blue/green laser materials

PROGRAM TITLE: Electronic and Photonic materials

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Technical Objectives:

This project is designed to assist industry with the development of blue/green semiconductor lasers, and other short-wavelength optical emitters, by providing data about the optical and optoelectronic properties of the materials that form the basis for these devices.

Technical Description:

Researchers who are growing wide-bandgap semiconductor materials need feedback about how process parameters affect the properties of their materials that are most relevant to the intended applications. In this project, several techniques of optical emission spectroscopy are used to investigate the properties of wide-bandgap semiconductor materials as a function of process parameters. The correlation of the observed properties with material structure and composition is examined in order to provide a more fundamental understanding of the data. The lasers will be used in high-density optical data storage systems; the non-laser blue/green emitters will be used in compact full-color displays (e.g., notebook computers, instrument panels).

Accomplishments:

Several optical emission spectroscopy systems have been set up to enable the experimental measurements needed for this project. These include: laser photoluminescence (PL) and photoluminescence excitation spectroscopy; time-resolved PL with a pulsed-laser source; and cathodoluminescence (CL) imaging and spectroscopy in a scanning electron microscope. Sample cooling to liquid-helium temperature, needed to observe spectral features that are not detectable at room temperature, is a capability of both the PL and CL experiments.

Collaborations have been established with researchers outside NIST who are producing state-of-the-art materials. Dennis Wickenden at Johns Hopkins University Applied Physics Laboratory is growing thin films of Aluminum Gallium Nitride (AlGaN) by metalorganic chemical vapor deposition. These films, which give rise to optical emission from the visible to the deep UV, are intended for UV lasers and visible display devices. Gene Cantwell at Eagle-Picher Research is growing Zinc Selenide (ZnSe) wafers as substrates for blue/green optical emitters. Experiments performed on both AlGaN and ZnSe specimens have demonstrated that the selected experimental techniques are applicable to these materials and provide significant information. Key experimental results are summarized below.

The PL and CL spectra of Al_xGa_{1-x} films (from Johns Hopkins Applied Physics Laboratory) were investigated as a function of composition (x), temperature, and type of excitation. The band-edge emission energy was found to depend linearly on x, as expected; the width of the band-edge emission peak, an indicator of residual stress, was found to increase slightly with x; and bound-exciton emission was associated with defects localized near the film-substrate interface. These results will assist growers in optimizing deposition conditions for the films.
Spectrally resolved CL images of Al_xGa_{1-x} films (from JHAPL) were observed at low temperature and room temperature. Images of the mixed composition films (Al content $x > 0$) showed networks of bright lines along directions of high crystal symmetry. These bright lines may be due to microcracks that arise from thermal expansion mismatch between the film and substrate. Images of some of the lower Al content films showed bright circular rings that may be due to screw dislocations. The extended defects identified by CL imaging may affect performance of the films in light-emitting and other photonic applications.
The variation of the low-temperature CL spectra of a ZnSe single-crystal boule was examined as a function of distance from the bottom of the boule. Peak shifts of some of the narrow emission peaks suggest a stress that varies with distance along the growth direction. An emission peak ascribed to dislocations was observed to be localized within the first millimeter from the bottom of the boule. Mapping the spatial distribution of stress and defects in the boule will help crystal growers produce material with lower stress and defect density. This is important for applications of the crystals as substrates for wide-bandgap LEDs and laser diodes.

PROJECT TITLE: Pulsed Laser Deposition (PLD) of Ceramic Thin Films

PROGRAM TITLE: Electronic and Photonic Materials

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Technical Description:

This project is intended to develop measurement capabilities to monitor, *in situ*, processing of thin films using PLD. This information is intended to allow industry in-process monitoring during film deposition and control of resultant thin film properties.

Technical Objective:

The objective of the work is to relate processing parameters to final film properties, thereby reducing the need for time consuming empirical derivation of optimal processing conditions and extensive post processing analysis currently used in industry.

Outcome:

The outcome of this work will be the development of *in situ* characterization techniques to allow real-time analysis of the details of the deposition process during PLD. The time dependent deposition parameters will be related to resultant film properties.

Accomplishments:

New or improved diagnostic methods were developed for real-time observations of key intermediate species during the PLD of electronic ceramic thin films. These methods were based on optical imaging and spectroscopy, coupled with mass spectrometry and their development was enhanced by chemical-dynamic model simulations of the PLD process. Improvements in process understanding, optimization and control are under development as a result of these advances.

Outputs:

Tangible outputs resulting from this project included seven invited talks, given at national and international symposia on thin film processing, together with eight research papers.

Technical papers:

"Imaging and Modeling of Pulsed Laser Ablation Plumes in Thin Film Deposition", P. K. Schenck, J. W. Hastie, A. J. Paul, and D. W. Bonnell, Optical Eng., in press, 1995; also Proceedings, SPIE Symposium, Laser-Induced Thin Film Processing, San Jose, Ca., Feb. 1, 1995.

"High Temperature Chemistry in Laser Generated Plumes", J. W. Hastie, D. W. Bonnell, A. J. Paul, J. Yeheskel, and P. K. Schenck, High Temp. Science and Materials. 33, 135 (1995).

PROJECT TITLE: Damage Accumulation in Actuator Materials

PROGRAM TITLE: Electronic and Photonic Ceramics

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Technical Description:

Actuator materials inherently undergo cyclic loading conditions which can lead to mechanical and electronic degradation. The degradation mechanisms and the interactions between mechanical, electronic, and thermal behavior in the material must be understood for reliability assessment and lifetime predictions to be meaningful.

Technical Objective:

This project is intended to identify failure mechanisms in cyclically loaded actuator materials, so that meaningful reliability estimates can be made for smart materials systems. The interactions between stress and piezoelectric properties, the generation of microcracks, and the effects of thermal excursions are all critical elements under investigation.

Outcome:

The outcome of this work will be an understanding of failure processes in lead zirconate titanate (PZT) actuator material and, thereby, tools to allow lifetime prediction for these materials used in hostile or critical applications.

Accomplishments:

We have determined that microcrack formation occurs during all loading conditions, but that it appears to be in competition with domain wall motion. Similarly, we have determined that the onset of the reported anisotropy in macrocrack lengths, parallel and perpendicular to the poling direction, corresponds to a discrete degree of polarization which, in turn, is correlated to the onset on non-180° domain wall motion. Finally, a decrease in microcrack density at $T \approx 180°C$ from that observed at RT with simultaneous increase in macrocrack growth appears to be tied directly to non-180° domain wall motion and concomitant reduction in residual stresses.

Output:

Technical Papers:

The output of this work, during the past year, includes the following technical papers:

M. D. Hill, G. S. White, and I. K. Lloyd, "An Examination of Factors Influencing Crack Extension in PZT During Cyclic Loading," submitted to the Proceedings of the 6_{th} Symposium on Fracture Mechanics of Ceramics," Karlsruhe, Germany (7/17-20/95).

M. D. Hill, G. S. White, and I. K. Lloyd, "Cyclic Damage in PZT," accepted by the J. Am. Ceram. Soc. (1995).

EVALUATED MATERIALS DATA

The objective of the Data Technologies program is to develop and facilitate the use of evaluated databases for the materials science and engineering communities. Both research- and application-directed organizations require readily available evaluated data to take advantage of the large volume of materials information developed on public and private sponsored programs. This information, particularly numeric data, is available in an ever increasing number of publications published worldwide. The necessity to consolidate and allow rapid comparison of properties for product design and process development underlies the database projects.

Evaluated Data projects are conducted in cooperation with the NIST Standard Reference Data Program Office and include compilation and evaluation of numeric data as well as recently initiated efforts directed at more effective distribution and use of data.

Database projects in MSEL include:

- Phase Diagrams for Ceramists (PDFC), conducted in cooperation with the American Ceramics Society;
- the Structural Ceramics Database (SCD), a compilation of evaluated mechanical and thermal data for nitrides, carbides, and oxides of interest to engineers and designers;
- a ceramic machinability database, coordinated with the Ceramic Machining Research Program;
- a high T_c superconductivity database developed in cooperation with the Japanese Agency for Industrial Science and Technology (see superconductivity);
- development and implementation of the STEP protocol for the exchange of materials data, under the auspices of the ISO 10313 activity;
- the NACE/NIST Corrosion Performance Database developed to provide a means to select structural alloys for corrosive applications; and
- the Crystal Data Center which provides fundamental crystallographic data on inorganic materials.

These projects are developed with the cooperation of the materials community and complement various research programs.

For additional information on the Evaluated Materials Data Program, contact S. J. Dapkunas at 301-975-6119 or e-mail to Dapkunas@micf.nist.gov.

PROJECT TITLE: Phase Diagrams for Ceramists

PROGRAM TITLE: Evaluated Materials Data

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Technical Description:

The Phase Equilibria Program sponsored jointly by the American Ceramic Society (ACerS) and NIST was formally established in 1982. The purpose of this cooperative effort is to provide qualified, critically evaluated data on thousands of chemical systems relating to ceramic materials research and processing, as well as a critical database to serve the ceramic community. This information serves as an objective reference for important processing data. Approximately $1.8M was raised by the ACerS from industry for the support of the program. The outputs of the program include printed volumes of evaluated phase programs as well as software packages containing bibliographic and graphics phase diagram data.

More recently, two stages have been planned for the development of an expert system that will guide the customer on the use and determination of phase diagrams. The first stage concentrates on developing software that will enable individuals to learn or review how to interpret and use unary, binary, ternary and quaternary phase equilibria diagrams. The second stage will concentrate on the implementation of an application that will provide instructions how one develops phase diagrams. The goal of this latter stage is to capture, using digital motion video and audio, the relevant expertise of scientists whose research focuses on the determination of phase-equilibria diagrams. In this way, the expertise, particularly the laboratory techniques and insights which do not appear in technical publications, would be preserved for future generations and, furthermore, would be widely distributable.

The software will make extensive use of digital video technology which allows motion video and audio signals to be digitally compressed and integrated with tradition software. Motion video and audio allow the user to visualize technical content to an extent not possible with a textbook. This would be particularly helpful in presenting crystallization sequences in ternary systems.

Technical Objectives:

The primary goal of this project is to deliver critically evaluated phase equilibria data to industrial and academic customers. A second goal is the development of innovative, distributable, multimedia software incorporating interactive digital video technology to assist in the interpretation and use of these diagrams. This Equilibria Expert (EE) will complement phase diagram work that has been part of almost sixty years of cooperation between NIST and the American Ceramic Society.

Outputs:

Evaluated data:

Volume 11, focusing on oxides, particularly those of interest for electronic applications, was completed and published in 1995. This compilation contains 685 commentaries and 871 diagrams, which covers the period up to 1988. The 1995 Cumulative Index was also published which contains the author and chemical systems indexes for Volumes I-XI of Phase Equilibria Diagrams, Annuals '91-'93, and the High Tc Superconductor Monograph.

Version 2.0 of the CD-ROM Database for Phase Diagrams for Ceramists was completed, and is being marketed through the ACerS. This database contains over 10,000 diagrams, including those previously published in Version 1.0. The Database allows search capabilities of bibliographic information as well as powerful graphics capabilities with regards to the diagrams.

Volume 12, focusing on oxides, was worked on in 1995 and will have an early 1996 publication date. This volume has 443 commentaries and approximately 800 diagrams. Also planned for publication in early 1996 is a new cumulative index which will contain the author and chemical systems indexes for Volumes I-XII of Phase Equilibria Diagrams, Annuals '91-'93, and the High Tc Superconductor Monograph.

Technical papers:

"A Multimedia Tutorial on Phase Equilibria Diagrams," E.F. Begley and C.G. Lindsay, Bulletin of the American Ceramic Society, Vol. 72, No.12, pp. 103-104, December 1993.

"Multimedia Computing in Materials Science and Engineering," E.F. Begley and C.P. Sturrock, to be published in the Journal of Materials Engineering and Performance.

PROJECT TITLE: Structural Ceramics Database

PROGRAM TITLE: Evaluated Materials Data

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Technical Description:

This project is designed to facilitate advances in materials science and technology by providing evaluated thermal, mechanical, and corrosion property data for the broad class of materials variously called advanced technical ceramics, structural ceramics, engineered ceramics, or fine ceramics.

Technical Objectives:

The objective of this project is to provide a reliable knowledge base for understanding material property and performance relationships. Use of the technically evaluated data enables industry to save substantial time and effort in the selection of materials.

Outcomes:

Several significant results are expected from this project: (1) a comprehensive set of evaluated data; (2) a software interface to the database; (3) standards and methodology for data evaluation; and (4) relations and models for properties and performance characteristics.

Accomplishments:

The Structural Ceramics Database (SCD) is distributed by the NIST Standard Reference Data Program as NIST Standard Reference Database Number 30. Currently, version 2 is being distributed, and the preparations for version 3 are well underway.

An agreement has been established with the Russian Research Center for Standardization, Information, and Certification of Materials to obtain property data from Russian language sources, and an agreement to exchange collections of property data has been established with the advanced materials effort at Tsinghua University in China. These agreements will enable U.S. efforts to access important data from Russian and Chinese language research reports that would not otherwise be readily available.

Outputs:

Database:

NIST Standard Reference Database Number 30: Structural Ceramics Database, Version 2, 1991.

Technical Papers:

"Material Specifications of Advanced Ceramics in Corrosion Studies", R. G. Munro, ASTM Journal of Testing and Evaluation, Vol. 23 (5), 395-398 (1995).

"Material Specifications of Advanced Ceramics and Other Issues in the Use of Property Databases with Corrosion Analysis Models," R. G. Munro, in Computerized Chemical Data Standards: Databases, Data Interchange, and Information Systems - 2nd Volume, ASTM STP 1298, C. E. Gragg and J. Mockus, eds. (American Society for Testing and Materials, Philadelphia, 1995).

"The Role of Corrosion in a Material Selector Expert System for Advanced Structural Ceramics," R. G. Munro, in Computerization and Networking of Materials Databases: Fourth Volume, ASTM STP 1257, C. Sturrock and E. F. Begley, eds. (American Society of Testing and Materials, 1995).

Impact:

Structural ceramics hold the key to advanced designs for high efficiency, high temperature heat exchangers, corrosion resistant materials, and electronic packaging materials. The SCD provides critical property data, functions as a knowledge base for understanding property-performance relationships, and serves as a guide to the areas most in need of supporting data or measurement technology.

PROJECT TITLE: STEP Materials

PROGRAM TITLE: Evaluated Materials Data

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Technical Description:

This project develops and maintains the materials-related aspects of the ISO STEP standard for computerized exchange of information on manufactured products.

Technical Objectives:

The immediate, near-term objective is to achieve ISO balloting approval of STEP Part 45 Materials as an ISO Standard (IS). The longer terms objectives are to have STEP accepted worldwide as a standard for exchange of information on materials in manufactured products and to assess its viability for accessing distributed sources of material information.

Outcomes:

The nearest-term outcome should be the promotion of STEP Part 45 Materials to an IS status. This should occur in 1996. The next nearest-term outcomes should be the use of its simplest constructs and concepts in STEP application protocols (APs) being developed by others. STEP APs using its simplest constructs are underway in the areas of automotive design; electronic circuit design; composites design and analysis; sheet metal dies design and planning; castings, forgings, and molded products design and manufacture planning; and numerical control machining planning. In the longer term, a possible outcome may be the use of STEP for accessing distributed sources of materials information.

Accomplishments:

1. Development of STEP Part 45 Materials to balloting for approval as an IS.

2. Proper incorporation of materials technology aspects into other STEP parts and APs.

Outputs:

John Rumble, Jr. and Joseph Carpenter, Jr., "Materials 'STEP' into the Future," Advanced Materials and Processes, Vol. 142, No. 4, October 1992, pages 23-27.

R. Swindells and J. Carpenter, "Industrial Automation Systems and Integration - Product Data Representation and Exchange - Part 45: Integrated Resources: Materials," ISO/CD 10303-45 (International Organization for Standardization, Geneva, Switzerland, September 2, 1993), 21 pages.

R. Swindells and J. Carpenter, "Industrial Automation Systems and Integration - Product Data Representation and Exchange - Part 45: Integrated Resources: Materials," ISO/DIS 10303-45 (International Organization for Standardization, Geneva, Switzerland, August 10, 1995), 34 pages.

N. Swindells, "Materials Property Data in STEP," in proceedings of CODATA Conference, September 1994.

N. Swindells and J. Carpenter, "Reducing the Costs of Collaborative Engineering with STEP," The ActionLine (newsletter of the Automotive Industries Action Group), October, 1995.

Highlights:

The Draft International Standard (DIS) version of Part 45 addressing Materials in STEP was completed and submitted for ISO balloting to IS status.

INTELLIGENT PROCESSING OF MATERIALS

Intelligent processing of materials (IPM) is the integration of: (1) on-line sensors of materials conditions or features; (2) sensors and controllers of process conditions which determine final desired material properties; and, (3) models which relate process conditions to desired properties. IPM research in the Ceramics Division is focused on the measurement of characteristics of powders and slurries. Through the measurement of particular characteristics and the understanding of how those characteristics relate to final properties, improved process control can be achieved.

Projects in this area are closely coupled with industrial and academic partners' research in the development of processes, such as the application of research techniques to commercial practice. NIST research addresses both the development of the measurement technique and the understanding of the phenomena on which the technique is based.

Two projects are underway:

1. The measurement of dispersion homogeneity in injection molding compounds by the use of nuclear magnetic resonance (NMR) is conducted in cooperation with industry to determine the role of processing parameters and binder and dispersion chemistry; and,

2. Electroacoustic measurement to determine loading and size distribution of powders in dense slurries.

For further information on IPM of ceramic materials, contact S. G. Malghan at (301) 975-6101 or e-mail malghan@micf.nist.gov

PROJECT TITLE: Injection Molding of Ceramics

PROGRAM TITLE: Intelligent Processing of Materials

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Technical Description:

Injection molding is widely used in the ceramics industry to mass produce component parts of larger assemblies. The ability to produce parts of intricate geometry to fine levels of detail renders this process attractive for advanced engine parts. Typically injection molding utilizes an organic binder to hold green parts together, and recently aqueous based binders have received attention due to reduced environmental concerns. Significant issues which require resolution to make this process suitable for production of reliable structural components include: understanding the relationship of uniformity of binder distribution to cracking during shrinkage, role of binder chemistry on its distribution and rheological properties, and mechanistic understanding of the powder-binder interactions.

Technical Objectives:

The objective of this project is to develop methods for the measurement and understanding of the chemical and physical parameters which effect powder and binder interactions and homogeneity as well as methods for the characterization of homogeneity.

Outcomes:

This research is expected to provide basic understanding of the role chemistry on the homogeneity of injection molding ceramic blends. This understanding will result in process changes which improve properties and process yield.

Accomplishments:

Techniques have been developed to characterize the distribution of binder in injection molded ceramic parts by NMR imaging. NMR spectroscopic techniques have been developed to identify the changes in molecular structure of organic binders as a result of aging. Methods to characterize powders during aqueous injection molding were developed. The interactions between powder and dispersant were delineated.

PROJECT TITLE: Analysis of Ceramic Powder

PROGRAM TITLE: Intelligent Processing of Materials

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Technical Description:

This project is designed to characterize Si-powder behavior in water slurries, under various processing parameters, by NMR spectroscopy and imaging.

Technical Objectives:

The goal of this project is to help US ceramic industry to define a set of favorable processing parameters in producing silicon nitride parts through reaction bonding directly from Si powders.

Outcomes:

Information related to the following areas will be obtained as a result of this study: (1) chemistry, impurity, and molecular structure of dispersants Darvan C and Betz 1190; (2) effects of these dispersants on silicon powder behavior in water slurry, 3-D MRI pictures and 2-D profiles of Si-powder distribution in water slurries at various concentrations and pH values.

Accomplishments:

Through interactions with consortium members, NMR imaging studies were performed for four types of slurries in which powders and dispersants use varies. These studies showed the impact of dispersant chemistry on processing of silicon and silicon nitride as well as provided data on the chemistry of dispersants of Betz 1190 was thus performed extensively by ^{1}H, ^{14}N, ^{15}N, ^{35}Cl, and ^{13}C NMR at 400.140 MHz, 28.894 MHz, 50.674 MHz (from a higher frequency spectrometer in University of Maryland), 39.193 MHz, and 100.577 MHz, respectively. The solutions analyzed are 30% Betz 1190 in water. The results show that Betz 1190 is actually only 74.1 % pure, in form of 2-hydroxy-3,3-ionene chloride. The other 25.9 % are either NH_4-containing or a larger polymer which has a different relaxation time (T_1). The ^{15}N spectrum shows that a pure component is indeed in an equal quantity of meso- and racemic- forms. ^{35}Cl NMR suggests a concentration dependence in chemical shift and dissociation constant. ^{1}H T_1 in solutions of pH 6 to 9 were also found to be constant.

Outputs:

Technical papers:

"NMR Characterization of Silicon Nitride: Slurry Homogeneity by T_2-Weighted Proton Imaging", Pu Sen Wang, submitted to Journal of Materials Research (1995).

"The Nitridation of a Silicon Powder Studied by XPS and Bremsstrahlung-Excited AES", Pu sen Wang and Thomas N. Wittberg, submitted to Surface and Interface Analysis (1995).

Impact:

NIST results led some of the Powder Processing Consortium members to reconsider the dispersants currently used. Decisions to change dispersants will depend on our future results which will be higher in powder concentrations.

PROJECT TITLE: Electroacoustic Characterization of Ceramic Powder Slurries

PROGRAM TITLE: Intelligent Processing of Materials

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Technical Description:

This project is focussed on the application of non-destructive electroacoustic techniques for characterizing particle size and charge in aqueous ceramic powder slurries, the development of data, measurement science and computational tools to support these applications, and the optimization of advanced ceramic processing by improvement of interfacial chemical control of slurry properties.

Technical Objectives:

The objective of this research is to develop measurement methods and an understanding of the surface density of ceramic powders of relevance to slury processing control.

Outcomes:

Two primary outcomes are expected to derive from this research: (1) development of electroacoustic analysis for process control in powder processing, (2) improved understanding of component interactions during aqueous processing of complex highly concentrated slurries.

Accomplishments:

Extensive testing and evaluation of a novel electroacoustic technique has demonstrated the capabilities of this method for characterizing ceramic powder systems. Improvements were derived from cooperative interaction with the instrument manufacturer.

Polyelectrolyte dispersants, commonly used additives in aqueous processing of powders, were studied and evaluated using electroacoustic methods in the model Si_3N_4/water system. Measurements in moderately concentrated slurries provided agglomerate size and zeta potential as a function of polymer concentration, molecular weight and suspension pH. This data will be used to optimize conditions for improved dispersion of Si_3N_4 powders.

Electroacoustic measurements, along with several other surface chemical techniques, were used to investigate the aqueous reaction-bonded silicon nitride system. This processing route begins with metallic silicon dispersed in aqueous suspension in a complex mixture with sintering aids, nitriding agent, dispersants and binders. The aging of unstable silicon in water was characterized under various conditions using electroacoustic measurements of the changing surface potential as a function of time and pH. The interaction of dispersants and binders with the primary silicon phase was examined extensively, leading to improvement of dispersion via chemical control of suspension conditions. This research is on-going.

A consortium was formed in September 1993 between NIST, five manufacturers that produce ceramic powders or components, an instrument company and a federal laboratory, in a joint effort to improve processing of ceramic powders. Participants in this Consortium on Intelligent Processing of Powders and Slurries include Cercom Inc. (Vista, CA), Coors Ceramics Co. (Golden, CO), Eaton Corp. (Southfield, Mich), Kerr-McGee Corp. (Oklahoma City, OK), St. Gobain/Norton Industrial Ceramics Co. (Northboro, MA), and instrument manufacturer, Matec Applied Sciences Inc. (Hopkinton, MA). The Metals and Ceramics Division of the U.S. Department of Energy's Oak Ridge National Laboratory is a participating partner.

Outputs:

"Electroacoustic Characterization of Particle Size and Zeta Potential in Moderately Concentrated Suspensions", Hackley, V.A. and Malghan, S.G., in *Ceramic Transactions*, Volume 56, Advanced Synthesis and Processing of Composites and Advanced Ceramics (American Ceramic Society, Westerville, OH, 1995) p 283.

"Techniques for Characterization of Advanced Ceramic Powders", Malghan, S.G., Wang, P.S. and Hackley, V.A., in *Chemical Processing of Ceramics*, Lee, B.I. and Pope, J.A., Eds., (Marcel Dekker, NY, 1994).

"Surface Chemical Interactions of Si_3N_4 with Polyelectrolyte Deflocculants", Hackley, V.A., Premachandran, R. and Malghan, S.G., *Key Engineering Materials*, **89-91**, 679 (1994).

"Intelligent Processing of Ceramic Powders and Slurries", Malghan, S.G., Hackley, V.A. and Wang, P.S., in *Ceramic Eng. and Sci. Proc.* (American Ceramic Society, Westerville, OH, 1994) p 527.

"The Surface Chemistry of Silicon Nitride Powder in the Presence of Dissolved Ions", Hackley, V.A. and Malghan, S.G., *J. Mat. Sci., 29, 4420* (1994).

Research results were also presented at two scientific meetings, the First International Symposium on Advanced Synthesis and Processing of Composites and Advanced Ceramics in Cocoa Beach, Florida, and the 1995 American Ceramic Society Annual Meeting in Cincinnati, Ohio.

Impact:

Research performed at NIST and reported to the consortium participants under the auspices of a Cooperative Research and Development Agreement has provided new and improved information regarding the aqueous suspension behavior of metallic silicon powders. This will have a direct impact on processing formulations for reaction-bonded silicon nitride. NIST testing and evaluation has provided feedback to the instrument manufacturer which resulted in system improvements. NIST measurements have provided necessary information regarding the application of electroacoustic methods to characterizing ceramic powders which is currently being employed for the analysis of materials used by consortium participants. The impact of NIST research in this area is demonstrated by the formation of new collaborations with industry which are currently in the development stage.

PROJECT TITLE: Environmental Stability of Infrastructural Cements

PROGRAM TITLE: Intelligent Processing of Materials

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Technical Description:

This project is designed to characterize the microstructural evolution during the hydration of cements as it affects highway infrastructural concretes, to characterize their microstructural degradation due to environmental effects, and to probe optimal additions of silica fume and other additives that would give superior performance.

Technical Objectives:

The objective is to assist in the production of advanced cementitious materials leading to highway infrastructural concretes of improved durability.

Outcomes:

Three major outcomes are expected to occur: (1) real-time, in-situ, non-destructive classification of the microstructural evolution during hydration of highway-infrastructure cements containing different pozzolanic additives; (2) assessment of aggregates, cement additives and hydration conditions for stability of next-generation infrastructural concretes; (3) improved optimization of cement and concrete formulations for the highway infrastructure. A future development will include experiments aimed at gaining a more fundamental understanding of the processes underlying the dormant induction period, well-known in the early stages of cement hydration, and the control of which is believed to be a key issue in the development of tailor-made high-technology concretes.

Accomplishments:

The effects, on the microstructural development during cement hydration, of adding pozzolanic additives to cements and concretes, in the form of silica fume, have been extensively studied using small-angle neutron scattering (SANS) and ultra-small-angle x-ray scattering (USAXS). A semi-fractal microstructural model, developed previously, has been modified to extract statistically-significant microstructural parameters from the SANS data. Different silica fume additives been characterized and compared, and the investigation has been extended to natural additives which are sometimes contained within aggregate minerals incorporated into concretes. These natural additives are known to engender natural pozzolanic reactions or alkali-silicate reactions that degrade performance.

Outputs:

R. A. Livingston, D. A. Neumann, A. J. Allen and J. J. Rush; "Application of Neutron Scattering Methods to Cementitious Materials", in Neutron Scattering in Materials Science II'. Ed. D. A. Neumann, T. P. Russell and B. J. Wuensch. MRS Symp. Proc. 376 (Materials Research Society, Pittsburgh, PA., 1995) 459-469.

A. J. Allen and R. A. Livingston; "Small-Angle Scattering Study of Concrete Microstructure as a Function of Silica Fume, Fly Ash or Other Pozzolanic Additions", (SP 153-62) in Proc. 5th CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete, June 1995. Ed. V. M. Malhotra. American Concrete Institute, Detroit, MI. 1179-1200 (1995).

R. A. Livingston and A. J. Allen; "Application of Small-Angle Neutron Scattering Method to the Study of the Durability of Historic Brick and Mortar", in Ceramics in Architecture', Proc. 8th CIMTEC World Ceramics Congress and Forum on New Materials. Ed. P. Vincenzini. CIMTEC, Faenza, Italy. Vol. A ???-??? (1995).

Impact:

The studies are enabling a quantitative assessment of the microstructural modifications underlying the variable effectiveness of different silica fume additives in forming cements and concretes of improved strength and durability.

MECHANICAL PROPERTIES OF BRITTLE MATERIALS

Mechanical properties are the source of some of the greatest benefits as well as the most serious limitations of ceramics. With their great strength-to-weight ratio, their relatively inert behavior in aggressive environments, and their ability to withstand much higher temperatures than metals or polymers, ceramics potentially offer major improvements for component design in a wide range of applications. On the debit side, brittle fracture, creep at elevated temperatures, environmentally enhanced failure, and the lack of techniques which can detect and quantify critical flaws before failure occurs, impede their more wide-spread uses. Problems in controlling and controlling failure of ceramics stems from three sources: lack of understanding of the failure processes, limited standard test techniques to permit inter-laboratory comparisons of material behavior, and inadequate statistical techniques for reliability and lifetime estimation. The Mechanical Properties program has components specifically addressing each of these issues.

Basic understanding of mechanical behavior of ceramics is being investigated for both ambient and elevated temperature applications. Effects of cyclic loading and microcrack formation/coalescence are being studied for lead zirconium titanate (PZT), a technologically important actuator material. Mechanical properties and failure processes in fiber-reinforced ceramic matrix composites are being investigated as a function of both temperature and fiber loading. Residual in stresses associated with crack bridging in both fiber-reinforced composites and microstructurally toughened alumina are being measured via micro-Raman techniques. In addition, environmentally enhanced fracture remains an important subject from both an experimental and a theoretical (see Theory and Modeling section) viewpoint. At elevated temperatures, the basic mechanisms responsible for creep in silicon nitride are being investigated.

An important aspect of the program is the development of standards. New standard test techniques for hardness and toughness are being developed and tested in round-robin experiments; these will eventually lead to ASTM and ISO standards. For elevated temperatures, a new creep specimen design has been developed which concentrates more of the applied load in the gage length and reduces the chance of failure from non-gage portions of the specimen.

Finally, techniques to predict lifetimes of ceramics under constant or cyclic loading conditions are being developed. The nonparametric bootstrap approach to lifetime prediction is being applied to a number of materials. A parametric model to predict lifetimes of PZT under cyclic loading is also under development. This latter model does not restrict the material to environmentally enhanced fracture behavior since experimental evidence has demonstrated that other, albeit unknown, processes are also active during cyclic loading.

For further information about the Mechanical Properties of Brittle Materials Program, please contact Edwin Fuller at 301-975-5795 or e-mail requests to erfuller@enh.nist.gov.

PROJECT TITLE: Creep of Ceramics

PROGRAM TITLE: Mechanical Properties of Brittle Materials

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Technical Description:

This project is designed for three purposes: (1) to develop standard testing methodologies for ceramics at elevated temperature, particularly for a tensile mode of loading; (2) to develop an engineering data base on high-temperature strength, creep, creep rupture properties, and crack growth behavior for advanced, structural ceramic materials; and (3) to investigate and elucidate effects of material microstructure on elevated temperature mechanical behavior.

Technical Objectives:

This research is designed to assist industry in the evaluation, design, and development of advanced, structural ceramics as high-temperature components in land-based heat engines for power generation or for vehicle applications, and in development of measurement methodologies for the evaluation of high-temperature mechanical properties.

Outcomes:

Major outcomes of this research are expected to include: (1) development of failure mechanism maps of relevant materials for the evaluation and design of ceramic engine components; (2) development of standardized tensile testing methodologies for material comparison; and (3) elucidation of the relationship between material's microstructure and mechanical behavior to guide material development and reliability assessment.

Accomplishments:

NIST and General Electric Corporation signed a CRADA in September, 1994 to investigate the creep, creep rupture, and strength properties of two commercial grades of *in-situ* toughened silicon nitride. The project represents one of the widest ranging attempts to gather data on single grades of engineering ceramic. We have combined room-temperature strength tests, intermediate-temperature (800 °C to 1000 °C) stress rupture tests, and high-temperature (1000 °C to 1400 °C) creep-rupture tests to produce failure mechanism maps for these materials. Although we have discontinued tests on one of the materials, due to poor specimen to specimen repeatability, the other material exhibits excellent and highly repeatable properties. Over one hundred samples of this material have been tested and selected creep samples are currently being examined by analytical transmission electron microscopy. Initial results indicate that the improved creep rupture behavior may be related to the atypical absence of cavitation within the narrow interfaces separating adjacent grains.

Through work on several other grades of silicon nitride, we have developed a new model for tensile creep deformation of silicon nitride. The model recognizes the fact that the silicon nitride grains themselves are undeformed by the creep process. Rather, redistribution of the silicate binding phase from cavitating multi-grain junctions to uncavitated pockets produces the observed extension. Sliding of the silicon nitride accommodates the redistribution. The model readily explains the hundred to thousand-fold increase in creep resistance in compression over tension. In compression, the applied stress opposes the volume increase of the interstitial silicate volume. When this mechanism is suppressed, the silicon nitride deforms by ordinary solution-reprecipitation creep.

We have used finite element techniques to develop two new tensile specimen geometries to add to a third already in use. Each represents a significant improvement over previous designs, allowing us to conduct tests at much higher stresses than previously possible. One specimen is specifically designed to be amenable to fabrication from existing engineering components. This test ability will make it possible to verify the engineering properties of the components themselves. We have experimentally verified that the three geometries yield identical creep properties.

We have selected one of the newly designed specimens for an inter-laboratory round robin for creep testing.

Outputs:

Technical papers:

William E. Luecke, Sheldon M. Wiederhorn, Bernard J. Hockey, Gabrielle G. Long, and Ralph F. Krause Jr., "Cavitation Contributes Substantially to Creep in Silicon Nitride," J. Am. Ceram. Soc, 78, 2085-2096 (1995).

S. M. Wiederhorn, W. E. Luecke, B. J. Hockey, and G. G. Long, "Creep-Damage Mechanisms in Si_3N_4," pp. 305-326 in Tailoring of High Temperature Properties of Si_3N_4 Ceramics, M. J. Hoffman and G. Petzow, eds. Kluwer Academic Publishers, Dordrecht, The Netherlands, 1994.

S. M. Wiederhorn, J. D. French, and W. E. Luecke, "High-Temperature Reliability of Structural Ceramics," pp. 145-155 in 5th International Symposium on Ceramic Materials and Components for Engines, D. S. Yan, X. R. Fu and S. X. Shi, eds. World Scientific Publishing Co., Singapore (1995).

S. M. Wiederhorn and W. E. Luecke "The Importance of cavitation to the creep of structural ceramics," to be published in Proceedings of the Symposium on Plastic Deformation of Ceramics, Snowbird Utah, August 7-12, 1994. Sponsored by The Engineering Foundation, 1995.

S. M. Wiederhorn, J. D. French, and W. E. Luecke, "A Comparison of Fracture Mechanism Maps with the Larson-Miller Method of Predicting Lifetime," Ceram. Eng. Sci. Proc, in press (1995).

J. D. French and S. M. Wiederhorn, "Tensile specimens from Ceramic Components," J. Am. Ceram. Soc, 1995, in press.

Technical papers submitted for publication:

William Luecke and J. D. French, "Strain measurement uncertainty during flag-based extensometry," submitted to J. Am. Ceram. Soc, 1995.

B. A. Fields and S. M. Wiederhorn, "Creep and Cavitation in a Siliconized Silicon Carbide," submitted to J. Am. Ceram. Soc. 1995.

Impact:

The creep and rupture data gathered in this project demonstrate that silicon nitrides can have the structural properties necessary for use in high-temperature heat engines.

PROJECT TITLE: Mechanical Test Development

PROGRAM TITLE: Mechanical Properties of Brittle Materials

Principal investigator(s):

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Technical Description:

This project is designed to created, developed, improved, refined, and standardized mechanical testing methods for brittle ceramic materials.

Technical Objectives:

This research is designed to assist industry and in-house programs by developing procedures for characterizing ceramic materials and for generating lifetime and reliability data. Standard test methods are prepared for ASTM and ISO. Our goal is to develop procedures that are as technically rigorous as possible while remaining practical and usable by industry.

Outcomes:

Guidelines and test procedures are created and/or refined so that accurate and precise results for mechanical property data can be obtained by NIST, industry, government labs, and academia. Wherever possible, standard practices or test methods are prepared in the appropriate forum. Emphasis is on preparing ASTM and ISO standards. This project also provides guidance and prestandarization work relevant to Standard Reference Material projects. Round robin projects are conducted as necessary.

Accomplishments:

Four ASTM standards have been adopted by Committee C-28 since 1991 in large part due to work done in this project:

1. ASTM C 1198-91 *Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance*,

2. ASTM C 1161-90 *Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature*,

3. ASTM C 1211-92 *Standard Test Method for Flexural Strength of Advanced Ceramic at Elevated Temperature*,

4. ASTM C 1239-94a *Standard Practice for Reporting Strength Data and Estimating Weibull Distribution Parameters.*

In addition, work on this task has contributed to the following standards which are now being balloted in ASTM:

6. ASTM C-xxxx *Standard Test Methods for the Determination of Fracture Toughness of Advanced Ceramics*,

7. ASTM C-xxxx *Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics*,

8. ASTM C-xxxx *Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics*,

9. ASTM C-xxxx *Standard Practice for Fractography and Characterization of Fracture Origins in Advanced Ceramics.*

Outputs:

Outputs include standard test methods and practices as listed above. Round robin projects and reports not only measure the robustness of the methods and furnish information for precision and bias statements, but also serve to spread knowledge about the methods. Technical papers and reports support the standards.

Technical papers published:

C. Tracy and G. Quinn, "Fracture Toughness by the Surface Crack in Flexure (SCF) Method," Ceram. Eng. and Sci. Proc., Vol. **15**, [5], 1994, pp. 837-845.

G. Quinn, R. Gettings, and J. Kübler, "Fracture Toughness of by the Surface Crack in Flexure (SCF) Method: Results of the VAMAS Round Robin," Ceram. Eng. and Sci. Proc., Vol. **15**, [5] 1994, pp. 846-855.

J. Swab and G. Quinn, "Results of a Round Robin Exercise on the Fractography of Advanced Structural Ceramics," Ceram. Eng. and Sci. Proc., Vol. **15**, [5], 1994, pp. 867-876.

G. Quinn, J. Kübler, and R. Gettings, "Fracture Toughness of Advanced Ceramics by the Surface Crack in Flexure (SCF) Method: A VAMAS Round Robin," VAMAS Technical Report **#17**, NIST, June, 1994.

G. D. Quinn and C. A. Tracy, "Fracture Toughness of Advanced Ceramics as Measured by the Surface Crack in Flexure Method," in **Ceramics: Charting the Future**, ed. P. Vincenzini, Proceedings of the 8th World Ceramic Congress, CIMTEC, Techna, Faenza, 1995.

J. J. Swab and G. D. Quinn, "Fractography and Characterization of Fracture Origins in Advanced Ceramics, A VAMAS Round Robin," pp. 569-576 in **New Horizons for Materials**, Proceedings of the 8th World Ceramic Congress, CIMTEC, Florence, Italy, ed P. Vincenzini, Techna, Faenza, 1995.

G. D. Quinn, J. J. Kübler, and R. J. Gettings, "Fracture Toughness of Advanced Ceramics: A New VAMAS Round Robin," pp. 577-584 in **New Horizons for Materials**, Proceedings of the 8th World Ceramic Congress, CIMTEC, Florence, Italy, ed P. Vincenzini, Techna, Faenza, 1995.

J. J. Swab and G. D. Quinn, "Fractography of Advanced Structural Ceramics: Results From Topic #2 of the VAMAS Round Robin Exercise," in Proceedings of Jan. 1995 Meeting ACS, Cocoa Beach, Ceram. Eng. and Sci. Proc., 1995, in press.
R. J. Gettings and G. D. Quinn, "Surface Crack in Flexure (SCF) Measurements of the Fracture Toughness of Advanced Ceramics," in Proceedings of Jan. 1995 Meeting ACS, Cocoa Beach, Ceram. Eng. and Sci. Proc., 1995, in press.

J. J. Swab and G. D. Quinn, "Fractography of Advanced Structural Ceramics, Results from the VAMAS Round Robin Exercise," VAMAS Report **#19**, June, 1995. (Also published as U.S. Army Technical Report, ARL-TR-**656**, Dec., 1994.)

C. R. Brinkman, G. D. Quinn, and R. W. McClung, "Overview of ASTM Standard Activities in Support of Advanced Structural Ceramics Development," Ceram. Eng. and Sci. Proc., 1995, in press.

G. D. Quinn, R. J. Gettings, and J. J. Kübler, "Fracture Toughness of Ceramics by the Surface Crack in Flexure (SCF) Method," in 6th International Symposium on Fracture Mechanics of Ceramics, Karlsruhe, July, 1995, in press.

G. D. Quinn, R. J. Gettings, and J. J. Kübler, "Fractography and the Surface Crack in Flexure (SCF) Method for Evaluating Fracture Toughness of Ceramics," in Proceedings of the Third Alfred Conference on Fractography of Glasses and Ceramics," July, 1995, in press.

J. J. Swab and G. D. Quinn, "The VAMAS Fractography Round Robin: A Piece of the Fractography Puzzle," in Proceedings of the Third Alfred Conference on Fractography of Glasses and Ceramics," July, 1995, in press.

Impact:

Refined and standardized test methods are now available. The ceramics community now has procedures that generate data of high quality and data which is readily comparable between laboratories. This has benefitted both the industrial and research communities. This was not the case 10 years ago when data was of mixed quality and could not be readily compared between laboratories.

Data suitable for design is now easily obtained and data bases are being developed on the basis of the standards.

Tangible benefits also include significant cost savings. For example, the adoption of standard specimen sizes and surface preparation treatments has driven the cost of flexure specimens (the *bread and butter* strength test for ceramics) from $15-20 per specimen to $8 per specimen, a 50% savings.

As another example, fractography of ceramics has heretofore been a highly interpretive *art*. Work in this project has standardized some of the more routine aspects of fractographic analysis. Consistent results and interpretations are more readily obtained and can be related better to mechanical property results. The standard practice for fractographic analysis is now used as a teaching aid in courses at several universities and by a course offered by The American Ceramic Society.

PROJECT TITLE: Fracture Behavior of Ceramic Matrix Composites

PROGRAM TITLE: Mechanical Properties of Brittle Materials

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Technical Description:

This project is designed to elucidate failure modes, damage generation, and damage accumulation in fiber-reinforced ceramic matrix composites. Fracture in the composite matrix, individual fibers, fiber bundles, and at the fiber/matrix interface is studied as a function of processing methodology and microstructure.

Technical Objectives:

This research project is designed to assist industry by assessing the applicability of fracture mechanics in the description of failure modes and reliability of fiber-reinforced ceramic matrix composites. Goals are to relate damage and fracture behavior to microstructure, and to develop a testing methodology to quantify damage.

Outcomes:

The major outcomes expected from this research are: (1) development of testing methodologies to quantify damage in fiber-reinforced ceramic matrix composites; and (2) reliable damage and failure property data for design and manufacture of fiber-reinforced ceramic matrix composites.

Accomplishments:

Strength, fracture toughness, and the failure mode in SCS-6 SiC-fiber reinforced Si_3N_4 matrix composites were investigated as a function of fiber volume fraction, controlled flaw size (indentation load) and temperature. Matrix-cracking stress and ultimate strength of the composites were found to decrease with increasing temperature, but their temperature sensitivity decreased with increased fiber volume fraction. The tendency for non-catastrophic failure increased with fiber volume fraction, while the tendency for catastrophic failure increased with temperature. The failure mode was demonstrated to be determined by the fiber bundle strength and the matrix cracking stress. Catastrophic failure at high-temperatures was found to be mainly a result of fiber strength loss.

A constrained shear test was used to investigate damage in SiC/SiC fiber-reinforced composites. A bonded interface technique has allowed for direct observation of shear-induced distributed damage in these materials. Load-displacement curves are directly related to the amount of generated damage.

Outputs:

Several research papers were produced from the technical advancements, which were made.

Technical papers published:

H. H. Xu, C. P. Ostertag, E. R. Fuller, Jr., L. M. Braun, and I. K. Lloyd, "Fracture Resistance of SiC-Fiber-Reinforced Si_3N_4 Composites at Ambient and Elevated Temperatures," J. Am. Ceram. Soc., **78** [3], 698-704 (1995).

H. H. Xu, L. M. Braun, C. P. Ostertag, R. F. Krause, Jr., and I. K. Lloyd, "Failure Modes of SiC-Fiber/Si_3N_4-Matrix Composites at Elevated Temperatures," J. Am. Ceram. Soc., **78** [2], 388-94 (1995).

Impact:

NIST measurements will provide reliable damage and failure property data for the design and manufacture of fiber-reinforced ceramic matrix composites. This data will be used to improve reliability assessment models for fiber-reinforced ceramic matrix composite.

PROJECT TITLE: Lifetime Prediction Methodology

PROGRAM TITLE: Mechanical Properties of Brittle Materials

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Technical Description:

This integrated experimental and analytical research project is designed to provide confirmed methodologies for service life estimation on a variety of engineering components used by industry for performing critical functions so that interruption can be avoided.

Technical Objectives:

The objectives of this project are to elucidate and to characterize time-dependent failure mechanisms in ceramic systems. This includes development of damage evolution laws and understanding of kinetics and driving forces leading to damage accumulation, including the role of environment, temperature, stress, and other physical properties.

Outcomes:

Outcomes include the adoption of reliable lifetime prediction methodologies by industry (e.g., aerospace, electronic, energy, etc.) leading to improved reliability, savings in maintenance, and enhanced safety.

Accomplishments:

Predictions of long-term reliability of residually stressed InP have been made. A non-parametric bootstrap method has provided new insight into uncertainties associated with lifetime predictions for materials with minimal environmentally enhanced crack growth. A new form of controlled crack growth has been observed in InP; presently, its implications for reliability predictions are not understood.

Output:

Transfer of the non-parametric bootstrap method to AT&T has been achieved. In addition, two technical manuscripts have been written:

Technical papers submitted for publication:

G. S. White and L. M. Braun, "Environmentally Enhanced Fracture Behavior of InP," J. Am. Ceram. Soc., submitted.

E. R. Fuller, Jr., G. S. White, L. M. Braun, and W. C. Carter, "Lifetime Predictions and Confidence Levels Using a Nonparametric Bootstrap Technique," J. Am. Ceram. Soc., to be submitted.

Impact:

AT&T has used the results of the nonparametric bootstrap method to evaluate and modify qualification procedures for InP optical couplings.

PROJECT TITLE: Micro-Raman Stress Measurements

PROGRAM TITLE: Mechanical Properties of Brittle Materials

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Technical Description:

This project is designed to develop micro-Raman techniques for characterizing macroscopic and microstructural stresses in select ceramic systems. Stresses are determined by shifts of peak positions in the Raman spectra. Lateral resolution of a few micrometers is obtained.

Technical Objectives:

This research project is designed to assist industry in the measurement of localized residual stresses which can control mechanical, electrical and optical properties over a wide range of industrial applications.

Outcomes:

Three major outcomes are anticipated for this work. The first is the development of standardized calibration procedures allowing the technique to provide quantitative stress values for a wide variety of material systems. The second is the adoption of stress measurement procedures by electronic packaging industry. The third is the securing of accurate stress data leading to improved models for material system design and reliability assessment.

Accomplishments:

Measurements have been made in a number of ceramic materials systems. In alumina, changes in stress associated with crack-bridging grains and stress variations within individual grains have been detected. Preliminary work has begun in which stress measurements in alumina grains have been correlated with microstructural simulations. Stress changes in metal-organic films have been correlated with changes in stress state in sapphire substrates. Changes in stress state have been observed in siliconized-SiC composites after high-temperature tensile creep deformation.

Most importantly, applied stress versus peak shift curves of single crystal sapphire specimens with known orientation have demonstrated that calibration curves are orientation dependent. Therefore, in general, a crystallite orientation must be known before its stress state can be accurately determined.

PROJECT TITLE: Micro-Tribology

PROGRAM TITLE: Mechanical Properties of Brittle Materials

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Technical Description:

This project is to provide measurement techniques and standards of surface properties (mechanical and tribological) of advanced materials and systems from nano-meter scale to macro-scale. The initial focus is on magnetic hard disk information storage industry.

The National Storage Industry Consortium (NSIC) consists of 34 companies, 25 universities and government laboratories. Its aim is to foster pre-competitive generic research in information technologies. We work closely with the Tribology research group of NSIC to explore surface chemistries, textural designs, and to develop test techniques for measuring the effects of mono-molecular films. During last year, we have set up a state-of-the-art laboratory facility to measure friction, stiction, and wear of magnetic hard disks. Several new techniques were developed to measure the effect of mono-molecular film on superflat disks.

Experiments were done on a model system as well as on the actual disks. Stearic acid monolayer and submonolayer were deposited on a copper surface and examined using Fourier Transform Infrared Spectroscopy, Time-resolved Raman Spectroscopy, and Ultra-soft X-ray Absorption Spectroscopy. The combination of these techniques allows the determination of the molecular orientation, inclined angle, and the nature of the bonding of the stearic acid with the surface. These molecular parameters were correlated with the friction and wear performance of the model system to elucidate surface protection mechanisms.

Various organic monolayers and mono-molecular films were dipped coated on the NSIC CH_x coated surfaces as well as the CN_x coated surfaces. The coated disks were evaluated for friction and durability studies both at NSIC and NIST. Some films were found to perform much better than the current commercial practice.

Measurement techniques were also developed to measure thin film properties using Atomic Force Microscope, nano-indentor, nano-scratch tester, and ball-on-inclined plane apparatus.

Technical objectives:

This research project is designed to assist industry in providing critical measurement tools necessary for the development of new generation of data storage technology of 62 $Mbit/MM_2$ (40 $Gbit/in^2$).

Outcomes:

Two anticipated outcomes are expected to occur: (1) development of laboratory scale test procedures to evaluate new materials, coatings, and lubricant chemistry of the magnetic hard disk system, (2) understanding of how mono-molecular films interact and protect the magnetic disks, (3) determine the relationship between thin film structures and their mechanical properties.

Outputs:

"A Spectroscopic Study of a Copper Surface Under Boundary Lubrication: Chemisorbed Stearic Acid Multilayers," Z. H. Hu, P. S. Wang, S. M. Hsu, J. of Lubrication Science, 7-4, 293-307, 1995.

"Asperity-Asperity Friction as Measured by a Two-Ball Collision Apparatus," T. N. Ying and S. M. Hsu, Tribology Trans, 39, 1, 33-42, 1996.

"Tribochemistry and Lubrication," S. M. Hsu and R. S. Gates, Invited paper in the Proceedings of the Tribochemistry Forum in International Tribology Conference, Yokohama, Japan, Japanese Society of Tribologists, 1995.

"Molecular Orientation and Bonding of Monolayer Stearic Acid on a Copper Surface in Air," D. A. Fischer, Z. Hu, and S. M. Hsu, submitted to Tribology Letters.

"Tribochemical and Thermochemical Reactions of Stearic Acid on Copper Srufaces in Air as Measured by Ultra-Soft X-ray Absorption Spectroscopy," D. A. Fischer, Z. Hu, and S. M. Hsu, submitted to Tribology Letters.

NANOSTRUCTURED MATERIALS

Materials with microstructures, e.g., grain sizes, on the order of nanometers have been cited as having potentially improved strength and, for ceramics, superplastic behavior which could be beneficial as a low-cost forming technique. Difficulties have been recognized in consolidating nano-sized ceramic powders to full density. This difficulty has limited researchers ability to fully evaluate these materials. A high-risk project to consolidate nanostructured materials without sintering aids has been undertaken in cooperation with Sandia National Laboratory and Rutgers University. The approach taken at NIST utilizes high pressure compaction with liquified gas as a powder lubricant.

For further information on nanomaterial consolidation, please contact S. G. Malghan at (301) 975-6101 or e-mail to malghan@micf.nist.gov

PROJECT TITLE: Low Temperature Fabrication of Transparent Silicon Nitride

PROGRAM TITLE: Nanstructured Materials

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Technical Description:

This project is designed to characterize and evaluate the effects of high pressure compaction on the consolidation and densification of nanosize powders, including silicon nitride and aluminum oxide.

Technical Objectives:

This project is designed to assist industry indirectly in the development of processing technology for the production of advanced nanostructured materials possessing mechanical and functional properties superior to those commonly found in ceramic materials.

Outcomes:

The major outcome expected is the development of a high pressure compaction fabrication process for making fully dense nanostructured ceramic materials. Another is the process for fabricating ceramic materials with controlled porosity for a wide range of nanoscale filtration applications. The technology may also permit the fabrication of transparent ceramics by virtue of their nanoscale microstructures.

Accomplishments:

In this work, we have demonstrated the concept that simple molecular liquids can enhance densification in the compaction process by providing a lubricating medium for the nanosize particles. For example, green density of Si_3N_4 compacts fabricated by high pressure compaction of nanosize amorphous powder using liquid N_2 can be enhanced by as much as 10% over dry powder compaction permitting attainment of nearly full random packing density of the green body. However, on sintering, full densification without grain growth was not achieved. Furthermore, crystallization of the amorphous Si_3N_4 powder also occurred complicating the microstructural development during the sintering process. Similar studies on γ phase nanosize alumina powder showed that increased compaction pressure results in a corresponding increase in green body density, specifically from 54% theoretical at 1 GPa to 65% theoretical at 2.5 GPa. From this work two conclusions can be drawn: (1) Increased compaction pressure enhances the γ-to-α phase transition, effectively increasing the nucleation rate or alternatively lowering the transition temperature to the 1000 °C range, and (2) increased compaction pressure leads to increased densification upon heat treatment.

Outputs:

Several research papers were produced as a result of the technical advancements made here:

W. Chen, A. Pechenik, S. J. Dapkunas, G. J. Piermarini and S. G. Malghan, "Novel Equipment for the Study of the Compaction of Fine Powders", J. Am. Ceram. Soc., 77 [4] 1005-1010 (1994).

Eduardo J. Gonzalez, Gasper Piermarini, Bernard Hockey and Subhas G. Malghan "Low Temperature Fabrication From Nanosize Ceramic Powders", Final Report to DOE, ORNL/Sub/92-22041/02, April 28, 1995

M. R. Gallas and G. J. Piermarini, "The Bulk Modulus and Young's Modulus of Nanocrystalline γ-Alumina," J. Am. Ceram. Soc., 77 [11] 2917-20 (1994).

M. R. Gallas, B. Hockey, A. Pechenick, and G. J. Piermarini, "Fabrication of Transparent γ-Al_2O_3 from Nanosize Particles", J. Am. Ceram. Soc., 77 [8] 2107-12 (1994).

A. Pechenik, G. J. Piermarini, and S. C. Danforth, "Low Temperature Densification of Silicon Nitride Nanoglasses," Nanostructured Materials, 2, 479 (1993).

Accomplishment:

Utilizing conventional low pressure compaction procedures, nanosize ceramic powders cannot be pressed to high density green bodies. The present work demonstrated that high pressure compaction is an effective method to produce transparent high density green bodies of nanosize ceramic powders. The high density and nanostructure of these green bodies reduces the temperature and time normally needed for sintering thereby retaining the original nanostructure. Retention of the nanostructure promises feasibility of high temperature forming by superplastic deformation permitting near net shaping of complex ceramic pieces.

STANDARD REFERENCE MATERIALS

Standard Reference Materials (SRMs) are required for the calibration of analytical instruments. Projects underway in the Division either address the needs of the ceramics industry specifically or utilize the Division's capabilities for more broadly applicable SRMs. SRM project addition and production is conducted in cooperation with the Standard Reference Materials Program Office. Standard reference materials projects in the Division include:

- calibration standards for x-ray diffractometers;
- ceramic calibration blocks for high microhardness measurement;
- size, chemistry, and phase content SRMs for use in the ceramics processing field;
- zirconia powder size standards for use in the plasma-spray coating industry; and
- standards for the measurement of glass properties.

These projects are conducted in cooperation with applicable industries and are an integral part of the Division research program.

For additional information on the Standard Reference Materials program, contact S. J. Dapkunas at 301-975-6119 or e-mail to Dapkunas@micf.nist.gov.

PROJECT TITLE: SRMs for Powder Diffraction

PROGRAM TITLE: Standard Reference Materials

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Technical Description:

In the most general sense, powder diffraction patterns consist of a set of intensity values, x-ray or neutron, measured over a range of crystallographic d-spacings. These patterns exhibit peaks which result from the interference of coherent scattering of incident radiation from the periodic crystal structure of the specimen. The form of the pattern indicates the qualitative, quantitative, crystallographic, and microstructural aspects of the sample. Therefore, proper analysis of diffraction patterns can provide considerable information about the specimen, giving rise to a wide range of applications for this technique.

In order to gain this information, the effects from aberrations which are a result of the optical character of the equipment must be deconvolved from the observations to yield the desired measurement data. A function of NIST powder diffraction SRMs is to allow for the description, and possible removal, of the effects of equipment optics. NIST SRMs accomplish this by providing traceability of user measurements to fundamental physical constants or, alternatively, to values determined through round robin studies.

The three variables associated with diffraction equipment which can be evaluated with NIST powder diffraction SRMs are: 1) the d-spacing or line position, 2) line intensity as a function of position, or instrument sensitivity, and 3) instrumental and sample contributions to the shape of reflection profiles. Additional powder diffraction SRMs are designed for quantitative analysis with the use of the internal standard method. In order to serve as an SRM for powder diffraction, a material must be environmentally stable, particularly with respect to certified property. The microstructure must be such that errors in the user measurements, such as preferred orientation and particle counting statistics, etc., are minimized. Certification measurements are designed to determine the certified property as accurately as possible and ensure that the SRM is homogeneous.

Line Position SRMs

There are two SRMs, certified with respect to lattice parameters, which are intended primarily for line position measurement; SRM 640b (silicon powder), and SRM 675 (fluorophlogopite). The latter is for large d-spacing measurements. The certified values of these SRMs are traceable to the fundamental length standard through the wavelength of the x-ray source used for the original measurements. We are presently pursuing a new generation of line position SRMs which will be certified via a robust linkage to the iodine stabilized HeNe laser length standard. This project has involved the construction of a multi-axis diffractometer with a rotational resolution in the arc-second range. In order to obtain independence from sample position errors, the equipment utilizes a graded multilayer parabolic mirror to focus the divergent beam from the conventional x-ray source into a high intensity parallel beam. The silicon powder to be used for the SRM is being prepared from electronic grade P type single crystal boules. The lattice parameters of the single crystal material are being investigated with respect to interstitial, vacancy and impurity concentration; these concentrations are considerably less than a part per billion. The surface character of the powder is also being considered; the surface energy will result in stresses on the particles which will effect the measured lattice parameter. We anticipate that the lattice parameters will be certified to the parts per million range.

Instrument Sensitivity SRM 1976

This SRM consists of a sintered α Al_2O_3 plate which is certified with respect to 12 relative intensity values from (25 to 145) degrees two theta (Cu Kα). It was designed for sensitivity, intensity as a function of two-theta, calibration of conventional x-ray diffraction equipment via conventional methods, though it is also certified with respect to lattice parameters. Use of the SRM involves the collection of a set of intensity data which are then compared to the certified values. The Al_2O_3 plates of this SRM exhibit uniform levels of preferred orientation which required the use of tolerance intervals for the statistical analysis of the certification measurements. Hence the data should be considered collectively to determine if trends indicate whether or not a calibration curve should be applied. The sensitivity of the equipment used for certification was determined to be correct through a round robin study sponsored by the International Centre for Diffraction Data, ICDD, thus anchoring the certified data of the SRM.

Line Profile SRMs

The observed diffraction profile from the diffractometer consists of a sample profile convolved with an instrument profile and superimposed with noise. We can think of the sample profile as being "embedded" in the observed profile and the aim of the problem is to numerically determine the sample profile in the most general and complete manner. The sample profile characterizes the broadening due to crystallite size and micro-strain effects within the sample. Although there exist several methods of determining the sample profile they often require specific assumptions concerning the functional form of the sample profiles being made or result in ill-conditioned solutions. The approach which has been developed to solve this problem is to apply the Maximum Entropy method (MaxEnt). The MaxEnt method incorporates the *a priori* information about the instrument profile, noise distribution as constraints and determines the solution which maximizes the entropy with respect to these constrains.

Once, the sample profile is determined the microscopic properties, such as the crystallite size and micro-strain can be determined using the standard methods such as the Warren-Averbach (WA) method. However, the corresponding crystallite size and micro-strain analysis has revealed limitations in the above method. These limitations are: 1) that the micro-strain can only be interpreted in terms of a Gaussian probability distribution function, 2) the crystallite size is sensitive to experimental errors in the data and 3) the probability distribution for crystallite size cannot be determined from the experimental data.

At present research work is concentrating on developing a generalized MaxEnt/Bayesian method which could be applied to deconvolving and separating overlapped profile peaks and adopted for crystallite size and micro-strain analysis.

The instrumental contribution to observed line profile shape from x-ray diffraction equipment can be characterized with SRM 660 (LaB_6). This material was selected through an ICDD sponsored round robin which found that it was free of peak broadening due to crystallite size and micro-strain effects. Refinements of the SRM's evenly spaced, high intensity diffraction lines yield precise values for parameters of the selected profile shape function. Due to its certified lattice parameters, SRM 660 can also be used for determining instrumental parameters through a Rietveld refinement. Under development is an SRM which will display the effects of particle size induced profile broadening

Quantitative Analysis SRMs

Conventional methods of quantitative analysis are based on the Reference Intensity Ratio, RIR, method. This method entails the determination of the intensities of various diffraction line profiles and relating them to quantitative data through the use of calibration mixtures. The baseline for this method is the presumed purity of the components of the calibration mixtures. A more accurate and precise method is Quantitative Rietveld Analysis, QRA, in which the powder diffraction data are analyzed with Rietveld method. This method entails the calculation of the pattern from crystallographic, microstructural and equipment characteristics. These characteristics are related to the form of the pattern through a series of model functions. The difference between the calculated and observed pattern is then minimized by sequentially refining the physical parameters contained in the model functions to obtain an accurate and precise description of the specimen. The baseline for this method is the quality of the fit between the calculated and observed patterns.

While both of these methods can be used to perform quantitative analysis without the use of an internal standard, this is predicated by the assumption that the specimen has no amorphous component. In order to perform an accurate quantitative analysis which includes the amorphous content, a standard of known phase purity must be used. Thus, a major focus of the work in this area has been the development of a measurement and certification method for the amorphous content of SRM 676, a non-orienting alumina powder which is presently certified with respect to lattice parameters and eight relative intensity values.

The experimental approach is based on the comparison of the phase abundance of two phase mixtures determined from the preparation procedure using an analytical balance, which includes the amorphous component, to that determined from the diffraction data, which does not. Specimens consisted of 50-50 mixtures of SRM 676 and the silicon of SRM 640b. This silicon was crushed and jet milled from single crystal, electronic grade boules. The data were collected via neutron time-of-flight, TOF, and conventional x-ray powder diffraction. The data were interpreted with the assumptions that: 1) all amorphous material in the silicon was confined to the surface and 2) that the surface area of the silicon was sufficiently small, relative to the alumina, that the amorphous component of the silicon could be ignored altogether. Analysis is the refinements indicated that the TOF data were unbiased and thus the amorphous content of the alumina was credibly determined. Further experiments which are currently being performed with specimens made from five lots of silicon wherein the surface area was systematically varied. This will allow for a rigorous test of possible systematic errors in the amorphous content determination.

SRM 674a consists of a set of five powders: α Al_2O_3, ZnO, TiO_2 (rutile), Cr_2O_3, and CeO_2, which range in x-ray mass attenuation coefficients from (126 to 2203) cm^{-1} (Cu Kα). The materials available with this SRM permit the minimization of absorption contrast between the standard and the specimen. SRMs 1878a (α Quartz) and 1879a (cristobalite) were certified with respect to amorphous content for analysis of silica containing materials in accordance with health and safety regulations. Quantitative analysis of the silicon nitride system can be performed with SRM 656 which consists of two powders, one high in α content while the other is high in β. They are certified with respect to α/β ratio and amorphous content.

Technical Objectives:

Improve the accuracy and precision of powder diffraction measurements.

Accomplishments:

New methods of improved accuracy and precision have been developed.

Outcome:

The ability of researchers to establish linkage between a result obtained from powder diffraction and a material property is improved.

PROJECT TITLE: Particle Size Distribution Measurements

PROGRAM TITLE: Standard Reference Materials

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Technical Description:

Powder materials are generated with specific physical characteristics of shape, size, and chemical inertness. The particle size distributions for these materials are measured using microscopic imaging techniques .

Technical Objectives:

This research project is designed to assist industries utilizing powder materials. Knowledge of the particle size distributions of the powders is often a key factor in their industrial performance. NIST makes available powders of known size for calibration and testing of commercial sizing equipment.

Output:

Standard reference materials of glass beads are now available in several ranges of particle sizes.

Impact:

Approximately 500 units per year of these size distribution standards are purchased by industry for use in their quality control test programs.

PROJECT TITLE: Development of Microhardness Standards

PROGRAM TITLE: Standard Reference Materials

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Technical Description:

Ceramic blocks with certified Knoop and Vickers hardnesses are prepared. One hundred of each Knoop and Vickers indenter ceramic hardness blocks are prepared. These are Standard Reference Materials (SRM) #2830 and #2831, respectively. Each block has five indentations made and measured by NIST. The hardness is certified by NIST. These SRM's will support two ASTM hardness standards being developed concurrently.

Technical Objectives:

The objective of this project is to provide the ceramic community with hardness SRMs for calibration of testing machines.

Outcomes:

Certified hardness blocks for sale by the SRM office. Supporting technical papers and reports document the procedures used to prepare and use the SRM's. Two ASTM standard test methods based on this work are being balloted.

Accomplishments:

The Knoop SRM #2830 run was completed in December, 1995. The blocks were made available for sale in January, 1996.

A new ASTM Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics, which relies in part on SRM 2830, was adopted as C 1326-96 in January, 1996.

A new ASTM Standard Test Method for Vickers Indentation of Advanced Ceramics, which relies in part on SRM 2831, was adopted as C 1327-96 in January, 1996.

A hardness round robin was conducted in 1994 and verified that both SRM's are completely satisfactory. The round robin also furnished useful precision and accuracy information that was incorporated into the ASTM standards.

A new VAMAS (Versailles Project on Advanced Materials and Standards) round robin has been organized for 1996 to study instrumented (recording) hardness. A borosilicate glass and prototype silicon nitride SRM 2830 specimens will be used.

Outputs:

Outputs include SRM #2830 and, in mid-1996, SRM #2831. Round robin projects and reports measured the robustness of the methods and furnished information for precision and bias statements in two new ASTM standards. The round robins also spread knowledge about the SRM's. Technical papers and reports support the standards:

R. J. Gettings, G. D. Quinn, A. W. Ruff, and L. K. Ives, "Development of Ceramic Hardness Reference Materials," pp. 617-624 in New Horizons for Materials, Proceedings of the 8th World Ceramic Congress, CIMTEC, Florence, Italy, ed P. Vincenzini, Techna, Faenza, 1995.

R. J. Gettings, G. D. Quinn, A. W. Ruff, and L. K. Ives "Hardness Standard Reference Materials (SRM's) for Advanced Ceramics," VDI Berichte 1194, 1995, pp. 255-264. Proceedings of the International Symposium on Hardness Testing in Theory and Practice," Dusseldorf, Nov. 1995.

R. Gettings, G. Quinn, W. Ruff, and L. Ives, "New Hardness Standard Reference Materials (SRM's) for Advanced Ceramics," Ceram. Eng. and Sci. Proc., Vol. 15, #5, 1994, pp. 717-826.

ASTM C 1326-96, Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics, January, 1996.

ASTM C 1327-96, Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics

Impact:

Hardness is a frequently measured property used to characterize one of the key attributes of ceramics. The state of the art of hardness testing of ceramics is such that much published data is unreliable or worthless. The advent and propagation of these SRM's in conjunction with two supporting ASTM standards will rectify this state of affairs. Reliable, accurate and precise data should become the norm.

These SRM's and the ASTM standards are expected to have a worldwide impact and already are influencing work on a draft ISO standard in ISO Technical Committee 206, Fine Ceramics.

PROJECT TITLE: Glass Standards and SRMs

PROGRAM TITLE: Standard Reference Materials

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Technical Description:

This project provides technical support in collaborations with industrial laboratories in the development of glass SRMs and related methodologies related to glass and glass products.

Glass SRMs and standards priorities are established through formal ballots by producer/user industries and other interests. Current activities span across a list of endeavors for new and replacement SRMs.

Glass SRMs and related measurements are very important to the Glass Industry for R&D, productivity, quality control, and also to other industries using glass in high technology applications such as electrical and electronics products, communication, TV, and electro-optics and fiber optics devices. Technical activities include the identification of appropriate candidate materials, procurement of materials, homogeneity studies, organization and coordination of round robins, measurements protocols, interpretation of data, and fitting the data to predictive models.

Technical Objectives:

The objective of this program is to assist industry in the development of measurements, standards and SRMs related to glass and glass products.

Outcomes:

In the near term, principal outcomes of this program involve the development of new glass SRMs for Viscosity for a Lead-Silica composition, Thermal Conductivity/Diffusivity of a Glass Ceramic, Glass Density SRMs for Soda-Lime and Borosilicate compositions and Quartz, and Viscosity Fixpoints for Annealing, Softening and Strain Point of Quartz. Over the longer term, the current list of priorities includes SRMs for Thermal Expansion, Heat Capacity, Glass Transition, Crystallization, and Remelt Temperatures, and Stress Analysis.

Accomplishments:

SRM 717A for the Viscosity of Borosilicate Glass was certificated in 1995, and this SRM is now available for sale by NIST.

This SRM will play an important role in R&D, quality control in manufacturing, pollution control, and in optimization of energy usage which is a large cost factor in glass melting.

Outputs:

The results of this research program and rationale for priorities are discussed and reviewed in full with industrial and academic collaborators on a semi-annual basis.

Technical Publications:

Certificate for SRM on Viscosity of a Borosilicate Glass, Sept. 1995

SUPERCONDUCTIVITY

A significant program in high-T_c (critical transition temperature) superconductivity is being conducted in MSEL and other Laboratories at NIST. The primary focus of the MSEL Program is on bulk superconducting materials for wire and magnet applications. In conducting this program, researchers in MSEL work closely with their counterparts in other NIST Laboratories, and collaborators in U.S. industry and other National Laboratories.

The primary thrusts of the program are as follows:

- Phase equilibria - Work is being conducted in close collaboration with the U.S. Department of Energy (DOE) and its national laboratories to provide the phase diagrams necessary for processing these unique ceramic materials. A prime objective is the development of the portions of the phase diagram for the Pb-Bi-Sr-Ca-Cu-O system relevant to production of the high T_c materials.

- Flux pinning - This project makes use of a unique magneto-optical imaging facility to examine flux pinning in a variety of materials. Much of this work is being conducted in conducted in collaboration with American Superconductor Corporation. In addition techniques for better interpretation of magnetic measurements are being developed.

- Damage mechanisms - Work is being conducted under a joint CRADA (cooperative research and development agreement) with American Superconductor Corporation as part of the "Wire Development Group" which involves a number of DOE National Laboratories and the University of Wisconsin to elucidate the effects of strain on the loss of current in superconducting wires. The primary tool being employed is the use of microfocus radiography available at the NIST beamline at the Brookhaven National Laboratory.

- Database - A high-T_c superconductor database has been developed in collaboration with the National Research Institute for Metals (NRIM) in Japan. The High-Temperature Superconductor Database (HTSD) includes evaluated open-literature data on numerous physical, mechanical, and electrical properties of a variety of chemical systems. The first version of the database is now for sale by the Office of Standard Reference Data.

- Crystal structure - Thermal neutron diffraction techniques and profile refinement analyses are being utilized to investigate crystal and magnetic structures, composition, and crystal chemical properties. This research is being carried out in collaboration with a number of industrial and university experts.

For further information about the High-T_c Superconductivity Program, please contact S. W. Freiman at 301-975-6119 or e-mail requests to Freiman@micf.nist.gov.

PROJECT TITLE: Phase Equilibria Relations of High T_c Superconductors

PROGRAM TITLE: Superconductivity

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Technical Objectives:

Conduct phase-equilibria studies of superconducting materials with emphasis on regions pertinent to the processing of bulk conductors. Determine the primary crystallization field of the bismuth-strontium-calcium-copper oxide "2212" superconductor in air and investigate the effect of silver impurities. Investigate phase relations of thallium 1223-type superconductors with emphasis on melting behavior and associated thallia vapor pressures. Conduct high-temperature X-ray powder diffraction studies of Pb-BiSCCO "2223" wire precursor material as a function of oxygen partial pressure.

Accomplishments

The approximate primary crystallization field of the BiSCCO "2212" superconductor has been determined in air and encompasses a mole fraction compositional range of (20 to 42)% $BiO_{1.5}$, (15 to 35)% SrO, (2 to 25)% CaO, and (20 to 43)% CuO. Ag enters the melts at saturation levels of (6 to 8)% and depresses the solidus by as much as 26°C. Cu also substitutes into Ag, lowering its melting temperature. The 2212 BiSCCO superconductor melts incongruently to liquid plus alkaline-earth-rich phases and exhibits a eutectic at 825°C (Bi:Sr:Ca:Cu = 41.7:16.1:22.3:19.9). Melt compositions and associated thallia vapor pressures have been determined for three different thallium 1223-type superconducting compositions; thallia vapor pressures were found to be highly variable with substituent type. In contrast, the compositions of the melts were relatively insensitive to substituent-type and characteristically Cu-, Pb-, and Bi-rich and alkaline-earth-poor. High-temperature X-ray diffraction studies of Pb-BiSCCO 2223 wire precursor were conducted on silver foil under varying oxygen partial pressure and at different heating rates. Melting, recrystallization, and orientation phenomena were found to be markedly dependent on heating rate, the presence of silver, and oxygen partial pressure.

Outputs:

"Melting Equilibria of the Bi-Sr-Ca-Cu-O (BSCCO) System in Air. I. the Primary Crystallization Phase Field of the "2212" Phase and the Effect of Ag Addition", W. Wong-Ng, L.P. Cook, and F. Jiang. In final preparation.

"Melting Equilibria of the Ba-Y-Cu-O System in the Vicinity of the $Ba_2YCu_3O_{6+x}$ Phase Field", W. Wong-Ng and L.P. Cook. Submitted to Journal ACerS.

"Comparative Melting and Vaporization Behavior of Doped Thallium 1223 Phases: Preliminary Results in Oxygen", L.P. Cook and W. Wong-Ng. Submitted to NIST Journal of Research.

"High-Temperature X-ray Studies of Growth and Texturing of Bi-2223 on Silver", A.R. Drews, J.P. Cline, T.A. Vanderah, and K. Salazar. In final preparation.

PROJECT TITLE: Microstructural Characterization of Bi-2223/Ag Tapes

PROGRAM TITLE: Superconductivity

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Technical Description:

Multifilamentary Bi-2223/Ag composite tapes are tensilely strained on axis from (0 to 1)%, and the damage is studied in-situ and nondestructively by means of synchrotron-radiation microradiography at 24.4 keV. An asymmetric Bragg diffraction microscope is used to obtain digital microradiographs with a pixel size of 0.5 μm. The crack density, the dimensions of the crack opening and the strain distribution are determined. Displacements as small as 0.2 μm can be resolved by sub-pixel interpolation.

Technical Objectives:

The broad objective is to characterize strain-induced damage in composite tapes and to relate it to electrical performance. A specific objective is to identify the microstructure which maintains a continuous superconducting path through tapes that are strained well beyond the elastic limit of the superconductor. Such high strain should crack the brittle superconductor and disrupt the superconducting path, but surprisingly the critical current is reduced by only one order of magnitude.

Outcomes:

This project is expected to reveal ways to increase the critical strain, defined as the strain at which the critical current begins to degrade, beyond the present value of about 0.5%, and thereby broaden the range of commercial applications for superconducting composite tapes.

Accomplishments:

A model microstructure of tapes strained past the critical strain, featuring no continuous superconducting path through the tape, was proposed based on microradiography and optical microscopy data. However, the calculated electrical resistance of the model tape disagrees strongly with measurements. To achieve agreement, while maintaining consistency with the imaging results, the existence of a narrow superconducting bridge across the cracks is proposed. Microradiographs of tapes strained to 1% show cracks extending across the full width of individual filaments and repeating about every 0.75 mm along the tape. Optical micrographs of a longitudinal section of one tape confirm these results, and show that a crack in one filament is generally accompanied by (and aligned with) cracks in every other filament in the section. The crack opening dimensions, determined from the microradiographs, account for most of the macroscopic strain, suggesting that the filament segments between the cracks may be undamaged.

These results, taken alone, suggest that there is no continuous superconducting path through the tapes, implying that they should exhibit electrical resistance due to current flow in the silver around the cracks. However, the measured resistance at low (ideally infinitesimal) current is over one order of magnitude smaller than that calculated for an appropriate model geometry. This large discrepancy suggests that there is a superconducting bridge across the cracks, which is consistent with the imaging results only if the bridge has significantly smaller cross section than a filament, making it hard to observe. It is reasonable to suspect that the bridge exists at the silver interface, where the microstructure may differ from inside the filament.

Impact:

In cooperation with industrial partners, a more realistic model for the microstructure of superconducting composite tapes, which has resulted from this project, has been used to obtain a better understanding of the relationship between the tape microstructure and the electrical properties.

PROJECT TITLE: Magneto-Optic Imaging of High Temperature Superconductors

PROGRAM TITLE: Superconductivity

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Technical Description:

A magneto-optic imaging system originally developed in Russia has been placed into operation at NIST in order to study flux pinning mechanisms in high temperature superconductors. High flux pinning densities are needed for increasing the electrical current carrying capabilities of high temperature superconductors. Magneto-optic imaging is proving to be highly useful in visualizing and evaluating flux pinning densities.

Technical Objectives:

To provide U.S. industry with evaluation techniques and data which will accelerate the commercialization of high temperature superconductors for electrical power transport.

Accomplishments:

Magneto-optic has been very useful in evaluating the performance of cast tapes of the superconductor $Bi_2Sr_2CaCu_2O_z$ (hereafter called BSCCO), a leading candidate material for electrical power applications.

Microscopic flux flow in undoped and Li-doped BSCCO cast tapes before and after neutron irradiation was studied by means of a magneto-optical imaging technique in order to correlate flux flow with the microstructure. Flux penetration was very nonuniform, indicating that current flow in the specimens is inhomogeneous. The neutron irradiation treatment increased flux pinning in both the undoped and Li-doped tapes. These results are important in understanding the effect of microstructure and irradiation on the critical current density of BSCCO.

More recently, the magneto-optical imaging system was used to study magnetization and demagnetization in composite tapes from American Superconductor Corporation that are composed of superconducting BSCCO filaments embedded in a silver matrix. BSCCO filaments located up to 112 μm below the unpolished surface of a composite tape could be imaged directly through the outer silver sheath. The magneto-optical images provided information on the morphology and alignment of the upper layer filaments in the tape, the relative depths of these filaments from the tape surface, and the homogeneity of the magnetic flux distribution within these filaments. These characteristics should prove useful in predicting the performance of the composite in service. Furthermore, our results demonstrate that magneto-optical imaging could be a routine diagnostic tool for these materials. These measurements demonstrate the utility of that magneto-optical imaging for nondestructive analysis of composites containing high T_c superconductors.

Outputs:

Technical Papers:

"Magnetic Flux Penetration in Undoped and Li-Doped $Bi_2Sr_2CaCu_2O_z$ Cast Tapes Before and After Neutron Irradiation," M. Turchinskaya, D. L. Kaiser, A. J. Shapiro, and J. Schwartz, Physica C **246**, 375 (1995).

"Magneto-transport Behavior of Polycrystalline $YBa_2Cu_3O_7$: A Possible Role for Surface Barriers," Shi Li, M. Fistul, J. Deak, P. Metcalf, G. F. Giuliani, M. McElfresh, and D. L. Kaiser, Phys. Rev. B. in press.

Impact:

The NIST measurements are enabling American Superconductor Corporation to monitor progress in developing tapes made of high temperature superconductors for transmission of electrical power.

PROJECT TITLE: Materials Property Database for High Temperature Superconductors

PROGRAM TITLE: Superconductivity

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Technical Description:

This project is designed to facilitate technological advances in materials science by providing evaluated thermal, mechanical, and superconducting property data for the broad class of materials commonly called high temperature superconductors.

Technical Objectives:

The objective is to provide industry a reliable knowledge base for understanding material property and performance relationships. Use of the technically evaluated data enables industry to save substantial time, effort, and precious funds.

Outcomes:

Several significant results are expected from this project: (1) a comprehensive set of evaluated data; (2) a software interface to the database; (3) standards and methodology for data evaluation; and (4) relations and models for properties and performance characteristics.

Accomplishments:

The first version of the database has been designated as NIST Standard Reference Database 62: High Temperature Superconductors (HTS), Version 1. All phases of the development and testing of the database have been completed, and the database packages are now being distributed by the NIST Standard Reference Data Program. This is the first publicly available materials property database on HTS materials.

An agreement to collaborate with a similar project in Japan, at the National Research Institute for Metals (NRIM) has been signed by representatives of NIST and NRIM to provide a more efficient coverage of the worldwide effort. This collaboration will enable U.S. efforts to access important data, particularly from Japan, that would not otherwise be readily available.

Outputs:

Database:

NIST Standard Reference Database Number 62: High Temperature Superconductors, Version 1, 1995.

Technical Papers:

Reference Relations for the Evaluation of the Materials Properties of Orthorhombic $YBa_2Cu_3O_x$ Superconductors, R. G. Munro and H. Chen, J. Am. Cer. Soc. (1995).

Impact:

High temperature superconductors form the most intensely studied class of materials among all the types of materials that may be considered advanced or high-technology materials. The average publication rate since the discovery of the materials in 1986 has been on the order of 5000 papers per year. The resulting abundance of data has created serious challenges to accessing the essential data and to resolving the wealth of inconsistencies that may be found among the sets of property data. The HTS database provides a significant effort to overcome these barriers to the key technological information.

SYNCHROTRON RADIATION

The Materials Microstructural Characterization Group in the Ceramics Division operates two x-ray experimental stations at the National Synchrotron Light Source (NSLS), where researchers from NIST, industry, universities and other government laboratories carry out state-of-the-art measurements on ceramic, semiconductor, photonic, metallurgical, biological, and other materials of high scientific or technological interest. X-ray measurement capabilities include ultra-small-angle scattering (USAXS), topography, diffraction-imaging microscopy, x-ray absorption fine structure spectroscopy (XAFS), standing-wave x-ray scattering (SWXR), and reaction-kinetic surface science measurements.

The range of studies performed over the past year include processing-microstructure relationships in plasma-sprayed ceramic deposits, sintering of nanostructured ceramics, strain-induced microcracking in high-T_c superconducting composite tapes, grain-size/pore-size trajectories within ceramic microstructures during processing, diffraction imaging of ZnSe, strain and bond distortion in ultra-thin semiconductor films, and the determination of atomic-scale and molecular-scale structures at technologically important surfaces and interfaces.

In addition, as part of a national facility, time on the NIST instruments at the NSLS is made available to qualified researchers based on peer-reviewed proposals. In the past year, researchers from chemical, aerospace, energy and materials production industries as well as from NIST laboratories, other government laboratories and universities have completed experiments that could not have been performed elsewhere in the United States. The long-term goal of research at these facilities is to enable researchers to address basic issues so that U.S. manufacturers can provide superior materials based on structural information not available elsewhere.

Looking toward the future, the NIST Materials Science and Engineering Laboratory (MSEL) has entered a Collaborative Access Team (CAT) agreement with the University of Illinois, Oak Ridge National Laboratory, and U.O.P. Corporation, the purpose of which is to build and operate x-ray instruments on sector 6 at the Advanced Photon Source (APS) now under construction at the Argonne National Laboratory. The CAT is called UNI-CAT in recognition of the University, National Laboratory, and Industrial components of this team. This partnership is particularly attractive to us at NIST because there is significant overlap with current and future NIST interests.

The APS will offer a 10^2-10^4 increase in brilliance compared to the best synchrotron x-ray sources of today. In the years to come, the APS will supplant the NSLS as this nation's premier x-ray source. The APS beamlines now being built by UNI-CAT incorporate the newest technology which will not only enable NIST scientists to improve significantly our real-time x-ray microscopy, ultra-small-angle x-ray scattering, *in-situ* x-ray topography and EXAFS capabilities, but will also offer opportunities for cutting-edge experiments in structural microcrystallography (and time-resolved structural scattering), surface/interface scattering, diffuse scattering, and magnetic scattering. NIST scientists will extend our present portfolio of characterization capabilities to include an even wider range of materials measurement of importance to materials scientists and to U.S. industry.

For further information on the Synchrotron Radiation Program, contact Gabrielle Long at 301-975-5975 or e-mail to gabrielle@enh.nist.gov.

PROJECT TITLE: Materials Science at the NSLS

PROGRAM TITLE: Synchrotron Radiation

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Technical Description:

The Materials Microstructural Characterization Group operates two x-ray experimental stations at the National Synchrotron Light Source (NSLS) where researchers from NIST, industry, universities and government laboratories carry out state-of-the-art measurements on ceramic, semiconductor, photonic, polymeric, biological, and other materials of scientific and/or technological interest. In addition, a new soft-x-ray materials-science end station for the study of materials containing carbon, nitrogen, oxygen and fluorine has been designed, built and commissioned. This experimental station, which delivers state-of-the-art intensity, resolution, and detection sensitivity, is the product of a collaboration between NIST, The Dow Chemical Co., the Physics Department at Brookhaven National Laboratory, and two industrial awardees of SBIRs. Problems which are currently being investigated include model catalyst systems, polymer surfaces and their interfaces, and self-assembled monolayers.

Technical Objectives:

The objectives are to investigate processing / microstructure / property relationships in ceramic materials of high technologocal interest; to perform state-of-the-art diffraction imaging of semiconductor, superconductor and photonic single crystals; to develop a model of tensile-strained high-TC superconducting tapes; to probe bulk and surface structures of important low-atomic-number materials in a reactive environment.

Outcomes:

Through the use of the NIST portfolio of leading-edge materials science experiments, researchers will address basic issues regarding processing/microstructure/property relationships; will determine the structure at the surfaces of materials containing carbon, nitrogen, oxygen, and fluorine, including active-site chemical analysis; will be able to understand the electrical conductance around cracks which develop during tensile straining; and will correlate the defect structures in single crystals with the product properties.

Accomplishments:

1. The effects, on the microstructural development during cement hydration, of adding pozzolanic additives to cements and concretes, in the form of silica fume, have been extensively studied using small-angle neutron scattering (SANS) and ultra-small-angle x-ray scattering (USAXS).

2. A model microstructure of tapes strained past the critical strain, featuring no continuous superconducting path through the tape, was proposed based on microradiography and optical microscopy data.

3. Small-angle scattering was applied to the quantitative characterization of the crack and pore microstructure of plasma-sprayed deposits. The anisotropic character of the microstructure has been observed and the methods and techniques for separating the intralamellar crack population from the interlamellar pore population have been developed, in which the surface areas and volume of each are derived.

4. A detailed chemical and structural understanding of the lubricant-to-substrate interface was obtained for stearic acid.

5. The defect density of selected areas of undoped ZnSe grown by SPVT was found to be as low as (103 to 104) cm$_{-2}$, which is comparable to defect densities now achievable for other compound semiconductor materials. Devices grown on wafers from this particular boule were observed to have the longest measured lifetime.

6. The characteristic microstructure of each of the classes of diamond has been identified and the relationship between substrate quality and film defects has been documented.

7. The strain and bond distortions in epitaxial InAs, InGaAs and thicker InAs films have been studied via x-ray standing-wave and x-ray-absorption fine structure measurements. The results indicate that, for both InAs and InGaAs monolayers, strain and bond distortions are accurately described by macroscopic elastic theory.

Outputs:

Technical Papers:

A. J. Allen and R. A. Livingston; "Small-Angle Scattering Study of Concrete Microstructure as a Function of Silica Fume, Fly Ash or Other Pozzolanic Additions", (SP 153-62) in Proc. 5th CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete, June 1995. Ed. V. M. Malhotra. American Concrete Institute, Detroit, MI. 1179-1200 (1995).

J. Ilavsky, H. Herman, C. C. Berndt, A. N. Goland, G. G. Long, S. Krueger, and A. J. Allen, "Porosity in Plasma-Sprayed Alumina Deposits" in Thermal Spray Industrial Applications, C. C. Berndt and S. Sampath, Eds. (ASM International, 1994) 709-714.

D. A. Fischer, Z. Hu, and S. M. Hsu, "Molecular Orientation and Bonding of Monolayer Stearic Acid on a Copper Surface in Air," for submission to Tribology Letters.

D. A. Fischer, Z. S. Hu, and S. M. Hsu, "Tribochemical and Thermochemical Reactions of Stearic Acid on Copper Surfaces in Air as Measured by Ultra Soft X-ray Absorption Spectroscopy, " for submission to Tribology Letters.

D. B. Eason, Z. Yu, W. C. Boney, J. W. Cook, Jr., J. F. Schetzina, D. R. Black, G. Cantwell, and W. C. Harsch, "High-brightness light-emitting diodes grown by MBE on ZnSe substrates," Proc. Of PCSI Conf., Phoenix, AZ, Jsn. 9 - 13, 1995.

D. Black, H. Burdette, and G. Cantwell, "X-ray diffraction imaging of ZnSe," Symp. On Nondestructive Wafer Characterization for Compound Semiconductor Materials, Reno, NV, May 21 - 26, 1995.

D. R. Black, H. E. Burdette and W. Banholzer, "X-ray diffraction imaging of man-made and natural diamond," Diamond and Related materials 2 (1993) 121 - 125.

L. Robins, M. A. Plano, M. D. Moyer, M. A. Moreno, L. S. Pan, D. R. Kania, and W. F. Banholzer, "The defect microstructure of thick homoepitaxial diamond films," Advances in New Diamond Science and Technology, S. Saito, N. Fujimori, O. Fukunaga, M. Kamo, K. Kobashi and M. Yoshikaswa, Eds. (MYU, Tokyo, 1994) p. 251

M. A. Plano, M. D. Moyer, M. A. Moreno, D. Black, H. Burdette, L. Robins, L. S. Pan, D. R. Kania, and W. F. Banholzer, "Properties of thhick homoepitaxial diamond films," in Diamond, SiC, and Nitride Wide-Bandgap Semiconductors, Materials Research Society Proceedings Vol. 339, C. Carter, Jr., G. Gildenblat, S. Nakamura and R. Nemanich, Eds., (MRS, Pittsburgh, 1994) p. 307.

D. Black, H. Burdette, R. Linares and P. Doering, "X-ray diffraction imaging of free-standing single-crystal CVD diamond films," in Proc. Fourth Int'l. Symp. On Diamond Materials, (May, 1995, Reno).

D. Black, H. Burdette, M. A. Plano, M. D. Moyer, M. Moreno, J. Ager, and A. Chen, "Observation of individual defects in a homoepitaxial diamond film," in Proc. Fourth Int'l. Symp. on Diamond Materials, (May, 1995, Reno).

PROJECT TITLE: UNI-CAT Construction at the APS

PROGRAM TITLE: Synchrotron Radiation

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Technical Description:

The NIST Materials Science and Engineering Laboratory has entered into a Collaborative Access Team (CAT) agreement with the University of Illinois, Oak Ridge National Laboratory and UOP Corporation, the purpose of which is to build and operate x-ray experiments on Sector 33 of the Advanced Photon Source (APS), now nearing completion at Argonne National Laboratory. In the years to come, the APS will become this nation's premier x-ray source for high-brightness hard x-radiation. The APS experimental stations now under construction will enable NIST scientists to improve significantly our ultra-small-angle x-ray scattering and our real-time x-ray imaging capabilities. There will also be opportunities for cutting-edge experiments in structural crystallography, diffuse scattering, surface/interface scattering, and magnetic scattering.

Technical Objectives:

The technical objectives are: (1) research at the cutting edge of the fields of materials science, physics, chemistry, biology, chemical engineering, polymer science and geology, with emphasis on close collaboration among scientists and engineers from universities, industries and national laboratories.

Outcomes:

The NIST portfolio of synchrotron radiation capabilities will be expanded to include: high-resolution diffraction and spectroscopy; diffuse x-ray scattering; surface and interface diffraction and scattering during reconstruction, relaxation, roughening transitions, melting, chemical reactions and phase transitions; magnetic x-ray scattering; as well as significantly improving our present capabilities in ultra-small-angle x-ray scattering, x-ray diffraction microscopy, and in situ XAFS.

Accomplishments:

On the UNI-CAT Insertion Device beam line, the first optical enclosure, the second optical enclosure and both experimental hutches have been built. The monochromator and the beam-conditioning table are in the final stages of construction.

PROJECT TITLE: Microstructure of single-crystal and thin-film diamond

PROGRAM TITLE: Synchrotron Radiation

Principal investigator(s):

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Technical Description:

This project makes use of x-ray diffraction imaging (topography) to characterize the defect microstructure of high-temperature/high-pressure (HTHP) diamonds, natural diamonds, and homoepitaxial chemical-vapor- deposited (CVD) diamond films grown on single-crystal material. Diamonds have an unusual combination of properties which would be extraordinarily useful in electronic device technology. CVD diamond offers an opportunity to create materials with both high quality and low costs. Limiting factors include the inability to grow single-crystal films by heteroepitaxy, and the general material quality. Presently, small diamond substrates are used to grow homoepitaxial films to study the properties of CVD diamond. One way to reduce the cost and increase the size of substrate crystals is known as the "tiling" method. In this technique, homoepitaxial CVD diamond films are grown and then removed from the single-crystal substrates. Several films are then arranged in a tiling to form a mosaic substrate for further CVD growth of a larger crystal. The effects on CVD diamond film quality of the recently-developed electrochemical liftoff method to produce the free-standing crystals for tiling are being investigated, and the tiling process is being examined to understand the effects of substrate misorientation, crystallographic quality, strain and surface finish on the quality of the product CVD crystal.

Technical Objectives:

The goals of this project are to: characterize the defect microstructure of the four classes of diamond; evaluate each as substrates for deposition of homoepitaxial CVD diamond films; investigate the relationships between defects in the substrates, growth processes and film quality; relate film quality to electronic properties; and evaluate new methods for growing large area single-crystal diamond films.

Outcomes:

It is expected that an increased understanding of the relationships between substrate quality, growth processes and CVD diamond properties will improve the development of high-quality diamond films for high-technology applications.

Accomplishments:

The characteristic microstructure of each class of diamond has been identified and the relationship between substrate quality and film defects has been documented.

Outputs:

Technical Papers:

D. R. Black, H. E. Burdette and W. Banholzer, "X-ray diffraction imaging of man-made and natural diamond," Diamond and Related materials 2 (1993) 121 - 125.

L. Robins, M. A. Plano, M. D. Moyer, M. A. Moreno, L. S. Pan, D. R. Kania, and W. F. Banholzer, "The defect microstructure of thick homoepitaxial diamond films," Advances in New Diamond Science and Technology, S. Saito, N. Fujimori, O. Fukunaga, M. Kamo, K. Kobashi and M. Yoshikaswa, Eds. (MYU, Tokyo, 1994) p. 251.

M. A. Plano, M. D. Moyer, M. A. Moreno, D. Black, H. Burdette, L. Robins, L. S. Pan, D. R. Kania, and W. F. Banholzer, "Properties of thick homoepitaxial diamond films," in Diamond, SiC, and Nitride Wide-Bandgap Semiconductors, Materials Research Society Proceedings Vol. 339, C. Carter, Jr., G. Gildenblat, S. Nakamura and R. Nemanich, Eds., (MRS, Pittsburgh, 1994) p. 307.

D. Black, H. Burdette, R. Linares and P. Doering, "X-ray diffraction imaging of free-standing single-crystal CVD diamond films," in Proc. Fourth Int'l. Symp. On Diamond Materials, (May, 1995, Reno, Nevada).

D. Black, H. Burdette, M. A. Plano, M. D. Moyer, M. Moreno, J. Ager, and A. Chen, "Observation of individual defects in a homoepitaxial diamond film," in Proc. Fourth Int'l. Symp. on Diamond Materials, (May, 1995, Reno, Nevada).

Impact:

The improved understanding of the relationships between substrate quality, growth processes and CVD diamond properties has had a positive influence on the development of high-quality diamond films for high-technology applications.

PROJECT TITLE: Strain and Relaxation in InAs and InGaAs Films on GaAs (001)

PROGRAM TITLE: Synchrotron Radiation

Principal investigator(s):

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Technical Description:

One of the primary obstacles hampering epitaxial growth is the fundamental limit of strain accommodation in the overlayer film. As more layers are added, the strain energy increases proportionally, and above a critical thickness, it becomes energetically favorable for the film to collapse through a network of misfit dislocations. In this project, x-ray standing wave measurements and x-ray absorption fine structure measurements are combined to determine the atomic displacements and the bond lengths within buried interfaces grown epitaxially on single crystal GaAs.

Technical Objectives:

The objective of this research is to determine the bond lengths within buried interfaces of InAs and InGaAs grown epitaxially on GaAs (001) to determine the strain and relaxation at the buried interfaces and to test the limits of elastic theory.

Outcomes:

Despite the fundamental importance of strain accommodation in electronic materials, relatively little work has been performed to understand the geometrical aspects. The present research is expected to resolve the ongoing controversy concerning the effects of strain on both lattice constants and on bond lengths in thin films.

Accomplishments:

The strain and bond distortions in epitaxial InAs, InGaAs and thicker InAs films have been studied via x-ray standing-wave and x-ray-absorption fine structure measurements. The results indicate that, for both InAs and InGaAs monolayers, strain and bond distortions are accurately described by macroscopic elastic theory. Thicker InAs films are found to collapse beyond a critical thickness of about 2 monolayers, accompanied by a large degree of structural disorder and bond-length relaxation.

Outputs:

Technical Paper:

J. C. Woicik, K. E. Miyano, J. G. Pellegrino, P. S. Shaw, S. H. Southworth, and B. A. Karlin, "Strain and relaxation in InAs and InGaAs films grown on GaAs (001)," Phys. Rev. B. (in press).

Impact:

The present research has resolved an ongoing controversy concerning the effects of strain on both lattice constants and on bond lengths in thin films. The research has been able to demonstrate, surprisingly, that macroscopic elastic theory can accurately account for the strain observed in an atomic monolayer.

THEORY AND MODELING

Design and processing of advanced materials, such as monolithic fine ceramics with heterogeneous microstructures, as well as fiber-reinforced metal/intermetallic/ceramic matrix composites, is labor intensive, costly and still in the developmental stage. Computational materials theory, modeling, and computer simulations provide powerful new paradigms for rationally designing, processing, characterizing, and prototyping these advanced materials. Such research ranges from the molecular design and understanding of these materials via first-principles quantum mechanical calculations to micro-physical modeling of heterogeneous microstructural features and properties to computer simulations of microstructural evolution and subsequent physical properties

Modeling and computer simulations at the mesoscopic or microstructural level is an exciting new aspect of computational materials theory. Such simulations, in orchestrated collaboration with experimental development, provide an efficient stratagem for minimizing costs, time-to-application in ultimate production strategies of ceramic components, and lifetime prediction methodologies. They generally involve four elements: (1) simulation and/or representation (usually digital) of a microstructure; (2) simulation of important processing and physical properties; (3) comparison with experimentally measurable properties and (4) application to design-improved microstructures for specific applications.

The projects in the Ceramics Division in materials theory, modeling, and computer simulations are focused in two areas: (1) first principles calculations of ceramic phase diagrams; parameterization of microstructural properties; and (2) modeling of microstructures.

For further information on the Theory and Modeling program, please contact E. Fuller at (301) 975-5795 or e-mail to erfuller@enh.nist.gov.

PROJECT TITLE: Modeling of Microstructures

PROGRAM TITLE: Theory and Modeling

Principal investigator(s):

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Technical Description:

This project is designed to elucidate the essential physics of microstructural evolution and to develop computationally efficient algorithms for simulations of microstructural development.

Technical Objectives:

This research is designed to assist industry in developing new paradigms for elucidating microstructural evolution through models of the essential physics and identification of computationally feasible algorithms. Particular objectives are to model large collections of microstructural objects so as to capture the complexities which occur in materials, and to develop and implement new mathematical techniques for modeling microstructural evolution.

Outcomes:

Major outcomes of this research are expected to include: (1) better, more useful, more robust and more predictive models of materials processes, microstructures, and microstructural evolution; (2) implementation of developed algorithms by industrial researchers; and (3) deeper understanding of the fundamentals of materials behavior.

Accomplishments:

Simulation of anisotropic grain growth in silicon nitride illuminated the role of microstructural development in increasing the toughness of this material. Statistics and process control parameters are being understood at a microstructural level.

The physical processes associated with reactive wetting were clarified through studies of diffusion and interface growth during reactive wetting. This research has provided physical insight for designers of joining techniques for ceramic materials. Such techniques are a key technological component in materials utilization.

Effects of stress, both applied and self-generated, were modeled for the sintering process. Sintering is the most important ceramic processing technique; this research represents the first time that stress has been reckoned with physically rigorous approaches in realistic powder compacts.

Classical thermodynamics associated with anisotropic surfaces and interface growth were developed. Beautiful and elegant mathematical techniques of convexification were used to unify the description of diverse fundamental aspects in the field of ceramics: multicomponent phase diagrams, anisotropic surface energies, and crystal growth shapes. This important topic now has a solid theoretical foundation which can only benefit the understanding and the application of technological materials.

Outputs:

Several important outputs were derived from this project. A letter of appreciation was received from the duPont and IBM corporations signifying their appreciation for our successful efforts. Several research papers were produced from the technical advancements, which were made.

Technical papers published:

J. W. Cahn and W. C. Carter, "Crystal Shapes and Phase Equilibria: A Common Mathematical Basis," Met. and Mat. Trans., in press.

R. M. Cannon, E. Saiz, A. P. Thomsia, and W. C. Carter, "Reactive Wetting Taxonomy" MRS Proc., **357**, 279-291 (1995).

J. W. Bullard and W. C. Carter, "Numerical Determination of Critical Strain Rate for Neck Rupture for Evaporation-Condensation Sintering of Isotropic Particles," in ***Proc. Sintering '95***, in press.

J. W. Bullard, E. J. Garboczi, W. C. Carter, and E. R. Fuller, Jr., "Numerical Methods for Computing the Mean Curvature of an Interface," Comput. Mater. Sci., Vol. 4, 103-116 (1995).

Technical papers submitted for publication:

A. W. Searcy, J. W. Bullard, and W. C. Carter, "Possible Explanations of Transient Neck Formation between Pairs of 110 Faceted Particles," J. Am. Ceram. Soc., submitted.

J-H Choi, D-H Kim, B. J. Hockey, S. M. Weiderhorn, C. A. Handwerker, J. E. Blendell, W. C. Carter, A. R. Roosen, "The Equilibrium Shapes of Cavities in Sapphire," J. Am. Ceram. Soc., submitted.

Impact:

DuPont is now using mathematical techniques developed at NIST to design better sintering processes. Many industrial researchers have asked that we model their particular problems and have asked for use of our developed codes.

PROJECT TITLE: Prediction of Phase Diagrams

PROGRAM TITLE: Theory and Modeling

Principal investigator(s):

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Technical Description:

This project is designed to calculate phase diagrams of technologically important ceramic materials from first principles and to develop the theoretical techniques necessary to further these calculations.

Technical Objectives:

This research is designed to assist industry by providing information on phase equilibria and order/disorder configurations of technologically important classes of solid-solution ceramics. Such information is critical to the application of these materials because their ferroelectric, magnetic, and transport properties can be sensitive functions of composition and quenched-in cation order. The information is provided via first-principles phase diagram calculations with experimental verification of the results. A technical objective is to assess the relative merits of various techniques for calculating formation energies of ceramic compounds.

Outcomes:

Major outcomes of this research are expected to include: (1) first principles phase diagrams of important relaxor ferroelectrics, (2) improved techniques for calculating formation energies of ceramic compounds.

Accomplishments:

A first-principles calculation of the $Pb(Sc_{0.5}Ta_{0.5})O_3$-$PbTiO_3$ (PST-PT) quasi-binary phase diagram was completed using a Potential-Induced-Breathing/Cluster-Variational-Method (PIB/CVM). Results from these calculations include:

(a) Predictions of three new unexpected ordered phases, their enthalpies of formation, and the maximum temperatures for which they are stable.

(b) Ground-state analysis for the PST-PT system based on an 18-site cluster.

(c) Calculation of a complete theoretical phase diagram for the PST-PT system.

Preliminary results of the calculations for PST-PT solid solutions were published in Ferroelectrics, and a final paper was published in Phys. Rev. B.

Outputs:

This project has produced several research papers from the technical advancements which were made.

Technical papers published:

B. P. Burton and R. E. Cohen, "Nonempirical calculation of the PST-PT $Pb(Sc_{0.5}Ta_{0.5})O_3$-$xPbTiO_3$ quasibinary phase diagram," Phys. Rev. **B52**, 792 (1995).

B. P. Burton and R. E. Cohen, "First principles study of cation ordering in the system $Pb(Sc_{1/2}Ta_{1/2})O_3$, and $(1-x)Pb(Sc_{1/2}Ta_{1/2})O_3$-$xPbTiO_3$," Ferroelectrics **164**, 201-212, (1995).

Technical papers submitted for publication:

P. D. Tepesch, A. F. Kohan, G. D. Garbulsky, G. Ceder, C. Coley, H. T. Stokes, L. L. Boyer, M. J. Mehl, B. P. Burton, K. Cho, and J. Jannopoulos, "A model to compute phase diagrams in oxides with empirical or first-principles energy methods and application to the solubility limits in the CaO-MgO system," J. Am. Ceram. Soc., submitted.

OTHER PROJECTS

PROJECT TITLE: Diamond film Research

PROGRAM TITLE: Other Projects

Principle NIST Investigators:

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Technical Description:

Carbide cutting tools coated with CVD diamond have lifetimes ten times or greater than those of uncoated carbide tools provided that the coating does not delaminate. At the present time, delamination failure is detected only by lifetime testing under machining conditions, a time consuming procedure. We are currently investigating whether nondestructure techniques, thermal wave imaging (Ceramics Division), scanning acoustic microscopy (Metallurgy Division) and contact acoustic nonlinearity (Materials Reliability Division) can be used to predict premature delamination failure. The use and analysis of thermal imaging data may lead to an new method of measuring thermal conductivity/diffusivity in CVD diamond.

Technical Objectives:

The objective of this project is to determine whether nondestructive procedures can be used to predict premature debonding failure of cutting tools coated with chemical vapor deposited (CVD) diamond.

Accomplishments:

As part of a working group on standardizing thermal conductivity measurements on CVD diamond, NIST is assisting in coordinating an interlaboratory comparison (round robin) of thermal conductivity measurement on a given set of specimens. The purpose of the round robin is to ascertain the most appropriate methods for measuring the thermal conductivities of chemical vapor deposited (CVD) diamond specimens. Measuring the thermal conductivity of CVD diamond can be difficult because its thermal conductivity is higher than any material at room temperature. Ten laboratories have participated to date. The results indicate a significant spread in values obtained by the different laboratories. It is believed that specimen inhomogeneity is the main cause of much of the variability. This activity is an outgrowth from a series of NIST sponsored workshops on characterizing diamond films that has been held at NIST over the past several years.

In order to provide a forum for exchange of scientific information, NIST serves as host to The Applied Diamond Conference, ADC'95, the 3rd International Conference on the Applications of Diamond Films and Related Materials. Registered attendance was 385 with participation from 21 countries. The topics covered included cutting tools, active electronics, cubic boron nitride, diamond-like carbon, economic analyses, electronic packaging, heat management, negative electron affinity, performance evaluation, sensors, optics, etc.

Outputs:

"Proceedings of the Applied Diamond Conference 1995, Applications of Diamond films and Related Materials: Third International Conference," NIST Special Publication 885 (USGPO, 1995) A. Feldman, Y. Tzeng, W.A. Yarbrough, M. Yoshikawa, and M. Murakawa editors.

NISTIR 5692, Supplement to NIST Special Publication 885, Proceedings of the Applied Diamond Conference 1995 (1995), A. Feldman, Y. Tzeng, W.A. Yarbrough, M. Yoshikawa, and M. Murakawa editors.

"The Promise of Diamond Optics," A. Feldman and D. C. Harris in *Laser Damage in Optical Materials: 1994*, H. E. Bennett, A. H. Guenther, M. R. Kozlowski, B. E. Newnam, M. J. Soileau, Editors, Proc. SPIE 2428, pp. 580-590 (1994).

"A Closed CVD System for the Deposition of Diamond Films," E. N. Farabaugh, G. R. Lai, A. Feldman, and L. H. Robins in NIST Special Publication 885, *Applications of Diamond Films and Related Materials: Third International Conference*, A. Feldman, M. Yoshikawa, Y. Tzeng, and M. Murakawa, Editors (USGPO, 1995) p. 361-364.

"Round Robin Thermal Conductivity Measurements on CVD Diamond," by A. Feldman in NIST Special Publication 885, *Applications of Diamond Films and Related Materials: Third International Conference,* A. Feldman, M. Yoshikawa, Y. Tzeng, and M. Murakawa, editors (USGPO, 1995) p. 627-630.

Outcomes:

There are two possible outcomes of this research: 1) development of a nondestructive adhesion test for diamond coated cutting tools to replace operational testing; 2) development of a new method to measure thermal conductivity/diffusivity.

PROJECT TITLE: Advanced Technology Program (ATP) Assessment and Support

PROGRAM TITLE: Other Projects

NIST Staff:

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Technical Description:

ATP general competitions which included materials topics and a focussed competition on Materials Processing for Heavy Manufacturing were held to select projects of a high risk nature which, if successful, would enhance the technological and competitive position of domestic industry. Ceramics Division staff, supported the ATP staff in development of the focussed competition and in the evaluation of proposals to both solications.

Technical Objectives:

The broad objective is to improve the capability of U.S. industry by the development of advanced materials and materials procession methods. This precompetitive research is directed toward the resolution of significant technical barriers of to the extent that industrial participants can proceed to commercialization after completion of the ATP portion of development with reduced risk.

Outcomes:

Projects selected for support address applications such as stationary power generation, solid oxide fuel cells and earth moving equipment. Materials under investigation include ceramic coatings, welded steel and aluminum components.

PROJECT TITLE: NIST Industry Fellow

PROGRAM TITLE: Other Projects

Principal investigator(s):

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Technical Description:

S. J. Dapkunas was detailed to the Pratt & Whitney Materials and Mechanics Engineering Department for a period of six months to develop an understanding of the institutional aspects of materials development in high technology industry.

Technical Objectives:

The objective of this project was to identify the barriers to transitioning a new material from laboratory concept to commercial production and to determine the potential role of NIST research in facilitating that transition.

Outcome:

Specific issues which must be addressed in the development of materials for gas turbine/jet engine applications were identified. These include timeliness for engine testing and robust processing with few steps. NIST contributions to the transition process consist of the development of immediately usable products such as Standard Reference Materials, test methods, data on material systems under development and models specific to material processing methods used by the engine industry.

Accomplishments:

A report summarizing findings was written and briefings held at NIST to inform staff and management of these results. As a consequence of this interaction, projects have been initiated to: determine the availability of specific thermodynamic data necessary for the modeling phase diagrams of superalloy systems; development of an SRM for ppb level measurement of sulfur in superalloys; and, measurement of thermal conductivity of microlaminate thermal barrier coatings.

Impact:

Projects of value to the gas turbine industry, which would not otherwise have been initiated, are underway.

RESEARCH STAFF
AND
FIELDS OF INTEREST

DIVISION OFFICE

Fogarty, Gerard	• Non-linear optical processes • Nature, genesis, distribution, and effects of irregularities in monolithic crystals and multilayers • High-resolution diffraction imaging
Freiman, Stephen W.	• Electronic ceramics • Mechanical properties • Superconductivity
Steiner, Bruce W.	• High-resolution diffraction imaging • Defects in monolithic crystals and multilayers • Non-linear optical processes

POWDER CHARACTERIZATION AND PROCESSING

Cline, James P.	• Standard reference materials • High-temperature x-ray diffraction • Microstructural effects in x-ray diffraction • Rietveld refinement of x-ray diffraction data
Hackley, Vincent A.	• Electrokinetic and electroactive measurement • Slurry rheology • Surface chemistry of powders.
Kelly, James F.	• Quantitative scanning electron microscopy • Image analysis • Microstructure analysis • Powder standards
Lum, Lin-Sien H.	• Powder characterization • Instrumental analysis
Malghan, Subhas G.	• Powder and dense slurry characterization • Colloidal processing and forming • Interfacial and surface chemistry of powders • Standards development
Minor, Dennis B.	• Analytical scanning electron microscopy of ceramics and particulates

	• Powder test sample preparation • Powder characterization
Pei, Patrick T.	• Spectroscopic and thermal characterization • Chemical coating • Powders characterization
Ritter, Joseph J.	• Chemistry of powders synthesis • Specialty powders synthesis • Nanomaterials
Wang, Pu Sen	• Solid-state nuclear magnetic resonance • Spectroscopic characterization

<u>MECHANICAL PROPERTIES</u>

Braun, Linda M.	• Raman stress measurements • Ceramic matrix composites • Toughening mechanisms
Carter, Craig W.	• Materials thermodynamics • Advanced mathematical and computational techniques • Computation of materials processes
Chuang, Tze-Jer	• Creep/creep rupture • Fracture mechanics • Finite-element modeling • Lifetime predictions
Fuller, Edwin, R., Jr.	• Influence of microstructure on fracture • Toughening mechanisms • Microstructural modeling and simulation
Gettings, Robert J.	• Mechanical properties • Hardness and fracture toughness
Hockey, Bernard J.	• Electron microscopy • High-temperature creep
Jahanmir, Said	• Ceramic machining • Mechanisms of material removal • Mechanics of contacts • Effects of machining on mechanical properties

Kauffman, Dale A.	•	Glass melting
Krause, Ralph F., Jr.	•	Creep in flexure and tension
	•	Fracture mechanics
	•	Hot pressing
	•	Chemical thermodynamics
Quinn, George D.	•	Mechanical property test standards
	•	Standard reference materials
	•	Creep testing
Roosen, Andrew J.	•	Microstructural modeling
	•	Computer simulation
Selinger, Robin L.	•	Microstructural modeling
	•	Computer simulation
Smith, Douglas T.	•	Surface forces
	•	Charge transfer at interfaces
	•	Adhesion and friction
	•	Colloidal processing
Wallace, Jay S.	•	Processing and microstructure
	•	Silicon nitride densification
	•	Thermal analysis

PHASE EQUILIBRIA

Burton, Benjamin P.	•	Calculated phase diagrams
	•	Dielectric ceramics
Cook, Lawrence P.	•	High-temperature chemistry
	•	Phase equilibria
McCormack, Ryan P.	•	Phase Equilibria Calculations
Vanderah, Terrell A.	•	Solid-state chemistry
	•	Phase equilibria of microwave dielectrics
Wong-Ng, Winnie	•	X-ray crystallography and reference patterns
	•	Phase equilibria/crystal chemistry of high-T_c superconductors
	•	Molecular orbital calculations

ELECTRONIC MATERIALS

Blendell, John E.	• Ceramic processing and clean-room processing • Sintering and diffusion controlled processes • Processing high-T_c ceramic superconductors • Activation chemical analysis
Bonnell, David W.	• Computer automation • Molecular-beam mass spectrometry • Thermodynamic modeling • Laser/plasma sputtering
Chiang, Chwan K.	• Electronic ceramics • Thermoelectric power measurements • Electrical measurements
Gonzalez, Eduardo	• Ceramic Processing • Nano-scale sintering • Texture analysis
Hastie, John W.	• High-temperature chemistry • Phase equilibria thermochemistry • Molecular-beam mass spectrometry • Thin-film deposition • Vapor deposition process control and modeling
Hill, Michael D.	• Mechanical properties of PZT • Ceramic processing
Paul, Albert J.	• Laser physics • Plasma diagnostics • Laser spectroscopy • Laser mass spectroscopy • Gasdynamic modeling
Piermarini, Gasper J.	• Ceramic processing and high-pressure sintering • Pressure-induced transformation toughening • High-pressure physical properties and structures • High-pressure x-ray diffraction and spectroscopy
Sessoms, Arthur B.	• Molecular beam mass spectroscopy • Thin film deposition

Schenck, Peter K.	• Emission and laser spectroscopy • Thin-film deposition • Computer graphics and image analysis • Laboratory automation • Plasma monitoring and control
Vaudin, Mark D.	• Electron microscopy • Microscopy and diffraction studies of interfaces • Computer modelling of grain-boundary phenomena • Dielectric films
White, Grady S.	• Thin films • Nondestructive evaluation • Subcritical crack growth • Stress measurements • Cyclic fatigue

OPTICAL MATERIALS

Bouldin, Charles E.	• X-ray absorption spectroscopy • Diffraction anomalous fine structure • GeSi heterojunction bipolar transistors
Farabaugh, Edward N.	• Chemical vapor deposition of diamond • Structure and morphology analysis • Scanning electron microscopy
Feldman, Albert	• Chemical vapor deposition of diamond • Thermal properties • Modelling thermal wave propagation • Thin-film optical properties
Kaiser, Debra L.	• Physical properties and structures of high-temperature superconductors • Interfaces in high-temperature superconductors • Chemical vapor deposition of ferroelectric oxide thin films
Robins, Lawrence H.	• Defect identification and distribution • Cathodoluminescence imaging and spectroscopy • Photoluminescence spectroscopy

	•	Raman spectroscopy
Rotter, Lawrence D.	•	Measurement of electro-optic coefficients
	•	Photorefractive effect
	•	Optical spectroscopy of thin films

MATERIALS MICROSTRUCTURE CHARACTERIZATION

Allen, Andrew J.	•	Small angle x-ray scattering
	•	Ceramic microstructure analysis
Black, David R.	•	Defect microstructure in diamond
	•	Polycrystalline diffraction imaging
	•	X-ray imaging of photonic materials
Burdette, Harold E.	•	X-ray optics
	•	X-ray diffraction imaging
	•	Crystal growth
	•	Instrumentation
Fischer, Daniel A.	•	X-ray absorption fine structure
	•	X-ray scattering
	•	Surface science
Long, Gabrielle G.	•	Small-angle x-ray and neutron scattering
	•	Ceramic microstructure evolution as a function of processing
	•	X-ray optics
Spal, Richard D.	•	X-ray optics
	•	Diffraction physics
	•	X-ray scattering
Woicik, Joseph C.	•	UV photoemission
	•	X-ray standing waves
	•	Surface and interface science

SURFACE PROPERTIES

Gates, Richard S.	•	Tribo-chemistry
	•	Surface chemical properties of ceramics

Hsu, Stephen M.	• Ceramic wear mechanisms • Engineered ceramic surfaces • Lubrication and machining of ceramics
McGuiggan, Patricia M.	• Microtribology • Surface force measurement
Ives, Lewis K.	• Wear of materials • Transmission electron microscopy • Machining of ceramics
Ruff, Arthur W.	• Wear of materials • Microstructure effects • Mechanical behavior

DATA TECHNOLOGIES

Begley, Edwin F.	• Database management methods • Engineering database structures • Digital video interactive technology
Carpenter, Joseph A., Jr.	• Functional ceramics applications • Technical assessments • Industrial liaisons
Cellarosi, Mario	• Glass standards
Clevinger, Mary A.	• Phase diagrams for ceramists • Computerized data
Dapkunas, Stanley J.	• Structural ceramics applications • Technical assessments • Coatings
Harris, Joyce F.	• Data acquisitions • Digitization and data entry
Munro, Ronald G.	• Materials properties of advanced ceramics • Data evaluation and validation • Analysis of data relations

GUEST SCIENTISTS AND GRADUATE STUDENTS

Armstrong, Nicholas	University of Technology, Sydney
Bell, Michael I.	Naval Research Laboratory
Belmonte, Manuel	Autonoma University, Spain
Bernik, Slavko	University of Ljubljana
Blackburn, Douglas	Consultant
Brower, Daniel	Optex Corporation
Bogatin, Oleg	Institute of Nonmetallic Materials
Cedeno, Christina	American Ceramic Society
Chen, Hailong	University of Tokyo
Chu, Steven	State University of New York at Stony Brook
Cordero Cabrera, Mario	Centro National de Metrologia (CENAM)
Dai, Yongshan	Smithsonian Institute
Diao, Jianli	University of Maryland
Domingues, Louis	Trans-Tech, Inc.
Drews, Andrew R.	Naval Research Laboratory
Fischman, Gary	FDA
Frederiske, Hans	Consultant
Green, Thomas	American Ceramic Society
Haller, Wolfgang	Abbott Laboratories
Harmer, Martin	Lehigh University

Hayward, Evans	American Ceramic Society
Hill, Kimberly	American Ceramic Society
Hwang, Cheol-Seong	Seoul National University
Ilavsky, Jan	State University of New York at Stony Brook
Jemian, Peter	University of Illinois at Urbana/Champaign
Jiang, Feng	University of Maryland
Job, Lenox	University of Maryland
Kerch, Helen	Department of Energy
Kim, Bong-Ho	Changwan National Laboratory
Krebs, Lorrie	Johns Hopkins University
Kruger, Jerome	Johns Hopkins University
Kubler, Jakob	Swiss Federal Research Laboratories
Larsen-Basse, Jorn	National Science Foundation
Lee, Byeong	Hanyang University
Levinson, Lionel	General Electric
Liu, Daqing	Columbia University
Liu, Haiyan	University of Maryland
Loezos, John M.	University of Maryland
Maruthamuthu, Pichai	University of Madras, India
Maskara, Alok	University of New Mexico
McMurdie, Howard	Joint Center for Powder Diffraction Studies
Mobley, Morris	Alfred University

Nagarajan, Mala	University of Maryland
Nagarajan, Venkatta	Indian Institute of Science
Ondik, Helen	American Ceramic Society
Orlando, Stefano	Instituto Materiali speciali del (CNR)
Paik, Ungyu	Changwan National Laboratory
Paulik, Steven	Northwestern University
Perez, Joseph	Penn State University
Peterson, Marshall	Wear Sciences
Pothier, Brian	Army Research Laboratory
Pradip	Tata Research Development Design Centre
Premachandran, Ramannair Sarasamma	Indian Institute of Technology, Madras
Raman, Rajkumar	BDM Federal
Ritchie, Kevin	University of Maryland
Roberts, Ellis	Consultant
Roth, Robert	Viper Group
Russell, Thomas	Naval Surface Warfare Center
Shechtman, Dan	Johns Hopkins University
Shen, Ming	University of Illinois
Smith, Wallace	Office of Naval Research
Swab, Jeffrey	Army Research Laboratory
Swanson, Nils	American Ceramic Society
Natarajan, Venkata	Indian Institute of Science

Wachtman, John	Consultant
Wang, Jun	University of Maryland
Wang, Tong	Jiao Tong University
Wang, Yen	University of Illinois
Wang, Yinglong	University of Maryland
Wei, Lanhua	Wayne State University
Xu, Huakun	University of Maryland
Yeheskel, Jacob	Nuclear Research Center of the Negev
Ying, Tsi-Neng	University of Maryland
Zhang, Guangming	University of Maryland
Zhang, Jun	Lanzhou Institute of Chemical Physics

OUTPUTS AND INTERACTIONS

CERAMICS DIVISION PATENTS

1995

Process for Multilayer Ceramic Modules (MLC) (D)	M. Cellarosi
A Method of Obtaining High Green Density Ceramics from Powders (P)	S. G. Malghan R. S. Premachandran
Chemically Assisted Process for the Machining of Ceramics U.S. Patent No. 5,447,466, September 5, 1995	J. Wang S. M. Hsu

1994

A Process for the Controlled Preparation of a Composite of Ultra-fine Magnetic Particles Homogeneously Dispersed in a Dielectric Matrix, U. S. Patent No. 5,316,699, May 31, 1994	J. J. Ritter
A Process for the Chemical Preparation of Bismuth Telluride and Bismuth Telluride Composite Thermoelectric Materials (P)	J. J. Ritter
A Method for Obtaining High Density Green Ceramics (P)	S. G. Malghan R. S. Premachandran
Method of Adhering Substrates, U.S. Patent 5,368,942 November 29, 1994	D. T. Smith A. Grabbe R. G. Horn D. E. Deckman
Methods of Reducing Wear on SiC Ceramic Surfaces U. S. Patent No. 5,344,577, Sept. 1994	S. M. Hsu
Ultraviolet Faraday Rotator Glass (D)	Dexter Cooper D. G. Blackburn D. A. Kauffman D. C. Cranmer
A Method of Obtaining High Green Density Ceramics from Powders (P)	S. G. Malghan R. S. Premachandran

1993

Oxygen-Containing Organic Compounds as Boundary Lubricants for Silicon Nitride Ceramics (D)	R. S. Gates S. M. Hsu
Method of Producing a Smooth Plate of Diamond U. S. Patent No. 5,221,501, July 25, 1993	A. Feldman E. N. Farabaugh
Asymmetric Bragg diffraction microscope U. S. Patent No. 5,259,013, November 2, 1993	R. Spal
Method for Fabrication of Dense Compacts from Nanosize Particles Using High pressures and Cryogenic Temperatures (P)	A. Pechenik G. J. Piermarini
Friction and Wear Resistant Coating for Titanium and Its Alloys (D)	S. M. Hsu J.-M. Gu
A Cutting Fluid Additive for Machining of Ceramics (D)	S. Jahanmir G. Zhang

(D) = INVENTION DISCLOSURE
(P) = PENDING DECISION BY PATENT OFFICE
(A) = INVENTION ALLOWED BY PATENT OFFICE

CONFERENCES AND WORKSHOPS SPONSORED

International Workshop on Instrumented Indentation
Sponsors: NIST Standard Reference Materials Program (SRMP) and Institute for Mechanics and Materials (IMM)
Attendance: 96
April 22-23, 1995, San Diego, CA
Organized by: D.T. Smith, NIST

Workshop in Rational Design and Processing of Advanced Ceramics
Sponsors: NIST Center for Theoretical and Computational Materials Science (CTCMS) and Institute for Mechanic and Materials (IMM)
Attendance: approximately 70
June 21-23, 1995, La Jolla, CA
Organized by: Edwin R. Fuller, Jr. NIST, Joanna M. McKittrick, University of California, San Diego, and Tze-Jer Chuang, NIST

Objective: To elucidate the new paradigm of mesoscopic compute simulations in the microstructural design and processing of advanced ceramic materials and to forge collaborations between experimental and computational material scientists to develop efficient stratagems for tailoring and processing advanced materials and components.

Workshop on Large Scale computational Issues in Materials Science
Sponsors: NIST Center for Theoretical and Computational Materials Science (CTCMS)
Attendance: approximately 50
August 28-30, 1995, Gaithersburg, MD
Organized by: W. Craig Carter, NIST and Rob Almgren, University of Chicago

Symposium on Fracture: Instability Dynamics, Scaling, and Ductile/Brittle Behavior
Symposium Q, 18995 Fall Meeting, Materials Research Society (MRS)
Sponsors: Materials Research Society and Office of Naval Research (ONR)
Attendance: approximately 300
November 27 to December 1, 1995, Boston, Massachusetts
Organizers: Edwin R. Fuller and Robin Selinger, NIST

Objective: To bring together diverse groups from physics theory, experimental materials science, and computer simulations to discuss common research in fracture behavior: dynamical instabilities in crack propagation; scaling phenomena and fractal geometry in both brittle and ductile materials; and atomic scale mechanisms/criteria for brittle vs. ductile behavior in bulk crystalline solids and at interfaces.

Workshop on High Temperature Superconductor Phase Diagrams II
Sponsor: NIST
Attendance: approximately 50
October 31 to November 2, 1994, Gaithersburg, MD
Organizers: Terrell A. Vanderah, NIST

Workshop on Materials and Measurements for Wireless Communications
Sponsor: NIST
Attendance: approximately 50
November 7-8, 1995, Gaithersburg, MD
Organizers: Terrell A. Vanderah, NIST

Applications of Diamond Films and Related Materials: Third International Conference
Sponsor: NIST
Attendance: approximately 200
August 21-24, 1995, Gaithersburg, MD
Organizers: A. Feldman, NIST

Workshop on Large-Scale Computation and Realistic Microstructure
Sponsor: NIST
Attendance: approximately 25
August 28-30, 1995
Organizers: Craig Carter, NIST

Objective: To advance the state of the art in techniques for computing realistic microstructures in solidification problems.

STANDARD REFERENCE MATERIALS

The Division provides science, industries, and government a central course of well characterized materials certified for chemical composition of physical or chemical properties. These materials are issued with a certification and are used to calibrate instruments, to evaluate analytical methods, or to produce scientific data which can be referred to a common base.

DESCRIPTION	SRM NUMBER
Abrasive Wear	1857
Alumina Glass Anneal Point	714
Alumina Melting Point	742
Barium Glass Anneal Point	713
Borosilicate Glass Composition	93A
Borosilicate Glass Thermal Expansion	731
Calibrated Glass Beads	1003B
Calibrated Glass Beads	1004A
Calibrated Glass Spheres	1017B*
Catalyst Package for Engine Simulation (IIID)	1817
Catalyst Package for Engine Simulation (IIID)	1817C
Catalyst Package for Engine Simulation (IIIE)	8501
Catalyst Package for Lubricant Oxidation	2567*
Chlorine in Base Oil	1818A
Container Glass Composition	621
Container Glass Leaching	623
Copper Thermal Expansion	736L1
Fused Silica Thermal Expansion	739L1
Glass Analytical Standard	1835
Glass Dielectric Constant	774
Glass Electrical Resistance	624
Glass Fluorescence Source	477
Glass Liquidus Temperature	773
Glass Refractive Index	1820
Glass Sand (High Iron)	81A
Glass Sand (Low Iron)	165A
Glass Stress Optical Coefficient	709
High Boron Glass Viscosity	717
Intensity XRD Set	674A
Lead Barium Glass Composition	89
Lead Glass Anneal Point	712
Lead-Silica Glass High Temperature Resistivity	1414
Lead Glass Viscosity	711

Line Profile	660
Low Boron Glass Composition	92
Mica X-Ray Diffraction	675
Neutral Glass Anneal Point	716
Opal Glass Composition	91
Particle Size Distribution Standard	1978
Refractive Index Glass	1822
Respirable Cristobalite	1879
Silicon X-Ray Diffraction	640
Silicon Nitride Particle Size	659
Silicon Nitride Powders for Quantitative Analysis by Powder Diffraction	656*
Soda Lime Flat Glass Composition	S620
Soda Lime Float Composition	1830
Soda Lime Float Viscosity	710A
Soda Lime Sheet Composition	1831
Soda-Lime-Silica Glass	710A
Sulfur in Oil	1819A
Toluene 5 ML	211C
Total Nitrogen in Oils	1836
Wear Metals in Oil	1084A
Wear Metals in Oil	1085A
X-Ray Diffraction Instrument Sensitivity	1976
X-Ray Diffraction Intensity	676
X-Ray Diffraction Intensity Set	674A

*New in FY-1995

SELECTED TECHNICAL/PROFESSIONAL COMMITTEE LEADERSHIP

Advanced Photon Source
Steering Committee
G. Long, Member

Alfred University
Center for Advanced Ceramics
S. Freiman, Member of External Advisory Board.

American Association for the Advancement of Science
Physics Section
B. Steiner, Representative of the Optical Society of America

American Ceramic Society
Glass Division
Committee on Glass Standards Classification and Nomenclature
M. Cellarosi, Chairman
Editorial Committee
S. Wiederhorn, Subchairman
Basic Science Division
E. Fuller, Vice-Chair
Editorial Committee
B. Lawn, Chairman

ASM International
Energy Division
S. Dapkunas, Past Chairman, Division Council Member
Journal of Materials Engineering and Performance
S. Dapkunas, Editorial Board
Journal of Thermal Spray Technology
S. Dapkunas, Editorial Board
Washington D.C. Chapter Yearbook Committee
J. A. Carpenter, Jr., Chairman

American Society for Engineering Education
Postdoctoral Review Committee
A. Feldman, Member

American Society for Testing and Materials
C14: Glass and Glass Product
M. Cellarosi, Chairman
G. Quinn, Member

American Society for Testing and Materials
C14.01: Nomenclature of Glass and Glass Products
M. Cellarosi, Chairman
C28. Advanced Ceramics
G. D. Quinn, Chairman
C28.05: Powder Characterization
S. Malghan, Working Group Chairman
D2: Petroleum Products and Lubricants
S. M. Hsu, Member
E29.01: Advanced Ceramics, Organizational Meeting
M. Cellarosi
E42: Surface Science
G. G. Long, Member
E49: Computerization of Material and Chemical Property Data
R. G. Munro, E. F. Begley, Members
E-08: Fracture & Fatigue
G. Quinn, Member
F-04: Medical and Surgical Materials and Devices
G. Quinn, Member
Fl:02: Lasers
A. Feldman, Subcommittee Editor

American Society of Mechanical Engineers
Research Committee on Tribology
S. Jahanmir, Member
S. Hsu, Member
Subcommittee on Tribology in Manufacturing
S. Jahanmir, Chairman

Applied Diamond Conference
A. Feldman, Executive Committee

Ballistic Missile Defense Organization Technology Applications Office (BMDO/TA)
Materials and Electronics Panel
J. Carpenter, Member

Center for Advanced Ceramic Technology, Alfred University
S. W. Freiman, Member of Oversight Committee

Diamond Films and Technology
    Editorial Advisory Board
        A. Feldman, Member

Dow Chemical Company Synchrotron Radiation Spectroscopy Team
        D. A. Fischer, Member and Outside Advisor

Federation of Materials Societies
        D. L. Kaiser, Trustee

High Temperature Science Journal
        J. W. Hastie, Editorial Board Member

IEEE Lasers and Electrooptics Society
    Washington-Northern Virginia Chapter
        B. Steiner, Treasurer

IGES/PDES Organization
    Materials Committee
        J. Carpenter, Chairman

International Center for Diffraction Data
    High Tech Materials Task Group
        W. Wong-Ng, Chairman
    Ceramic Subcommittee
        W. Wong-Ng, Chairman
    Editorial Board, Powder Diffraction File
        W. Wong-Ng, Consulting

International Conference Series on High Temperature Materials Chemistry
    Organizing Committee
        J. W. Hastie, Member

International Energy Agency
    Subtask 8 International Standards
        S. Malghan, Overall Task Leader on Powder Characterization
    Subtask 8 Powder Characterization Subgroup
        S. Malghan, U. S. Task Leader

International Standards Organization (ISO)
    Technical Committee 206 Fine Ceramics
    Working Group 1, Particle Size
        S. Malghan, Member

Working Group 2, Flexure Strength
G. Quinn, Chairman
Working Group 3, Hardness
G. Quinn, Member

Technical Committee 184 Industrial Automation Systems and Integration,
Subcommittee 4 Industrial Data and Global Manufacturing Programming Languages
Working Group 3, Project 4 Materials
J. Carpenter, Jr., Chairman

International Union of Pure and Applied Chemistry Commission II-3
High Temperature and Solid State Chemistry
J. W. Hastie, Titular Member

Journal of Non Crystalline Solids
Editorial Board
S. Freiman, Member

Journal of Physical Chemical Reference Data
D. W. Bonnell, Editorial Board Member

Journal of Smart Materials
Editorial Board
S. Freiman, Member

Materials Research Society
G. Long, Council

Minerals and Metallurgical Processing Journal
Editorial Board
S. G. Malghan, Member

National Materials Advisory Board, National Academy of Sciences
Committee on Superhard Materials
A. Feldman, Member
Committee on High Temperature Coatings
S. J. Dapkunas, Member

National Science Foundation
Instrumentation Development Advisory Panel
D. A. Fischer, Member
Young Investiators' Award Selection Panel
J. A. Carpenter, Jr., Member

National Synchrotron Light Source
Housing Committee
C. E. Bouldin, Member
User Executive Committee
Daniel Fischer, Chairman
EXAFS Special Interest Group
C. E. Bouldin, Chairman
Proposal Study Panel on Imaging
B. Steiner, Member
Housekeeping Committee
J. C. Woicik, Member

Organizing Committee for Annual Users' Meeting
D. A. Fischer, Member

NIST Cold Neutron Research Facility
G. G. Long, Program Advisory Committee Member

Powder and Bulk Engineering Journal
S. Malghan, Member Editorial Advisory Board

Powder X-Ray Diffraction Data
W. Wong-Ng, Consulting Editor

Research Advisory Committee (RAC)
G. Long, Chairman

Society of Photooptical Instrumentation Engineers
Kingslake Award Committee
B. Steiner, Member

Society of Tribologists and Lubrication Engineers
Education Committee
S. Jahanmir, Member
Fellows Committee
S. Jahanmir, Member

Stevens Institute of Technology
Materials Science and Eng. Dept.
S. Jahanmir, Member of Advisory Board

Superconductor Applications Association
E. Fuller, Jr., Member of Advisory Board

Tribology International
Editorial Advisory Board
S. Jahanmir, Member

Tribology Letters
Editorial Advisory Board
S. M. Hsu, Member

University of Delaware
Dept. of Mechanical Engineering
S. Jahanmir, Member of Advisory Board

University of Florida Fine Particles Research Center
S. G. Malghan, Member, Advisory Council

U. S. Department of Energy
Review Panel for Office of Basic Energy Sciences on "Structural Ceramics and Mechanical Behavior of Ceramics: Emphasis on Mechanical Behavior"
E. Fuller, Jr., Chairman
Review Panel for Office of Basic Energy Sciences' Materials Science Program in X-ray Absorption Spectroscopy
D. A. Fischer, Member

U. S. Bureau of Mines Communication Research Center
S. G. Malghan, Member, Advisory Panel

Versailles Project on Advanced Materials and Standards (VAMAS)
Technical Working Area on Ceramics
G. D. Quinn, U. S. Representative and International Chairman
Technical Working Area on Wear Test Methods
S. Jahanmir, Co-chairman
Technical Working Area on Materials Databanks
J. Carpenter, Member

INDUSTRIAL AND ACADEMIC INTERACTIONS

INDUSTRIAL

ACTIS, Inc.

An agreement exists between NIST and ACTIS, Inc. for a joint research and development activity related to comprehensive computerized tribology databases. These databases are evaluated by NIST and marketed by ACTIS, Inc. Other participants in the program are DOE, U.S. Army, U.S. Air Force, ASME and STLE.

Advanced Technology Materials

G. Stauf and P. Van Buskirk of ATM are collaborating with C.-S. Hwang, L. H. Robins, L. D. Rotter, M. D. Vaudin and D. L. Kaiser in studies of the processing/structure/property relationships in $BaTiO_3$ thin films deposited by MOCVD.

Specimens of silicon carbide single crystal provided by ATM have been examined for defect content by cathodoluminescence (L. H. Robins) and x-ray diffraction imaging (D. R. Black).

Raman spectroscopy was used to characterize the structure of barium titanate thin films grown by Advanced Technology Materials, EMCORE, and NZAT. Interactions with these companies were initiated by D. L. Kaiser and L. D. Rotter in conjunction with their work (see above) on the MOCVD growth and electro-optic properties of barium titanate films (see above).

Allied Signal Corporation

S. G. Malghan is conducting collaborative studies with J. Pollinger of Allied Signal Ceramic Components on the interactions of powder-binder-sintering aid in gelcasting of silicon nitride powders.

Aldrich Chemical Company

J. J. Ritter provided Aldrich with documentation on the use of chemical flow reactors to generate commercial quantities of YBCO precursor powders.

American Ceramic Society

P. Schenck is collaborating with the American ceramic Society (ACerS) and the industrial sponsors of the NIST-ACerS phase diagram optimization program, in the development of a graphical phase diagram database for ceramic and other inorganic systems.

American Superconductor

M. D. Vaudin is involved in a collaboration to investigate by means of x-ray diffraction techniques the preferred crystallographic orientation of BiSSCO 2223 superconductor in Ag-swaged wires and tapes.

Flux flow in silver-sheathed BiSCCO 2223 wires produced by American Superconductor is being correlated with microstructure by means of a novel magneto-optical imaging technique used by M. Turchinskaya and D. L. Kaiser.

R. D. Spal is studying the effect of mechanical strain on the electrical properties of silver-sheathed bismuth superconductor (BSCCO-2223) tapes.

The efforts above are parts of a larger project to investigate correlations between the mechanical and electrical properties of the BiSSCO wires and tapes the textures induced in them by fabrication.

American Xtal Technology, Inc.

Collaboration in the characterization of the crystalline order of gallium arsenide substrates grown with a greatly increased degree of regularity by a new commercial process, vertical gradient freeze, is being carried out by B. W. Steiner with M. Young of AXT.

Argonne National Laboratory

U. Balachandran of ANL collaborates with W. Wong-Ng in the melting characterization of the high-T_c 2223 phase in the Bi(Pb)-Sr-Ca-Cu-O system.

AT&T Bell Laboratories

J. C. Woicik collaborates with C. King of Bell Labs on the growth, characterization and the consequences of strain in SiGe and other group IV semiconductor quantum structures, and with J. Patel on standing-wave x-ray studies of Pb on Ge.

G. S. White, L. M. Braun, W. C. Carter, and E. R. Fuller, Jr. are collaborating with Bell Labs on lifetime predictions for InP in water and in 50% RH.

T. Vanderah collaborates with T. Siegrist of Bell Labs in the structural characterization of microwave dielectric ceramics.

Battelle Columbus Laboratories

W. Glaeser at Battelle and A. W. Ruff are involved in a joint activity to prepare a wear atlas from selected literature and research findings at Battelle Columbus Laboratories, NIST, and the German Bundesanstalt fur Materialprufung.

Brookhaven National Laboratory

B. W. Steiner and G. Fogarty are collaborating on the characterization of crystalline order and its relation to the magnetic coupling in rubidium calcium trifluoride crystals with J. Hill of Brookhaven National Laboratory.

Caterpillar Company

The Surface Properties Group at NIST is working with Caterpillar Company in several areas. F. Kelly of Caterpillar is working with S. M. Hsu in the area of advanced lubrication, diesel particulate reduction and engine simulations. He is also working with A. W. Ruff to improve the wear resistance of tractor under-carriage linkage. S. M. Hsu is also working with K. Bruk, B. Hockman, and R. Nevinger of Caterpillar on the design of ceramic valve seat inserts.

Ceradyne, Inc.

J. Mangles of Ceradyne collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Cincinnati Milacron, Inc.

C. Yoon of Cincinnati Milacron collaborates with S. Jahanmir in the Ceramic machining Consortium.

Containerless Research, Inc.

D. Bonnell is collaborating with S. Krishnan of Containerless Research regarding levitation of liquid metals, pyrometric, emissivity, and laser ellipsometric measurements at high temperatures, and related studies.

Continuous Electron Beam Accelerator Facility (CEBAF)

D. Bonnell is collaborating with L. Phillips on development of new technologies for producing superconducting RF accelerator cavities as part of the goal of a world-wide consortium of government, academic and industrial groups. In addition, there is also a joint interest in possible adoption of NIST-developed PLD deposition techniques as an attractive alternate route to fabricating niobium alloy-based thin film superconducting RF cavities.

Corning, Inc.

Merle Adler of Corning collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Crystacomm

Characterization of defects in InP wafers is carried out in a collaboration between G. Antypas of Crystacomm and D. R. Black.

Crystallume

L. H. Robins and D. R. Black collaborate with M. A. Plano and others in cathodoluminescence and x-ray topography characterizations of strain and defect generation in thick diamond films CVD-grown homoepitaxially on a synthetic diamond substrates.

Cummins Engine Company

S. M. Hsu is working with J. Wang, M. Naylor, and T. Gallant of Cummins Engine on the lubrication of new materials, evaluation of chemistries and development of advanced lubrication concepts for future engines. New chemistries are being developed and these chemistries are being evaluated in prototype engines by Cummins.

Deltronic Crystal Industries

G. Fogarty and B. W. Steiner collaborate in the growth and characterization of crystalline order of barium titanate and strontium barium niobate with R. Uhrin, M. Garrett, and J. Martin of Deltronic Crystal Industries.

DOW Chemical Company

G. Mitchell and B. Dekoven of Dow have begun a research collaboration with D. A. Fischer to study polymer surfaces and metal-polymer interfaces using ultra soft x-ray absorption spectroscopy. The materials studied include poly(acrylic acid), poly(butyl methacrylate), polystyrene, polycarbonate, poly(ethylene terephthalate), and model acrylic coatings.

T. Parsons of Dow collaborates with S. Jahanmir in the Ceramic Machining Consortium.

DuPont

P. Schenck, J. Hastie, D. Bonnell, and A. Paul are collaborating with D. Kountz, A. Lauder, and other members of the DuPont Superconductivity Group as part of a CRADA to develop process control technology for laser and sputter deposition of complex oxide thin films.

L. P. Cook is collaborating with DuPont in their (partially ATP-supported) research on problems in processing and characterizing thallium-containing high-T_c superconductors, especially as related to the phase equilibria of these materials and their interaction with ferroelectrics in thin-film devices.

Eagle-Picher

A collaboration has been initiated between Eagle-Picher Research Laboratory (G. Cantwell) and NIST (L. H. Robins and D. R. Black) to characterize defects and dopant impurities in zinc selenide single crystals, being developed (in part under an ATP grant) as substrates for blue-green light-emitting diodes and laser diodes.

Eaton

J. Edler of Eaton is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

EG&G Instruments

B. W. Steiner collaborates in the growth, device processing, and characterization of crystalline order of mercuric iodide with L. van den Berg of EG&G Instruments.

Eonic, Inc.

B. French of Eonic collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Exxon Research and Engineering

G. Meitzner and J. Sinfelt of Exxon are collaborating with D. A. Fischer to study the electronic structure of adsorbed carbon monoxide and hydrocarbons on platinum-supported catalysts using near-edge spectroscopy above the carbon K-edge.

Eastman Kodak Company

D. T. Smith has been collaborating informally with R. Sharma of Kodak's Polymer Research Laboratory to study the surface charging properties of thin insulating films of potential industrial interest to Kodak.

Edge Technology Inc.

Artificial diamonds to be used as machine tools were supplied to D. R. Black, who correlated (for purposes of quality control) topographic examination of the crystals to optically observed defects.

Ford Motor Company

K. Carduner and M. Rokosz of Ford Motor Company have been active with P. S. Wang in the application of NMR spectroscopy and imaging to characterization of ceramic materials. Efforts so far have involved data exchange of Si-29 CP/MAS NMR for phase composition determination of silicon nitride and carbide powders; in the future, we plan to exchange imaging capabilities.

R. Allor of the Ford Scientific Research Lab is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

Gas Research Institute

R. G. Munro continued interactions with the Gas Research Institute to provide evaluated data fo the use of advanced ceramics in heat exchangers and gas-fueled engines.

General Electric

W. Banholzer of GE has been collaborating to evaluate a single-crystal diamond wafer by spatially resolved cathodoluminescence spectroscopy (L. H. Robins) and by x-ray diffraction imaging (D. R. Black).

K. Kumar of GE Superabrasives is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

D. Hurley of the GE Corporate Research Center and D. R. Black are performing defect characterization on isotopically-controlled man-made diamond crystals. This is part of a larger study of the relationship of defects to ultrasonic measurements on these materials.

V. Krishnamurthy of the GE Corporate Research Center and J. J. Ritter explored the potential of the Cyclic Dip and Fire process as a low-cost approach to apply ferrite films for use in integrated microwave circulator devices.

General Motors Corporation

R. Fouts of GM's AC Delco Systems collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Hauptman-Woodward Medical Research Institute, Inc.

George DeTitta is the co-investigator with W. Wong-Ng for the development of a standard reference material for the alignment of single crystal x-ray diffractometers.

IBM, Almaden Research Laboratory

W. Wong-Ng interacts with T.C. Huang of IBM on investigations of the x-ray property of PZT and $BaTiO_3$ thin films prepared at NIST using the laser deposition technique.

International Centre for Diffraction Data

W. Wong-Ng is Chairperson of the Ceramics Subcommittee and the High-Tech Materials Task Group of the ICCD. Efforts have been initiated to organize the inorganic materials of the

X-ray Powder Diffraction File (PDF) into minifiles according to their functions, properties or structure. W. Wong-Ng also serves as a consulting editor for the PDF.

Johns Hopkins University Applied Physics Laboratory

D. Wickenden of the JHUAPL is collaborating with L. H. Robins in a project for characterizing defects and dopant impurities in thin films of gallium nitride and $Al_xGa_{1-x}N$ by spatially resolved CL. These materials are being developed for short-wavelength laser and LED devices. The specimens, grown via MOCVD at JHUAPL, are being characterized at NIST.

D.T. Smith is collaborating with R. Cammarata on mechanical properties measurements (using nanoindentation) of nanoscale iron films.

Kennemetal Inc.

S. M. Hsu is cooperating with the machining group at Kennemetal (R. F. Upholster) on jointly developing chemically assisted technology of ceramics. Samples were exchanged and many discussions were held. Some of the more promising chemistries may be tested at their facility.

G. D. Quinn, L. K. Ives, R. Gettings and A. W. Ruff have collaborated with Kennemetal in modeling tungsten carbide disks which will be used as Vickers hardness standard reference materials.

Kobe Steel Electronic Research Center

Characterization of defects in natural diamond substrates and in doped homoepitaxial diamond films is currently the subject of a collaboration between D. R. Black and B. Fox of Kobe Steel.

Lawrence Livermore Laboratories

As part of a study with P. Johnson of Lawrence Livermore Laboratories (LLL) of the cohesive strength of grain boundaries in Ni_3Al as a function of grain boundary misorientation and symmetry, alloy stoichiometry and boron concentration, grain orientations in test bars have been measured by M. D. Vaudin) prior to mechanical testing.

Collaboration on the crystal growth of potassium dihydrogen phosphate for high power lasers is being carried out by B. W. Steiner with J. DeYoreo and C. Ebbers of LLL.

Marlow Industries

J. J. Ritter has held discussions with Marlow, a major U.S. supplier of thermoelectric elements, to disclose chemical methods used to synthesize and modify bismuth telluride-based thermoelectric materials.

Matec Applied Sciences

This cooperative research is related to the development of electrokinetic sonic amplitude measurement for dispersion of powders in dense slurries. Research at NIST under the direction of S. G. Malghan will be utilizing hardware and software developed by Matec Applied Sciences for on-line measurement of dispersion.

Morgan-Matroc

In collaboration with Morgan-Matroc, damage in PZT-8 due to cyclic loading has been investigated. Morgan-Matroc supplied the specimens and information regarding their piezoelectric properties and G. S. White, M. D. Hill, C.-S. Hwang provided information on the mechanical response to loading.

Nanophase Technology Inc.

J. Parker of Nanophase Technology is collaborating with G. G. Long and S. Krueger (Reactor Radiation Division) in the investigation of microstructure evolution during densification of nanophase ceramics.

NASA Consortium for Commercial Development

Cooperative research is aimed at understanding fundamentals of zeolite nucleation, growth of CdTe single crystals and strain development during growth of GaAs. G. G. Long, D. R. Black, H. E. Burdette, and S. Krueger (Reactor Radiation Division) are working with E. Coker, H. Wiedemeier and D. Larson of the consortium on this research.

National Institute of Statistical Sciences (NISS)

A collaboration between NISS, the Ceramics Division, and the (NIST) Statistical Engineering Division has explored cross-disciplinary applications of statistics and statistical concepts to materials science and the formulation of a research agenda leading to high-impact advances. An outgrowth of this collaboration was a workshop, *Statistics and Materials Science: Microstructure - Property - Performance Relations*, which was held at NIST on July 26-28, 1993.

National Renewable Energy Laboratory

Carbon films deposited by R. Pitts via solar methods at the National Renewable Energy Laboratory were examined by L. H. Robins for diamond content by Raman spectroscopy.

National Research Institute for Metals (Japan)

R. G. Munro and J. R. Rumble, Jr (NIST Office of Standard reference Data) are collaborating with S. Nishijima, Y. Asada and K. Hoshimoto of NRIM on development of a comprehensive materials property data base for high-T_c superconductors.

J. A. Carpenter, Jr. participates with Y. Monma of NRIM and G. Dean of the National Physical Laboratory (United Kingdom) in the VAMAS Inventory of Data Reduction Models Project.

Naval Research Laboratory

S. Lawrence and B. Bender of NRL are collaborating with J. S. Wallace on the thermochemical treatment of polymer-derived SiC fibers and the degradation mechanisms of these fibers during high temperature heat treatments.

M. D. Hill and G. S. White are is collaborating with Dr. Sadananda at NRL investigating cyclic fatigue of piezoelectric material. Mechanical properties are investigated at NIST and TEM is done at NRL.

B. W. Steiner collaborates with M. Bell and R. Whitlock of NRL in a study of the role of crystalline irregularities on the performance on quartz-crystal resonators.

T. A. Vanderah collaborates with M. Osofsky and other physicists in the Materials Science and Technology Division of NRL in the crystal growth and characterization of high-temperature superconductors and microwave dielectric ceramics.

D. L. Kaiser and T. A. Vanderah are collaborating with E. F. Skelton and S. B. Qadri of NRL to study structural inhomogeneities in superconducting $YBa_2Cu_3O_{7-x}$ single crystals by means of high spatial resolution x-ray diffraction.

Naval Surface Warfare Center

Tom Russell of NSWC has been working with G. J. Piermarini on the study of energetic materials and Buckminsterfullerenes at high pressure.

A joint research project with D. R. Black has been developed to supply and characterize copper single crystal substrates for use as substrates for heteroepitaxial growth of diamond films.

Northrop Grumman

D. R. Black, H. E. Burdette and G. G. Long are collaborating with D. Larson of Northrop Grumman on the characterization of earth-grown and space-grown CdZnTe crystals.

NZ Applied Technologies Corporation

G. Fogarty and B. W. Steiner collaborate with J. Zhao of NZ and M. Cronin-Golomb of Tufts University in the characterization of crystalline order of SBN layers grown on magnesium oxide.

L. D. Rotter has performed electro-optic, poling and second harmonic generation studies on $BaTiO_3$ and $Sr_xBa_{1-x}NbO_6$ (SBN61) thin films grown by J. Zhao of NZ.

Oak Ridge National Laboratory

A Collaborative Access Team (CAT) agreement has been established between MSEL (G. G. Long), the University of Illinois at Urbana-Champaign (H. Chen) and the UOP Research Corporation on the design and construction of a beamline at the Advanced Photon Source (APS) at Argonne National Laboratory.

OPTEX Inc

W. Wong-Ng collaborated with ATP-funded company OPTEX of Rockville, Maryland, on x-ray diffraction studies of undoped and doped optical SrS thin films for optical data storage, to characterize these films for correlations with processing parameters.

L. H. Robins uses CL spectroscopy to detect rare-earth ions in rare-earth doped SrS:(Eu,Sm) thin films prepared by OPTEX. The goal of this project is to better understand how the rare-earth ions, as well as other impurities and defects, determine the optical emission properties of the films, and to correlate the optical emission properties with film preparation conditions.

Polaroid Corporation

P. K. Schenck is collaborating with K. S. Kim of Polaroid to study the mechanism of laser ablation transfer being utilized in a state-of-the-art printer.

Research Triangle Institute

A joint research project was developed with D. R. Black to study the microstructure of diamond substrates to be used for homoepitaxial growth of diamond films by chemical vapor deposition.

Rockwell International

A. Feldman collaborated with S. Holly of Rockwell International to organize the Diamond Optics IV conference sponsored by the Society of Photo-Optical Instrumentation Engineers (SPIE).

W. C. Carter assisted in development of computational models for woven composite behavior.

Russian Academy of Sciences Institute of Solid State Physics

A collaborative program to elucidate the nature of disorder in oxide crystals has been initiated by B. W. Steiner with V. Shekhtman of the Institute of Solid State Physics.

Russian Research Center for Standardization, Information and Certification of Materials

R. G. Munro and J. R. Rumble, Jr. (NIST Office of Standard Reference Data) are collaborating with A. D. Koslov to establish evaluated property data for selected oxide and carbide structural ceramics worldwide.

Russian Academy of Science

Cooperative activities under the NIST-Russian Academy of Science Agreement have continued in the areas of tribology and materials science. Current emphasis is on a joint US-USSR book on tribology (A. W. Ruff and S. Jahanmir). Future benefits to NIST and the Division include exchange of tribology data, exchange of computer software for surface analysis, and future exchange of technical staff.

A collaborative program between the Russian Academy of Sciences and the NIST Ceramics (D. L. Kaiser) and Metallurgy Divisions is underway to map flux distributions in high-temperature superconductors by a magneto-optical technique.

SAC International Ceramics, Ltd.

A. Solomah is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

Sanders Corporation

B. W. Steiner and G. Fogarty collaborate on the crystal growth of barium titanate single crystals with M. Cronin-Golomb of Tufts University and T. Pollack of Sanders.

Sandia National Laboratory

A cooperative program has been established between Mark Smith at SNL and P. Pei, G. Long and D. Smith of NIST to evaluate processing effects on the structure and properties of thermal spraydepostied coatings.

Schmidt Instruments

A collaborative research project was initiated with D. R. Black to grow heteroepitaxial diamond films and characterize their crystal perfection.

Southwest Research Institute

R. Page of SRI is collaborating with S. Kruger (Reactor Radiation Division) and G. G. Long on the microstructure evolution of alumina during densification.

St. Gobain Norton

V. Pujari of St. Gobain Norton and S. G. Malghan are studying the characteristics of agitation milled silicon nitride powders in aqueous environment. St. Gobain Norton has provided silicon nitride powder samples for NIST to compare the performance of agitation ball milling to large-scale processing by conventional methods.

V. Pujari is also cooperating with S. M. Hsu on the effects of machining on tensile strength of silicon nitrides. Tensile bars were received from St. Gobain Norton, subjected to chemically assisted machining, and then tested for strength.

K. Grey of St. Gobain Norton is learning to use the NIST (A. Feldman and H.P.R. Frederikse) photothermal radiometry facility for the purpose of setting up such a facility at St. Gobain Norton for thermal conductivity measurements on diamond films. We plan to collaborate in the analysis of the data generated at St. Gobain Norton.

S. M. Hsu is working with B. McEntire of St. Gobain Norton to jointly evaluate ceramic materials for valve seat insert application. The program is a joint program with the Gas Research Institute (GRI), Caterpillar, and Southwest Research Institute (SWRI) in which a full-scale engine test is being conducted at SWRI to evaluate different materials for the valve seat inserts in a gas-fueled Caterpillar 3500 series engine.

The Cerbec Division of St. Gobain Norton has furnished silicon nitride bearing balls which will be used by G. D. Quinn, R. Gettings, A. W. Ruff and L. K. Ives to make Knoop hardness standard reference materials.

S. Subramanian of the World Grinding Technology Center of St. Gobain Norton collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Smithsonian Institute

W. Wong-Ng in collaboration with Y.S. Dai of the Smithsonian Institute has studied the twin structure of Ba-Ti-Zn-(F,O) as well as the characterization of Na-doped $Ba_2YCu_3O_{6+x}$ superconductors.

Tata Research Design and Development Center

Under a Indo-US program, a collaborative project is in progress to study Al_2O_3-ZrO_2 suspensions and their evaluation.

Torrington Company

S. Redder of Torrington is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

Tower Oil and Technology Company

E. Nachtman of Tower Oil collaborated with S. Jahanmir in the Ceramic Machining Consortium.

Trans-Tech, Inc.

T. A. Vanderah, R. S. Roth of the Viper group, and T. Negas of Trans-Tech, Inc. collaborate on microwave dielectric materials processing, phase equilibria and crystal structure data for these materials. Measurement of electrical properties is a joint effort between NIST and Trans-Tech.

J. J. Ritter collaborating with Trans-Tech under a CRADA to develop a simple, effective process for making a variety of ferrite films.

U.O.P. Research Corporation

A Collaborative Access Team (CAT) agreement has been established between MSEL (G. G. Long), the University of Illinois at Urbana-Champaign (H. Chen) and the U.O.P. Research Corporation on the design and construction of a beamline at the Advanced Photon Source (APS) at Argonne National Laboratory.

U. S. Air Force

J. J. Ritter provided B. Poindexter, a dentist at Bolling Air Force Base, information on the efficacy of ceramic coatings for potable water condenser surfaces.

U. S. Army Research Laboratory

G. D. Quinn is working closely with ARL in the preparation of a standard practice for the characterization of fracture origin in ceramics.

U.S. Bureau of Mines at Albany

The goal of this collaboration between N. Gokcen of the U.S. BoM and W. Wong-Ng is to investigate the effect of high oxygen pressure on the structural and superconducting properties of the superconductors, $Ba_2RCu_3O_{6+x}$ (R=neodymium and yttrium), and of $(Ca,Sr)CuO_2$.

W. R. Grace & Company

J. Chakraverty of the Diamonite Plant collaborates with S. Jahanmir on the Ceramic Machining Consortium.

West Advanced Ceramics, Inc.

R. West collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Xsirius, Inc.

Collaboration in the characterization of sapphire substrates for high temperature superconducting devices is ongoing between B. W. Steiner, U. Laor of the Nuclear Research Center of the Negev (Israel), and W. Graham, President of Xsirius.

ACADEMIA

Alabama A&M University

B. W. Steiner collaborates with R. Lal and A. Batra of Alabama A&M on the growth in space and on the ground of triglycine sulfate crystals.

Auburn University

A. Feldman collaborated with Y. Tzeng of Auburn to organize the First International Conference on the Applications of Diamond Films and Related Materials, ADC'91.

Boston University

L. H. Robins has collaborated with J. Lin of Boston University to examine by cathodoluminescence spectroscopy the defect content of thin boron-doped diamond films grown by the electron cyclotron resonance microwave plasma assisted CVD method.

Cleveland State University

S. Duffy of Cleveland State is collaborating with T.-J. Chuang in the area of continuum damage mechanics on continuous fiber reinforced ceramic composites for high-temperature applications.

Columbia University

P. Somasundaran of Columbia has been collaborating with S. G. Malghan on a research project to study basic parameters affecting the preparation of dense suspensions of silicon nitride powder containing sintering aids.

Dartmouth College

Real-time imaging of dislocation motion in pure and doped ice is currently underway between D. R. Black and F. Liu of Dartmouth.

East China University of Chemical Technology

This is a joint effort to use finite-element techniques to analyze creep behavior of ceramic c-rings at elevated temperatures. D. Wu and Z.-D. Wang of ECUCT are developing the finite-element model for C-rings and a computational algorithm for creep and T.-J. Chuang is providing a theoretical framework and experimental data to support the program.

Free University of Brussels

J. Hastie and D. Bonnell are collaborating with J. Drowart of Brussels on a survey of ionization cross section usage in high-temperature mass spectrometry.

Florida State University

A collaborative study between J. Schwartz of Florida State and D. L. Kaiser and F. W. Gayle (Metallurgy Division) is underway to correlate microstructure with microscopic flux flow during magnetization of $Bi_2Sr_2CaCu_2O_{8+x}$ superconducting tapes by means of a magneto-optical imaging technique.

Georgia Institute of Technology

S. Danyluk of the School of Mechanical Engineering of Georgia Tech collaborates with S. Jahanmir in the Ceramic Machining Consortium.

Harvard University

G. Golovchenko of Harvard and J. C. Woicik are collaborating on standing-wave x ray and scanning tunneling microscopy studies of Bi on Si(111).

Howard University

G. Walrafen of the Chemistry Department of Howard University and G. J. Piermarini continue a long-standing collaboration on study of materials at high pressures. They are currently studying liquid state of H_2O in the superpressed state at elevated temperatures and pressures.

Indian Institute of Technology, Kanpur, India

P. Ghosh of IIT-Kanpur and J. Hastie are collaborating on a study of ion-molecule reactions in RF plasmas.

Indira Gandhi Institute (IGI) Kalpakkam, India

A collaborative activity is underway between Dr. Mathews of IGI and J. Hastie for the mass spectrometric investigation of materials at very high temperatures generated by laser heating.

Iowa State University

Characterization of defects in icosahedral AlPdMn is being performed by D. R. Black and A. Goldman and S. Kycia of Iowa State.

Johns Hopkins University

J. Kruger and L. Krebs of JHU are collaborating with G. G. Long and C. Majkrazak (Reactor Radiation Division) on in-situ polarized neutron reflectometry studies of the nature and structure of passive films.

D. Shechtman of JHU is collaborating with A. Feldman and E. Farabaugh in the high-resolution TEM analysis of CVD diamond nucleation and growth.

Massachusetts Institute of Technology

R. Hallock and W. Rhine of MIT interacted with W. Wong-Ng to characterize the $BaTiO_3$ precursor material, $BaTi(O)(C_2O_4)_2 \cdot 5H_2O$, by an x-ray diffraction method.

National Tsing Hua University

S. Lee and J.-L. Chu Tsing Hua are collaborating with T.-J. Chuang on creep life prediction.

North Dakota State University

G. J. McCarthy of the North Dakota State University is collaborating with W. Wong-Ng in the preparation of standard reference for high-T_c superconductor and related materials.

Northwestern University

S. M. Hsu is collaborating with M. Fine of Northwestern on optimization of ceramic wear resistance by introducing compressive stress into the surface and interfaces.

K. T. Faber and several of her students at Northwestern are collaborating with E. R. Fuller, Jr. on a research project focused on the understanding and control of materials which undergo process-zone phenomena around propagating cracks.

D.L. Johnson of Northwestern is collaborating with G. G. Long and A. Allen of the University of Maryland on microwave-assisted reaction-bonded silicon nitride.

Pennsylvania State University

S. M. Hsu is collaborating with Profs. Duda, Klaus, Philips, and Christen on a variety of projects. Duda and Klaus are working on lubrication, development of lubricants for alternative fuels and ceramic lubrication. Philips is working on synthesis of nano-sized particles of ceramic materials using a microwave assisted plasma reactor. Christen is working on computer simulation of grain growth.

S. G. Malghan and P. S. Wang are collaborating with R. German of PSU in their consortium on powder injection molding.

M. D. Hill is interacting with E. Cross of PSU in investigations of PZT. NIST is providing information on mechanical testing of PZT and is reviewing training on machining electrical measurements.

R. G. Munro (NIST) and J. Hellmann (PSU) are continuing their extended collaboration on the indentification of important high-temperature ceramic materials and properties issues that may be addressed in the development of the NIST Structural Ceramics Database.

Purdue University

D. L. Kaiser is collaborating with M. McElfresh of Purdue University to study magnetization and current transport in superconducting $Yba_2Cu_3O_{7-x}$ single crystals.

Rensselaer Polytechnic Institute

Collaboration on the diffraction imaging of semiconducting multilayers in order to determine the genesis and influence of disorder on photonic device performance is being carried out by B. W. Steiner and H. Wiedemeier and K. Rajan of RPI.

Rice University

D. Bonnell is collaborating with the High Temperature Group in the Chemistry Department of Rice on levitation and thermophysical properties of liquid metals.

Rutgers University

S. C. Danforth of Rutgers has provided S. G. Malghan with nano-sized Si_3N_4 powder for processing at NIST using cryogenic compaction.

S. M. Hsu is collaborating with Profs. Niesz and Wachtman on microstructural design for wear resistance on silicon nitrides. Variation of grain size, shape and interface strength are being examined. The materials processed are evaluated both at NIST and at Rutgers.

H. Han and G. Skandan of Rutgers are collaborating with G. G. Long and S. Krueger (Radiation Reactor Division), and A. Allen of the University of Maryland in the study of nanophase powders and processing.

Seoul National University

J. E. Blendell is collaborating with D.-Y. Kim of Seoul National University on the wetting of grain boundaries in Al_2O_3.

Stanford University

W. Spicer of Stanford is collaborating with J. C. Woicik in the use of x-ray standing-wave studies, surface-EXAFS, and ultraviolet photoemission of metal/semiconductor interfaces and semiconductor surfaces.

State University of New York/Stony Brook

Collaborative researh on the relationship of processing to microstructure of plasma sprayed coatings has been under way between J. Ilavsky of SUNY and G. Long of NIST.

Stevens Institute of Technology

T. Fischer of the Materials Science and Engineering Department is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

Texas A&M University

J. Mayer of the Mechanical Engineering Department is collaborating with S. Jahanmir in the Ceramic Machining Consortium.

Tufts University

B. W. Steiner and G. Fogarty collaborate with M. Cronin-Golomb of Tufts on the observation of optical mixing and related optical phenomena in non-linear optical crystals. Other collaborators include R. Uhrin, M. Garrett, and J. Martin of Deltronic Crystal Industries and J. Zhao of NZ Applied technologies.

University of California at Berkeley

A. M. Glaeser of UC Berkeley is collaborating with T.-J. Chuang on microdesign of interfacial defects for creep crack growth experiments.

University of California at Santa Barbara

Joint experiments between J. N. Israelachvili and P. McGuiggan of UC Santa Barbara and D. T. Smith) are being conducted to investigate frictional properties of silica surfaces under dry conditions and with a variety of thin (< 10 nm) intervening liquid films.

University of Dayton Research Institute

Using UDRI's x-ray photoelectron spectrometer (XPS) and Auger electron spectrometer (AES), T. Wittberg of UDRI is conducting studies on surface structures and reactivities with P. S. Wang.

University of Grenoble

B. P. Burton has been working with A. Pasturel of the University of Grenoble on first-principles phase diagram calculations of BCC-based ordering in Ni-Al-Ti and Fe-Be alloys.

University of Illinois

M. McNallan of UI is collaborating with S. M. Hsu on wear mechanisms of ceramic materials and the definition of surface quality in terms of strength as a result of machining damage.

A Collaborative Access Team (CAT) agreement has been established between MSEL (G. Long), the University of Illinois at Urbana-Champaign (H. Chen) and U.O.P. Research Corporation on the design and construction of a beamline at the Advanced Photon Source (APS) at Argonne National Laboratory.

University of Maryland

A collaborative study between A. Roytburd of the University of Maryland (UMD) and D. L. Kaiser and F. W. Gayle (Metallurgy Division) involves theoretical aspects of the effect of twin boundary and grain-boundary defects on flux flow during magnetization of high temperature superconductors.

L. Chang of UMd is collaborating with E. F. Begley and C. G. Lindsay on appropriate instructional techniques for a computer-based tutorial in phase diagram interpretation. UMd will also provide a testbed of students for evaluation of the tutorial.

B.-H. Chen and B. Eichgorn of UMd are collaborators with W. Wong-Ng in the structural investigation of possible new superconductor related single-crystal materials.

M. D. Hill, G. S. White, and S. W. Freiman are collaborating with I. Lloyd of UMd investigating mechanical and electrical effects of cyclic loading of PZT. Hill is using the research as partial fulfillment of requirements for a PhD in materials science.

R. K. Khanna is collaborating with S. G. Malghan on spectroscopy of powders.

S. Jahanmir, as Adjunct Professor in the Department of Mechanical Engineering at UMd, is presently supervising three students (L. Job, K. Ritchie, and M. Nagarajan) who are working toward their M.S. degrees. These students work at NIST on ceramic machining as Guest Researchers.

G. Zhang of the Mechanical Engineering and the Systems Research Institute of UMd collaborates with S. Jahanmir in the Ceramic Machining Consortium.

University of Michigan

J. Gland of Michigan and D. A. Fischer are collaborating on the study of hydrogenolysis of aniline on the Pt(111) surface.

J. Schwank of Michigan is carrying out specialized characterization of conductive ceramic powders by ESCA and Auger spectroscopy in collaboration with J. J. Ritter. These powders are synthesized at NIST for NASA.

University of Pennsylvania

P. Davies of the University of Pennsylvania has been working with T. A. Vanderah and R. S. Roth of the Viper Group on high-resolution transmission electron microscopic images of microwave dielectric materials.

University of Rochester

J. Chakraverty of the Diamonite Plant collaborates with S. Jahanmir in the Ceramic Machining Consortium.

University of South Carolina

R. G. Munro (NIST) and T. Datta (U. So. Car.) are collaborating on the evaluation of materials properties for high-temperature superconductors.

University of Southampton

D. Bonnell and J. Hastie have been working with I. R. Beattie of the University of Southampton to adapt transpiration mass spectrometry to the quantitative determination of volatiles from spent reactor fuel pins. This association has developed into a detailed interaction to refine details of system design and instrument selection for Project Osprey.

University of Toronto

S. Jahanmir is collaborating with T. Coyl of the University of Toronto in machining of ceramic composites. C. Ryan, a graduate student from the Materials Science and Engineering Department, is working on his thesis on machining at NIST with S. Jahanmir.

University of Virginia

D.T. Smith is collaborating with H.N.G. Wadley of UVa on nanoindentation studies of Al/Al_2O_3 and Al/Cu nanoscale multilayer systems.

University of Washington

L. Sorenson of UW and J. C. Woicik are using diffraction anomalous fine structure measurements to study strained semiconductor layers and 123 semiconductors.

B. W. Steiner, J. C. Woicik, and J. G. Pellegrino (Thermophysics Division) also collaborate with Professor Sorenson
on strain relaxation in III-V photonic crystals.

D. Castner and B. Ratner of the University of Washington and D. A. Fischer have used the polarization dependence of carbon and fluorine NEXAFS to understand the orientation of fluorocarbon groups and proteins on polymeric biomaterials used in medical implants.

G. D. Quinn is collaborating with M. Jenkins in the creation of a fracture toughness standard test method for ceramics for ASTM Committee C28.

University of Western Ontario

A collaboration was initiated this year between H. H. Schloessin and R. A. Secco of the Geophysics Department of Western Ontario with R. D. Spal. This work involves study of geological samples by means of x-ray diffraction topography and the asymmetric Bragg diffraction microscope.

University of Wisconsin

A joint activity is underway between S. Babcock, X. Cai, and D. Larbalestier of the University of Wisconsin and D. L. Kaiser to characterize the microstructural, magnetic and electrical transport properties of single crystals and bicrystals of superconducting $YBa_2Cu_3O_{6+x}$.

M. D. Vaudin is collaborating with J.-L. Wang and S. Babcock of Wisconsin in the determination of misorientations across twist bicrystals of ceramic superconductors.

Worcester Polytechnic Institute

I. Bar-On of Worcester Poly is conducting a joint ASTM Committee C28-E08 project to write a fracture toughness standard.

FACILITIES

FACILITIES

POWDER CHARACTERIZATION AND PROCESSING

High Temperature X-ray Diffraction - J. P. Cline

The x-ray diffraction facility at NIST consists of a high-temperature machine of theta-two theta geometry equipped with an incident beam monochrometer and a position sensitive proportional counter. The incident beam monochrometer removes the $K\alpha2$ radiation and results in diffraction profiles that are more sensitive to effects of sample character. The position sensitive detector allows for data collection at a rate two orders of magnitude faster than conventional detectors. The furnace is an enclosed high vacuum chamber capable of reaching 3000 K, and is equipped with a mass flow controller for atmospheric control. This equipment is used for the study of high-temperature phase equilibria, high-temperature reaction kinetics, sintering of monolithic ceramics, and strain development during sintering of ceramic composites. Additional equipment consists of four automated and updated Philips diffractometers which are used for certification of standard reference materials (SRMs), studies on the effects of microabsorption and extinction, and the development of the Rietveld method for a conventional, sealed tube, x-ray diffraction equipment.

Electrokinetic Measurements - V. A. Hackley and S. G. Malghan

The Matec ESA-8000 system has the unique capability for measuring colloidal properties in dense slurries. The analytical capabilities of the ESA system include performance in the following modes: potentiometric titration, conductometric titration, time-series titration, and concentration series titration. In the selected mode, the equipment can monitor: electrokinetic sonic amplitude, zeta-potential, electrophoretic mobility, electrical conductivity, isoelectric point, surface charge density, and phase angle of the material with the specified experimental conditions. The latest addition to this laboratory is a Acousto-Sizer by Matec Applied Sciences.

Slurry Rheology - S. G. Malghan and V. A. Hackley

The RTI rheometer allows for viscosity as well as rheology characterization of ceramic slurries. Rheological measurements are more informative and flexible with respect to the various slurry properties: Newtonian, pseudoplastic, plastic, dilatant, and thixotropic. The modeling of these rheological properties as a function of sample treatment and surface chemical properties is paramount in developing and improving the slurry processing technology.

Physical Properties Characterization Laboratory - L. Lum, D. B. Minor, P. T. Pei and S. G. Malghan

The physical properties characterization laboratory is equipped with state-of-the-art techniques for the measurement of particle size distribution, specific surface area, specific gravity, tap density, and porosity. The particle size distribution is measured by three techniques -- gravity sedimentation by Sedigraph, centrifugal sedimentation by Joyce-Loeble, and laser diffraction by Horiba LA-900. The range of particle size distribution covered by these techniques is (0.01 to

200) μm. The specific surface area determination is carried out by nitrogen adsorption and the BET method. The porosity of powders and ceramics is measured by mercury intrusion.

Colloidal Processing of Powders - S. G. Malghan, D. B. Minor and P. T. Pei

The focus of this laboratory is to develop data and understanding of non-oxide powders processing in aqueous environment. The laboratory is equipped with instruments and equipment for studying deagglomeration, dispersion, suspension stability, slurry casting, and green body microstructure evaluation.

Agitation Milling of Powders - D. B. Minor and S. G. Malghan

High energy agitation milling of silicon nitride powders is carried out with a minimum contamination by the use of a specially designed milling system. This milling device allows for the size reduction of silicon nitride powder by milling at high slurry densities in approximately 1/6th to 1/10th of the time required by the conventional tumbling ball mill. The mill is lined with silicon nitride and the media are made of silicon nitride materials; hence, external sources of contamination can be minimized.

Nuclear Magnetic Resonance (NMR) - P. S. Wang

The solid state NMR facility includes a Bruker MSL-400 NMR system capable of studying almost all NMR-active nuclei in the periodic table in both solid and liquid states as well as performing NMR imaging in proton and carbon-13 frequencies. Currently, the operation parameters for both states at proton, deuterium, carbon-13, and aluminum-27 have been defined and proved by documented NMR spectra of organic and inorganic molecules. The equipment has been tuned to Si-29, Cu-63, and Y-89.

Scanning Electron Microscope/Image Analysis (SEM) Facility - J. F. Kelly

This laboratory is equipped with an Amray 1830 digital scanning electron microscope with LaB_6 source and a Leitz optical microscope. The SEM is equipped with a solid-state backscatter detector and an ultrathin-window x-ray detector. A Kevex Delta V EDS x-ray analysis and image analysis system is interfaced to both the SEM and optical microscopes. Automated imaging capabilities enable rapid size and shape analysis of a variety of imaged features, including ceramic powder particles and second phase regions in composite structures. Fracture stages have been developed for real time observation and measurement of *in-situ* crack propagation in ceramic specimens. The addition of an interior mounted phosphor screen with video camera imaging provides the capability of imaging single-grain electron backscatter diffraction patterns from bulk specimens. This permits the measurement of crystallographic orientation in ceramic specimens.

Thermal Analysis Facility - J. S. Wallace and J. E. Blendell

This facility includes equipment for measurement of behavior of ceramic materials in a wide range of atmospheres and temperatures. The equipment is comprised of a computer-controlled differential pushrod dilatometer capable of measuring thermal expansion or sintering shrinkage in vacuum, inert, oxidizing or reducing conditions from room temperature to 1600°C. The atmosphere can be monitored using either a zirconia oxygen cell or an external mass spectrometer using its own associated computerized data acquisition system.

The second major piece of equipment is a simultaneous thermal analysis (STA) system which is capable of performing simultaneous thermogravimetric and differential thermal analysis from room temperature to 1700°C. Atmospheres can be varied from vacuum to single and mixtures of gases using a four-channel mass flow controller. The STA is also connected to the mass spectrometer system and it's associated data acquisition system. The quadrapole mass spectrometer system has a capability of analyzing to 512 AMU.

Chemical Laboratory Facilities - J. J. Ritter

Chemical synthesis of powders is carried out in a well equipped laboratory, which consists of controlled atmosphere glove boxes, preparative chemical vacuum systems, and a chemical flow reactor. A range of powders can be synthesized for exploratory purposes.

Ceramics Powders Processing Laboratory - J. S. Wallace and J. E. Blendell

A processing laboratory for processing and sintering well controlled ceramic powders has been assembled. This facility consists of: equipment for chemical powder synthesis routes, attrition mills, ball mill, jet mill, pressure slip caster, uniaxial presses, cold isostatic press, spray dryers, drying ovens, hot presses, air furnaces to 1700°C, controlled atmosphere furnaces with associated gas flow systems and oxygen sensors for temperatures to 1600°C, graphite furnace for temperatures to 2300°C, and a hot-isostatic-press/gas-pressure sintering furnace capable of 2300°C and 200 MPa using graphite elements and insulation.

Nano-Size Powders Processing - E. Gonzalez, G. J. Piermarini and S. G. Malghan

This is a new facility which consists of equipment for powder handling in inert environment, compaction of nano-size powders, and sintering under environment control. The compaction equipment was designed to facilitate the application of wide range of pressures (up to 5 GPa), temperatures (cryogenic to 1000°C), and environments. The size of green ceramic produced in this system is 3.0 mm diameter.

SURFACE PROPERTIES

Wear Tests - S. M. Hsu

A state-of-the-art friction and wear testing laboratory is available for the evaluation of materials under different applications and conditions. Contact geometries include pin-on-disk, cross cylinders, ball-on-flat, ball-on-balls, flat-on-flat, and ring-on-block. Various motions and operating conditions are available to simulate many industrial applications. Environmental control includes temperature (room temperature to 1200°C), vacuum, and humidity.

Surface Analysis - S. M. Hsu and R. S. Gates

Many modern specialized instrumentation are available for the analysis of surface properties of materials. Mechanical property measurement include hot hardness tester, Vicker's indenter, nano-indentor, scratch test, and controlled-depth micro-scratch test. Chemical property measurement include time-resolved micro-Raman spectroscopy, FTIR microscopic spectroscopy, GC-MS, SEM with EDX analysis, IR and UV spectroscopies with API compound identification files. A specially designed, organo-metallic specification facility is also available to detect surface reaction products at ppm level. Access to conventional surface analysis such as XPS, ESCA, Auger, etc are also available through external contracts.

STM/AFM - S. M. Hsu

A digital commercial scanning tunnelling microscope (STM) and atomic force microscope (AFM) is available to measure surface properties at atomic level.

Time-Resolved Micro-Raman - S. M. Hsu

This versatile facility consists of a pulsed Nd-YAG laser, a CW Ar-ion laser, a triple monochromator, and a gated intensified diode array detector. This facility, therefore, provides a wide variety of Raman analysis techniques in both time-resolved and continuous operation modes, using either visible or ultra-violet excitation sources for either operation mode. In addition, either bulk macro-Raman or 5-μm-resolution micro-Raman analyses are available.

MECHANICAL PROPERTIES

Surface Forces Laboratory - D. T. Smith

The surface forces laboratory consists of a semi-clean-room preparation facility and a crossed-cylinders surface force apparatus. The crossed-cylinder apparatus permits measurements of atomic-scale forces between surfaces. It can be operated with a variety of liquid or gaseous environments, thus allowing investigations of the effects of chemical changes on the forces between two surfaces. The apparatus includes several unique features that were developed and built by the surface forces group. First, sensitive custom electrometer circuits were built into the apparatus to allow *in-situ* measurements of surface charges resulting from contact electrification. Second, the apparatus has been modified to permit the sliding of one surface over the other under constant applied load.

Instrumented Microindenter - D. Smith

This apparatus is designed to bridge the gap between conventional hardness testers and the nano-indentation facility. It measures continuous load-displacement curves for indentations in the load range 10 g to 2 kg, and is of particular use in studying the mechanical properties of thermal-spray and other ceramic coatings with relatively coarse microstructures.

Analytical Electron Microscopy - B. J. Hockey

Several transmission and scanning electron microscopes are available for analysis of the changes in microstructure as a result of creep.

Glass Melting - D. A. Kauffman

Extensive glass melting and annealing facilities for production of melts up to 1600°C are available. Batch sizes up to about (2.5 to 3) kg can be produced using this equipment. Special facilities for melts containing heavy metals such as thallium and lead are also available.

Creep Apparatus - R. F. Krause, Jr. and S. M. Wiederhorn

The creep measurements laboratory possesses nineteen controlled-temperature furnaces (800 °C to 1700°C), seven laser extensometers, ten optical long-distance microscopic extensometers, and twenty loading frames (fourteen pneumatically driven, four screw driven, and two direct weight). Among these loading frames fourteen can be used in tension, three in tension or compression, one in flexure, and one as a sintering forge.

Hot-Pressing Apparatus - R. F. Krause, Jr.

A graphite heating-element furnace (2300°C maximum) which be can operated in vacuum or an inert gas atmosphere is mounted in a hydraulic loading frame (0.5 MN maximum). Ceramic powders can be hot pressed in graphite dies, (50, 75, 100, and 125) mm diameter.

Nano-Indentation Facility - D.T. Smith

The Ceramics Division nano-indentation facility consists of a Nano Indenter II indentation machine, manufactured by Nano Instruments, Inc., and related computer and optical components. The indenter, under computer control, is capable of measuring loading-unloading curves with displacement resolution better than 0.1 nm and load resolution better than 200 nN. Nomarski interference contrast (NIC) optics and translation stages with placement precision better than 1 μm permit the measurement of material properties such as hardness and Young's modulus in selected volumes as small as 10^{-17} m^3.

Optical Microscopy Laboratory - G. D. Quinn

The optical microscopy laboratory is equipped with conventional reflected light microscan inspection of polished specimen as well as stero-binocular microscopes and camera stands for fracture surface analysis. A WILD M-10 stereo discussion microscope system with video monitoring and instant photography capability is very well suited for fractography.

ELECTRONIC MATERIALS

Level 10 Clean Room - J. E. Blendell

A Level 10 Clean Room has been constructed for the processing of ceramics in a controlled environment where the presence of air contaminants at low levels can seriously affect the final product's properties. The room is provided with separated work stations to allow simultaneous conduct of experiments.

Thermal Wave Analysis Facility - A. Feldman and G. S. White

This facility is used for characterizations based on variations of thermal diffusivities. Equipped with both an Ar-ion and CO_2 laser, the facility permits analyses by infrared and mirage methods. It is especially useful as a nondestructive method of detecting flaws in near-surface regions of ceramics.

OPTICAL MATERIALS

Optical Characterization - L. H. Robins and A. Feldman

Facilities include a Cary spectrophotometer for measuring optical transmittance in the spectral range 0.2 μm to 2.5 μm, optical spectrometers for measuring photoluminescence and Raman spectra, and an argon ion laser.

Magneto-Optical Imaging of High Temperature Superconductors - D. L. Kaiser, F. W. Gayle (Metallurgy) and A. Shapiro (Metallurgy).

The facility consists of a magneto-optical imaging system with attached video equipment. It is used to measure real-time flux distributions in high-temperature superconductors as a function of temperature, (7 - 300) K, and applied magnetic field, (0 to $\pm$ 65) mT.

Electro-optic Thin-film Characterization - L. D. Rotter

The facility consists of a vibration-isolated optical table, argon-ion and helium-neon laser sources, polarizing components, lenses, optical stages, optical detectors, and electronic signal processing equipment for measuring the electro-optic coefficients and optical birefringence of thin ferro-electric films.

Metalorganic Chemical Vapor Deposition (MOCVD) System - D. L. Kaiser

A specialized system was constructed for the deposition of oxide thin films from metalorganic precursors. It has been used to deposit $BaTiO_3$ films on 1.5 cm x 1.5 cm substrates.

Diamond Film Deposition - E.N. Farabaugh and A. Feldman

Facilities consist of three hot-filament CVD reactors and a microwave-enhanced CVD reactor. The hot-filament reactors can accommodate substrates up to 2.5 cm × 2.5 cm square. The microwave reactor can accommodate substrates up to 10 cm in diameter. The reactant gases are hydrogen, methane, oxygen, argon, and ethyl alcohol which contains boron of doping. Growth rates typically range from (0.1 to 0.6) μm/h.

MATERIALS MICROSTRUCTURE CHARACTERIZATION

Synchrotron Radiation Beamlines - G. G. Long

The Materials Microstructure Characterization Group operates two beamstations on the X23A port at the National Synchrotron Light Source at Brookhaven National Laboratory in New York. These two beamstations offer access to dedicated instrumentation for small-angle x-ray scattering, x-ray diffraction imaging (topography) and EXAFS.

Small-angle x-ray scattering can be carried out in the energy range from (5 to 11) keV. The minimum wavevector is 4×10^{-3} nm^{-1} and the wavelength resolution is $\Delta\lambda/\lambda = 10^{-4}$, allowing anomalous small-angle scattering with excellent resolution. Diffraction imaging of single crystals and powders is carried out with monochromatic photons between (5 and 30) keV. An energy-tunable, x-ray image magnifier enables imaging of microstructure down to less than 1 μm. EXAFS experiments are also performed over an energy range from (5 to 30) keV.

Small-angle scattering measurements on ceramic and metallurgical materials are being used to characterize the microstructure in the 2 nm to 1 μm size range as a function of starting chemistry and processing parameters. Diffraction imaging is being used to study imperfections and strains in single crystals and powder compacts. EXAFS is being used to study the structure of strained semiconductor interfaces and metal multilayers. A combination of EXAFS and diffraction will provide a capability for site-specific local structure determination in crystals.

SANS - Ceramics Furnace - G. Long

The SANS-Ceramic furnace is a unique facility that has been recently commissioned. This system allows simultaneous *in-situ* densification studies of ceramic powders and SANS measurements. The experimental system has been designed to carry out densification studies of oxide powders at temperatures up to 2000°C. In addition, the furnace will be equipped with a dilatometer.

PHASE EQUILIBRIA

Single Crystal X-ray Diffraction Facility- W. Wong-Ng

Currently, this research facility is primarily used to characterize single crystals in terms of crystal symmetry, lattice parameters and detailed structure.

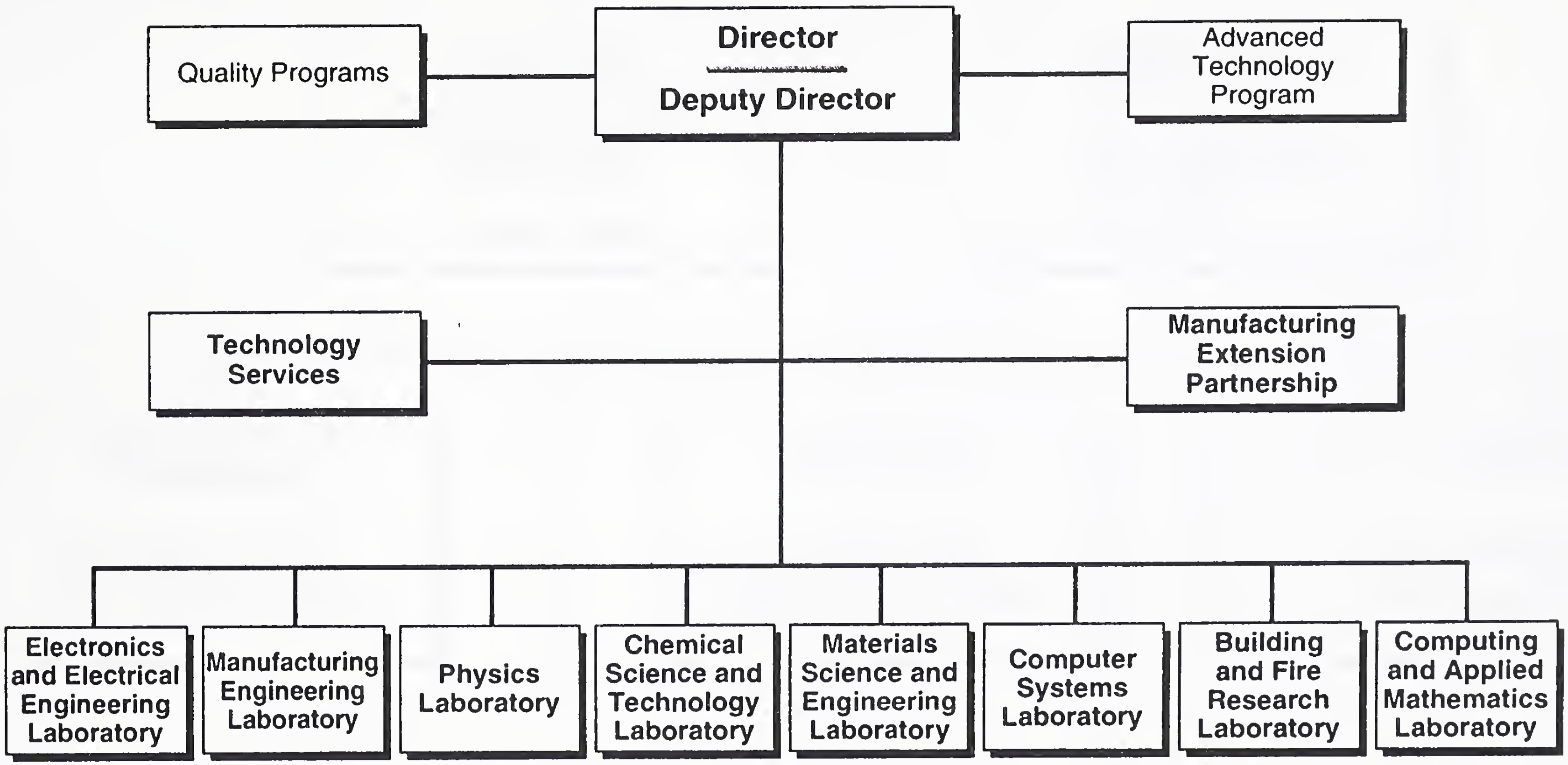
Phase Equilibrium Facilities for High-T_c Superconductor Systems Bi(Pb)-Sr-Ca-Cu-O and Ba-R-Cu-O - W. Wong-Ng and L.P. Cook.

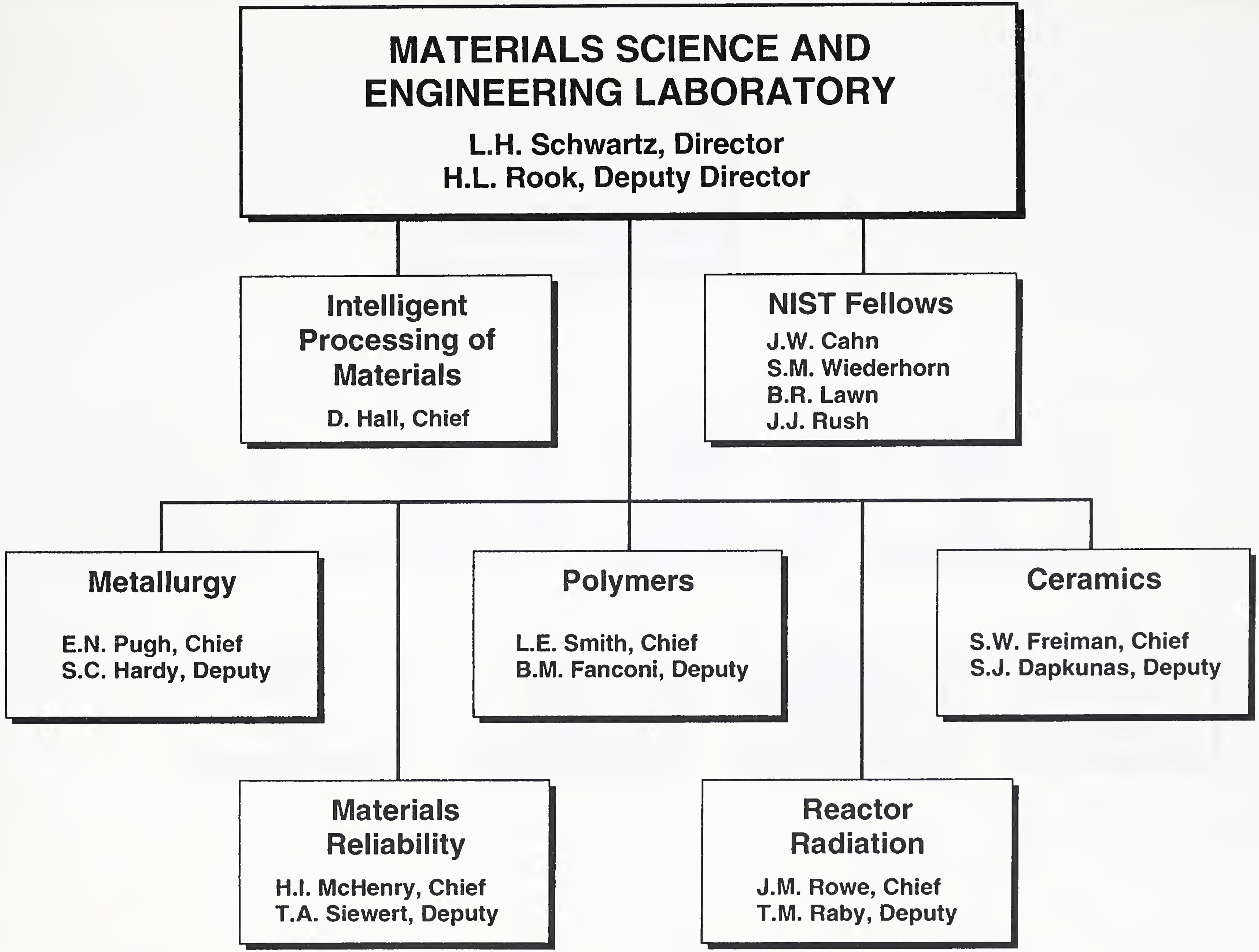
This facility consists of two controlled-atmosphere, quenched furnaces and a Mettler thermal analyzer for performing DTA/TGA experiments. Both solid and melt can be quenched into liquid nitrogen cooled helium atmosphere so that the high temperature structure can be captured and analyzed. Liquid composition can also be estimated quantitatively with a wicking method during the quenching experiments.

APPENDIX

National Institute of Standards and Technology

Organizational Chart




MATERIALS SCIENCE AND ENGINEERING LABORATORY
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Intelligent Processing of Materials
D. Hall, Chief
NIST Fellows
J.W. Cahn
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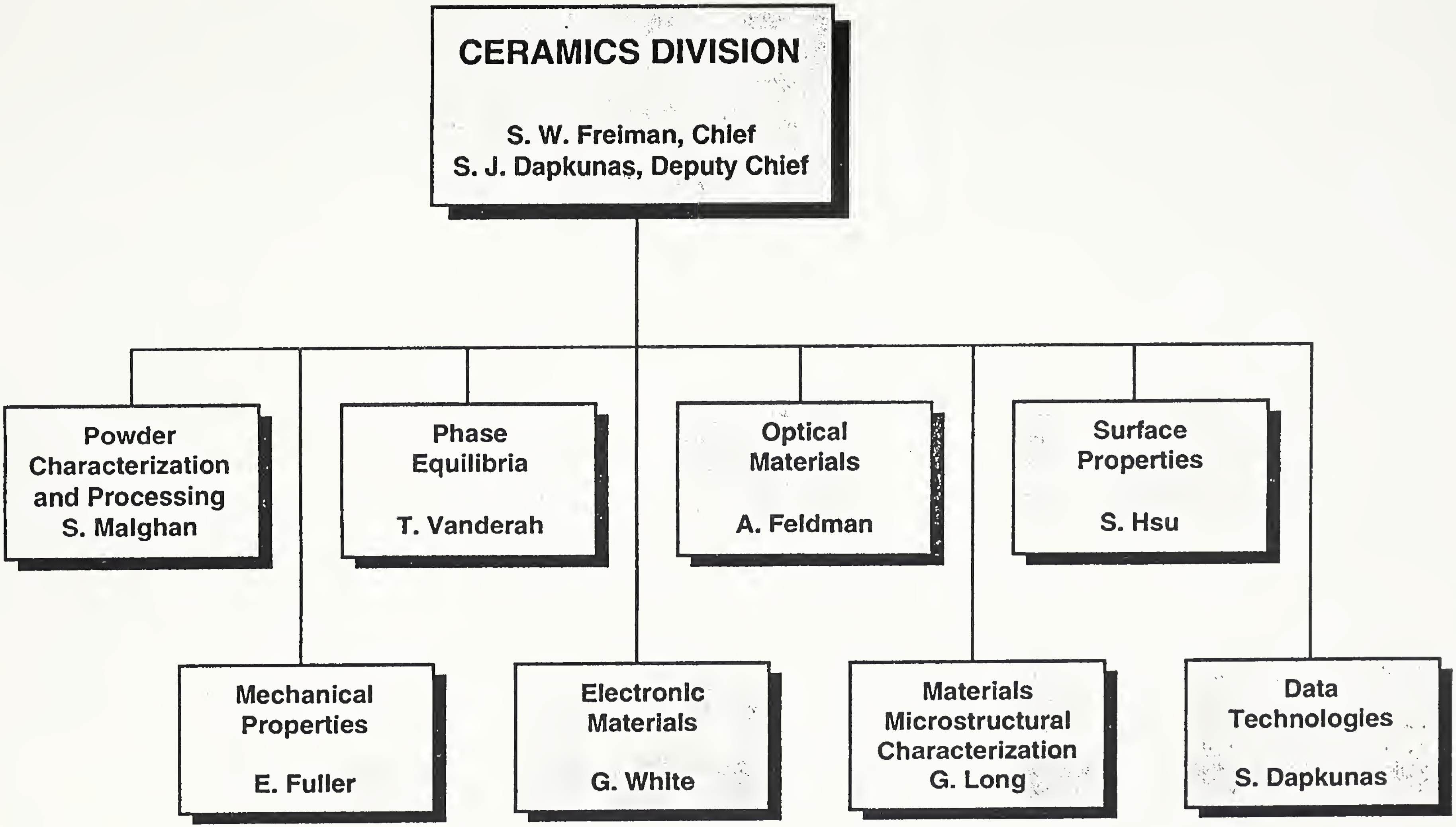
J.M. Rowe, Chief
T.M. Raby, Deputy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