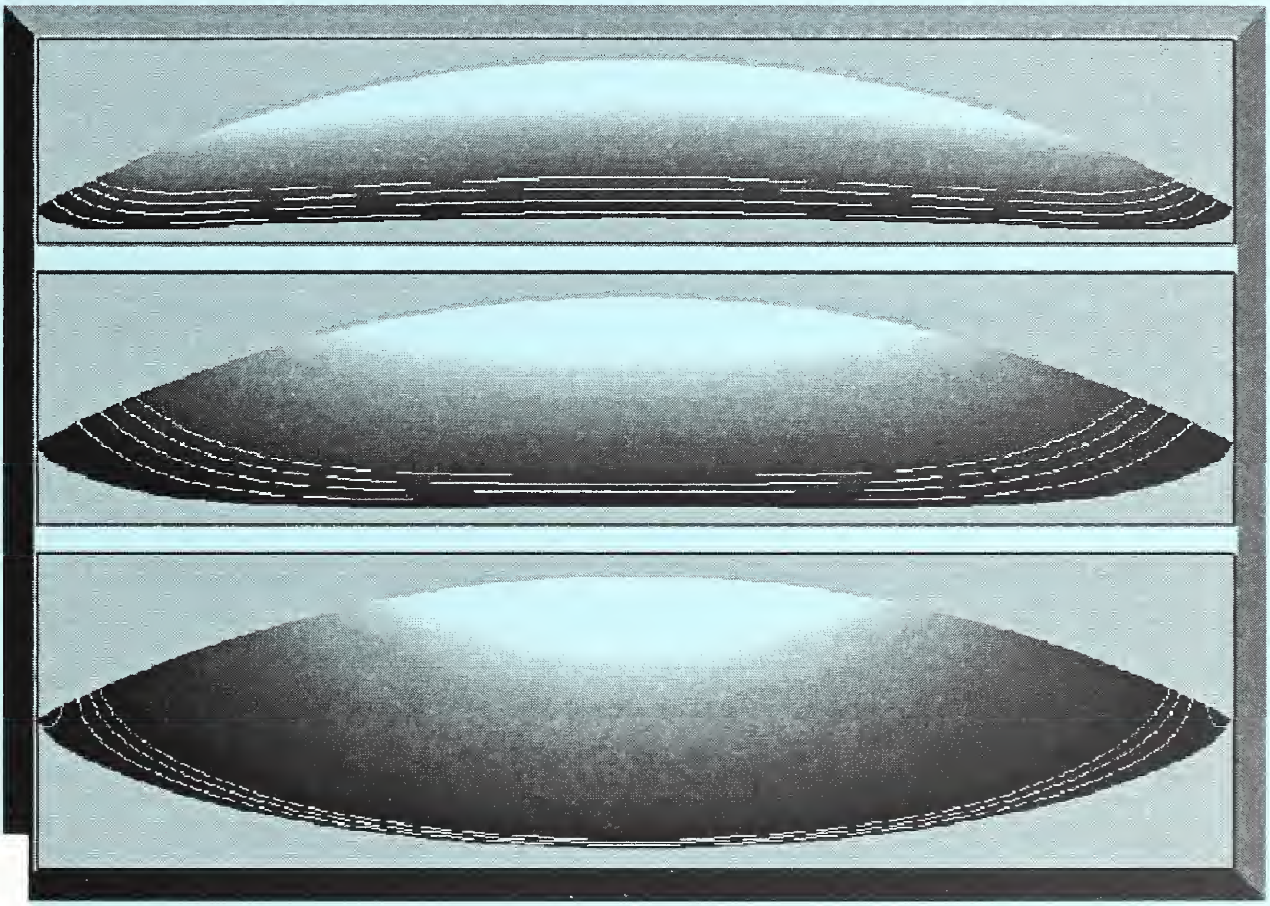
MSEL

Materials Science and Engineering Laboratory

METALLURGY



NISTIR 5965
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities
1996

Metallurgy

Reaction of a molten metal droplet with a metallic substrate, showing partial dissolution of the substrate by the melt. The white lines are isoconcentrates of solute in the liquid. The figures are output of a model which is being developed to analyze reactive wetting of solders on electronic components. These studies are clarifying the interpretation of solderability tests used by the electronics industry.

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METALLURGY

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NISTIR 5965
U.S. Department of Commerce
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Technical Activities 1996

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OVERVIEW

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The Metallurgy Division is working closely with materials suppliers and users to develop the measurement and standards infrastructure needed in diverse technological areas - from steelmaking to the fabrication of nanostructured multilayers for magnetic recording heads. As a result of global competition and the consumer's expectations of ever-increasing performance and reliability at lower cost, U.S. industry is making fundamental changes in the ways that new products are developed and manufactured and in the ways that materials are used. At the core of these changes is the need by both the metals producers and users for new measurements that make possible more accurate predictions of materials performance, manufactureability, and long-term reliability.

The Metallurgy Division maintains core competence in a range of materials science and metallurgy disciplines within the structure of five separate groups: Electrochemical Processing, Magnetic Materials, Materials Performance, Materials Structure and Characterization, and Metallurgical Processing. The Division's projects require a wide range of expertise and, therefore, cut across formal group boundaries. Likewise, many major MSEL programs involve collaboration among scientists from several Divisions.

Programs in the Metallurgy Division address a range of measurement-related needs within major industrial sectors using metals and alloys: aerospace, automotive, electronics, and stationary power generation, as well as some of the needs of suppliers of steel, aluminum, nickel-based superalloys, and other specialty metals. These programs include:

- Industry-NIST consortium led by NIST for the development of integrated design tools for the casting of aerospace alloys.
- Measurements and standards for electronic packaging, including solderability measurement and optimization, measurements for the development of lead-free solder alloys and high-temperature, fatigue-resistant solder alloys, development of integrated design tools for solder joint design, and stress measurements in electronic packaging using scanning acoustic microscopy.
- In-situ measurements for the processing control and optimization of thermal barrier coatings produced by thermal spray processes.
- Measurement, micromagnetic modeling, and standards for conventional and nanoscale magnetic materials.
- Development of an understanding of the effects of processing and structure on the giant magnetoresistance (GMR) of multilayer, nanostructured films.

- Modeling of deformation processing and formability of lightweight materials for automotive applications, including the development of tests for mechanical property variability in high-strength, low alloy steels and aluminum alloys.
- Development of a national Rockwell C hardness scale, traceable to NIST standards.

Particular topic areas are chosen by MSEL/Metallurgy Division in close cooperation with industry, considering the potential impact of NIST contributions on these major civilian technology areas. Descriptions of all Metallurgy Division programs and projects can be found in this report.

Most of the Division's projects focus on developing an understanding of the relationships among processing, structure, and properties from a fundamental point of view. Such fundamental research is required to establish the science and technology base necessary for new measurements methods and new materials. This approach means that measurement techniques are designed and carried out within the context of general models of metals behavior, not simply from empirical rules for a limited class of materials. By starting from this base, today's measurements may be leveraged in the future to describe the behavior of a wider range of metal alloys under a wider range of conditions than are currently needed.

A small fraction of Division resources continues to be employed on programs supported by other federal agencies or private organizations in which there is a demonstrated need for NIST expertise. Examples of such programs are: the study of corrosion issues for aging aircraft supported by Wright Laboratories, the development of replacement alloys for mercury-containing amalgams supported by the American Dental Association, and the examination of the limits of nitrogen incorporation into corrosion-resistant nitrogenated stainless steels supported by the U.S. Army Research Office.

DENTAL AND MEDICAL MATERIALS

The Dental and Medical Materials Program provides basic materials science, engineering, test methods, and standards to sectors of the health-care industry for the development of new or improved materials and delivery systems. The focus of this program is the development of improved dental restorative materials with greater durability, wear resistance and clinical acceptability. This MSEL program consists of nine separate projects on dental restoratives in the Metallurgy, Polymers, and Ceramics Divisions; the Metallurgy Division project described below is aimed at developing metallic replacements for mercury-containing amalgams.

Dental research directions in support of the goals are established in collaboration with the American Dental Association (ADA), the National Institute of Dental Research, and guest scientists from the U.S. Navy and the U.S. Public Health Service. NIST has hosted research associates from ADA since 1928. Currently, the ADA Health Foundation sponsors 32 research associates at NIST in the Metallurgy and Polymers Divisions. The collaborative relationship between that professional association and the federal government is unique, and continues to develop and transfer important new technologies to dentistry and medicine.

Project Title: **ADVANCED RESTORATIVE DENTAL MATERIALS**

Investigators: Gery R. Stafford, Christian E. Johnson, and David R. Kelley

Technical Description:

The occupational and environmental hazards associated with the use of mercury-containing dental alloys are a recurring source of public concern. Since dental amalgams have performed exceedingly well over more than one hundred years, the development of a direct filling material still based on the common constituents of dental amalgams, other than mercury, is desirable. The approach is based on three main processes: (1) the cold-welding of oxide-free silver; (2) the *in situ* formation of Ag_xSn_y intermetallics by the room temperature fast diffusion of silver and tin; and (3) the homogeneous precipitation of silver by Sn(II) in solution. This project is focused on acid-assisted consolidation of chemically precipitated silver powders and property measurements of hand-consolidated test compacts prepared with the tools and procedures normally employed by dentists. The observed values of flexural strength for the silver compacts were equal or superior to mercury amalgams. Corrosion resistance, microleakage and marginal toughness values of the compacts were found to be superior to those of amalgams. Wear and biocompatibility studies on the hand-consolidated compacts are in progress.

Technical Objectives:

- Demonstrate that the critical mechanical properties of metallic dental restoratives can be achieved in hand consolidated specimens without the use of mercury.

- Strive to reduce the time required to place the restoration to match that of amalgam, without sacrificing mechanical properties.

Anticipated Outcome:

- Development of silver-based compacts which have the advantage of being mercury-free and thereby provide an alternative metallic dental restorative in the event that mercury-containing restoratives are curtailed.

Accomplishments for FY 1996:

- A study on the effects of agglomerate size on the consolidation and transverse rupture strength (TRS) has revealed that no significant increase in TRS values was obtained by reducing the agglomerates size below 500 mesh, 31 μm.

- Modification of the silver precipitation process resulted in less agglomeration of the powder during precipitation. This has minimized the sieving of the dried powder to the extent that 200 mesh, 74 μm, powder agglomerates consolidate to the same TRS values as previously obtained with 500 and 625 mesh powders.

- Present precipitation processing of silver powders provides a material that will withstand 750°C heat treatment with virtually no sintering. This has resulted in a 5% increase in the consolidation density due to the increased deformability of the heat treated powders.

Impact and Technical Highlights:

- We have achieved 75-80% theoretical density of pure silver by the acid-assisted hand consolidation of precipitated silver powder. These compacts, which were consolidated using normal dental tools and procedures, have transverse rupture strengths of 100-140 MPa, compared to 115 MPa for a typical dental amalgam.

Outputs:

Publications:

Dariel, M. P., Levin, L., Ratzker, M., Stafford, G. R., Eichmiller, F. C., "Cold-Welding Effects in Silver Compacts After Acid-Assisted Consolidation, Powder Metallurgy Conference, Washington, DC (1996).

Presentations:

Johnson, C. E., "Alternative Dental Restorative Materials from Chemically Precipitated Silver by a Two-Solution Technique," IADR Annual Meeting - Advances in Dental Materials Sciences, San Francisco, CA, 1996.

ELECTRONIC PACKAGING AND INTERCONNECTION

Major elements of the U.S. microelectronics business group are the semiconductor and the electronic interconnection industries. Combined, these two industries deliver the power and functionality of semiconductor technology to the hands of the users. They also face changing consumer expectations, product evolutions, design cycles, and international competition with a pace and urgency never before seen in world commerce.

In response to the importance of electronic packaging to the microelectronics industry, this program is focusing on industry's most pressing challenges surrounding the utilization of advanced materials and material processes in **semiconductor packaging**, **electronic interconnection**, and **assembly**. Initiated in 1994, the program complements semiconductor fabrication activities supported by the NIST National Semiconductor Metrology Program and is linked via the world-wide web (www).

MSEL's program deals with industry's most pressing materials issues which are associated with the product and technology priorities contained within leading industry roadmaps. Roadmapping activities sponsored by major industry associations, namely *The National Technology Roadmap for Semiconductors*, *The National Technology Roadmap for Electronic Interconnects*, and more recently *The National Electronics Manufacturing Initiative*, have led to a portfolio of projects dealing in matters such as electrical, thermal, and mechanical characteristics of thin film materials; solders, solderability and solder joint design (www link); interfaces and adhesion; moisture measurement and control; and electromigration. These projects are conducted in conjunction with collaborators from numerous industrial consortia, individual companies, academia, and other government agencies.

The mission of MSEL's program is to develop and deliver to the U.S. electronics and electronic materials industries measurement tools and data for materials and processes used in semiconductor packaging, module interconnection and component assembly. The strategy we have developed to implement this program is based upon three primary needs.

- Develop techniques and procedures for making *in situ*, in process and in use measurements on materials and material assemblies having micrometer and submicrometer scale dimensions.

- Record and quantify the divergence of material properties from their bulk values as dimensions are reduced and interfaces are approached.

- Develop fundamental understanding of materials needed for future packaging, interconnection and assembly schemes.

More information about this program, and other NIST activities in electronic packaging, interconnection and assembly, is contained in *Electronics Packaging, Interconnection and Assembly at NIST: Guide and Resources* (www link), NISTIR 5817. This publication presents a complete inventory of NIST's activities in this area and lists information on how to contact project leaders.

Project Title: **LEAD-FREE SOLDERS**

Investigators: Carol A. Handwerker, Ursula R. Kattner, William J. Boettinger, John R. Manning, Frank Gayle, Maureen Williams, Leonard Smith, and Jonice Adams

Technical Description:

Scientists from NIST working with a broad-based industrial consortium organized through the National Center for Manufacturing Sciences (NCMS) are devising critical tests and providing materials property data to evaluate Pb-free solder alloys with respect to a wide range of manufacturing, performance, and environmental standards.

NIST and NCMS have a multi-year CRADA describing NIST participation in this $8M research and development program, which is supported almost entirely by in-kind contributions from each of its members. In addition to NIST, members include AT&T, Rockwell, Texas Instruments, Ford, GM-Hughes, GM-Delco, Hamilton Standard/United Technologies Research Center, Rensselaer Polytechnic Institute, and Sandia National Laboratories. NIST contributions to this industry-led program have included phase diagram determinations, materials fabrication, and test development for manufacturing performance and mechanical failure assessments.

Technical Objectives:

- Develop critical materials property test methods and data for alloy development.
- Determine the full range of possible alloy compositions consistent with manufacturing requirements for multi-element solders through calculation and experimental determination of phase diagrams.
- Identify new alloys for evaluation based on phase diagram and mechanical failure data.
- Fabricate candidate solder alloys for testing by consortium members.

Anticipated Outcome:

- New lead-free solder alloys identified that conform to specific manufacturing, performance and environmental standards.
- Lead-free alloys developed by consortium will be available for use by industry and their performance established before national or international legislation is passed that restricts lead use.
- Guidelines for alloy testing established for development of solder alloys that minimize expensive and time-consuming manufacturing and reliability trials.
- Recommended test methods will be applied to the design of other classes of lead-free alloys with longer fatigue life.

Accomplishments for FY 1996:

- The relationship between alloy composition and a new failure mode for plated-through-hole joint geometries was established. These results were used to identify the most promising lead-free solder alloys for use in through-hole applications.
- Reliability test vehicles were evaluated for the eight down-selected candidate alloys using cross-section metallography of solder joints and electrical monitoring data.

Impacts and Technical Highlights:

- An extensive analysis of multicomponent phase diagrams has been performed to determine which Pb-free candidate alloy systems have acceptable melting ranges as required for manufacturing.
- Solidification experiments were used to determine non-equilibrium phase formation in the solder during cooling from the liquid state. These experiments demonstrated that the effective solidus temperatures of some multicomponent solder alloys could be over 70°C lower than given by the equilibrium phase diagram.
- NIST has been asked to provide guidance on test methods and thermodynamic/phase diagram data in a new NCMS consortium for the development of fatigue-resistant solder alloys for high reliability, high temperature automotive applications.

Outputs:

Presentations:

Handwerker, C. A., "Solder Wetting and Spreading, Korean Ceramic Society Meeting, Seoul National University, Seoul, Korea, October 23, 1995

Handwerker, C. A., "Wetting of Solder Interconnects," Chonnam National University in Kwangju, Korea, October 24, 1995

Project Title: **HIGH TEMPERATURE SOLDER FOR MICROELECTRONICS**

Investigators: Frank W. Gayle, Ursula R. Kattner, William J. Boettinger, Maureen Williams, Leonard Smith, and Jonice Adams

Technical Description:

In this project, scientists from the Metallurgy Division are working with a broad-based industrial consortium organized through the National Center for Manufacturing Sciences (NCMS) to develop critical tests and evaluate performance of candidate microelectronics solder alloys for applications at high temperatures. The need for these new solder alloys is being driven by the automotive, telecommunications, and avionics industries, all of which have new demands for performance of interconnects beyond the current capabilities of lead/tin eutectic alloys. There is a particular focus on thermal fatigue of the alloys since this is regarded as the primary failure mechanism for solders in high temperature applications.

In addition to NIST, members of the consortium include Delco Electronics, Ford Motor Company, Allied Signal, Indium Corporation of America, Heraeus Cermalloy, Johnson Manufacturing, EMPF, Ames Laboratory, Rensselaer Polytechnic Institute, and Edison Welding Institute. NIST contributions to this industry-led program have included phase diagram determinations, materials fabrication, and test development for manufacturing performance and mechanical failure assessments.

NIST activities include the proposal and evaluation of candidate alloys based on available phase diagram data, the calculation of required multicomponent phase diagrams to determine the intermetallic phases over the manufacturing and operating temperature ranges, and the measurement and characterization of a wide range of mechanical, chemical, and thermal properties.

Technical Objectives:

- Identify candidate alloys based on an evaluation of available phase diagram data.
- Calculate selected multicomponent phase diagrams to determine the phases and melting behavior over the manufacturing and operating temperature ranges. Perform experiments to evaluate the solidification behavior of candidate alloys using metallographic analysis.
- Characterize the properties of the candidate solder alloys through a range of mechanical and thermomechanical tests.

Anticipated Outcome:

- New high temperature alloys identified with improved thermal fatigue performance for demanding applications.
- Guidelines for alloy testing established for development of alloys with high thermal fatigue resistance.

Accomplishments for FY 1996:

- Thermodynamic properties of 10,500 compositions were examined by NIST, resulting in over 600 candidate eutectic solder compositions with melting points in the desired temperature range.
- Evaluation and down selection of these alloys based on physical metallurgy principles and environmental considerations resulted in 83 candidates for the Consortium study.
- NIST has established the database for alloy down selection for the Consortium.
- A new approach to improving thermal fatigue resistance through doping at the ppm level of pure elements has been proposed at NIST. Initial experimental results are promising.

Impacts and Technical Highlights:

- A new concept has been proposed by NIST for thermal fatigue management through alloy composition variation which appears promising in preliminary experiments.
- Eighty-three promising candidate alloys have been identified and will now be evaluated by the Consortium in a series of experiments to determine manufacturability and thermal fatigue resistance.

Outputs:

Presentations:

Gayle, Frank W., "A Phase Diagram Approach To Development Of New High Temperature Solders." July 30-31, 1996, US Navy Electronics Manufacturing Production Facility, Indianapolis, IN.

Gayle, Frank, W., "NIST capabilities in Solder Science and Engineering," December 12, 1995, NCMS, Ann Arbor, MI. High Temperature Fatigue-Resistant Solder Consortium.

Gayle, Frank W., "Concepts In Alloy Design For Solders," February, 1996, Northwestern University, Evanston, IL, High Temperature Fatigue-Resistant Solder Consortium.

Project Title: **ELECTROCHEMICAL CHARACTERIZATION OF SOLDERS**

Investigators: Gery R. Stafford and Sandra W. Claggett

Technical Description:

The loss of solderability of printed wiring boards and component leads during storage is a major problem that costs the electronics industry millions of dollars each year. It is clear that surface oxidation is involved, but the nature of the various oxides and their role in the degradation process are obscure. This project focuses on the electrochemical evaluation of artificially aged copper as well as some of the organic coatings which are being considered for surface protection against aging.

Technical Objectives:

- Assist the electronic packaging industry in establishing benchmarks for electrochemical tests of standard surfaces. Electrochemical examination of the surface oxides of copper after exposure to four different accelerated aging procedures should provide information about the oxidation state and amount of oxide present on the surface.

- Initiate the electrochemical evaluation of imidazole-coated copper in borate buffer electrolyte. Imidazole coatings have been developed to protect copper surfaces from oxidation during storage.

- Take part in an industry-led round robin test program to determine the reproducibility and repeatability of the sequential electrochemical reduction analysis.

Anticipated Outcome:

- Electrochemical signature determined for each of the aged test coupons, thereby providing a comparison of the effects of accelerated aging conditions based on both the oxidation state and quantity of surface oxide present.

- Procedures for calibration of serial electrochemical reduction analysis determined for a range of surface finishes, including copper, imidazole-coated copper, and lead-tin coated copper.

Accomplishments for FY 1996:

- Experiments demonstrated that the amount and valence of oxide formed on copper coupons aged under different conditions can be determined by galvanostatic reduction. Reproducibility within a given set of coupons is quite good. Cu_2O was the primary species detected for all levels of aging; however, two of the aging conditions resulted in 1-2 monolayers of CuO on the outer surface.

- Several imidazole-coated copper printed wiring boards which had been exposed to air for one hour at 125 °C were examined by cathodic chronopotentiometry. The oxide coating on all of the samples tested consists of a single compound which appears to be Cu_2O. The charge density for oxide reduction varies somewhat between samples, ranging from about 10 to 15 mC/cm^2. This is considerably less than that obtained for copper aged without the imidazole coating.

- The Electrochemical Processing Group took part in the round robin test program to determine the reproducibility and repeatability of the sequential electrochemical reduction analysis. Copper and tin coupons were used in the evaluation. Five measurements were made at two current densities. The data was complied and submitted to the program coordinator.

Impact and Technical Highlights:

- Electrochemical reduction appears to be a promising technique for determining the oxidation state of copper, both qualitatively and quantitatively. This allows us to evaluate various accelerated aging treatment as well as the effectiveness of organic protective coatings. These measurements can be performed directly on printed wiring boards and component leads and may eventually be used to assess solderability.

Project Title: **SOLDERABILITY MEASUREMENTS AND OPTIMIZATION**

Investigators: J. R. Manning, W. J. Boettinger, F. W. Gayle, C. A. Handwerker, S. C. Hardy, D. Josell, U. R. Kattner, J. A. Warren, and M. E. Williams

Technical Description:

To meet the need for improved solder joint reliability, NIST is developing measurements, test techniques and scientific guidelines that U.S. manufacturers can use to evaluate components for solderability before committing them to the production line. For this purpose, measurements and modeling of wetting balance solderability tests, area-of-spread solderability tests, and solder wetting phenomena are underway.

Technical Objectives:

- Develop improved solderability tests using steam aging techniques to allow reliable accelerated testing.

- Analyze solder wetting phenomena that influence area-of-spread and wetting-balance solderability tests.

- Identify conditions that lead to poor reproducibility in presently-used solderability tests.

Anticipated Outcome:

- Improved solderability test methods which lead to increased manufacturability and reliability in microelectronic devices. Such increased reliability and predictability for solder joints will be essential for U.S. industry in producing surface mount and ball grid array interconnects, where small size scales and limits on visual inspection of the solder joint make rework of improperly soldered connections difficult or impossible.

- Predictive models that identify reasons for discrepancies among various experimental test methods. Improved reliability in testing will be obtained by introduction of needed correction terms and by identification of critical solderability parameters.

Accomplishments for FY 1996:

- Accelerated solderability tests using steam-aging methods have been analyzed in collaboration with the Institute for Interconnecting and Packaging Electronic Circuits (IPC) Solderability Subcommittee and Wetting Balance Task Group.

- Research has been conducted to identify possible sources of errors present in the interpretation of wetting balance tests. The relationship between the force measured during the test and the observed changes of contact angle and meniscus shape has been studied for a model hot silicone oil system and for solder. Discrepancies between the measured quantities and those expected based on the simple, but widely used, instantaneous static equilibrium assumption have been described.

- The discrepancies between the measured and expected quantities were analyzed to allow application of correction terms to wetting balance results. For rising menisci, the force discrepancy was found to be of the same order of magnitude as the shear stress exerted on the sample by fluid rising up the sample. For static menisci, force and meniscus height discrepancies may be explained by Marangoni flow caused by temperature gradients in the fluid for the oil but not for the solder experiments. The latter discrepancies are likely due to other factors such as solder oxide or flux.

- The dynamics of area-of-spread solderability tests have been studied using numerical simulation of the diffusive transport that occurs in the solder during reactive wetting. Analysis was performed for the simple case where no intermetallic is present but where partial dissolution of the substrate occurs.

Impacts and Technical Highlights:

- Results from a NIST-designed accelerated test using steam-aging techniques to allow more rapid testing have been incorporated into testing criteria for ANSI-J-STD 002 (Component Lead Solderability) currently under consideration by IPC.

- The results of a study identifying possible sources of error present in the interpretation of wetting balance tests, which inherently involve rising liquid menisci, have been published in the ASME Journal of Electronic Packaging. Identification of error sources will allow the application of needed correction terms for wetting balance solderability tests.

Outputs:

Publications:

Moon, K. W., Boettinger, W. J., Williams, M.E., Josell, D., Murray, B., Carter, W.C., Handwerker, C. A., "Dynamic Aspects of Wetting Balance Tests," accepted into the Journal of Electronic Packaging (1996).

Presentations:

Boettinger, W. J., "Reactive Wetting of Solder," Department of Materials, Swiss Federal Institute of Technology, Lausanne, Switzerland, August 13, 1996.

Handwerker, C. A., “Reactive Solder Wetting” Ceramic Microstructures ‘96 Conference in Berkeley, CA, June 25, 1996.

Handwerker, C. A., “Issues in Reactive Wetting,” Third International Workshop on Interfaces: Wetting, Fracture and Chemistry of Interfaces, University of Santiago, Santiago, Spain, Sept. 17, 1996.

Project Title: **SOLDER INTERCONNECT DESIGN TEAM**

Principal Investigators: James Warren, Carol A. Handwerker, Daniel Josell, and W. C. Carter

Technical Description:

The NIST Solder Interconnect Design Team, with support from NIST's Center for Theoretical and Computational Materials Science, has been formed to address several pressing issues in the design and fabrication of circuit boards. Having met frequently over the past three years, in partnership with both academic and industrial researchers, the Team has established an agenda for solving modeling problems concerning equilibrium solder joint shape, and the consequential thermal and mechanical properties of the formed joint. Our ultimate goal is to provide the industrial community with a suite of useful software tools for solder interconnect design, and to provide solved test problems (available electronically on the World Wide Web). With this in mind we are actively supporting the development of software that will interface the public domain program Surface Evolver, which has been shown to be quite capable at computing equilibrium solder meniscus shapes.

Technical Objectives:

- Develop modeling tools for predicting the geometries of small-scale solder joints with a wide range of starting configurations of interest to industry.

- Develop computational methods for providing solder geometries to other models of processing and reliability.

- Establish an industry-academia-national laboratory working group on solder joint design for the exchange of information and collaboration on topics of special importance.

- Provide a forum for discussion of the calculations and models and access to software through the Internet/www.

- Organize workshops and symposia for the SIDT and others in the electronic packaging community to promote collaboration and bring the community toward a consensus on the features required for a useful solder modeling system.

Anticipated Outcome:

- Improved software tools for the design of solder interconnects with higher manufacturing yields and reliability.

Accomplishments for FY 1996:

- NIST held two workshops for the Solder Interconnect Design Team.

- Members of the Design Team developed a 3D enmeshment of a surface evolver calculation imported into the ANSYS and PHYSICA finite element packages (by team members K. Brakke and C. Bailey, respectively).

- NIST provided support and guidance to the University of Wisconsin in developing a user-friendly interface to Surface Evolver.

Impacts and Technical Highlights:

- Two Solder Interconnect Design Team workshops were held this year at NIST to bring together scientists and engineers from industry, universities and national laboratories to share progress made in software development. Recent developments, including formulations of standard solder joint problems and feedback on software modifications, are shared through the Internet.

Outputs:

Publications:

Schwartz, L. H., Glotzer, S. C., Hall, D.E., Roosen, A. R., and Warren, J. A., "Materials Science in the Information Age," Technology in Society, Vol 18. 151-164 (1996).

Warren, J. A., Carter, W. C., Josell, D., Handwerker, C.A., Moon, K-W, "Modeling Solder Interconnects: A NIST Program," BIPC Printed Circuits, 1996, Technical Proceedings, S9-4 1-7 (1996).

Presentations:

Handwerker, C. A., "Numerical Methods for Calculation of Solder Joint Geometries," IPC's 39th Annual Meeting in San Jose, CA, March 6, 1996.

Handwerker, C. A., "Program Overview," Solder Interconnect Design Workshop, NIST, August 22, 1996.

Project Title: **STRESS MEASUREMENT IN ELECTRONIC PACKAGING**

Principal Investigator: Eva Drescher-Krasicka

Technical Description:

The accumulation of stress in electronic chips can have several different sources. Of these, the most important appear to be (1) condensation of moisture in plastics which can lead to "popcorning" during assembly processes and (2) thermal fatigue during use from the unavoidable mismatch of the coefficients of thermal expansion between the silicon die, the metal frame, and the polymer filler. If the stresses are high enough, cracking and delamination may occur in electronic packages.

Scanning acoustic microscopy (SAM) can be used routinely to detect popcorn cracks and delamination in electronic chip packages. There is a need, however, for fast and reliable detection of residual stresses in electronic and microelectronic components before measureable cracking occurs. This measurement need can be met by new (SAM) techniques developed at NIST for *in-situ* assessment of stress in electronic packaging.

In a collaboration with Texas Instruments and Sandia National Laboratories, NIST scientists are developing calibration techniques for this SAM technique, known as Scanning Acoustic Imaging of Stress (SAIS). Electronic packages containing arrays of Auburn University's Silicon (stress) Sensor Chips were examined before and after exposure to thermal and mechanical shock. Finite element analysis provided from Sandia and from Texas Instruments are qualitatively confirming SAIS results.

Technical Objectives:

- Develop a test methodology to measure residual stresses in multilayered electronic packaging containing Silicon Sensor Chips and calibrate the SAIS technique based on sensor readings.

- Apply Scanning Acoustic Imaging of Stress for stress detection in electronic chips without mechanical defects in order to evaluate the level of residual stress before mechanical failure (cracks, delaminations).

Anticipated Outcome:

- Technique developed to detect areas with high residual stresses in electronic chip components.
- Quantitative levels of residual stresses in electronic packaging obtained from calibrated images of SAIS.
- Comparison of stresses measured from calibrated acoustic images with the stresses calculated using finite element modeling and analytical solutions.

Accomplishments for FY 1996:

- Longitudinal and shear waves were used to detect cracked areas and stressed areas around silicon dies. Theoretical and experimental studies on delamination detection by acoustic microscopy suggest that a phase inversion technique for delamination detection is affected by high residual stresses in the area of delamination. This is presently being pursued as a joint venture between NIST and Texas Instruments (Dr. Tom Moore).
- The acoustic images obtained using a 150 MHZ surface leaky acoustic wave were found to agree qualitatively with finite element analysis in the area close to the die. This experiment was done by applying the surface leaky acoustic wave on a milled sample. The sample, thinned to 600 microns, was provided by Texas Instruments. The SAIS results confirmed FEM calculations showing the maximal principal stresses peak at the corners of the die.

Impacts and Technical Highlights:

- In cooperation with Sonix Inc., NIST is preparing software and test procedure guidelines for routine measurement and control of residual stress in electronic components.

Project Title: **SOLDER JET PRINTING FOR MICROELECTRONICS APPLICATIONS**

Investigators: F. W. Gayle, L. C. Smith, M. E. Williams, and J. Adams

Technical Description:

The printing of solder patterns onto chips or circuit boards using ink jet printer technology is a novel means to avoid cumbersome screening techniques and environmentally unfriendly electroplating methods. In addition, "solder jet" technology is flexible (patterns can be input by keyboard) and capable of very fine pitch between solder deposits (< 100 μm). These capabilities are needed for the next generation of microelectronic packaging.

The NIST Metallurgy Division is working closely with ATP Awardee MicroFab Technologies, of Plano, Texas, and a consortium including Delco Electronics, AMP, Universal Instruments, Texas Instruments, Eastman Kodak, and Motorola, to bring solder jet technology closer to commercial reality. NIST scientists are focusing on materials compatibility issues involving the liquid solder and the jetting apparatus, and reactions between the jetted solder and the various substrate materials used in microelectronic interconnects.

Technical Objectives:

- Provide technical assistance to MicroFab in issues of materials compatibility.

- Collaborate with MicroFab and its customers in determining system requirements for reliable solder jet operation and for product (solder joint) performance.

- Characterize the behavior of solder jet deposits onto different substrates under a range of deposition conditions, including joints fabricated through reflow.

Anticipated Outcome:

- Materials compatibility issues developed for next generation devices for creating solder patterns.

- Better understanding of solder joint formation after reflow for solder deposited using jet technology.

Accomplishments for FY 1996:

- Contamination of incoming solder has been identified as a major cause of poor reliability of the solder jets. A test for screening solder for iron contamination in the form of $FeSn_2$ intermetallic particles has been developed in the Metallurgy Division.

- A number of specimens provided by MicroFab were cross sectioned and examined for wetting, wetting reactions, and dissolution of substrates under various jetting conditions.

- Processing windows for substrate temperature, jet height, etc., were found which allow necessary wetting of substrates but without excessive interfacial reaction. Certain conditions unfavorable to reflow were also found.

Impacts and Technical Highlights:

- Research at the Metallurgy Division has identified temperature regimes which are suitable for deposition onto Au-coated silicon, enabling good metallurgical bonds to form without compromising joint integrity with excessive intermetallic growth.

- The solder quality measurement technique which we developed has been used to determine quality of solders used in the program. The Consortium has requested that we use this technique to screen their new supplies of solder outputs:

Outputs:

Presentations:

Gayle, Frank, W., "Failure Mechanisms in Solder Jetting." May, 1996 Presentation at Solder Jet Consortium, at GM Delco, Kokomo, IN.

Gayle, Frank, W., "Materials Compatibility Issues in Solder Jetting." August 8-9, 1996, Eastman Kodak, Rochester, NY.

Gayle, Frank, W., "Solder Jetting - from Development to Application" presented to U.S. Senate Task Force on Manufacturing, November 1, 1996.

EVALUATED MATERIALS DATA

The objective of the Evaluated Materials Data 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 databases are developed in cooperation with the NIST Standard Reference Data Program Office and, often, coordinated with the activities of other laboratories and scientific/technical societies. Research consists of the compilation and evaluation of numeric data as well as recently initiated efforts directed at more effective distribution and use of data. Database activities reflect laboratory programs with scientific capabilities required for appropriate data evaluation. The Metallurgy Division is currently involved in one major database project: the NACE/NIST Corrosion Performance Database developed to provide a means to select structural alloys for corrosive applications.

Project Title: **CORROSION DATA PROGRAM**

Investigators: C. P. Sturrock, C. D. Flanigan, and B. Mashayekhi (NACE Research Associate)

Technical Description:

In 1978, NIST and Battelle Columbus Laboratories estimated that corrosion costs the U.S. economy 4.2% of the gross domestic product. Today, this would be over $200 billion per year or more than $1000 per person each year. Also, it was estimated that a significant portion of this cost could be prevented by using existing knowledge of corrosion. To make this knowledge more readily available to industry, NIST joined with NACE International (formerly the National Association of Corrosion Engineers) in 1982 to form the NACE-NIST Corrosion Data Program (CDP). The overriding goal of the CDP is to enhance the cost effective practice of corrosion prevention by collecting, organizing, evaluating and disseminating in computerized form, corrosion and materials performance information.

Technical Objectives:

- Develop materials performance informatics that are:
  - accurate and practical in technical content,

- relevant to industry's engineering needs,
- convenient and easy to use,
- representative of the latest advances in computing technology, and
- available at reasonable cost to the user.

- Organize workshops and symposia to bring the materials performance information community towards consensus on materials performance informatics and related standards.

- Collect experimental data (from laboratory and field exposure tests) and examine for trends via automated induction, using such tools as connectionist models, machine learning, pattern recognition, etc.

Anticipated Outcomes:

- Reliable materials performance informatics which are used by industry to improve the design of their products and production processes, strengthening their competitive position while improving customer and employee safety.

- Standards and recommended practices for the development of materials performance informatics.

- Identification of previously unknown patterns and/or correlations implicit within materials performance data.

Accomplishments for FY 1996:

- The CHEM•COR series of expert systems addressing the handling and storage of chemical process industry corrosives was completed for public distribution in FY 1996 with the conversion of modules 6-12 to the LAN version. These expert systems were developed under a nine year project with funding and corrosion expertise provided by the Materials Technology Institute, a consortium of some 40 companies that sponsors nonproprietary research in materials performance of interest to the chemical process industries. Material selection and performance advice is provided by the individual expert systems for the following corrosives:

CHEM•COR 1:	Concentrated sulfuric acid
CHEM•COR 2:	Acetic acid
CHEM•COR 3:	Formic acid
CHEM•COR 4:	Hydrogen chloride
CHEM•COR 5:	Chlorine
CHEM•COR 6:	Sodium hydroxide
CHEM•COR 7:	Phosphoric acid
CHEM•COR 8:	Once-through waters

CHEM•COR 9:	Hydrogen fluoride
CHEM•COR 10:	Dilute sulfuric acid
CHEM•COR 11:	Nitric acid
CHEM•COR 12:	Ammonia

- An advanced materials performance expert system available on microcomputers, known as the AUSCOR program, was completed and released for review and evaluation by the project sponsor, the Materials Technology Institute. The AUSCOR program can predict the performance of some 28 stainless steel and nickel-based chromium-bearing alloys in actual service across a wide range of aqueous environments. AUSCOR is considered an advanced system because part of its reasoning is based on mixed potential theory, which provides for variations in oxidizing capacity, temperature and velocity of the electrolyte, galvanic coupling of the alloy in question with other metals, changes in the active-passive behavior of the alloy, and numerous other factors generally considered only by the most informed and experienced corrosion experts in the industry.

- The POWER•COR software series addressing corrosion issues in the electric power industry was completed in FY 1996. These modules were developed in a six year project with funding and corrosion expertise provided by the Electric Power Research Institute, a consortium of some 600 utility companies, and address the following topics:

POWER•COR 4:	Fatigue crack growth rates for structural materials in nuclear environments
POWER•COR 5:	Corrosion in wet flue gas desulfurization systems
POWER•COR 6:	Corrosion in service water systems

- Three papers describing evaluation of various inductive learning methods using typical corrosion databases were published in FY96. Of the methods studied, which included classical and nonparametric statistical methods, connectionist models, and methods originating in machine learning, the decision tree approach was found to provide the best combination of accuracy and scrutability in predicting an output from a set of known values of inputs.

- To better serve users, the original COR•SUR (metals) and COR•SUR2 (nonmetals) databases are being updated from a DOS environment to a Windows-based application. Sales of the original database have exceeded 2000 copies, and they are believed to be the most widely distributed corrosion database. The new COR•SUR will contain information on more than 50 metals exposed to almost 1000 environments, while the new COR•SUR2 allows access to over 40 nonmetallics in almost 900 chemical environments. Over 500 footnotes provide additional information.

Impact and Technical Highlights:

- One of the member companies of the Materials Technology Institute reported that its occasional use of one of the 12 CHEM•COR expert systems saved two to three times its investment in the entire series.

- A workshop was conducted near the end of the fiscal year to consider future Metallurgy Division database projects.

Outputs:

Publications:

Sturrock, C. P. and Bogaerts, W. F., "Computer Learning Systems in Corrosion," Computers in Corrosion Control - Volume V, NACE International, Houston, TX, 1996.

Sturrock, C. P., and Bogaerts, W. F., "Classification and Prediction of Corrosion Phenomena via Cluster Analysis," Computers in Corrosion Control - Vol. V, NACE International, Houston, TX, 1996.

Sturrock, C. P. and Bogaerts, W. F., "Reasoning About Corrosion from Cases Instead of Rules," Modeling Complex Data for Creating Information: Real and Virtual Objects, J. E. Duboit and N. Gershon, eds., Springer-Verlag, Berlin, 1996.

Presentations:

Sturrock, C. P., "Classification and Prediction of the Corrosion Behavior of Nickel-Containing Alloys from Field Test Data," ASTM Fifth International Symposium on Networking of Materials Databases, Tsukuba, Japan, Nov. 6, 1995.

Sturrock, C. P., "Automation for Discovery, Learning and Engineering of Corrosion Knowledge," keynote address, EUROCORR '96, Nice, France, Sept. 25, 1996.

HIGH TEMPERATURE SUPERCONDUCTIVITY

A significant program in 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 carrying out this program, researchers in MSEL work closely with their counterparts in other NIST Laboratories, and collaborators in U.S. industry, universities, and other National Laboratories.

The primary thrusts of the program are as follows:

- Phase equilibria. Work is being performed 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. Use is made of a unique magneto-optical imaging facility to examine flux pinning in a variety of materials, with much of this work being conducted in collaboration with American Superconductor Corporation. In addition techniques for better interpretation of magnetic measurements are being developed. Structure and dynamics of flux lattices and melting phenomena, critical to applications, are investigated with small-angle neutron scattering techniques.

- Damage mechanisms. Work is being carried out 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 temperature 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 scattering techniques and profile refinement analyses are being utilized to investigate crystal and magnetic structures, composition, dynamics and crystal chemical properties. This research is being carried out in collaboration with a number of industrial and university experts and researchers at National Laboratories.

Project Title: MAGNETIC PROPERTIES OF SUPERCONDUCTORS

Investigators: L. J. Swartzendruber, L. H. Bennett, R. D. Shull, F. W. Gayle, A. J. Shapiro, D. E. Mathews, H. J. Brown, and R. V. Drew

Technical Description:

In collaboration with scientists from universities, industry, and other Divisions at NIST, superconducting materials are prepared and their microstructure and magnetic properties determined. The properties of impurities which form during processing of superconductors, or which are intentionally added to modify the properties, are also investigated. Measurements performed include ac and dc magnetization as a function of temperature and applied magnetic field, hysteresis loops, flux penetration and viscosity, critical fields, and critical temperatures. Microstructure studies are performed using scanning and transmission electron microscopy. Emphasis is on high temperature superconducting materials.

Technical Objectives:

- Improve present magnetic measurements and develop new measurements for superconductors.

- Provide support to the Ceramics Division in the determination of phase diagrams important for the processing of high temperature superconductors.

- Investigate the magnetic properties of new superconducting materials and of materials prepared under varying processing conditions.

- Determine the effect of various microstructural features such as inclusions, compositional modulation, and precipitates, on the flux pinning in high temperature superconductors.

- Develop relationships between ac and dc susceptibilities and the impurities and phase distributions present in the material.

Anticipated Outcome:

- Improvements in the ability of manufacturers and researchers to interpret magnetic measurements in high-temperature superconductors.

- Improvements in the ability of manufacturers to characterize and control the characteristics and quality of material they produce.

- Increased critical current densities by improvements in flux pinning.

- Better control over the flux pinning properties of materials for shielding and levitation bearings.

Accomplishments in FY 1996:

- The effect of lanthanide additions on the micro crack density and critical currents in melt processed $YBa_2Cu_3O_{6+x}$ was determined.

- The effect of impurity compounds which are present in various proportions in the high temperature thallium superconductor $ThBa_2Ca_2Cu_3O_x$ was measured

Impacts and Technical Highlights:

- Commercial devices using high temperature superconductors are currently available. Many of these devices are being fabricated using laser ablation, a method which was developed by NIST in cooperation with the Johns Hopkins Applied Physics Laboratory.

- Our explanation of "inverse levitation" in terms of the effect of flux pinning on the magnetic properties of high temperature superconductors opens up many possibilities (e.g., magnetic bearings) for the use of these materials in devices.

- An instrument, developed by NIST in cooperation with the Institute of Solid State Physics in Russia, for observing the flux distribution in superconductors and other magnetic materials won an IR100 award and has been commercialized by a U.S. Company.

- An explanation of the reason for a measurement artifact, often referred to as a "positive Meissner effect," and the effect of impurity compounds on magnetic measurements of superconductors enables better interpretation of magnetic measurements of high temperature superconductors.

Outputs:

Publications:

Turchinskaya M., Kaiser, D.L., Shapiro, A.J., Riley Jr., G.N., and Christopherson, C., "Nondestructive Magneto-optical Imaging Analysis of Superconducting $(Bi,Pb)_2Sr_2Ca_2Cu_3O_X$ Composites", J. Mater. Res., vol. 11, No. 7, 1957 (1996).

Seyoum, H.M., Melamud, M., Wong-Ng, W., Bennett, L.H., Swartzendruber, L.J., Cook, L., and Brown, H.J., "Effect of Barium Cuprate on High Temperature Superconductors," submitted to J. Appl. Phys. (1996).

INTELLIGENT PROCESSING OF MATERIALS

Intelligent processing of materials (IPM) is the conversion of materials into value-added products using model-based control of processing variables. Information for real-time process control is provided by on-line sensors which measure material characteristics and/or processing conditions. Intelligent processing will enable industry to economically produce materials with improved quality, consistent properties, and enhanced functionality. The IPM Program makes important contributions to three MSEL strategic thrusts: advanced processes, advanced materials, and measurement technology.

The IPM concept is the principal approach used to achieve the MSEL goal to "Foster the development and implementation of technologies for advanced processing of materials." The central elements of IPM are 1) process understanding expressed in terms of a process model, 2) real-time information on processing parameters and material condition obtained with on-line process sensors, and 3) a model-based sensing and control strategy to achieve the desired characteristics in the finished product. IPM projects advance each of the elements, and joint projects with industry are integrating these elements into improved processing capabilities.

The IPM Program is an important contributor to the MSEL goal to "foster the use of advanced materials in commercial products." Advanced materials are materials with microstructures which are designed and controlled to provide superior properties and performance for specific functions. Microstructural control is perhaps the most important application of IPM. The idea is to model microstructural evolution during processing, sense microstructural changes in real time, and use a model-based control strategy to achieve the desired microstructure in the finished product. Microstructural consistency is essential to the commercialization of advanced materials because it assures reliable properties and performance of the material.

The IPM Program also contributes to MSEL's measurement technology goal. A major focus of the IPM projects is process sensors, which our industrial collaborators repeatedly identify as a crucial need. Sensor technology is a core competence of MSEL which has its roots in sensor development for nondestructive evaluation of materials. Unique MSEL capabilities are being used to measure thermophysical properties of multi-component metals at elevated temperatures; these data are combined with model enhancements and then incorporated in industrial software for metal casting. In addition, magnetic sensors are being developed to non-destructively assess the variability in yield stress of steels.

In the Metallurgy Division, many of the projects in the Intelligent Processing of Materials Program are part of the Consortium on Casting of Aerospace Alloys, which links NIST to the leading companies in the aeropropulsion industry, including engine manufacturers and the foundries which supply them, as well as a company which produces software for modeling the investment casting process. These projects contribute in several ways to the enhancement of the software, for example by providing precise measurements of the thermophysical properties of alloys at high temperatures, by developing models which allow use of thermodynamic data bases to predict alloy compositions, and by identifying thermal conditions which lead to formation of defects in the castings. In each project, the collaboration of NIST investigators with the industrial scientists assures that the data and models developed are directly applicable to the industrial problems.

Project Title: **SOLIDIFICATION PATH FOR CASTING OF MULTICOMPONENT AEROSPACE ALLOYS**

Investigators: William J. Boettinger, Ursula R. Kattner, Sam R. Coriell, and Ann Bolcavage

Technical Description:

This project provides a method to predict the fraction solid (and heat content) vs. temperature relationship for multicomponent superalloys. This information is necessary for accurate macroscopic heat flow modeling used to determine soundness of casting. The information also predicts the identity and volume fraction of all phases present in the microstructure. A combination of multicomponent phase diagram calculations with a kinetic analysis of solidification microsegregation is being applied to superalloys. Models will be developed in a form to be implemented into a commercial software code for castings as part of the NIST Consortium on Casting of Aerospace alloys.

Technical Objectives:

- Develop thermodynamic data base for Ni-base superalloys for the calculation of tie-line information required for the solidification models.

- Develop solidification kinetic models for multicomponent superalloys to treat the dendritic aspects of solidification.

- Integrate the thermodynamic data base and solidification kinetic models into commercial casting software.

Anticipated Outcome:

- Improved quality of simulation of investment castings by industry.

- More reliable prediction of casting defects.

- Reduction of casting reject rate.

Accomplishments for FY 1996:

- An algorithm has been developed that couples a microscopic solidification model to a macroscopic heat balance code developed for castings. The algorithm employs subroutines that deliver phase diagram data for multicomponent alloys to the micromodel. Since the coupling occurs during the run time of the macroscopic code, the effect of local cooling history is included.

- The phase diagram subroutines, which are used for the micro/macro coupling, have been enhanced to: (a) deal with an arbitrary number of components, (b) allow SGTE compatible representation of the specific heat C_p and magnetic effects in a format compatible with that used to calculate the Gibbs energy (permitting enthalpy vs. temperature predictions), (c) employ Redlich-Kister polynomials for binary interactions and (d) allow use of the compound energy model (important to describe the γ' (Ni_3Al) phase).

- Liquidus and solidus data and partition coefficients have been measured for several Ni-Al-Cr-Ta alloys to permit refinement of the thermodynamic data base used in the above subroutines.

- A model for secondary arm spacing and equiaxed solidification in multicomponent alloys with different liquid diffusion coefficients was formulated in collaboration with M. Rappaz of EPFL in Switzerland.

Impacts and Technical Highlights:

- NIST algorithm for micro/macro coupling of the solidification path has been incorporated into ProCAST^{TM}, a commercial software code as part of the NIST Consortium on Casting of Aerospace Castings.

Outputs:

Publications:

Bolcavage, A., and Kattner, U. R. “Reassessment of the Calculated Ni-Nb Phase Diagram,” J. Phase Equilibria **17** , pp. 92-100 (1996).

Kattner, U. R., Boettinger, W. J., and Coriell, S. R., “Application of Lukas Phase Diagram Programs to Solidification Calculations of Multicomponent Alloys,” Z. Metallkunde, **87**, p.522 (1996).

Presentations:

Boettinger, W. J., "Multicomponent Alloy Solidification," Annual Meeting, Consortium on Casting of Aerospace Alloys, NIST, May 2, 1996.

Boettinger, W. J., "Prediction of Solidification Path for Ni-Base Superalloys," AeroMat'96 (7th Annual Advanced Aerospace Materials and Processes Conference), ASM International, Dayton, OH, June 6, 1996.

Boettinger, W. J., "Modelling Multicomponent Solidification Using a Thermodynamic Phase Diagram Approach," Department. of Materials, Swiss Federal Institute of Technology, Lausanne, Switzerland, August 22, 1996.

Project Title: **MODELING OF ALPHA CASE THICKNESS IN TITANIUM CASTING**

Investigators: W. J. Boettinger, S. R. Coriell, and U. R. Kattner

Technical Description

The surfaces of Ti investment castings are contaminated with oxygen because of reaction with the oxide mold material. This brittle surface layer, called alpha case, must be removed before use. Prediction of the thickness of the contaminated layer would allow modification of mold design and gating to reduce the alpha case. It would also provide a proper estimate of the extra dimension to be added to the casting to allow for alpha case removal.

Technical Objectives:

- A diffusion-based model, which uses temperature-time history at points of interest along the surface of a casting obtained from a thermal model, will be developed to predict the oxygen penetration normal to the surface.

- The results of this modeling effort will be a module of code that can be appended to a heat flow model of the casting.

Anticipated Outcome:

- This research will produce an enhancement of a commercial software code used to model castings. This enhancement will predict the thickness of alpha case over the surface of a Ti investment casting.

- Investment casters will be able to determine the optimal extra dimension to be added to Ti castings to allow for alpha case removal.

Accomplishments in FY 1996:

- To predict the penetration of oxygen into a Ti casting, a diffusion model was developed that used the simulated temperature-time data as input. It was tested and compared to experimentally measured alpha-case thicknesses obtained from a step wedge casting. Good agreement was found between the predicted and measured α-case thickness, which ranged from 0.2 mm to 0.8 mm.

- A report summarizing the results of project has been prepared and this project has been completed.

Impact and Technical Highlights:

- The diffusion model has been added to the ProCAST™ code for modeling of castings and delivered to members of the NIST Consortium on Casting of Aerospace Alloys. The code was also delivered to United Technologies Research Center (A. Giamei).

Outputs:

Presentations:

Boettinger, W. J., "Alpha-case Formation in Ti Castings," Annual Meeting, Consortium on Casting of Aerospace Alloys, NIST, May 2, 1996.

Project Title: **GENERATION OF GRAIN DEFECTS NEAR CORNERS AND EDGES IN CASTINGS**

Investigators: R. J. Schaefer, W. J. Boettinger, M. D. Vaudin, and D. R. Black

Technical Description:

This project seeks to characterize the grain defects which occur in single-crystal nickel-based metal castings, identify the processing conditions which lead to their formation, and identify steps which industry could take to reduce their incidence. The defects under study are regions of the casting in which the crystal structure is significantly misaligned, which makes the parts unacceptable for the demanding aeropropulsion applications for which they are manufactured. The trend toward increasing size and complexity of single crystal components results in increasing incidence of these defects. There are a variety of mechanisms by which such misalignments can occur, and several of them are accentuated by the need for crystals to grow around corners or through regions of changing cross section. Crystallographic analysis helps to identify the mechanism of formation of specific defects, and modeling helps to identify the thermal conditions under which they form. The work on this project is carried out as one of the activities of the Consortium on Casting of Aerospace Alloys, that links NIST to a group of industries, universities, and government agencies.

Technical Objectives:

- Develop a classification system, based on the experience of the Consortium's industrial members, to identify the specific types of defects which cause single-crystal superalloy castings to be rejected during inspection.

- Characterize the geometry and crystallography of the different types of defects and identify solidification mechanisms which could lead to the production of such features.

- Identify thermal conditions during solidification of the casting which cause defects to form by the action of the proposed solidification mechanisms.

- Identify modifications of casting practice which could reduce the incidence of specific types of defects.

Anticipated Outcome:

- Improved guidelines for the design of single crystal casting processes to avoid the conditions which lead to defect formation.

- More precise criteria for classifying defects in single crystal castings and thus for identifying the conditions which are responsible for their formation.

Accomplishments for FY 1996:

- A model for lateral spreading of dendrites on sloping mold surfaces was developed that included steady-state and transient prediction of supercooling at the advancing dendritic solidification front.

- Dendrite misorientations were determined by electron backscattering patterns (EBSP) in a highly misaligned region of a large superalloy single crystal sample.

- Excellent agreement was demonstrated between the shape of the solidification front predicted by an analytical model and that predicted by a cellular automaton - finite element model developed by workers at Ecole Polytechniqe Federale de Lausanne (Switzerland).

Impacts and Technical Highlights:

- Criteria were proposed for prediction of mold geometries and growth conditions leading to defect formation in single crystal castings.

Outputs:

Publications:

Gandin, Ch.-A., Schaefer, R. J., and Rappaz, M., "Analytical and Numerical Predictions of Dendritic Grain Envelopes," Acta Met. et Materiala **44**, 3339-3347 (1996).

Presentations:

Schaefer, R. J., "Defect Formation in Single Crystal Superalloy Slabs," Semi-Annual Meeting, Consortium on Casting of Aerospace Alloys, Boulder, CO. November 6, 1995.

Schaefer, R. J., "Defect Formation Mechanisms in Single Crystal Superalloys," Annual Meeting, Consortium on Casting of Aerospace Alloys, NIST, May 2, 1996.

Project Title: **POROSITY IN CASTINGS**

Investigators: William J. Boettinger, Robert J. Schaefer, Sam R. Coriell, Ursula R. Kattner, and Rodney D. Jiggetts

Technical Description:

Porosity is a common defect in castings. The amount and geometry of porosity vary considerably among the various casting methods. This project is focused on two extremes: porosity found in die castings and porosity found in investment castings of superalloys. In die castings, used for example for brake components, the contiguity of the porosity with the external surface can cause leakage. Thus, one part of the project is focused on methods to establish the geometry of porosity. In investment castings, the porosity is typically more isolated. Here models are sought to describe the location and volume fraction of microporosity using an accurate description of the pressure at each point in the liquid in the mushy zone of a casting. The pressure varies due to the fluid flow required to feed the solidification shrinkage. Such a calculation requires a knowledge of the density of the liquid and solid as a function of temperature and liquid or solid composition, a description of the solidification path (temperature and composition of liquid and solid as a function of fraction solid) and a description of the permeability of the mushy zone.

Technical Objectives:

- Develop the use of hot isostatic pressing (HIP) as a diagnostic tool for the evaluation of the connectivity of porosity in castings.

- Develop a melt flow model during solidification of multicomponent superalloys to treat the pressure drop required to feed solidification shrinkage.

- Integrate porosity formation models into commercial casting software as part of the NIST Consortium on Casting of Aerospace Alloys.

Anticipated Outcome:

- Improved measurement of the way in which porosity in castings is interconnected, indicating both the origin of the porosity and the influence of the porosity on performance in hydraulic applications.

- More reliable prediction of casting defects and reduction of casting reject rate.

Accomplishments for FY 1996:

- Hot isostatic pressing cycles needed to close porosity in aluminum die castings were developed.

- The response of porosity in die castings to HIP treatment was determined in terms of the origin of different types of porosity.

- A method was developed to compute the pressure drop required to feed the density change in a dendritic mushy zone during directional solidification of multicomponent alloys. The magnitude of the drop depends on the temperature gradient and the solidification velocity and also the nonequilibrium freezing range of the alloy.

- The fraction of microporosity was predicted for a four component Ni-base superalloy using the above pressure calculation and a simple model for pore nucleation.

Impacts and Technical Highlights:

- Discussions have been initiated with members of the NIST Consortium on Casting of Aerospace Alloys on ways to couple this approach to commercial software

- Data on fraction solid vs. temperature for Al-Cu-Si alloy were delivered to Allied Signal for use in modeling of die casting in their ATP program on automotive parts.

Project Title: **THERMOPHYSICAL DATA FOR CASTINGS**

Investigators: Ared Cezairliyan, John McClure, Tsuyoshi Matsumoto, and Debasis Basak

Technical Description:

This project focuses on the accurate determination of selected thermophysical properties of high temperature alloys of technological interest, particularly Ni-base superalloys important to the NIST Consortium on Casting of Aerospace Alloys. Millisecond- and microsecond-resolution pulse-heating techniques are used to make measurements in both solid and liquid phases up to about 300 K above their melting region. Work focuses primarily on measurements of selected key properties, such as enthalpy, specific heat, heat of fusion, electrical resistivity, hemispherical total emissivity, and normal spectral emissivity.

Technical Objectives:

- Obtain accurate thermophysical properties data on selected multicomponent alloys of technological interest, primarily those used in the aerospace industry, in support of modeling of casting processes.

- Develop methods of estimating properties of an alloy from the properties of its constituent elements.

- Obtain accurate thermophysical properties data on selected well-characterized binary and ternary alloys in support of validation of the estimation methods.

Anticipated Outcome:

- Generation of accurate thermophysical properties data on alloys needed by the casting industry.

- Development of reliable methods for estimating properties of multicomponent alloys from the properties of constituent elements.

Accomplishments in FY 1996:

- Definitive measurements of the properties (enthalpy, specific heat, electrical resistivity, normal spectral emissivity, and solidus temperature) of a nickel-base alloy (Mar-M247) were made in the solid phase near the melting region. This alloy is used for fabrication of blades in some superalloy jet engines.

- Preliminary experiments were performed on the binary alloy 53Nb-47Ti to measure enthalpy and electrical resistivity of the solid alloy near the melting region, heat of fusion, and enthalpy, specific heat, and electrical resistivity of the liquid alloy up to near 2500 K (about 300 K above the melting region).

Impacts and Technical Highlights:

- NIST-generated thermophysical properties data on selected alloys are used in modelling of casting processes by major U.S. industrial companies which cast alloys for aerospace applications.

- Accurate data on solidus and liquidus temperatures will significantly improve the phase diagrams of some high-temperature alloys.

Project Title: **MAGNETICS FOR STEEL PROCESSING**

Investigators: L. J. Swartzendruber, G. E. Hicho, D. E. Mathews, R. V. Drew, and R. D. McMichael

Technical Description:

Sheet steels, especially high strength low alloy steels, are extensively used in the construction of automobiles and other structures which require a high strength to weight ratio combined with low cost and formability. Knowledge of the yield strength of such materials, before, during, and after fabrication is critical for their efficient use. Since magnetic properties can be measured nondestructively at any time during the use of these steels, the use of magnetic properties to characterize the mechanical and metallurgical properties of steel products is being investigated. Magnetic properties can be measured rapidly and nondestructively, whereas direct mechanical measurements are time consuming, require special sample preparation, and must be done destructively. Extensive measurements have demonstrated that, for specific types of steel, a close correlation can be shown between mechanical properties and a combination of measurable magnetic properties. This creates the potential for the use of magnetic sensors for real-time on-line determination of a steel's mechanical properties.

One of the barriers to the application of magnetic methods to continuous on-line measurements during steel production is the requirement for close contact between the magnetic sensor and the steel, a contact difficult to achieve on rapidly moving sheet steel in a steel mill. The development of improved sensors that can operate at an increased surface-sensor distance would provide for a greatly expanded use of magnetic methods in steel property control and would contribute to improving quality and reducing production costs of sheet steel.

Technical Objectives:

- Collect experimental data on the magnetic and mechanical properties of a wide variety of commercial steels as a function of thermomechanical treatment and environmental exposure (e.g. hydrogen content).

- Isolate and measure the individual microstructural features (e.g. dislocations, grain size, and precipitates) that contribute to the mechanical and magetic properties of a steel. Determine if a combination of magnetic properties can distinguish between these various features.

- Develop sensors for measuring the magnetic properties of steel in various geometries.

- Use state-of-the-art analog to digital conversion and digital processing techniques to process sensor outputs.

- Use magnetic methods to determine the ferrite and austenite contents in stainless steels.

Anticipated Outcome:

- Improved understanding of the relationship between the magnetic and microstructural properties of steel.

- Reduction in the number of costly and time-consuming mechanical tests required.

- More consistent production of large quantities of steel with well controlled and uniform mechanical properties.

- Production of lighter weight mechanical structures though improved control of the mechanical properties of high strength low alloy steels.

- Better control of the magnetic properties of low carbon steels used in the production of low cost motors and transformers.

- Development of methods for testing for the presence of in-service property degredation in steel structures (e.g., hydrogen embrittlement).

Accomplishments in FY 1996:

- The relationship between mechanical deformation and the magnetic properties were determined for a commercially-produced, ultra-low-carbon sheet steel.

- The applicability of multi-parameter models for relating yield strength to magnetic properties was extended to low carbon sheet steel and a high strength low alloy sheet steel.

- A patent (U.S. Patent Application Serial No. 08/503,263) was awarded to NIST for the measurement of steel hardness using a Barkhausen signal method. The rights to this patent are assigned to the American Iron and Steel Institute under the terms of a CRADA.

- A system for combining Barkhausen and ultrasonic measurements in a single hand-held instrument was proposed (in conjunction with George Alers at the Boulder Lab). The development of such an instrument is currently being pursued.

- The changes in magnetic properties in a high yield strength AISI 4340 steel were measured as a function of hydrogen charging.

Impacts and Technical Highlights:

- Participants in an industry-government study of magnetic measurement of yield strength became aware of the capabilities of magnetic test methods and the restrictions on their in-situ application based on equipment requirements. In particular, the requirement of knowing the distance between the magnetic sensor and moving steel sheet significantly complicates instrument design.

- The effect of plastic deformation on the magnetic properties of ultra low carbon sheet steel has been measured and made available for use in the production of low cost electrical equipment, such as motors and relays.

Outputs:

Publications:

Swartzendruber, L. J., Hicho, G. E., Chopra, H. D., Leigh, S., Adam, G., and Tsory, E.. "Effect of Plastic Strain on Magnetic and Mechanical Properties of Ultra Low Carbon Sheet Steel," Journal of Physics, in press.

MAGNETIC MATERIALS

Magnetic materials are pervasive throughout our society. They are used, for instance, in magnetic recording media and devices, in all motors, in all transformers, on credit cards, as permanent magnets, as magnetic sensors, on checks, in theft control devices, in automotive and small engine timing devices, in xerographic copiers, in magnetic resonance imaging (MRI) machines, in microwave communications, in magnetic separation, and in magnetic cooling. Magnetic materials include metals, ceramics and polymers at different size scales ranging from large castings to particulates, thin films, multilayers and nanocomposites.

In the present trend to make devices smaller, thereby reducing weight or increasing storage density, new magnetic materials are constantly being developed. One critical need for implementation of these materials is the development of the measurement science needed for their characterization, in terms of both material properties and performance. This is the focus of the Magnetic Materials Program. Proper measurements of key magnetic properties, determination of the fundamental science behind the magnetic behavior of these new materials, analysis of the durability and performance of magnetic devices and development of standard reference materials are key elements of this program. Some information is only obtainable by the use of unique measurement tools at NIST like the NBS reactor, or the magneto-optic indicator film apparatus for observation of magnetic domain motion. Of particular interest is understanding the magnetic behavior of low dimensional systems, in which one or more characteristic dimensions have been reduced to nanometer sizes. For these new materials, however, it is not known whether their exciting novel behavior is due to new physics or to a logical extension of large-size behavior to small dimensions. Consequently, implementation of this new type of material into marketable products is significantly delayed. NIST is providing the measurement science to answer this critical unknown.

Areas of present study include the following:

- processing of magnetic multilayers for optimal giant magnetoresistance effect measurement and modeling of the enhanced magnetocaloric effect in nanocomposites

- observation and micromagnetic modeling of magnetic domains for understanding magnetization statics and dynamics in advanced and conventional materials

- nanotribology of magnetic hard disk, measurement of stiction, friction, and wear at the nanometer scale

- development of magnetic sensors of mechanical properties for incorporation as *in situ* controls in a steel mill (see description above)

- development of a measurement system for the preparation of an absolute magnetic moment standard

Project Title: **MAGNETIC PROPERTIES OF NANOMATERIALS**

Investigators: R. D. Shull, R. D. McMichael, L. J. Swartzendruber, L. H. Bennett, J. Weismueller, H. J. Brown, H. D. Chopra, R. V. Drew, and D. E. Mathews

Technical Description:

When the characteristic length scale of a material (e.g., grain size, layer thickness, interparticle separation distance, composition modulation wavelength) is reduced to become comparable to the magnetic exchange length, then novel magnetic behavior has often been found to occur. The existence of such new behavior depends critically on the material chemistry and morpholgy. The type of novel behavior and how it depends on the above two variables are the principal unknowns which presently limit the implementation of this new type of material into marketable products, and which is the focus of this NIST activity. In collaboration with scientists at other organizations within and outside of NIST, nanocrystalline and nanocomposite materials are prepared and magnetically characterized, thereby developing the measurement science necessary for understanding these novel materials.

Technical Objectives:

- Determine what magnetic properties are unique to the nanometer scale, and what their origins are, thereby determining which new properties represent new physics.

- Experimentally establish whether magnetic domains exist in magnetic nanomaterials, and if they exist, determine their statics and dynamics.

- Experimentally measure the magnetic correlations between magnetic entities in nanocrystalline and nanocomposite materials.

- Determine magnetic characteristics of nanocomposites with different morphologies.

- Provide information on the magnetic properties of nanomaterials to industry.

Anticipated Outcome:

- Improved prediction capability of magnetic properties of magnetic nanomaterials in different morphologies.

- Improved capability to engineer magnetic properties by design.

- Development of improved characterization techniques for magnetic nanomaterials, thereby leading to improved quality control by manufacturers.

- Better control over flux dynamics in small magnetic devices.

Accomplishments in FY 1996:

- The first nanometer-sized intermetallic compound, Fe_2P, was successfully introduced into the pores of a silica gel to form a bulk magnetic nanocomposite in collaboration with Vanderbilt University. These materials were found to possess a remarkably high blocking temperature of near 250 K. This means that these materials are good candidates for the first superparamagnetic magnetic storage material.

- In a collaboration with the University of Saarlandes (Germany), a surprising "reduction" in magnetization was found with increasing Fe concentration in vapor-condensed Y-Fe nanocomposites for Fe contents in excess of 10 atomic percent. It is still not understood whether this anomaly is due to the unique "magnetic sponge" morphology of this material or to the nanometer-thickness of the Fe-containing planes.

- In collaboration with the University of Saarlandes and Queen's University (Ontario, Canada), the first SANS (small angle neutron scattering) data were measured on a magnetic nanocrystalline material containing a very small number of pores and thereby possessing a majority scattering which is of magnetic origin. This data will now enable us for the first time to get information on the magnetic fluctuations in nanocrystalline materials.

- A U.S. Patent for the concept of Magnetic Nanocomposite Refrigerants was awarded to NIST.

- A SBIR solicitation for the construction of a magnetic refrigerator using the results of NIST research on magnetic nanocomposites the last few years was awarded.

- The Third International Conference on Nanostructured Materials (NANO'96), at which over 250 participants from industry, universities, and government laboratories from more than 25 countries attended, was organized by NIST.

Impacts and Technical Highlights:

- NIST is now considered an international leader in magnetic nanocomposite processing and properties. Consequently, NIST now holds leadership positions in this area in several research-coordinating organizations, including The Minerals, Metals and Materials Society (TMS), the Materials Research Society (MRS), and the International Committee of

Nanostructured Materials (ICNM). NIST is also now consulted by many outside organizations for advice in the area, including as well the design of university courses and of scientific research programs.

- As a result of NIST research on magnetic nanocomposite refrigerants, many groups around the world have initiated research activities in the area, including in China, Germany, France, Great Britain, Japan, and the United States. The NIST research has also accelerated US research on other types of magnetic refrigerants.

Outputs:

Publications:

Atzmony, U. Levine, Z., McMichael, R. D., Bennett, L. H.,"Magnetic viscosity investigations in nanograin iron powder," *J. Appl. Phys.*, **79**, 5456 (1996).

"Metastable Phases and Microstructures" MRS Symposium Proceedings, edited by R. D. Shull, published by the Materials Research Society, Pittsburgh, PA **400** (1996).

Herbst, J. F., Fuerst, C. D., McMichael, R. D., "Structural, magnetic and magnetocaloric properties of $(Hf_{0.83}Ta_{0.17})Fe_{2+x}$ materials," *J. Appl. Phys.,* **79,** 5998, (1996).

Luckhart, C. M., Milne, S. B., Stock, S. R., Shull, R. D., Wuttig, J. E. "Nanocomposites Containing Nanoclusters of Selected First-Row Transition Metal Phosphides",, Nanotechnology: Molecularly Designed Materials, eds. Chow, G. M., Gonsalves, K. E., Am. Chem. Soc. Publ., Washington, D.C. (1996) p. 195.

Zachariah, M. R., Shull, R. D., McMillin, B. K., Biswas, P., "In Situ Characterization and Modeling of the Vapor-Phase Formation of a Magnetic Nanocomposite", Nanotechnology: Molecularly Designed Materials, eds. Chow G. M., Gonsalves, K. E., Am. Chem. Soc. Publ., Washington, D.C. (1996) p. 42.

Shull, R. D., "Viewpoint Update: Nanocrystalline and Nanophase Materials", J. Nanostructured Materials **7**, No. 1/2, 265 (1996).

Weissmueller, J., Shull, R. D., G. Rixecker, G., McMichael, R. D. Bennett, L. H. "Relating magnetic properties and microstructure in inert-gas condensed Y-Fe alloys," *Nanostructured Materials,* 7 161 (1996).

Presentations:

Shull, R. D., "Applications of Magnetic Nanocomposites", Workshop on Applications of Nanostructured Materials, Berlin, Germany, September 5, 1996

Shull, R. D., "Multicomponent Nanocrystalline Magnetic Materials", Materials Science Colloquia Speaker, University of Virginia, Charlottesville, VA, April 8, 1996.

Shull, R. D."Nanoscale Magnetic Materials-An Overview of Processing and Properties", Symposium on Synthesis and Processing of Nanocrystalline Powder, TMS Annual Meeting, Anaheim, CA, February 6, 1996.

Shull, R. D., "Nanocomposite Magnetic Refrigerants", Department of Physics and Astronomy Seminar Speaker, University of Delaware, Newark, DE, December 12, 1995.

Shull, R. D.,"Preparation and Properties of Nanostructured Materials", First Argentina-USA Bilateral Symposium on Materials Science and Engineering (FAUSASMAT'95), Buenos Aires, Argentina, November 15, 1995.

Shull, R. D., "Magnetic Properties of Fe-Oxide/Silica Nanocomposites", Symposium on the Processing and Properties of Nanocrystalline Materials, TMS Fall Meeting, Cleveland, Ohio, October 31, 1995.

Shull, R. D., "Magnetic Refrigeration", ARO Workshop on Nanoscale Design of Magnetic Materials", Research Triangle Park, NC, September 17, 1996.

Shull, R. D., "NIST Nanostructured Materials Activities", Departement des Materiaux Colloquium, Ecole Polytechnique Federale de Lausanne, Lausanne, Switzerland, September 6, 1996.

Project title: **GIANT MAGNETORESISTANCE MATERIALS**

Investigators: W. F. Egelhoff, Jr., P. J. Chen (guest researcher), H. D. Chopra (guest researcher), and R. D. Gomez (part-time contractor).

Technical description:

Our research is aimed at providing a scientific understanding of the manufacturing processes for giant magnetoresistance (GMR) materials and using that understanding to develop new, improved manufacturing processes that yield improved GMR materials. Our research is providing U.S. companies with a significant competitive edge.

Technical objectives:

- Establish the world's foremost laboratory for the study of GMR materials.
- Understand the manufacturing methods for GMR products at the scientific level.

- Develop improved manufacturing methods for GMR products.

- Help guide U.S. companies in the development of improved GMR products.

Anticipated outcome:

- Improved GMR products which will expand the market for U.S. companies.

- Increased demand for GMR products as their performance is improved.

- World leadership by U.S. companies in this market as a result of our research.

Accomplishments of FY 1996:

- The Magnetic Engineering Research Facility (MERF) at NIST, the most elaborately instrumented thin-film deposition facility in the world, maintains high productivity, with an operational status in excess of 80% of available time.

- Once again this year, research at MERF set a new record for the largest GMR value ever recorded in the type of sample (spin valve) best suited to commercial products.

- It was discovered that one of the key process variables is the carefully controlled use of oxygen as a surfactant in the manufacturing environment. It was the discovery of oxygen as a surfactant that allowed the new record to be set.

Impacts and Technical Highlights

- Research at MERF has been described as "invaluable" to U.S industry in the latest quarterly report to the ATP from the National Storage Industry Consortium, an industry group funded by the ATP to develop GMR products.

- Nonvolatile Electronics, Inc., the only company in the world with a GMR product already on the market, has written that no one "has contributed more to the basic understanding of the growth and properties of GMR structures than" (NIST).

- The understanding of the key process variables involved in producing films with the record-setting GMR values has been transmitted from NIST scientists to U.S. companies.

- During the past year NIST began a DARPA-funded collaboration with Motorola to develop a new generation of Dynamic Random Access Memory (DRAM) chips based on GMR materials.

Outputs:

Publications:

Egelhoff, Jr., W. F., Ha, T., Misra, R. D. K., Kadmon, Y., Nir, J., Powell, C. J., Stiles, M. D., . McMichael, R. D., Lin, D. -L., Sivertsen, J. M., Judy, J. H., Takano, K., Berkowitz, A. E., Anthony, T. C., Brug, J. A.,"Magnetoresistance Values Exceeding 21% in Symmetric Spin Valves," J. Appl. Phys. **78**, 273 (1995).

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Egelhoff, Jr., W. F., Chen, P. J., Powell, C. J., Stiles, M. D., McMichael, R. D.,"Growth of GMR Spin Valves Using In as a Surfactant," J. Appl. Phys. 79, 2491 (1996).

McMichael, R. D., Egelhoff, Jr., W. F., Ha, M.,"Thermal Degradation of GMR Spin Valve Films," Proc. Mat. Res. Soc. Symp., 384, 397 (1995).

McMichael, R. D., Egelhoff, Jr., W. F., Bennett, L. H.,"Thermal Degradation of Exchange Biased Films in Spin Valves," IEEE Trans. Mag. 31, 3930 (1995).

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Egelhoff, Jr., W. F., Chen, P. J., Powell, C. J., Stiles, M. D. McMichael, R. D., Lin, C. -L., Sivertsen, Judy, J. H., Takano, K., Berkowitz, A.E., Anthony, T.C. Brug, J.A.,"Optimizing the GMR of Symmetric and Bottom Spin Valves," J. Appl. Phys. 79, 5277 (1996).

Egelhoff, Jr., W. F., Chen, P. J., Powell, C. J., Stiles, M. D., McMichael, R.D., Lin, E. -L., Sivertsen, J. M., Judy, J. H., Takano, K., Berkowitz, A.E., Anthony, T.C., Brug, J. A.,"The Trade-off between Large Magnetoresistance and Small Coercivity in Symmetric Spin Valves," J. Appl. Phys. 79, 8603 (1996).

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McMichael, R. D., Watanabe, T. Dura, J. A., Borchers, J. A., Egelhoff, Jr., W. F., Chen, P. ., . Brown, H. J., "Origins of the Coercivity Increase in Annealed Symmetric Spin Valves," IEEE Trans. Mag. 32, 4636 (1996).

Chopra, H.D., Hockey, B. J., Chen, P. J., Salamanca-Riba, L., Hua, S. Z., Wuttig, M., Egelhoff, Jr., W. F., "Detailed Structural Characterization of Symmetric Spin Valves using High-Resolution Transmission Electron Microscopy," J. Appl. Phys. (accepted)

Nikitenko, V. I. Gornakov, V. S.,. Dedukh, L. M., Kabanov, Yu. P., Khapikov, A.F., Bennett, L. H., Chen, P. J., McMichael, R. D., Donahue, M. J., Swartzendruber, L. J., Shapiro, A. J., Brown, H. J., Egelhoff, Jr., W, F, "Magneto-optical Indicator Film Study of the Magnetization of a Symmetric Spin Valve," IEEE Trans. Mag. 32, 4639 (1996).

Hua, S. Z., Lashmore, D. S., Chopra, H. D., Swartzendruber, L. J., Egelhoff, Jr.,W. F., "Domain Dynamics in GMR Polycrystalline CoNi/Cu Multilayer Films," J. Appl. Phys. (accepted).

Chopra, H. D., Hua, S. Z., Lashmore, D. S., Swartzendruber, L. J., Egelhoff, Jr., W. F., "*In-situ* Observation of Domain Dynamics in GMR Co/Cu Multilayer Magnetic Films," J. Appl. Phys. (accepted).

Chopra, H. D., Chen, P. J., McMichael, R. D., Egelhoff, Jr., W. F., Berkowitz, A. E., Takano, K., "Magnetoresistive Studies of NiO/Co/Cu-Based Symmetric Spin Valves," J. Appl. Phys. (accepted)

Gornakov, V. S. Nikitenko, V. I., Bennett, L. H. Brown, H. J., Donahue, M. J., Egelhoff, Jr., R., W. F., McMichael, R. D., Shapiro, A. J., "Experimental Study of Magnetization Reversal Process in a Non-symmetric Spin Valve," J. Appl. Phys. (submitted)

Presentations:

Egelhoff, W. F., "Optimizing the GMR of Spin Valves," 40th Annual Magnetism and Magnetic Materials Conference, Philadelphia, November 7, 1995.

Egelhoff, W. F., "Recent Studies of GMR Spin Valves at NIST," Physics Department, Virginia Commonwealth University, Richmond, February 2, 1996.

Egelhoff, W. F., "Recent Studies of GMR Spin Valves at NIST," Motorola Corporate Research Laboratories, Phoenix, March 27, 1996.

Egelhoff, W. F., "Optimized Growth of GMR Spin Valves," National Storage Industry Consortium Quarterly Workshop, Stanford, March 28, 1996.

Egelhoff, W. F., "Recent Studies of GMR Multilayers at NIST," National Storage Industry Consortium Annual Meeting, Lake Tahoe, NV, June 5, 1996.

Egelhoff, W. F., "XPS and Auger Electron Forward Scattering: A Structural Tool for Ultra-Thin Films, Epitaxy, Surface Segregation, and Interdiffusion," 18th International Conference on Surface Physics, Wroclaw, Poland, June 11, 1996.

Egelhoff, W. F., "Status and Plans for GMR Research at NIST," DARPA/Motorola Workshop on Magnetoresistive Random Access Memory, Arlington, July 25, 1996.

Egelhoff, W. F., "Surfactants in Epitaxy," International Workshop on Determination of Surface Morphology by High Resolution Diffraction," Hildesheim, Germany, September 26, 1996.

Project Title: **PROCESSING AND MICROMAGNETICS OF THIN FILMS**

Investigators: R. D. McMichael, W. F. Egelhoff, P. J. Chen, and M. J. Donahue, H. J. Brown, and D. E. Mathews

Technical Description:

This project is focused on the effects of processing on the characteristics of magnetic thin films such as giant magnetoresistance, coercivity, and magnetization dynamics. In particular, the thermal stability of multilayer structures exhibiting giant magnetoresistance is of concern to companies that manufacture recording heads for ultra-high-density magnetic data storage, other magnetic field sensors and non-volatile magnetic computer memory. Good thermal stability will allow incorporation of these metal films into existing semiconductor fabrication processes, which typically include annealing steps. Good thermal stability will also allow magnetoresistive sensors to tolerate heating at the high current densities required for rapid signal detection. This project is being carried out in cooperation with the National Storage Industry Consortium.

Technical Objectives:

- Develop measurement methods and imaging techniques needed to characterize multilayer thin magnetic films.

- Develop measurement techniques for and evaluate the effects of thermally induced damage to multilayer magnetic thin film structures.

Anticipated Outcome:

- Measurement techniques developed for evaluation of magnetic film properties.

- Thermal stability of magnetic multilayer structures enhanced through an understanding of the relationships among film composition, structure and diffusion.

Accomplishments of FY 1996

- Oxidation effects in NiO-biased, symmetric spin valves were found to penetrate the center, 'free' layer, giving rise to increased coercivity following annealing.

- An apparatus was designed, assembled, and tested to measure reversible and irreversible effects of thermal treatments on electrical resistance of thin films in vacuum, in magnetic fields and at temperatures up to 870K.

- Thermally induced magnetoresistive changes in NiO-biased Co/Cu multilayers were found to be almost completely reversible at temperatures up to 470K, while irreversible thermal degradation occurs at higher temperatures.

Impacts and Technical Highlights:

- Thermal stability measurements have provided guidance for companies in the National Storage Industry Consortium and in particular, its ATP-sponsored HEADS project for ultra-high density recording.

Outputs:

Publications:

McMichael, R. D., Watanabe, T., Dura, J. A. Borchers, J. A., Chen, P. J. Brown, H. J., Egelhoff, Jr., W. J., "Origins of Coercivity Increase in Annealed Symmetric Spin Valves," IEEE Trans. MAG, **32**, 4636 (1996).

McMichael, R. D., Egelhoff, Jr., W. F., Bennett, L. H., "Thermal Degradation of exchange-biased films in spin-valves," IEEE Transactions on Magnetics, **31**, 3930 (1995).

McMichael, R. D., Egelhoff, Jr., W. F., Ha, M.,"Improved thermal stability of GMR spin valve films," Mat. Res. Soc. Symp. Proc. **384**, 397 (1996).

Project Title: STANDARDS IN COMPUTATIONAL MICROMAGNETICS

Investigators: R. D. McMichael and M. J. Donahue.

Technical Description:

Micromagnetic modeling techniques are being developed and evaluated for predicting hysteretic behavior and magnetic domain configurations in small film elements. This project focuses on providing a fundamental understanding of micromagnetic computation techniques, including evaluation of algorithms and of calculated results, and comparison of calculated to experimentally determined magnetic domain structures. Accurate micromagnetic modeling is important both for understanding of magnetic material properties and for design of magnetic devices.

Technical Objectives:

- Provide tools for evaluation of micromagnetic computational techniques currently in use in the micromagnetic modeling community.
- Develop fast, accurate and reliable algorithms for micromagnetic computation.
- Provide experimental verification of micromagnetic calculation results.

Anticipated Outcome:

- Reliability of micromagnetic modeling techniques improved through rigorous testing.
- Standard problems developed for evaluation of micromagnetic software with experimental verification of calculated results.
- Computer code for micromagnetic calculations made publicly available.

Accomplishments in FY 1996:

- NIST sponsored a computational round-robin for a standard micromagnetic problem, with six submitted solutions from industry, academia and government.
- A critical comparison was performed of discrete representations of exchange energy for micromagnetic modeling.
- More accurate and faster techniques were developed for computation of magnetostatic effects in micromagnetic modeling.

Impacts and Technical Highlights:

- NIST's computational round-robin on a micromagnetic standard problem yielded surprisingly different results. This was the first time such comparisons had been attempted between independently developed computer codes. The large differences between the submitted results have caused many in the micromagnetic modeling community to rethink the reliability of micromagnetic modeling technique.

Outputs:

Publications:

Donahue, M. J., McMichael, R. D., "Exchange Energy Representations in Computational Micromagnetics," Physica B, to be published (1997).

Presentations:

Egelhoff, W. F., "Opportunities in Graduate Research in the Physical Sciences," Faculty Fellows Lecture Series, Hampden-Sydney College, Hampden-Sydney, VA, March 11, 1996.

METALS DATA AND CHARACTERIZATION

The performance of metals during use and their behavior during processing can be understood and predicted only with the availability of a detailed body of information on their physical properties and microstructure. The value of this information is greatly enhanced if it is developed within the context of models or theories which describe how the measured properties of a metal will vary with changes in composition, microstructure, temperature, geometry, or other parameters. The Metals Data and Characterization Program includes activities which refine the technology for measuring the properties and behavior of metallic materials, and which correlate these properties and behavior to alloy microstructures.

The large majority of metals are used in applications based on their mechanical properties, with other applications based on electronic, magnetic, optical or other functional properties forming smaller but nonetheless critical markets. Whatever the application, satisfactory long-term performance of metallic components demands chemical and microstructural stability, sometimes in the face of harsh environments. This program identifies those property measurements and microstructure characterizations which are critical to U.S. industry for both the processing and the performance of metals, and carries them out within the context of the NIST mission of providing data and standards. A significant part of the program is the use of advanced microscopy techniques to characterize the microstructures which form the basis of the measured properties.

The measurements of mechanical, chemical, magnetic, and optical properties carried out under this program have an impact in a number of different technology sectors:

- Precision measurements of Rockwell Hardness of specially prepared steel test blocks are leading to the development of national standards for measurement methods and calibration procedures, as well as production of calibrated test blocks for sale to industry as Standard Reference Materials. Availability of this national standard will lead to increased acceptance of this most-often-specified material property in domestic and international trade.

- In work involving a collaboration with ALCOA and USCAR, models are being developed and verified for the press-and-sinter and powder-forge processes for metal matrix composites, with the goal of developing lightweight materials for automotive applications.

- High-speed optical techniques are being developed and applied to the measurement of thermophysical properties of solid and liquid metals at high temperatures. This work provides accurate benchmark data which are used by the metal casting industry in modeling the solidification of aerospace alloys.

- A new project has been initiated to study problems associated with the corrosion of aging aircraft. By identifying the rate at which corrosion damage depends on environmental conditions, this project has the potential to allow more rational planning of inspection intervals and thus significantly reduce the costs of using older aircraft in both commercial and military applications.

Project Title: **THERMOPHYSICAL PROPERTIES**

Investigators: Ared Cezairliyan, John L. McClure, Tsuyoshi Matsumoto, Debasis Basak, and Daniel Josell

Technical Description:

This project is involved with the development of new techniques for the accurate measurement, primarily at high temperatures, of selected thermophysical properties of materials, in both solid and liquid phases, utilizing rapid (millisecond- and microsecond-resolution) pulse-heating (volume and surface) techniques.

Technical Objectives:

- Develop new techniques for the accurate measurements of selected thermophysical properties of solid materials at high temperatures (up to their melting region) utilizing millisecond-resolution resistive self-heating techniques.

- Develop new techniques for the accurate measurements of selected thermophysical properties of liquid materials at high temperatures (several hundred degrees above their melting region) utilizing microsecond-resolution resistive self-heating techniques.

- Develop a laser pulse technique for the accurate measurement of thermal diffusivity of multilayered thin films at high temperatures.

Anticipated Outcome:

- Advancement of the state-of-the-art of thermophysical measurements at high temperatures.

- Generation of accurate benchmark data on selected key materials.

- Development of high-temperature thermophysical standards.

Accomplishments for FY 1996:

- Modifications and refinements were made of the novel high-speed laser polarimeter developed during the previous year. The laser polarimeter is now capable of measuring, with unprecedented accuracy, normal spectral emissivity of metals and alloys at high temperatures (above 1500 K) without requiring a blackbody configuration for the specimen.

- Applicability of the laser polarimetry technique to the non-contact detection of phase transformations in metals and alloys at high temperatures was demonstrated for the first time. The preliminary measurements included detection of structural phase transformations (titanium, zirconium, iron, and hafnium) and melting (nickel, zirconium).

- A new accurate technique for the measurement of hemispherical total emissivity was developed. The new technique is a combination of transient and brief steady-state techniques, less than one second in total duration, for measurements on electrically-conducting materials at high temperatures (above 1500 K). The brief steady-state period is used in the determination of emissivity.

- The laser pulse system was used to measure thermal diffusivity of homogeneous as well as multilayered materials. Extensive measurements were performed on specimens consisting of copper and iron layers and several industrially important multilayered materials. Additional measurements were conducted on pure molybdenum specimens to further assess the operation of the system. Modifications to the specimen holder and refinements to the computer programs were made.

Impacts and Technical Highlights:

- NIST developed, improved, and successfully used a new technique, the first and the only one of its kind, for the measurement of normal spectral emissivity of metals and alloys at high temperatures in rapid pulse-heating experiments. This technique will enable measurement of the true temperature of a specimen where it is either very difficult or impossible to have a blackbody configuration for the specimen to permit direct radiometric measurements of its true temperature.

- NIST developed a new technique for the measurement of hemispherical total emissivity of metals and alloys in subsecond-duration experiments. This technique is likely to provide, with unprecedented accuracy, emissivity of electrically-conductive materials at temperatures above 1500 K.

- An accurate knowledge of thermal diffusivity will play an important role in the selection, use, and assessment of thin films and specifically thermal barrier coatings in high temperature applications, such as in jet engine blades.

Outputs:

Publications:

Cezairlilyan, A., Krishnan, S., and McClure, J. L., "Simultaneous Measurements of Normal Spectral Emissivity by Spectial Radiometry and Laser Polarimetry at High Temperatures in Millisecond-Resolution Pulse-Heating Experiments: Application to Molybdenum and Tungsten," Int. J. Thermophys. **17**, p. 1455 (1996).

Cezairliyan, A., and Righini, F., "Issues in High-Speed Pyrometry," Metrologia **33**, p. 229 (1996).

Kaschnitz, E., and Cezairliyan, A., "Radiance Temperatures at 100 nm of Niobium and Molybdenum at Their Melting Points by a Pulse-Heating Technique," Int. J. Thermophys. **17**, 1069 (1996).

Kaschnitz, E., and Cezairliyan, A., "Radiance Temperatures (in the Wavelength Range 530-1500 nm) of Iron and Cobalt at Their Melting Points by a Pulse-Heating Technique," High Temp.-High Press., in press.

McClure, J.L., and Cezairliyan, A., "Radiance Temperatures (in the Wavelength Range 525-906 nm) of Vanadium at Its Melting Point by a Pulse-Heating Technique," Int. J. Thermophys., in press.

Josell, D., Cezairliyan, A., Van Heerden, D., and Murray, B.T., "An Integral Solution for Thermal Diffusion in Periodic Multilayer Materials: Application to Iron/Copper Multilayers," Int. J. Thermophys., in press.

Presentations:

Cezairliyan, A., "Thermophysical Properties of High Temperature Liquid Metals and Alloys," NASA Microgravity Materials Science Conference, Huntsville, Alabama, June 10, 1996.

Project Title: MAGNETIC PROPERTIES AND STANDARD REFERENCE MATERIALS

Investigators: L. J. Swartzendruber, R. D. Shull, A. J. Shapiro, D. E. Mathews, R. V. Drew, and L. H. Bennett

Technical Description:

A wide variety of magnetic measurement techniques are used to characterize the details of magnetic structures and magnetization processes in metals. Magnetic properties of materials important to the scientific and industrial community are being determined and the methods for measurement of these properties are being improved. To make meaningful comparisons between magnetic measurements made in different laboratories, it is necessary to develop and produce standard reference materials for instrument calibration.

Technical Objectives:

- In cooperation with universities and industry, determine methods for characterizing the accommodation and after effect properties in materials used in magnetic recording and permanent magnets.

- In cooperation with industry determine the magnetic properties of materials used in the control of flux in devices used for induction heating.

- Develop an instrument for the absolute measurement of magnetic moment and use this instrument to reissue SRM 722.

- Determine the parameters necessary to fully characterize magnetic materials. These parameters can then be used in models which correctly predict the behavior of the magnetic material under actual operating conditions. Extend the parameters to include magnetostriction and time dependence.

Anticipated Outcome:

- Improved characterization of magnetic recording media resulting in lower overall net cost per unit of storage.

- Improved characterization of permanent magnets and other materials for magnetic devices resulting in more efficient use of such materials.

- Improved calibration of magnetic measurement instruments giving NIST traceability at lower costs. Facilitation of commerce in magnetic materials through improved agreement between producer and consumer on the measurements of magnetic properties.

Accomplishments in FY 1996:

- Measurements were performed on nanoscale particulate recording materials used in floppy diskettes, in a cooperative program with the George Washington University Institute for Magnetics. The nature of the interrelationships between the accomodation and time decay properties was demonstrated.

- Magnetic measurements were made on a large number of Fe-Si-Al alloys. The optimum heat treatment for minimizing the coercive field of an Fe-13 w/o Si alloy was determined.

- A system for measuring absolute magnetic moments was designed. The equipment necessary to assemble the instrument was ordered and the material for fabrication of nickel ball standard reference material was obtained.

- The magnetic properties of mechanically-alloyed nanostructured composites of permalloy-silver (in collaboration with the University of Maryland Baltimore County) and of iron-copper (in collaboration with Dartmouth College) were measured. It was determined that microstructural and magnetic properties of these systems are a strong function of milling time.

- Studies were carried out of magnetic resonance images (MRI) from metallic biomedical implants, which give rise to artifacts which impair interpretation of MRI of patients. The studies showed how these artifacts arise, and how the implant's magnetic susceptibility affects the size and shape of the artifact.

- The properties of nanocomposites containing nanoclusters of Fe_2P or γFe_2O_3 were studied. Processing methods for maximizing the yield of desired species and avoiding undesired materials, such as Fe_2SiO_4, were discovered.

Impacts and Technical Highlights:

- Measurements of the magnetic accommodation and magnetic after effect have important implications for the use of nanoscale particulate recording materials for longtime storage of data on commercial magnetic tapes and floppy disks. These processes are also important for the stability of high-performance hard magnets for use in motors and other devices.

Outputs:

Publications:

Bennett, L.H., Wang, S.P., and Donahue, M.J., "Artifacts in Magnetic Resonance Imaging from Metals," J. Appl. Phys., **79**, 4712 (1996).

Shull, R. D., Cline, J. P., Baker, I., Liu, F., "Identification of a High Temperature Magnetic Phase Transition in Ball-Milled Nanocrystalline Fe-Cu Alloys", J. Appl. Phys., **79**, 6028 (1996).

Swartzendruber, L.J., Bennett, L.H., Vajda, F., and Della Torre, E., "Relationship Between the Measurement of Accomodation and After-Effect," J. of Physics B, in press.

Project Title: **MICROSTRUCTURAL STUDIES OF COMPLEX PHASES**

Investigators: Leonid A. Bendersky, Terell Vanderah, Robert S. Roth, and R. M. Waterstrat

Technical Description:

Improved new materials with higher dielectric constants and high saturation magnetization values are looked for in different systems, and in particular in the $BaO:Fe_2O_3:TiO_2$ system. Such materials potentially can be used in a wide variety of electronic devices for microwave communication. In a recent work by Vanderah et al [1] on phase equilibria in the $BaO:Fe_2O_3:TiO_2$ system at 1250-1270°C the existence of sixteen ternary compounds has been reported. Most of the newly found compounds apparently have a new structure type and require complete structural determination. Structural studies by x-ray diffraction were not successful for some of the compounds because of (a) insufficient quality of single crystals; (2) presence of defects and (3) difficulties in establishing a correct space group. Therefore, high-resolution electron microscopy (HREM) was employed to study these compounds. Considering the complexity of the structures (from 200 to 600 atoms per unit cell) and the presence of disorder, the work is extremely challenging. The work is in close collaboration with MSEL's Ceramics Division.

Additional research is also continuing in the development of an understanding of complex phase transformations in multicomponent systems that form intermetallic compounds. This research area is closely related to the Metallurgy Division's past programs in rapid solidification, phase diagram calculations, and electronic packaging and represents a core competence in the Division.

Technical Objectives:

- Establish structural correlation between the phases obtained as a single crystal (from an off-stoichiometric melt) and as a polycrystalline specimen (by solid-state reaction).

- Understand structural and growth defects.

- Provide structural models derived from the high-resolution imaging as an input model for powder neutron diffraction refinement.

- Understand general principles controlling the formation of these compounds (packing of Ba/O and O_4 layers, distribution of metal atoms on interstitial sites, etc.)

Accomplishments for FY 1996:

- Problems of specimen preparation for HREM has been resolved.

- High-resolution images and electron diffractions (three main zone axes) of good quality were obtained for six compounds (M, E, F, K, I and J phase).

- A previously unknown space group of the E phase has been established.

- A group of compounds with a new type of structural architecture was discovered. The new type consists of two type of structural blocks: polytypic perovskite and block with a triple ordered unit cell.

- Unusual disorder (not detected by x-ray diffraction) was discovered to exist for a few compounds. The disorder reflects poor phase correlation between the ordered blocks.

- A set of general structure-building principles for these ternary phases has been established.

- A model of the M phase (over 400 atoms/cell) was constructed. Preliminary multi-slice simulations produced a reasonable correspondence with an experimental images.

- Magnetic measurements of Curie temperature and B vs. H characteristics were obtained for these compounds (with R. D. Shull).

Impact and Technical Highlights:

- The $BaO:Fe_2O_3:TiO_2$ phase diagram is of immediate interest to U.S. industry involved in the production of ceramics for wireless communications systems, e.g. for microwave circulators and isolators. Understanding of the structures and defects in the $BaO:Fe_2O_3:TiO_2$ compounds will provide a basis for tailoring microstructures and properties of such materials.

Outputs:

Publications:

L. A. Bendersky, J. K. Stalick, R.Portier and R.M.Waterstrat, "Crystallographic Structures and Phase Transformations in the ZrPd Alloy", J. Alloys and Compounds, 236, 19 (1996).

I. Levin, L. A.Bendersky, D.G.Brandon and M.Ruhle, "Cubic to Monoclinic Phase Transformations in Alumina", submitted to Acta Metall. Mater. (1996)

S.Mitao and L.A.Bendersky, "Morphology and Growth Kinetics of Discontinuous Coarsening in Fully Lamellar Ti - 44 Al (at%) Alloy," submitted to Acta Metall. Mater. (1996)

L.A.Bendersky and R.M. Waterstrat, "Incommensurate Structure of the Phase Zr_3Rh_4", accepted to J.Alloys and Compounds, (1996).

L.A.Bendersky, T.A.Vanderah and R.S. Roth, "Structural Features of "$Ba_4Fe_4Ti_3O_{16}$" and "$Ba_{26}Fe_{20}Ti_{21}O_{98}$" as Revealed by High Resolution Electron Microscopy", J. of Solid State Chemistry, 125, 281 (1996).

Traiber, A. J. S., Turchi, P. E. A., Waterstrat, R., Allen, S. M., "Electronic Structure and Phase Equilibria in Ternary Substitutional Alloys," J. Phys. Condens. Matter, 8, 6357 (1996).

Presentations:

L. A. Bendersky and R. M. Waterstrat, "Incommensurate Structure of the Phase Zr_3RH_4," Meeting on Electron Crystallography, Stockholm, 1996.

Project Title: **MECHANICAL AND THERMAL PROPERTIES OF MULTILAYERS**

Investigators: T. Foecke, D. Josell, W. C. Carter (Ceramics Division), A. Cezairliyan, D. Read (Materials Reliability Division), D. Shechtman (Technion, Israel), M. R Stoudt, and D. vanHeerden

Technical Description:

Because they contain a high density of interfaces, multilayered materials may possess technologically important properties. For example, the giant magnetoresistance (GMR) effect in nanoscale magnetic thin films has significantly led to major changes in the magnetic data storage industry (see the Magnetic Materials Program). In the area of mechanical properties, nanoscale multilayers (nanolaminates) hold the promise of extremely high strength and wear resistance, and may have commercial potential as free-standing components and, in particular, as coatings. Similarly, the thermal properties of multilayers have made them attractive as thermal barrier coatings. This project is developing the science base to address both of these potential applications.

Technical Objectives:

- Develop an understanding of the mechanical behavior of free-standing multilayers, including:
  - the creation and motion of dislocations and their interaction with interfaces and cracks.
  - the role of the interface characteristics (chemical and structural sharpness, and crystallographic and epitaxial relationships).
  - the influence of layer thickness and the relative amounts of the constituent metals.

- Investigate the effects of nanolaminate coatings on the resistance of metals and alloys to fatigue initiation.

- Measure the thermal diffusivity of multilayer thermal barrier coatings (TBCs) at operating temperatures (1000-1400° C).

- Determine the thermal resistance associated with the presence of an interface in a material and, hence, evaluate the efficiency of a multi-layered material as a TBC.

- Determine the relationships between capillarity factors (interface and grain boundary free energies) that determine the stability of multilayer structures to grain-boundary-induced hole formation.

Anticipated Outcomes:

- A fundamental understanding developed of how layering, constituent materials, and interfacial characteristics of a structure determine its properties and performance.

- Predictive tools established that can be used to design materials for specific applications.

- Insights into basic mechanical properties of nanolayered materials applied to reliability issues in the magnetic recording and microelectronics industry. These insights address such problems as thermomechanical fatigue, adhesion, and abrasive wear resistance in fine layered structures.

- An assessment of the influence of nanolaminate coatings on the resistance of metals and alloys to fatigue initiation.

- The thermal conductivity measurements for multilayer materials at high temperatures used to assess the usefulness of the multilayer thermal barriers over standard monolithic thermal barriers already in use.

- Measurement of the ultimate tensile strength for several multilayer systems will permit analysis of the potential of these materials for structural purposes and of theoretical models for plasticity in multilayer structures.

- An understanding gained of the factors that determine the stability of nonreactive multilayer structures operating at high temperatures.

Accomplishments for FY96:

- First successful straining of a cross-sectional multi-layer in the TEM led to:

  - First in situ observations of dislocation generation and motion in metallic nanolaminates.

  - Observation of effect of interfaces on dislocation motion.

  - First observation of dislocation pileups in a nanomaterial.

  - First video images of an operating Frank-Read dislocation source in any material.

  - Observation of the pinning of mobile Orowan dislocation bows on interfacial misfit dislocations, and observation that this pinning dominates Peierls-type lattice resistance to dislocation motion.

- A single-bath, pulsed potential electrochemical system to deposit multilayer films on cylindrical fatigue testpieces was designed and assembled.

- Collaborative efforts on measurements of thermal diffusivity have been established with Howmet, Battelle, and EPRI. Results obtained on industry-provided material have been shared with industrial partners.

- Model system multilayer thermal barriers of iron/copper and alumina/molybdenum have been fabricated using an electron beam evaporator deposition system at NIST.

- A closed form integral solution has been obtained for heat flow through a multilayer thermal barrier. It has been used to study data obtained from preliminary tests on multilayer thermal barriers.

- Aluminum/titanium multilayers with a variety of layer thicknesses and compositions have been fabricated and tested.

- A collaborative effort with D. Read (NIST, Boulder) to study the mechanical properties of multilayer thin films was established.

Impacts and Technical Highlights:

- Observations of pileups and the effect of interfacial dislocations on glissile dislocations has forced the revision of the theoretical treatments of nanolaminate mechanical behavior by most modeling groups. A Quicktime movie of the operating Frank-Read dislocation source has been produced and distributed upon request over the Internet. Reviews of the observation have been extremely positive.

- A second year of a Binational United States-Israel Grant (with D. Schechtman) to study polymorphism in multilayer structures was approved, and D. Josell visited the Technion as part of the first year collaboration. Work on silver/titanium and zirconium/titanium multilayers fabricated at NIST has been conducted at the Technion as part of the collaborative effort.

Outputs:

Publications:

Foecke, T., "A Sample Geometry for Controlled In Situ TEM Straining Experiments on Brittle Materials," Scripta Materialia, "in press".

Foecke, T., "Direct Observation of the Generation and Motion of ‘Orowan’ Bows in Single Crystal Cu/Ni Nanolaminates," Journal of Materials Research, "in press".

Foecke, T., Weihs, T. P., and Anderson, P.M., " Proceedings of the Workshop on Nanoscale Layered Materials for Protective Coating and Structural Applications," NIST-IR 5890, 1996.

Foecke, T. and vanHeerden, D., "Experimental Observations of Deformation Mechanisms in Metallic Nanolaminates," Proceedings of the Symposium on Chemistry and Physics of Nanoscale and Non-equilibrium Materials, TMS, Pittsburgh, PA, "in press".

Was, G. S. and Foecke, T., "Deformation and Fracture in Microlaminates," Invited Review Article, Thin Solid Films, in press.

Josell, D., Cezairliyan, A., van Heerden, D., and Murray, B.T., “An Integral Solution for Thermal Diffusion in Periodic Multilayer Materials: Application to Iron/Copper Films,” to be published in International Journal of Thermophysics.

Josell, D., Cezairliyan, A., van Heerden, D., and Murray, B.T., "Thermal Diffusion Through Multilayer Coatings: Theory and Experiment," to be published in Nanostructured Materials as proceedings of Nanostructured Materials Conference, Spring 1996, Hawaii.

Josell, D. and Carter, W. C., "Implications and Applications of Zero Creep Experiments for Multilayer Stability," to be published in proceedings of TMS symposium Creep and Stress Relaxation of Microstructures, Cincinnati, OH, October, 1996.

van Heerden, D., Josell, D., and Shechtman, D., "The Formation of Fcc Titanium in Titanium-Aluminum Multilayers," Acta Metallurgica et Materialia **44**, 297 (1996).

van Heerden, D., Josell, D., and Shechtman, D., "Tensile Tests of Low Density Multilayer Thin Films," Materials Research Society Symposium Proceedings **403**, Polycrystalline Thin Films: Structure, Texture, Properties and Applications, Eds. H.J. Frost et al., 139 (1996).

Presentations:

Foecke, T., "Deformation of Nanolaminates," ASME Mechanics and Materials Conference, The Johns Hopkins University, Baltimore, MD, June 16-20, 1996.

Josell, D., Thermal Diffusion In Multilayer Materials, "Theory And Experiment For Fe/Cu And Alumina/Mo Multilayers On Mo Substrate," 3rd International Conference on Nano- structured Materials, Hawaii, 7/8/96-7/12/96.

Josell, D., "Implications and Applications of Zero Creep Experiments for Multilayer Stability." TMS Fall Meeting, symposium on Creep and Stress Relaxation in Microstructures, Cincinnati, Ohio, 10/6/96 - 10/8/96.

Josell, D., "Multilayer Materials: Theory and Experiment for Thermal Transport, Zero Creep and Structural Stability." Technion in Haifa, Israel, 11/6/96.

Project Title: **HARDNESS STANDARDS**

Investigators: S. R. Low, D. J. Pitchure, W. S. Liggett (Statistical Engineering Division, ITL), E. N. Pugh, and R. E. Ricker

Technical Description:

In the United States and throughout the world, the Rockwell hardness test is the most often specified material property test for manufacturing process control, product quality control, and product acceptance. The NIST Metallurgy Division is working in collaboration with the Precision Engineering Division of MEL, the Standards Reference Materials program, the National Voluntary Laboratory Accreditation Program (NVLAP), the Statistical Engineering Division of ITL, ASTM and ISO to standardize Rockwell hardness measurements in the United States, to define a system for traceability of Rockwell hardness measurements to U.S. national standards, and to promote the development of uniform international Rockwell hardness standards.

Technical Objectives:

- Define the U.S. Rockwell hardness scales (e.g., HRA, HRB, HRC, Superficial N and T) based on material science and metrology.
- Develop uniform and stable hardness blocks as a means of transferring the Rockwell scales to industry.
- Provide calibrated test blocks (low, medium and high level blocks) for sale to industry as Standard Reference Materials (SRMs).
- Provide certified Rockwell indenters meeting tight geometry and performance criteria for sale to industry as Standard Reference Materials.
- Experimentally determine the effects of the variables of the Rockwell test cycle (i.e., indenter velocity and dwell times) on the hardness measurement, and understand these effects based on material science.
- Collaborate with the hardness standardization laboratories of other countries in an effort to unify Rockwell hardness testing worldwide.

Anticipated Outcome:

- Standard measurement methods, calibration procedures and standard reference materials that enable U.S. industry to conduct Rockwell hardness tests that are traceable to national and international standards, enabling compliance with international quality standards such as ISO 9000.

- Improved national and increased international trade with U.S. manufacturers because trading partners can assure each other that they are using the same nationally and internationally accepted standard measurements to assess the quality of products for quality control and acceptance testing.

- Assurance for U.S. manufacturers that Rockwell hardness tests cannot become a technical barrier to international trade in countries which wish to inhibit the importation of U.S. manufactured goods.

- Improved national and more unified international hardness testing standards.

Accomplishments for FY 1996:

- Calibrated approximately 100 sets of Rockwell C scale hardness blocks at three hardness levels to be sold as NIST Standard Reference Materials.

- Conducted hardness intercomparisons between NIST and the national hardness standardization laboratories of Japan, China, Italy, and Germany.

- Initiated interaction in the area of hardness standardization between NIST and the national laboratories of Brazil and Mexico.

- As chair of the Traceable Hardness Standards task group at ASTM, NIST has led the effort to develop requirements for traceability of Rockwell hardness measurements to U.S. national standards.

- Joined the U.S. delegation to ISO for hardness testing.

- Initiated a study of the Rockwell B hardness scale (HRB), and the material used in the manufacture of HRB test blocks.

Impact and Technical Highlights:

- The first U.S. national Rockwell C Scale hardness standards have been calibrated, and will be available as SRMs in the next fiscal year.

- ASTM is currently revising their Rockwell hardness testing standards to include requirements for traceability to national standards utilizing the SRMs to be produced by this program.

Outputs:

Publications:

Low, S., Pitchure, D., and Liggett, W., "Development of the NIST Standard Reference Material for Rockwell C Scale Hardness," Proceedings of the 5th Joint Italy-USA Seminar on Cooperation in Metrology, Equivalence of National Standards, Dissemination of SI Units, to be published, Italy.

Song, J. F., Smith, J. H., and Vorburger, T. V., "A Metrology Approach to Unifying Rockwell Hardness Standards", Proceedings of the 9th International Symposium of Hardness Testing in Theory and Practice, Duesseldorf: VDI-Verlag, Berichte NR. 1194, November 23-24, 1995.

Song, J. F., Rudder, Jr., F. F., Vorburger, T. V., and Smith, J. H., "Stylus Techniques for the Direct Verifications of Rockwell Diamond Indentors," Proceedings of the 9th International Symposium of Hardness Testing in Theory and Practice, Duesseldorf: VDI-Verlag, Berichte NR. 1194, November 23-24, 1995.

Song, J. F., Rudder, Jr., F. F., Vorburger, T. V., and Smith, J. H., "Calibration Uncertainties of Rockwell Diamond Indentors," NIST Journal of Research, Vol. 100 No. 6, November 1995.

Presentations:

Low, S. R., "Development of the NIST Standard Reference Material for Rockwell C Scale Hardness," 5th Joint Italy-USA Seminar on Cooperation in Metrology, Equivalence of National Standards, Dissemination of SI Units, Gaithersburg, MD, Feb. 26-29, 1996.

Low, S. R., "Realization of Hardness Scales," International Symposium on Hardness Measurements, Instituto de Pesquisas Technológicas, Sào Paulo, Brazil, Sept. 23-24, 1996.

Low, S. R., "Manufacturing and Certification of Hardness Standard Blocks," International Symposium on Hardness Measurements, Instituto de Pesquisas Technológicas, Sào Paulo, Brazil, Sept. 23-24, 1996.

Low, S. R., "Hardness Intercomparisons," International Symposium on Hardness Measurements, Instituto de Pesquisas Technológicas, Sào Paulo, Brazil, Sept. 23-24, 1996.

Project Title: **LIGHTWEIGHT MATERIALS FOR AUTOMOTIVE APPLICATIONS**

Investigators: R. J. Fields, R. B. Clough, R. deWit, A. C. Fraker, and E. N. Pugh

Technical Description:

The development of automobiles with substantially higher energy efficiency and lower emissions has become the goal of the U.S. automotive industry. Regardless of any possible changes in the fuel system or power train, a reduction in curb weight on the order of 50% is required to reach this goal. The automobile industry is pursuing the possibilities of reducing weight by replacing mild steel with high strength low alloy steels, thus permitting the use of thinner sections, and by employing lightweight materials such as aluminum alloys, particle-reinforced aluminum composites, and magnesium alloys. Although materials exist that fulfill current design requirements, a major technical challenge is the development of cost-effective manufacturing processes. In the case of magnesium alloys, corrosion is also an important issue. The aim of this project is to assist automobile manufacturers and their suppliers in the development of modeling, measurement, and characterization tools needed for the incorporation of the candidate materials into a new generation of fuel efficient vehicles.

Technical Objectives:

- Develop models of the press-and-sinter and powder forge processes for metal matrix composites.

- Validate powder consolidation models for commercial aluminum matrix composites.

- Assist industry in applying these models to accelerate die design and optimize preforming/forging conditions.

- Develop measurement methods to characterize multi-axial deformation of high strength aluminum and steel, leading to models for metal forming.

- Develop measurement techniques for assessing mechanical property variations in high strength aluminum and steel.

- Analyze dislocation patterning during forming by use of in-situ synchrotron, high resolution transmission electron microscopy (HREM), and small angle neutron scattering (SANS) experiments to guide modeling of forming processes.

- Provide reproducible electrochemical corrosion testing procedures for magnesium alloys which will yield results representative of automotive service.

Anticipated Outcomes:

- Improved models for forming lightweight materials, which will enable the automotive industry to produce automobiles which are lighter and more fuel efficient without sacrificing performance.

- Improved markets for U.S. producers of high strength steel, aluminum alloys, aluminum metal matrix composites, and magnesium alloys as a result of the removal of technical barriers to the use of their products by automobile manufacturers.

Accomplishments for FY 1996:

- A model for the plastic compaction of mixed hard and soft powders was developed, extended to the fully multi-axial case, and presented at the International Union of Theoretical and Applied Mechanics Symposium on Porous and Granular Media.

- The model was validated by experiment for industrially relevant case of aluminum powder with SiC reinforcement. The measurements, which have been made to date only under hydrostatic conditions, were in excellent agreement with the model predictions.

- A reproducible electrochemical test method for magnesium alloys was established.

Impacts and Technical Highlights:

- At the request of the U.S. Automotive Manufacturing Partnership (USAMP), NIST has signed a CRADA with the automobile manufacturers and their suppliers allowing them to use the research on particle-reinforced aluminum in their program to develop a low-cost consolidation process. In return, NIST will receive material and feedback from industrial researchers.

Outputs:

Presentations:

Clough, R. B., "Yield Surface for Porous Metal Matrix Composites," IUTAM Symposium on Mechanics of Granular and Porous Materials," invited paper, Cambridge University, Cambridge, England, July 15-17, 1996.

Fields, R. J., "NIST's Program on Al MMCs," presented to USCAR, Cambridge Modeling Group, and MATSYS, Inc. in Detroit on Oct. 27, 1995 and at ALCOA Tech Center, Dec. 4, 1995.

Fields, R. J., "NIST Intramural Research in Support of ATP Springback Predictability Program," Motor Vehicle Manufacturing Technology Meeting, Detroit, 4/18/96.

Project Title: **PERFORMANCE OF STRUCTURAL MATERIALS**

Investigators: R. B. Clough, R. deWit, R. J. Fields, T. Foecke, D. E. Harne, G. E. Hicho, E. N. Pugh, and J. H. Smith

Technical Description:

Using its expertise in testing and materials characterization, the Materials Performance Group provides assistance to industry and other Federal agencies on a broad range of problems related to the service performance of structural metals and alloys.

Technical Objectives:

- Assist the U.S. Department of Transportation in the development of the technical basis for safety regulations governing the design, manufacture and testing of new, advanced high pressure gas cylinders.

- Provide elevated temperature stress-lifetime data for copper tubes joined with new lead-free solders to be used for plumbing in new building construction, and to assist the Copper Development Association (CDA) in the development of design codes.

- Assist ATP contractor, the Wyman-Gordon Company, to correlate heat treatment, and hence microstructure, with low cycle fatigue properties of a Ni-based alloy (superalloy) which will be used in large, near-net-shape forgings for use in land-based gas turbines.

Anticipated Outcomes:

- New standards established for the design and manufacture of both high strength steel and composite gas cylinders, and improved standards in the case of Al cylinders.

- Improved retest methods developed for steel and Al gas cylinders.

- Enable CDA to establish safe codes for the use of new lead-free plumbing solders for copper-tube joints for water systems in commercial and residential buildings.

- Superalloy microstructures identified that lead to good low cycle fatigue resistance.

Accomplishments for FY 1996:

- Evaluation of sustained load cracking (SLC) in aluminum cylinders.

- Development of fracture criteria and tests for high strength steel cylinders.

- Evaluation of NDE methods for periodic retesting of steel cylinders.

- Development of design and testing criteria for composite cylinders.

- Revision of the technical specifications for the construction of high pressure cylinders.

- Completion of testing of one (Sn-5Sb) of three candidate plumbing solders for copper tube joints, and start of statistical analysis of the data and the development of a physical model for predicting creep life. Substantial progress has also been made in the testing of the other two lead-free solders.

Impact and Technical Highlights:

- Changes in the manufacture of aluminum cylinders have virtually eliminated the occurrence of failures in newly produced aluminum cylinders.

- The adoption of ISO worldwide standards for high strength steel and aluminum cylinders will permit the U.S. cylinder manufacturers to produce cylinders that are accepted for worldwide use.

- Established CRADA with the Wyman-Gordon Company to conduct collaborative work on microstructure and low-cycle fatigue resistance for superalloy samples. Also arranged for the Wyman-Gordon Company to provide a Research Associate (M.A. Morra) to work at NIST on the project.

Outputs:

Publications:

Clough, R. B. and McDonough, "The Measurement of Fiber Strength Parameters in Fragmentation Tests by Using Acoustic Emission," Composites Science and Technology, 1996.

Barnett, John T., Clough, R. B., and Kedem, B., Power Considerations in Acoustic Emission," Technical Research Report, TR 95-42, Univ. of Maryland.

Brand, P. C., Prask, H. J., Blackburn, Fields, R. J., and Proctor, T., "Possible Standard Specimens for Neutron Diffraction Residual Stress Measurements," Neutron Scattering in

Materials Science II, eds. D.A. Neumann, T.P. Russell, and B.J. Wuensch, MRS Symposium Proceedings 376, pgs. 429-33, 1996.

Brand, P. C., Hicho, G. E., and Prask, H. J., "Residual Stress in Railroad Track Measured by Means of Neutron Diffraction," NIST-IR 5912, 16 pgs., 1996..

Choi, C.S., Starke, W., Barker, J., and Fields, R.J., "Neutron Diffraction Study of Austempered Ductile Iron," Metall. Trans., 27A, pp. 923-928, 1996.

deWit, R., Fields, R. J., Low, III, S. R., Harne, D. E., and Foecke, T., "Fracture Testing of Large-Scale Thin-Sheet Aluminum Alloy," Fatigue and Fracture Mechanics: 27th Symposium, ASTM STP, American Society for Testing and Materials, Philadelphia, "in press".

deWit, R., "Diffraction Elastic Constants of a Cubic Polycrystal," Journal of Applied Crystallography, "in press".

Lu, J., James, M. R., and Mordfin, L., "Comparative Study of Different Techniques" Chapter 9 in Handbook of Measurement of Residual Stresses, J. Lu, editor, pp. 225-231, (Society for Experimental Mechanics, Bethel, CT, 1996).

Rana, M. D., Smith, J. H., and Tribolet, R. O., "Technical Basis for the Flawed Cylinder Test Specification to Assure Adequate Fracture Resistance of ISO High Strength Steel Cylinders," eds. K. K. Yoon, S. Bhandari, J. M. Bloom, H. Mehta, and G. Wilkowski, ASME PVP, Vol. 342, Fatigue and Fracture, "in press".

Presentations:

deWit, R., "The Overall Elastic Constants of a Cubic Polycrystal," Mechanics-Materials Linkage 1996 IMM Summer School, Institute for Mechanics and Materials, University of California, San Diego, July 22-Aug. 2, 1996, La Jolla, CA.

Smith, J. H., "Metrology of Materials Properties Tests," ASQC/NIST Measurement Quality Conference, Rockville, MD, Spring 1996.

Fields, R. J., "Effect of Eta-phase Precipitation on the Low Cycle Fatigue Behavior of Alloy 706," ATP Public Meeting on Materials Processing for Heavy Manufacturing, Gaithersburg, MD, Sept. 24, 1996.

Project Title: **PERFORMANCE OF METALS IN CORROSIVE MEDIA**

Investigators: A. C. Fraker, E. N. Pugh, R. E. Ricker, M. R. Stoudt, L. Bendersky, and F. W. Gayle

Technical Description:

The Materials Performance Group conducts a range of studies in this area in support of U.S. industry and other Federal agencies. The emphasis of these studies is on the effects of corrosion on the mechanical behavior of metals and alloys, and the activities center on testing, test method development, modeling and life prediction.

Technical Objectives:

- Collaborate with U.S. Air Force scientists at the Wright Laboratories to mitigate the effects of corrosion on the performance of aging military aircraft.

- Assist CRADA partner, Sandusky International, Inc., the only U.S. manufacturer of the large suction roll shells used in the pulp and paper industry, to develop improved test methods to assess the resistance of specialized duplex stainless steels to corrosion fatigue in the complex environments encountered in the pulp and paper industry.

- Assist the Science Applications International Corp (SAIC) in determining the corrosion resistance of electrode and other materials used in equipment for the production of "activated water."

- Provide technical assistance to the Nuclear Regulatory Commission (NRC) in the evaluation of degradation mechanisms in spent nuclear fuel rods and cladding materials.

Anticipated Outcomes:

- Standard tests and life prediction methodology developed that allow the U.S. Air Force to lower costs and improve safety associated with the operation of the aging Air Force fleet.

- Global competitiveness of Sandusky International strengthened as a result of the development of improved duplex stainless steels.

- Information provided to aid the NRC in making decisions related to the storage and transport of spent nuclear fuel.

Accomplishments for FY 1996:

- Developed competence in the area of corrosion-related failures in aging military aircraft through participation in two major conferences (Air Force Fourth Aging Aircraft Conference and the Seventh FAA-NASA International Conference on Continued Airworthiness of Aircraft Structures) and visiting the U.S. Air Force Air Logistics Center in Oklahoma City where the Air Force inspects and refurbishes its oldest fleet of aircraft, the KC-135s.

- Critically reviewed the existing literature on the corrosion behavior of aluminum alloys used for aerospace applications to gain insight into aging aircraft problems.

- Designed a system for measuring corrosion fatigue crack propagation in a simulated pulp and paper processing environment, and conducted preliminary experiments.

- Supervised the conduct of electrochemical tests by SAIC staff on a series of candidate materials for use in equipment for the production of activated water, and assisted in the interpretation of the data.

Impact and Technical Highlights:

- A CRADA was initiated with Sandusky International, Inc. for the development of improved measurements to evaluate the resistance of their proprietary duplex stainless steels to corrosion fatigue.

Outputs:

Publications:

Chohayeb, A., Fraker, A., Eichmiller, F., Waterstrat, R., and Boyd, J., "Corrosion Behavior of Dental Casting Alloys Coupled with Titanium," Medical Applications of Titanium and Its Alloys: The Material and Biological Issues, ASTM 1272, S. A. Brown and J. E. Lemons, 1996.

Clough, R.B., Shapiro,A.J. Bayba, A.J., and Lucey, G.K. "Boundary Layer Fracture in Composite Solder Joints." Journal of Electronic Packaging, Dec. 1995, vol. 117, 270-4

Bandyopadhyay, S., Miller, A. E., Chang, H. C., Banerjee, G., Yuzhakov, V., Yue, D.-F., Ricker, R. E., Jones, S., Eastman, J. A., Baugher, E., and Chandrasekhar, "Quasi-periodic Quantum Dot Arrays Produced by Electrochemical Synthesis, Nanotechnology, "in press".

Bruening, R. and Mordfin, L., "Infrared Thermography Standards for Nondestructive Testing," International Conference on Thermal Sensing and Imaging Diagnostic Applications, SPIE Proceedings Vol. 2245, pp. 220-221 (SPIE, Bellingham, WA, 1995).

Imam, M. A. and Fraker, A. C., "Titanium Alloys as Implant Materials," Medical Applications of Titanium and Its Alloys: The Material and Biological Issues, ASTM STP 1272, S. A. Brown and J. E. Lemons, eds., 1996.

Polvani, R. S., Fraker, A. C., and Evans, C. J., "Using a Potentiostat to Model In-Process Electrochemical Dressing," Proceedings International Manufacturing Engineering Conference, University of Connecticut, Storrs, CT, Aug. 7-9, 1996. Manufacturing Engineering - 2000 and Beyond, ed. Ioan D. Marinescu, 1996.

Polvani, R. S., Evans, C. J. and Fraker, A. C., "Effects of Coolant Chemistries on Electrochemical In-Process Wheel Dressing," IDA/Lawrence Livermore National Laboratory Supertech 96 Meeting, submitted September 1996.

Ricker, R. E., Miller, A. E., Chang, H. C., Yue, D.-F., Banerjee, G., and Bandyopadhyay, G., "Nanofabrication of a Quantum Dot Array: Atomic Force Microscopy of Electropolished Aluminum," Journal of Electronic Materials, Vol. 25, No. 10, 1996.

Ricker, R. E. and Bertocci, U., "A Review of Electrochemical Noise and its Application to the Study of Stress Corrosion Cracking," New Techniques for Characterizing Corrosion and Stress Corrosion, The Minerals, Metals, and Materials Society, Warrendale, PA, pp. 235-245, 1996.

Stoudt, M. R., Fink, J. L., and Ricker, R. E., "Evaluation of the Propensity of Replacements for Halon 1301 to Induce SCC in Alloys used in Aircraft Fire Suppression and Distribution Systems," Journal of Materials Engineering and Performance, Vol. 5, No. 4, pp. 507-515, 1996.

Presentations:

Ricker, R. E., "The Influence of Fracture Surface Morphology and Ligament Formation on the Propagation of Transgranular Stress Corrosion Cracks," invited paper presented at the Second International Conference Corrosion Deformations Interactions, Nice, France, Sept. 1996.

Stoudt, M. R., "Corrosion Fatigue of Duplex Stainless Steel in De-Inked Paper Processing White Water--The Current Issues," Annual Program Review, NIST, Gaithersburg, Md., Aug. 1996.

Project Title: MAGNETIC IMAGING AND MODELING

Investigators: A. J. Shapiro V. I. Nikitenko,* V. I. Gornakov,* R. D. McMichael, M. J. Donahue, H. D. Chopra, L. H. Bennett, R. D. Shull, and R. D. Mathews
*Guest Scientists, Institute for Solid State Physics, Russian Academy of Sciences

Technical Description:

As a result of cooperative work between NIST and the Institute for Solid State Physics of the Russian Academy of Sciences, a new non-destructive method was developed for real time characterization of magnetic domain structure in a wide range of technologically important magnetic materials such as spin-valves, ulrathin multilayer, and granular systems. The domain structure is imaged through the interaction of polarized light with a transparent magneto-optical indicator film (MOIF) which is placed on top of the magnetic sample. Since the polarization of light is affected by magneto-static field of the sample (Faraday effect), its magnetic domain structure can be identified using a polarizing microscope through local changes in light polarization in the indicator film. Magnetic force microscopy (MFM) and a magnetic fluid decorating method (MFDM) add an extra dimension to these MOIF studies. The MFM allows very high magnification observation of chosen regions and the MFDM provides the opportunity for studying magnetic domain boundaries at relatively high magnification in a scanning electron microscope.

Technical Objectives:

- Develop magnetic-optical imaging techniques for routine non-destructive quality control of non-transparent magnetic and high-temperature superconducting materials.

- Measure using the MOIF technique real-time magnetization reversal processes in spin-valves, ultrathin multilayer, and granular systems.

- Investigate domain wall nucleation and motion in magnetic materials as a function of their microstructure and processing variables.

Anticipated Outcome:

- MOIF method is expected to become a standard non-destructive quality control imaging technique for the next generation of magnetic materials for sensors and storage devices.

- MOIF studies will contribute to the fundamental understanding of remagnetization process in artificial magnetic materials.

Accomplishments for FY 1996:

- Static and dynamic (ac) magnetization and remagnitization data were obtained for different technological stages of spin-valve structure manufacturing.

- Magnetic domain structures of granular and multilayer nanostructured systems were measured and their relationship to film parameters and defects were investigated.

- Imaged inhomogeneous stray fields due to magnetization ripple using magnetic force microscopy in NiO-coupled thin film coupled to NiO, and identified this stray field as a mechanism for coercivity transfer to unpinned films.

Impacts and Technical Highlights:

- Argonne National Laboratory, Oak Ridge National Laboratory, General Electric Company, Intermagnetic General Corporation, and the University of Wisconsin used the MOIF technique to develop long high-temperature superconductor wires for electric generators and to optimize the wire manufacturing processes.

- The MOIF system was deemed as one of the 100 most technologically significant new products of the year by R&D Magazine.

Outputs:

Publications:

Bennett, L.H., McMichael, R.D., Swartzendruber, L.J., Hua, S., Lashmore, D.S., Shapiro, A.J., Gornakov, V.S., Dedukh, L.M., Nikitenko, V.I., "Dynamics of Domain Structure in Magnetic Multilayers," IEEE Transactions on Magnetics. Volume 31, Number 6/2, (1996).

Nikitenko, V.J., Gornakov, V.S., Dedukh, L.M., Kabanov, Yu P., Khapikov, A.E., Bennett, L.H., Chen, P.J., McMichael, R.D., Donahue, M.J., Swartzendruber, L.J., Shapiro, A.J., Brown, H.J., Egelhoff Jr., W.F., "Magneto-Optical Indicator Film Study of the Magnetization of a Symmetric Spin Valve," IEEE Transactions on Magnetics. Vol. 32, No. 5/2, 4639 (1996).

Donahue, M.J., Bennett, L.H., McMichael, R.D., Swartzendruber, L.J., Shapiro, A.J., Nikitenko, V.I., Gornakov, V.S., Dedukh, L.M., Khapikov, A.F., Matveev, V.N., Levashov, V.I., "Complementary Imaging of Granular Co-Ag Films with Magneto-optical Indicator Film Technique and Magnetic Force Microscopy," J. Appl. Physics. Vol. 79, No. 8/A, 5315, (1996).

Nikitenko, V.I., Gornakov, V.S., Dedukh, L.M., Khapikov, A.F., Bennett, L.H., McMichael, R.D., Swartzendruber, L.J., Shapiro, A.J., Donahue, M.J., Matveev, V.N., Levashov, V.I.

"Magneto-optical Indicator Film (MOIF) Microscopy of Granular and Layer Structures," J. Appl. Phys., Vol. 79, Number 8/B, 6073 (1996).

Project Title: **DEVELOPMENT OF SCANNING ACOUSTIC MICROSCOPY FOR TOMOGRAPHY OF STRESS**

Principal Investigator: Eva Drescher-Krasicka

Technical Description:

The Metallurgy Division has developed Ultrasonic Computer Tomography of Stress based on the interference of polarized modes propagating through stressed and unstressed areas in the sample. Although the theoretical solutions for longitudinal and leaky modes do not presently exist, an effort to develop them is in progress in cooperation with J. R. Willis from the Department of Mathematics and Theoretical Physics at the University of Cambridge and J. A. Simmons at NIST. The shear wave interference for tomography of polarized modes was first solved by R. A. Kline, L. Chiang and E. Drescher-Krasicka but this solution is based on the assumption that the split shear components after the reflection create only two interfering waves. This was corrected by J. R. Willis and E. Drescher-Krasicka in a recently published paper in Nature Vol. 384/1996.

Technical Objectives:

- Solve analytically or numerically the propagation and reflection problem for longitudinal and surface/leaky acoustic waves in media under stress.
- Use the wave propagation solutions for interpretation of the ultrasonic imaging of stress.

Anticipated Outcome:

- Reconstruction of the values of the principal stresses as a function of position in a material.
- Scanning Acoustic Microscopy developed as a quantitative tool for measurement of residual stresses.

Accomplishments for FY 1996:

- Obtained quantitative distribution of the shear stresses by Scanning Acoustic Imaging of Stress in diametrally compressed aluminum disks 3mm thick and 40mm in diameter under loads from 2000N to 2450N. The values of of maximal shear stress were determined at every point of the image by comparison with a two-dimensional analytical solution for this loading geometry.

- Obtained qualitative distributions of stress in welds and in electronic packaging as a function of thickness based on frequency variable surface-leaky wave scanning.

- Obtained U.S. Patent Number 5549003, Ultrasonic Computer Tomography of Stress, issued August 27, 1996.

Impact and Highlights:

- NIST proved the feasibility of the ultrasonic technique for the qualitative mapping of the distribution of stress across a sample (examples: welds, electronic packaging).

Outputs:

Publications:

Drescher-Krasicka, E., and Willis, J. R., "Mapping Stresses with Ultrasound" Nature, Vol. 384, No. 6604, 1996, pp. 52-55.

Drescher-Krasicka, E., Rowcliffe, D. J., and Dutton, R., "An Approach to the Characterization of Bi-Material Interfaces by Acoustic Microscopy." Proceedings of ICCE/3, ed. Hui, D., New Orleans, July 96, pp. 223-225.

Drescher-Krasicka, E., "New Approach to Acoustoelasticity," ICIFUAS11/Poitiers, France, July 1996, submitted to the "Journal de Physique."

Presentations:

Drescher-Krasicka, E., "Characterization of Bi-Material Interfaces by Acoustic Microscopy," ICCCE/3, New Orleans, July 1996.

Drescher-Krasicka, E., Three lectures at Sonix Inc.: Basics of Stress Imaging by Scanning Acoustic Microscopy.

Project Title: **ELECTRON MICROSCOPY**

Investigator: John Bonevich

Technical Description:

Atomic structure and compositional characterization of materials can lend crucial insights into their properties. For example, direct observation of localized structures by transmission electron microscopy (TEM) provides an important information feedback to the optimization of crystal growth and processing techniques. A wide variety of structures may be observed such as crystal structure and orientation, grain size and morphology, defects, stacking faults, twins and grain boundaries, second phase particles (their structure, composition and internal defect structure), compositional variations and the atomic structure of surfaces and interfaces. To this end, the Metallurgy and Ceramics Division TEM Facility consists of three transmission electron microscopes, a specimen preparation laboratory, and an image processing/computational laboratory. The state-of-the-art JEOL3010 TEM has atomic scale resolution as well as detectors for analytical characterization of thin foil specimens; a thin window X-ray detector for compositional analysis and an energy selecting imaging filter (IF) for compositional mapping at atomic resolution.

An active collaboration with the Chemical Science and Technology Laboratory (CSTL) has been initiated for the development of holographic measurement methods using their new 300 keV field-emission TEM. The TEM employs a highly coherent electron source that has allowed Metallurgy Division scientists to develop and apply electron holography, in addition to having the capability of forming ~1 nm probes with 1 nA currents. Holograms record the phase distribution of electron waves interacting with matter and provide a quantitative measure of electromagnetic phenomena such as the magnetic fields *inside* superconductors (the flux-line lattice) and electric fields emanating from pn junctions. Holography also quantitatively measures specimen thickness, surface topography, mean inner potentials of materials, dislocation strain fields, nano-diffraction and electron microscope lens aberrations.

Technical Objectives:

- Characterize the structure and chemistry of materials at the atomic scale to better understand and improve their properties.

- Observe the electric and magnetic domain structure of materials to better characterize their structure and property relationships.

- Develop and apply new measurement techniques in electron microscopy.

Anticipated Outcome:

- Feedback of structure and compositional information from electron microscopy as a guide to optimization of crystal growth and other materials processing techniques.

- Quantitative measurements for mean inner potential databases obtained by electron holography.

Accomplishments for FY 1996:

- Developed and refined a software package for the reconstruction and analysis of electron holograms. This software, called HolograFREE, was made freely available to the scientific community through the Internet. The software incorporates advanced window/masking techniques in Fourier space to eliminate pronounced smearing of data and allows for precise alignment of the phase in reconstructed holograms.

- Measured the mean inner potential, surface topography and internal void structure of nanoscale TiO_2 particles by electron holography. The particles were shown to have hollow voids with faceted internal surfaces with potentials of 12.5 eV.

- Created a centralized User Facility for the preparation and processing of TEM specimens. The Facility has rapid thinning ion mill, two dimplers, a precision polishing wheel and other preparation equipment.

- Developed Fresnel and Foucault microscopy techniques using the imaging filter (IF) to image magnetic contrast in recording media and spin-valve materials. These experiments allow observation of magnetic contrast with higher resolution and sensitivity than conventional TEMs.

- Refined the theoretical treatment of vortex image contrast in superconductors. The results show that the London model of a vortex can be a useful computational tool whereas the Clem model gives better agreement with the experimental data.

Impacts and Technical Highlights:

- The software package HolograFREE is being used to reconstruct and analyze electron holograms in academic and corporate research laboratories in the U.S. (Northwestern, Stevens Institute of Technology, Lawrence Berkeley National Laboratory, NIST), Japan (Hitachi), and Europe (Philips, Bologna University).

Outputs:

Publications:

Book chapter entitled Electron Holography of Electromagnetic Fields in: *Introduction to Electron Holography*, eds. L. Allard, et al., (Elsevier Press, Amsterdam, in press).
Bonevich, J. E., Pozzi, G., and Tonomura, A., "Electron Holography of Electromagnetic Fields" in: Introduction to Electron Holography, eds. L. Allard, et al., (Elsevier Press Amsterdam, in press).

Bonevich, J., James, A., Capacci, D., Pozzi, G., Harada, K., Kasai, H., Matsuda, T., Tonomura, A., "On the Influence of Specimen Thickness on TEM Images of Superconducting Vortices," Proc. of 54th Microscopy Society of America, ed. G.W. Bailey, (San Francisco Press, San Francisco, 1996), p. 724.

Turner, S., Bonevich, J., E., Maslar, J. E., Aquino, M. I., Zachariah, M. R., "Transmission Electron Microscopy and Electron Holography of Nanophase TiO_2 Generated in a Flame Burner System," Materials Research Society Proceedings, (in press).

METALS PROCESSING

The properties of metals and their alloys depend strongly on their processing history. The conditions under which materials are processed and fabricated into useful items determine the distributions of phases, grain structure, alloy compositional segregation, and defects in the final products. These distributions are crucial in determining the alloy strength, ductility, magnetic response, homogeneity, and other properties important for commercial applications. The Metals Processing Program focusses on measurements and predictive models needed by industry to provide improved process control, develop improved alloy properties, tailor material properties for particular applications, and reduce costs.

Major successes in applying measurements and modeling to processing applications have been achieved in interactions with the aerospace, powder metallurgy, and electronics industries. For example, cooperative research and development projects with industry (Crucible Compaction Metals, Fluxtrol Manufacturing) have resulted in significant improvements in process control for atomization of steel and superalloy powders. Predictive models for solidification and microstructural evolution during processing have been incorporated by industry into design systems for casting of aerospace alloys and production of defect-free electronic materials, helping to reduce rejection rates arising from defective parts. Current measurement and modeling activities in this program emphasize work on rapidly solidified steels, aerospace castings, electrodeposited thin films, alloy coatings, and electronic materials. Frequent interactions and collaborations with industrial producers are maintained, along with participation in industrially-oriented consortia. Standard reference materials are prepared, including electrodeposited thickness standards, microhardness standards, and homogeneous alloy samples produced by atomization, rapid solidification, and hot isostatic pressing processes.

Measurements and predictive models for processing being pursued in this program are of three kinds:

- Measurements and models are developed to help design materials production processes, such as measurements and evaluations to provide alloy phase diagrams, which are the roadmaps alloy designers use to predict the alloy phases that can be produced under specific processing conditions. These evaluations are playing key roles in NIST collaborations with industrial consortia on electronic solders and casting of superalloys for aerospace applications.

- Measurements are made under dynamic conditions to monitor, in real time, properties of materials while they are actually being produced and to determine difficult-to-measure process parameters while the process is occurring. In collaboration with the NIST program on intelligent processing of materials, special fast-response sensors, simulations and imaging techniques have been developed for application to powder atomization and spray deposition processes, and workshops have been held to transfer these techniques to industry. Here, dynamic models of the process are important both for design of manufacturing procedures and for applications of real time feedback and control.

- To evaluate the adequacy of process models, it is important to measure the properties of the final materials and relate them to the process conditions. A current emphasis in this respect is on relating impact resistance and corrosion resistance of rapidly solidified nitrogenated stainless steels to processing conditions and alloy microstructure. Norfolk Southern (railroad switches), DoD (armor applications), and Crucible Materials Corporation (corrosion-resistant alloys) have collaborated with NIST in various aspects of this work.

In all of this work, the objective is to help U. S. Industry apply measurements and predictive modeling to produce improved materials at reduced cost.

Project Title: **PROCESSING OF ADVANCED MATERIALS**

Investigators: F. S. Biancaniello, R. J. Schaefer, S. D. Ridder, R. D. Jiggetts, U. R. Kattner, M. E. Williams, R. E. Ricker, and J. L. Fink

Technical Description:

Advanced powder metallurgy processing methods are being evaluated to determine the effect of process measurements and control on high performance materials. Predictive models and thermodynamic assessments are developed to aid in microstructure, composition and property control. This research is an outgrowth of a highly successful NIST/Industry Consortium project where intelligent processing techniques were applied to production of rapidly solidified alloy powder by controlled atomization of liquid superalloys. Two areas of recent emphasis are (1) research on nitrogenated stainless steel, including support for an on-going industrial ATP project, to predict effects of rapid solidification on these high-strength corrosion-resistant alloys and (2) application of rapid solidification methods to produce standard reference materials with improved homogeneity. In addition, methods are developed for advanced alloy preparation, and samples are produced both for studies within NIST and for nationwide research programs.

Technical Objectives:

- Provide industry with measurements, predictive models and methodologies needed to apply the processes investigated in this project to advanced alloys.

- Develop techniques to prepare improved standard reference materials and research samples.

- Apply phase diagram information and kinetic models to develop methods for predicting solubility and phase stability in alloys. Develop guidelines for eliminating the tendency to form undesirable alloy phases.

Anticipated Outcome:

- Development of improved measurements and process models for producing alloys with improved properties. Improved understanding of metal alloying effects. Enhancement of the thermodynamic data base for improved accuracy in predictive modeling of metals processing.

- Availability of more homogeneous standard reference materials.

- Identification and modeling of new and improved processing methods so that industry can more efficiently produce advanced alloys.

Accomplishments for FY 1996:

- Molten metal samples of nitrogenated stainless steel were extracted during simulated atomization runs in order to measure nitrogen uptake as a function of time and temperature under nitrogen atmospheres. These measurements made it possible to establish kinetic rates as well as to test process models and thermodynamic solubility predictions for these alloys used in industrial applications.

- Produced powders of white cast iron and 446 stainless steel for use in standard reference materials having improved homogeneity and microstructural refinement.

- Conducted pitting and crevice corrosion tests which indicated that nitrogenated stainless steels prepared at NIST have unusually good corrosion resistance.

- Applied empirical models and thermodynamic data to develop methods for predicting nitrogen solubility and phase stability in atomized stainless steel alloys.

Impacts and Technical Highlights:

- Data developed on solubility and rate of absorption of nitrogen in stainless steel were used by industry as part of an ATP project on corrosion-resistant steels.

- Molten metal atomization by use of high velocity inert gas in combination with HIP consolidation of the resulting rapidly-solidified powder was shown to be a viable processing technique to prepare standard reference materials having special homogeneous properties.

- Specimens of nitrogenated stainless steels produced at NIST for testing at Aberdeen Proving Grounds show promise as armor for amphibious vehicles, armored personnel carriers and tanks. These alloys allow numerous applications since they simultaneously can be very strong, hard, and ductile, thus having excellent impact resistance, and also have outstanding corrosion resistance.

- Special solder alloys prepared at NIST allowed development of a comprehensive data base for a nationwide industrial research program on alternatives to lead-based solders. This program was initiated by the National Center for Manufacturing Sciences in collaboration with ten industrial companies involved in microelectronics.

Outputs:

Publications:

Agren, J. Cheynet, B., Clavaguera-Mora, M. T., Hack, K., Sommer, F., and Kattner, U. R., "Extrapolation of the Heat Capacity in Liquid and Amorphous Phases," CALPHAD, **19** (1995) 449-480.

Biancaniello, F.S., Gayle, F. W., Suryanarayana, C., Froes, F. H., "Minimization of Nitride Formation During Mechanical Alloying of Ti-Al-Nb," Proceedings International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials, Quebec, July 1995, ed. F. H. Wonbier, Materials Science Forum, Vols. 225-227, Trans. Tech. Publications, Switzerland, pp. 483-488 (1996).

Biancaniello, F.S., Gayle, F. W., Fields, R. J., Schaefer, R. J., and Janowski, G. M., "Processing and Structure of Ball Milled Nitrogenated Stainless Steel," Processing and Properties of Nanocrystalline Materials, Edited by C. Suryanarayana, J. Singh, and F. H. Froes, The Minerals, Metals, and Materials Society, 1996.

Project Title: **SOLIDIFICATION MODELING**

Investigators: Samuel R. Coriell, James A. Warren, and William J. Boettinger

Technical Description:

Numerical and analytic models are being developed to predict solute segregation during solidification as a function of processing conditions. These segregation patterns play an important role in determining the properties of the solidified crystals and castings. The effects of fluid flow on the breakdown of the crystal-melt interface from planar to nonplanar cellular shapes, and on the growth of fully-dendritic shapes are being studied. The fluid flow research is relevant to materials processing in microgravity.

Technical Objectives:

- Perform solidification modeling, especially of fluid flow, for application to microgravity solidification research including: monotectic composite solidification, dendritic growth and faceted crystal growth.

- Develop a unified numerical technique for the simulation of dendritic growth that includes tip kinetics, solute redistribution and coarsening.

Anticipated Outcome:

- Improved scientific understanding of completed and planned experiments being carried out at a number of research laboratories involved with microgravity research. These experiments probe the fundamental role of melt convection or its absence during solidification. These include experiments on directional solidification of bismuth-tin alloys performed in microgravity and on monotectic composite alloys of aluminum-indium at the University of Alabama in Birmingham.

- Optimized commercial growth conditions identified to produce high-quality lead bromide-silver bromide crystals as a non-linear optical material.

- Supercomputer calculations of a single dendrite which permit construction of improved approximate models for castings with better prediction of microstructures and defects.

Accomplishments for FY 1996:

- A model for the effect of density changes during composite solidification allows the prediction of the volume fractions of the transformed phases. The model has been applied to the aluminum-indium monotectic system.

- A stagnant boundary layer model for the effect of natural convection on dendritic growth has been developed. It predicts the relationship between dendrite growth velocity and tip radius as a function of bath supercooling and agrees with experimental results.

- Stability calculations of the effect of shear flows on macrostep formation in crystals grown from solution have shown that flow in the direction of step motion is destabilizing while flow up the steps is stabilizing. This is in qualitative agreement with classic experiment on the growth of ADP crystals and is applicable to KDP crystals for laser fusion and to protein crystal growth.

- The possibility of dendrite fragmentation during remelting of a partially liquid/solid alloy structure was demonstrated using 2-D simulations with the phase-field, diffuse interface, numerical technique. A model for dendrite fragmentation is needed to predict the columnar to equiaxed transitions in castings and the formation of stray grains during dendritic single crystal growth of gas turbine blades.

- Investigations into 3D simulations of dendritic growth, fragmentation and coarsening have begun, using the phase-field method. These calculations are extremely computationally demanding, requiring the most sophisticated supercomputers, and will be done in collaboration with the NIST Information Technology Laboratory. Three-dimensional simulations are essential to accurately predict experimentally observed structures.

Impact and Technical Highlights:

- Observations and calculations of solutal convection driven interface instabilities in lead bromide doped with silver bromide have been carried out in collaboration with a group at Northrup-Grumman. X-ray studies of this optoelectronic material show that the instabilities deteriorated the crystal quality.

- A definitive test of morphological stability theory in tin-bismuth alloys was carried out during the first MEPHISTO space experiment in collaboration with a group at Grenoble. Calculations of the growth of instabilities under transient conditions have provided understanding of the instability behavior; excellent agreement between theory and experiment has been obtained.

- NIST phase-field research continues to stimulate worldwide application of this technique to a variety of solidification problems.

Outputs:

Publications:

Biloni, H., and Boettinger, W. J., "Solidification," in Physical Metallurgy, 4th edition, edited by P. Haasen and R. W. Cahn, 1996, North Holland, Amsterdam, p. 669-842 (1996).

Boettinger, W. J., and Warren, J. A. "Prediction of Solidification Microstructure using the Phase-Field Method," Proceedings of the General COST512 Workshop on Modelling in Materials Science and Processing, Ed. By M. Rappaz and M. Kedro, European Commission, Brussels, p. 11-20 (1996).

Coriell, S. R., Murray, B. T., Chernov, A. A., and McFadden, G. B. "Effects of Shear Flow and Anisotropic Kinetics on the Morphological Stability of a Binary Alloy," Metallurgical and Materials Transactions **27A**, p. 687-694 (1996).

Coriell, S. R., Murray, B. T., Chernov, A. A. and McFadden, G. B., "Step Bunching on a Vicinal Face Growing in a Flowing Solution," J. Crystal Growth, in press.

Favier, J. J., Lehmann, P., Drevet, B., Garandet, J. P., Camel, D., and Coriell S. R. "A Study of Morphological Stability during Directional Solidification of a Sn-Bi Alloy in Microgravity, in Materials and Fluids under Low Gravity, Lecture Notes in Physics, Vol. 464, ed. L. Ratke, H. Walter, and B. Feuerbacher (Springer-Verlag, Berlin, 1996), p. 77-94.

Sekerka, R. F., Coriell, S. R., and McFadden, G. B., "The Effect of Container Size on Dendritic Growth in Microgravity," J. Crystal Growth, in press.

Wheeler, A. A., McFadden, G. B., and Boettinger, W. J., "Phase-Field Model for Solidification of a Eutectic Alloy," Proc. Royal Soc., Series A, **452** p. 495-525 (1996).

Losert, W., Shi, B. Q., Cummins, H. Z., and Warren, J., "Spatial Period Doubling of Dendritic Arrays in Directional Solidification," Physical Review Letters, Vol. 77, 889 (1996).

Warren, James, A., and Murray, B.T., "Simulations of Ostwald Ripening: a Phase-Field Method," Modelling and Simulation in Materials Science Vol. 4, 219 (1996).

Boettinger, William, J., and Warren, James, A., "The Phase-Field Method: Simulation of Alloy Dendritic Solidification During Recalescence," Met. Trans., Vol. 27A, 657 (1996).

Warren, James, A., and Boettinger, William, J., "Prediction of Dendritic Microsegregation Patterns using a Diffuse Interface Phase-Field Model," in Modeling of Casting, Welding and Advanced Processes VII., Cross, M., and Campbell, J., eds.

Presentations:

Boettinger, W. J., "Modeling Dendritic Solidification of Alloys Using a Diffuse Interface Phase-field Method," Department of Materials Science and Engineering, University of Arizona, Tucson, AZ, November 9, 1995.

Boettinger, W. J., "Alloy Solidification: From Fundamental Studies to SuperAlloys," Burgess Memorial Lecture, Washington Chapter ASM, March 12, 1996.

Coriell, S. R., "Interface Morphology during Crystal Growth," 3rd Microgravity Fluid Physics Conference, Cleveland, Ohio, June 12-15, 1996.

Coriell, S. R. "The Effect of a Shear Flow on the Morphological Stability of a Vicinal Face: Growth from a Supersaturated Solution," 31st COSPAR Scientific Assembly, Birmingham, England, July 13-19, 1996.

Coriell, S. R., "Step Bunching on Vicinal Stepped Faces Growing from Solutions and Melt," Tenth American Conference on Crystal Growth and the Ninth International Conference on Vapor Growth & Epitaxy, Vail, Colorado, August 4-9. 1996.

Boettinger, W. J., "Prediction of Solidification Microstructure Using the Phase-Field Method," Department. Of Materials, Swiss Federal Institute of Technology, Lausanne, Switzerland, August 20, 1996.

Boettinger, W. J., "Prediction of Solidification Microstructure Using the Phase-Field Method," 1996 General Workshop of COST 512 on Modeling in Materials Science and Processing, European Commission, Davos, Switzerland, Sept. 29, 1996.

Warren, James, A., American Society of Mechanical Engineers, San Francisco, CA, November, 1995, "Wavelength Selection During Directional Solidification of a Binary Alloy."

Warren, James, A., Materials Research Society Fall Meeting, Boston, MA, December, 1995, "Wavelength Selection During Directional Solidification of a Binary Alloy."

Warren, J. A., American Physical Society, St. Louis, Missouri, March, 1996, "Prediction of Dendritic Growth and Microsegregation Patterns in a Binary Alloy using a Phase-Field Method."

Warren, James, A., Univ. of Chicago, Chicago, IL, April, 1996, "Prediction of Dendritic Growth and Microsegregation Patterns in a Binary Alloy using a Phase-Field Method."

Project Title: **MODELING AND SIMULATION OF MATERIALS PROCESSING**

Investigator: J. A. Simmons

Technical Description:

Materials science and engineering is a field of practical application and technology that is ripe for the application of new mathematical techniques spawned from the revolution in computer technology. The goal of this project is to identify areas of practical importance, lay out the theoretical basis for approach to these areas, foster interactions with computational mathematicians and experimental materials scientists, and develop new technologies for solving previously intractable materials science problems.

Because understanding of materials science is becoming more quantitative and computational capabilities are growing with the advent of large-scale parallel processing, modeling of material microstructure development as a function of processing conditions has become one of the most promising areas for new materials science theory. There are new opportunities both to apply presently available mathematical techniques to materials processing and to develop new mathematics for this technology.

Recent project work has emphasized applications to microstructural evolution, including modeling of solid-state diffusive transformations and incorporation of stress effects. This work is now being redirected to focus on the application of leaky ultrasonic waves to tomographic descriptions of internal stresses in acoustic microscopy.

Technical Objectives:

- Model nonlinear elastic effects on leaky waves in the presence of internal stresses and to collaborate with experimental materials scientists in developing ultrasonic tomographic reconstruction of internal stress distributions in thin films and near interfaces.

- Model effects arising from dynamic processing parameters and environmental conditions on complex metal/ceramic alloys.

Anticipated Outcome:

- On-line methods developed for measuring internal stress distributions in thin films and coatings, leading to improved reliability in new materials products based on thin film technology.

- Predictive mathematical models developed that can be incorporated into the control of materials processing systems. This will lead to improvement of material products and reduction of materials processing costs.

Accomplishments for FY 1996

- Collaboration with E. Drescher-Krasicka and J. R. Willis of Cambridge University has led to a theory for describing residual stresses in acoustic microscopy using polarization effects produced by nonlinear elastic effects.

- A new Markov chain theory for nonequilibrium systems was developed. This theory addresses stochastic control in the processing of materials undergoing diffusive ordering and phase transformations in crystalline solids. While extensive computational development is required to implement the theory, the formulation provides a new method for finding processing regimes which optimize the probability of producing high performance microstructures and predicting the risk of structural breakdown of microstructure as a result of environmental conditions.

- Probability superposition methods--used in many lattice entropy calculations for finding equilibrium phase structures in alloys--were reviewed using both combinatorial Moebius function and linear convex methods, and the theory for applying interior point methods to replace entropy maximization in highly ordered structures was given.

Impacts and Technical Highlights:

- Collaboration with E. Drescher-Krasicka and J. R. Willis of Cambridge University has permitted Dr. Drescher-Krasicka to develop new experimental methods for visualizing internal stresses in thin films.

- A Markov chain approach to predictive modeling of processing for microstructure control was presented at the symposium on the "Mathematics of Thermodynamically Driven Microstructural Evolution". This symposium was coorganized by J. A. Simmons and sponsored by TMS, SIAM, NIST and ARPA. The Proceedings of this symposium are being jointly published by TMS and SIAM. This symposium has had a significant impact in establishing new collaborations between mathematicians and materials scientists.

Outputs:

Publications:

Simmons, J. A. "The Mathematics of Processing for Material Microstructure," Proceedings of TMS Symposium on the Mathematics of Thermodynamically Driven Microstructural Evolution, The Minerals, Metals, and Materials Society, 1996.

Presentations:

Simmons, J. A. "A New Approach to Diffusive Phase Transformations," TMS Symposium on the Mathematics of Thermodynamically Driven Microstructural Evolution, Cleveland, Ohio, Nov. 1, 1995.

Project Title: **POWDER ATOMIZATION AND SPRAY DEPOSITION**

Investigators: S. D. Ridder and F. S. Biancaniello

Technical Description:

Research in this project is focused on measurement and modeling tools for process understanding and control of metal powder and coatings produced by atomization and plasma spray deposition processes. Previous studies on powder atomization resulted in the development of advanced process sensors and the incorporation of these sensors into an expert system based process controller. The processing tool chosen as a demonstration platform was the NIST Supersonic inert Gas Metal Atomizer (SiGMA). The current project activities are focused on the transfer of the process control technology to a commercial atomizer at Crucible Research Center near Pittsburgh and the installation of a new plasma spray deposition system at NIST. The technology transfer work involved software and hardware conversion to move the control system from a

Macintosh environment to a PC computer running LabVIEW for Windows and documentation of the software and hardware requirements. The resulting control system is easily portable to other computer systems, other atomizers, and other industrial processes. The focus of the plasma spray deposition project, once installed, will be the development of measurement tools to provide diagnostic and control capabilities to the production of industrially important spray coatings such as ceramic-based Thermal Barrier Coatings (TBC's) and metallic-based corrosion and wear reducing layers.

Technical Objectives:

- Develop diagnostic tools for measuring process conditions of gas atomizers and plasma spray systems. This includes off-line analysis tools (e.g. high-speed cinematography, holography) and real-time sensors suitable for process control.

- Develop mathematical modeling techniques to provide predictive calculations of process conditions and product characteristics of gas atomizers and plasma spray systems. This includes Computational Fluid Dynamics (CFD) of supersonic jets and gas plasma jets, process simulators, statistical analysis of process performance, and calculations of composition and phase stability within the resulting powder and spray deposition products.

- Develop expert system driven process control strategies with generic applicability to a wide range of metal processing equipment and computer platforms.

- Provide industry with the methodology necessary to incorporate technology developed in this project into the production environment.

Anticipated Outcome:

- Robust process sensors for monitoring and control of atomizers and plasma spray systems.

- New mathematical modeling tools to aid in equipment design and improve process efficiencies.

- Expert system driven process controllers with hardware and software supplied and supported by third party companies with established national distribution network.

Accomplishments for FY 1996:

- Operated new process controller on SiGMA system suitable for transfer to an industrial gas atomizer.

- Work is progressing with the installation of data acquisition hardware on the atomizer at Crucible Research Center.

- A User Reference Manual has been completed by NIST that documents the software and hardware components of the SiGMA control system.

- System hardware components have been purchased and the electrical utilities have been installed within the spray deposition booth.

- A NIST-SBIR-funded thermal imaging sensor was demonstrated on the spray plume of the SiGMA system.

Impacts and Technical Highlights:

- CFD code and gas flow diagnostics equipment developed by NIST under this project is scheduled to be used by industry to redesign gas jet geometry and operating parameters to improve efficiency of powder production processes. The estimated cost savings are in the millions of dollars.

- The thermal imaging sensor developed under a NIST SBIR grant has the potential to improve several materials processing systems by providing temperature and emissivity data in harsh industrial environments at relatively low cost.

- The User Reference Manual written this year provides the necessary documentation for efficient implementation of the NIST-developed process control system.

Project Title: **ELECTRODEPOSITION OF ALLOYS**

Investigators: Gery R. Stafford and Sandra W. Claggett

Technical Description:

Aluminum and its alloys can only be electrodeposited from aprotic nonaqueous solvents or molten salts. Many of these have physical properties, such as high vapor pressure or melting temperature, which make them unsuitable for most technological applications. Organic chloroaluminates, which are obtained when certain anhydrous organic chloride salts such as 1-methyl-3-ethyl-imidazolium chloride (MeEtimCl) are combined with $AlCl_3$, are viable alternatives since they are liquids at room temperature and exhibit negligible vapor pressure. This project, in collaboration with Professor Charles Hussey at the University of Mississippi, focuses on the electrodeposition of aluminum and some of its alloys, from the $AlCl_3$-MeEtimCl room temperature electrolyte.

Technical Objectives:

- Characterize the microstructure, morphology and chemical purity of pure aluminum, electrodeposited at room temperature from a 60:40 and 66:33 $AlCl_3$:MeEtimCl molten salt electrolyte.

- Determine the electrochemical behavior of Ni(II) and Co(II) in this electrolyte and evaluate these modified electrolytes as media for electrodepositing Al-Ni and Al-Co alloys. Compare structure and morphology with deposits made from high temperature, inorganic chloroaluminates.

Anticipated Outcome:

- $AlCl_3$:MeEtimCl assessed as a room temperature alternative media for electrodepositing aluminum and its alloys. Problem areas identified and recommendations made on use of this electrolyte.

Accomplishments for FY 1996:

- Determined that aluminum deposited from both 67:33 and 60:40 $AlCl_3$:MeEtimCl electrolytes is contaminated with Cl^-. The viscosity of this room temperature system is higher than that of the high temperature analogs and electrolyte entrapment appears to be a significant problem.

- Demonstrated that the addition of benzene to the $AlCl_3$-MeEtimCl electrolyte reduced the viscosity and increased the ionic conductivity of the melt. Deposits made from benzene containing electrolytes are Cl^--free and have much improved morphologies over those made from pure melt.

- Determined that Al-Ni and Al-Co alloys can be electrodeposited from benzene-free $AlCl_3$-MeEtimCl electrolytes at potentials positive of the aluminum deposition potential. The mechanism appears to be similar to that observed in the inorganic chloroaluminates where aluminum incorporation is driven by the free energy of alloy formation.

Impact and Technical Highlights:

- Demonstrated that the chemical purity and deposit morphology of aluminum is dramatically improved by the addition of benzene to an $AlCl_3$-MeEtimCl molten salt electrolyte. The elimination of Cl^- from these aluminum electrodeposits is a first step toward making these electrolytes suitable for technological applications.

Outputs:

Publications:

Mitchell, J. A., Pitner, W. R., Hussey, C. L., Stafford, G. R., "Electrodeposition of Cobalt-Aluminum Alloys from the Aluminum Chloride-1-Methyl-3-Ethylimidizolium Chloride Room-Temperature Molten Salt," Tenth International Symposium on Molten Salts, **96**, 189 Meeting Electrochemical Society, Los Angeles, CA (1996).

Mitchell, J. A., Pitner, W. R., Hussey, C. L., Stafford, G. R., "Electrodeposition of Cobalt and Cobalt-Aluminum Alloys from a Room-Temperature Chloroaluminate Molten Salt," J. Electrochem. Soc., 143, 3448 (1996).

Pitner, W. R., Hussey, C. L., Stafford, G. R., "Electrodeposition of Nickel-Aluminum Alloys from the Aluminum Chloride-1-Methyl-3-Ethylimidizolium Chloride Room-Temperature Molten Salt," J. Electrochem. Soc., 143, 130-138 (1996).

Stafford, G. R., and Moffat, T. P., "The Electrochemistry of Titanium in Molten $2AlCl_3$:NaCl," J. Electrochem. Soc., 142, 3288-3296 (1995).

Presentations:

Stafford, G. R. "The Electrodeposition of Corrosion Resistant Aluminum Alloy Coatings," Norwegian University of Science and Technology, Trondheim, Norway, 1996.

Project Title: **ELECTRODEPOSITED STANDARD REFERENCE MATERIALS**

Investigators: Carlos R. Beauchamp, Hilary B. Gates, Christian E. Johnson, David R. Kelley, Jasper L. Mullen, and Shari L. Tobery

Technical Description:

There are two major efforts related to research and production of Electrodeposited Standard Reference Materials.

The first encompasses the production of coating thickness standards for the organic and inorganic coating thickness industry. These standards consist of pre-figured sets of coupons of fine-grained copper with thickness ranging from 2.5 µm to 2 mm, which has been electrodeposited onto low carbon steel substrates. The uniform coatings are then overplated with a thin protective layer of chromium and the coupon's total coating thickness is then certified. They are primarily intended for use in calibrating coating thickness measurement instruments based on the magnetic induction principle which are used for the non-destructive measurement of non-magnetic coatings over magnetic substrates.

The second effort addresses the present lack of a low value hardness standard required by the electronic industry. This industry uses hardness measurements as its quality control criterion for the operating conditions of gold electrolytes during the production of electrodeposits on printed circuit boards. The low load gold microhardness standards being developed at NIST are electrodeposited from a 24 karat gold electrolyte to a minimum deposit thickness of 25 µm. The test area will be 1 cm^2 with the substrate material being metallographically prepared copper with a flash of nickel on the surface acting as a diffusion barrier. They are primarily intended for the calibration of microhardness test instruments.

Technical Objectives:

- Manufacture and certification of Primary Calibration Standards employed in the production of Coating Thickness Standards.

- Prototype development of a gold microhardness standard to be certified at a load of 0.245 Newton (25 gram) using a Knoop indenter and having a microhardness value of 70, with a hardness variation of less than 5% across the test surface.

Anticipated Outcome:

- Completion of the production and certification of the replacement Primary Standards used during the certification process of SRMs 1300's series.

- Polished 24 karat gold electrodeposits produced that comply with the required hardness criteria.

Accomplishments for FY 1996.

- All the necessary data required for the certification of the Primary Standards for the Coating Thickness project has been collected.

- A system was developed for obtaining thick gold deposits (500µm) after evaluating alternate gold electrolytes as candidates for the electrodeposition of the standard.

- The labor intensive step of metallographically preparing the gold surface was eliminated by replacing this step with one involving diamond turning of the deposits.

Impacts and Technical Highlights:

- The completion of the Primary Standards for the SRMs 1300's series allows the renewal of the production of these standards, which had been halted for the last three years. The renewed production will offer customers Secondary Standards with an expanded uncertainty analysis, tighter quality control assurances, and expected reduced uncertainties over the complete measurement protocol.

- The hardness standards will be the first electrodeposited gold microhardness standards developed and will fill the present void for hardness standards at such low values.

Project Title: **ELECTRODEPOSITED CHROMIUM FROM TRIVALENT ELECTROLYTES**

Investigators: Christian E. Johnson and Jasper L. Mullen

Technical Description:

Chromium is widely used as an electrochemically applied coating on metal for wear resistance, to reduce friction, or for a desired appearance. In present commercial electroplating processes, chromium is deposited from electrolytes in which it is in the toxic hexavalent (Cr^{+6}) state. Chromium coatings presently being deposited from non-toxic trivalent electrolytes (Cr^{+3}) are used solely for decorative applications, in the form of thin coatings (1mm-5μm). This project focuses on the characterization of structure and properties of chromium coatings that are electrodeposited from a trivalent electrolyte, and are thick enough (50 μm-250 μm) to be suitable for engineering applications.

Technical Objectives:

- Determine the effects of electrolyte composition and operating parameters on the resulting deposit composition and properties.

- Determine the effects of thermal treatment on structure and properties of the electrodeposited coatings.

- Compare the structure and properties of chromium coatings from trivalent and hexavalent electrolytes.

Anticipated Outcome:

- Processing conditions identified which cause the properties, hardness and wear resistance, of heat-treated chromium deposits from trivalent electrolytes to be equal or superior to deposits from hexavalent electrolytes.

Accomplishments for FY 1996:

- It was determined that the carbon content in chromium deposits from trivalent electrolytes can be varied from 2 weight percent to 3 weight percent when the mole ratio of metal to the carbon containing species in solution is varied from 8/1 to 4/1, respectively.

- The structure of chromium deposits from trivalent electrolytes was determined and found to range from amorphous, as-deposited, to crystalline body-centered-cubic, sometimes having a precipitated second phase of Cr_7C_3 or $Cr_{23}C_6$ depending on carbon content and heat treatment temperature.

- The maximum hardness of heat-treated chromium deposits form trivalent electrolytes was found to be 1800 Knoop compared to 950 Knoop for deposits from hexavalent electrolytes when measured at a load of 0.98 Newton.

- Wear resistance of heat-treated chromium deposits from trivalent electrolytes was found to be two and one-half times better than deposits from hexavalent electrolytes when determined in a lubricated abrasive wear environment.

Impacts and Technical Highlights:

- It has been demonstrated that electrodeposition of chromium by the trivalent process will produce coatings which, with heat treatment, are harder and more wear resistant than coatings deposited by the hexavalent process. This technology provides the metal plating and finishing industry with a non-toxic alternative to Cr^{+6} systems for electroplating thick coatings suitable for engineering applications.

Outputs:

Publications:

Johnson, C. E., "Cost Effective, Nontoxic Process for Depositing Thick, Highly Wear Resistant Chromium Coatings," Materials Technology, Vol. 11, **2**, (1996).

Project Title: **ELECTROCHEMICAL PROCESSING OF NANOSCALE MATERIALS**

Investigator: Thomas P. Moffat

Technical Description:

A variety of nanostructured materials may be synthesized by electrochemical deposition. The research effort at NIST is currently focused in two areas: a) electrodeposition of low dimensional structures, such as multilayers and b) in-situ characterization of the structure and dynamics associated with technically relevant processes that occur at solid/electrolyte interfaces. The role of inorganic and organic adsorbates on the evolution of thin film microstructures and morphologies is of particular interest. In-situ scanning tunneling microscopy (STM) has been shown to be useful to investigate a variety of technologically relevant surface phenomena (e.g., electrodeposition, corrosion, and etching.) The microscope is capable of imaging surface structure and associated real time dynamics at the solid/electrolyte interface over a size scale ranging from the atomic to the micron level.

The commercial success of the electroplating industry stems largely from the remarkable influence of electrolyte additives on the physical properties of the deposited film. Since the chloride ion is a ubiquitous species in most commercial copper electroplating processes, initial STM studies in this project have been conducted with copper in acid chloride electrolytes.

Technical Objectives:

- Develop and optimize methods for producing well-defined metallic thin films via electrochemical processing. Study the evolution of epitaxial structure and morphology during film growth. Apply the knowledge gained to the growth of multilayered materials with a well-defined modulation, orientation, and defect structure.

- Develop the use of scanning probe microscopy (SPM) to characterize the structure and dynamics of the electrode/electrolyte interface. Explore the feasibility of using SPM as a nanostructural synthesis tool via implementation of spatially localized electrochemical reactions.

- Investigate the mechanisms by which organic molecules and inorganic anions influence the microstructural and morphological evolution of electrodeposited metal films.

- Develop an inexpensive means for high resolution patterned electrodeposition.

Anticipated Outcome:

- Guidelines developed for the electrochemical fabrication of strained-layer superlattices that exhibit technically interesting magnetic and mechanical properties.

- Insight provided through SPM studies into the way adsorbates influence microstructural evolution. The commercial success of electroplating technologies stems largely from the remarkable influence of electrolyte additives on the physical properties of the deposited films.

- Technology available for inexpensive, low capital cost, patterned electrodeposition of metals.

Accomplishments for FY 1996:

- Heteroepitaxial deposition of nickel and cobalt on copper was explored in order to develop the capability to produce a variety of Cu-(Ni,Co,Fe) metallic superlattices with rigorously defined modulation orientation. To date, NIST research has yielded a series of well-defined Cu/Ni and Cu/Co multilayers on Cu(100). The mechanical and magnetic properties of these materials are being examined by several other researchers at NIST. It was found that in contrast to the specular films grown on Cu(100), deposition on Cu(111) and Cu(110) leads to roughened surfaces. The origin of this instability is being investigated and studies on the potential use of surfactants to control interfacial morphology have been initiated.

- The capability of using in-situ STM was developed at NIST to study the structure and dynamics of the deposition/dissolution of Cu, and the influence of anion adsorption and Pb underpotential deposition on step dynamics.

- The use of self-assembling monolayers was demonstrated as masks for pattern plating. Micro level features were easily and inexpensively synthesized by contact printing of the SAM's followed by electrodeposition in a typical chemical laboratory environment. In the past year an effort has been initiated to extend this technology into the submicron regime.

Impact and Technical Highlights:

- STM studies at NIST revealed that the surface of the copper electrode is covered by a layer of oxidatively adsorbed chlorine at electrode potentials typically associated with copper deposition and dissolution. The adlattice exerts a strong influence on the adatom binding and activation energy at steps and thus plays a dominant role in determining the evolution of surface morphology.

Outputs:

Publications:

Moffat, T. P., "An In-Situ STM Study of Cu and Ni Electrodeposition,"In-Situ Electron and Tunneling Microscopy of Dynamic Processes, **404**, MRS, Pittsburgh, PA (1996).

Moffat, T. P., "Electrochemical Production of Single Crystal Cu-Ni Strained-Layer Superlattices on Cu(100)," J. Electrochem. Soc., 49, 3767-3770 (1995).

Moffat, T. P., "Electrochemical Synthesis of Strained-Layer Metallic Superlattices," Nanostructured Materials in Electrochemistry, The Electrochemical Society, Inc., Pennington, NJ (1995).

Moffat, T. P., "STM Study of Adsorption and Electrodeposition on Copper in Acid Chloride Media," (in press).

Moffat, T. P., Yang, H., "Patterned Metal Electrodeposition Using an Alkanethiolate Mask," J. Electrochem. Soc., 142, L220-L221 (1995)

Presentations:

Moffat, T. P., "Electrochemical Synthesis of Strained Layer Metallic Superlattices," Gordon Conference on Electrodeposition, Colby-Sawyer College, 1996.

Moffat, T. P., "Electrochemical Synthesis of Strained-Layer Metallic Superlattices," Columbia University, New York, NY, 1996.

Moffat, T. P., "Electrochemical Synthesis of Strained-Layer Metallic Superlattices," The Pennsylvania State University, University Park, PA, 1995.

Moffat, T. P., "An In-Situ STM Study of Anion Adsorption Processes on Cu(100), "Symp. On Scanning Probe Microscopy for Electrode Characterization and Nano-meter-Scale Modification, 188th Electrochemical Society Meeting, Chicago, IL, 1995.

Moffat, T. P., "Electrochemical Synthesis of Cu-Ni Superlattices," Symp. On Molecular Structure of the Solid-Liquid Interface and its Relation to Electrodeposition, 188th Electrochemical Society Meeting, 1995.

Other Outputs in FY 1996:

Presentations:

Handwerker, C. A., "Thermodynamics and Kinetics of Anisotropic Interfaces," Korea Advanced Institute of Science and Technology, Taejon, Korea., October 19,1995.

Handwerker, C. A., "Anisotropic Grain Growth in Ceramics," Korean Ceramic Society Meeting, Seoul National University, Seoul, Korea, October 22, 1995.

Other Outputs in FY 1996:

Presentations:

Hundgerden, [illegible] Institute of Science and Technology [illegible]

[illegible] Grain Growth in [illegible] Seoul National University, Seoul, Korea [illegible]

[illegible]

CONFERENCES AND WORKSHOPS
FY 1996

Mathematics of Thermodynamically Driven Microstructural Evolution
October 30 - November 2, 1995
J. R. Manning, J. A. Simmons
100 attendees

A four-day symposium was organized at the TMS/ASM Materials Week Meeting, Cleveland, Ohio, to bring together mathematicians and materials scientists to focus on the Mathematics of Thermodynamically Driven Microstructural Evolution. The symposium was jointly sponsored by ASM International, The Society of Industrial and Applied Mathematics (SIAM), The Minerals, Metals, and Materials Society (TMS), Defense Advanced Research Projects Agency (DARPA), and NIST.

μMAG Micromagnetic Modeling Workshop
November 8, 1995
M. J. Donahue, R. D. McMichael
60 attendees

A workshop held in conjunction with the 40th Annual Conference on Magnetism and Magnetic Materials in Philadelphia, PA, where the first solutions to a standard micromagnetic problem were presented and compared.

Symposium on the Synthesis and Properties of Metastable Phases and Structures
December 1-5, 1995
R. D. Shull
150 Attendees

A Symposium which formed part of the Fall Meeting of the Materials Research Sociey in Boston, Mass. The symposium summarized the status of the field at the time.

Measurement of Sulfur in Superalloys
March 22, 1996
R. J. Schaefer
21 Attendees

A workshop involving aircraft engine manufacturers, suppliers of superalloy materials and components, analytical instrument manufacturers, and scientists from NASA and NIST in discussions of the role of sulfur in superalloys and the problems of measuring sulfur at very low concentrations.

INTERMAG96
April 9-12, 1996
R. D. McMichael, Organizing Committee
800 Attendees

A major international magnetics conference sponsored by the IEEE to provide a forum for presentation of new developments in applied magnetics, related magnetic phenomena, and information storage techniques.

Solder Interconnect Design Team
April 24-26, 1996, & August 26-30, 1996
J. A. Warren and C. A. Handwerker
25 Attendees

Two workshops of the Solder Interconnect Design Team were held at NIST. The Team is a group of researchers from industry, academia, and government labs which works in an informal collaboration to identify and encourage the development of software for the modeling of the formation and reliability of solder interconnects. In addition to NIST, members include DEC, Ford, Edison Welding Inst., AT&T, Motorola, Heraeus, AMP, Rockwell, Delco, U. of Colorado, Susquehana U., U. Colorado, U. Mass, U. Wisc., U. Loughborough, Greenwich U., Marquette, RPI, and SUNY Binghamton.

Workshop on Hysteresis Modeling and Micromagnetism
May 20-22, 1996
L. H. Bennett, R. D. McMichael

This workshop, held at GWU/VA campus (Ashburn, VA), brought together workers in the field of hysteresis modeling ranging from very physical simulations such as micromagnetic modeling to general mathematical studies of hysteresis.

Third International Conference on Nanostructured Materials
July 7-12, 1996
R.D. Shull
230 attendees

An international conference, held in Kona, Hawaii, summarizing the status in the field in all types of nanostructured materials.

Metallurgy Division Data Strategies
September 1996
C. P. Sturrock, organizer
26 attendees

A NIST Workshop to review the state-of-the-art and practice in delivery of materials data, assess the needs of the technical community, and determine how the Metallurgy Division can help to meet those needs. Representatives from industry, government, and academia attended.

Second International Conference on Corrosion Deformation Interactions
Sept. 24-26, 1996
R. E. Ricker, International Advisory Committee
700+ attendees

The objective of this meeting was to enable the reduction of corrosion failures by corrosion-deformation interactions by bringing together scientists working in this area from all over the world to discuss, debate, and improve our understanding of these failure processes. This meeting was held as part of the EuroCorr '96 which had over 700 attendees from over 30 different nations. EuroCorr is the annual meeting of the European Federation of Corrosion and EuroCorr '96 was hosted by the two French member societies - Centre Français de l'Anticorrosion (CEFRACOR) and Société de Chimie Industrielle (SCI). The Corrosion Deformation Interactions (CDI) Conference was the largest of the 15 sessions held at this meeting and consisted of 47 papers and 59 posters.

PATENTS
FY 1996

Granted

Ultrasonic Computer Tomography Of Stress
Eva Drescher-Krasicka (Metallurgy)
U. S. Patent No. 5,549,003 issued 08/02/96.

Composite Solder
R. B. Clough (Metallurgy), 3 others (non-NIST)
U.S. Patent No. 5,520,752, issued 05/28/96

Pending

Electrochemical Fluidized Bed Coating of Powders
D. S. Lashmore (non-NIST), D. R. Kelley and C. E. Johnson (Metallurgy) and G. L. Beane (non-NIST)
Disclosure Filed

Acid Assisted Cold Welding and Intermetallic Formation and Dental Applications Thereof
D. S. Lashmore (non-NIST), M. P. Dariel (non-NIST), C. E. Johnson (Metallurgy), M. Ratzker (non-NIST), T.Guiseppitti (non-NIST) and F. Eichmiller (non-NIST)
Disclosure Filed

Steel Hardness Measurement System and Method of Using Same
G.E. Hicho and L. J. Swartzendruber (Metallurgy)
Application No. 08/503,263

TECHNICAL/PROFESSIONAL COMMITTEE LEADERSHIP ACTIVITIES

Aluminum Association
S. D. Ridder, Standards Committee

American Academy of Mechanics
R. deWit
R. J. Fields

American Ceramic Society
C. A. Handwerker, Basic Science Division, Chairman

American Electroplaters and Surface Finishers Society
C. E. Johnson, Hard Chromium Committee
Alloy Deposition Committee
Aerospace and Light Metals Committee
Research Board

American Institute of Mining, Metallurgical and Petroleum Engineers (TMS of AIME)
The Minerals, Metals, and Materials Society (TMS)
W. J. Boettinger, Solidification Committee
Bruce Chalmers Award Committee
C. A. Handwerker, J. R. Manning
Electronic Packaging and Interconnection Materials Committee
R. E. Ricker, Chairman, Committee on Corrosion and Environmental Effects
International Ambassador's Advisory Board for TMS International
S. D. Ridder, Solidification Committee
Powder Metallurgy Committee
R.D. Shull, Chairman, The Chemistry and Physics of Materials Committee
Acta Metallurgica Gold Medal and Hume Rothery Award Committee
SMD Awards Committee
EMPMD Division Council
SMD Division Council

American Physical Society
J. A. Warren, March Meeting Organization Committee
Division of Computational Physics, Publicity Committee

American Society of Mechanical Engineers
R. B. Clough, Materials Characterization Committee

American Society for Metals (ASM)
T. Foecke, Organizing Committee, TMS/ASM Fall Meeting
S. D. Ridder, Processing Committee

American Society of Mechanical Engineers
A. Cezairliyan, Thermophysical Properties Committee

American Welding Society
J. H. Smith, Committee B-4 on Mechanical Testing of Welds

ASM International
Alloy Phase Diagram Committee
U. Kattner,

Corrosion and Environmental Effects Committee
R. E. Ricker, Chairman

Journal of Phase Equilibria Commission
W. J. Boettinger
F. W. Gayle

Materials Database Committee
C. P. Sturrock

Washington, DC Chapter
M. R. Stoudt, Treasurer
R. J. Fields
G. E. Hicho
S. R. Low

ASNT
E7:04: Acoustic Emission
J. A. Simmons

ASTM
B2: Nonferrous Metals and Alloys
S. D. Ridder

B7: Light Metals
W. J. Boettinger

B8: Metallic and Inorganic Coatings
C. E. Johnson

B8.10: General Test Methods
C. E. Johnson

B8.10.03: Microhardness Testing
C. E. Johnson, Liaison to E04

B9: Metal Powders and Metal Powder Products
J. R. Manning

C26: Nuclear Waste
A. C. Fraker

D30: High Modulus Fibers and Their Composites
M. R. Stoudt

DS-56D: Metal and Alloys in the Unified Numbering System
G. E. Hicho

E4: Metallography
E4.05: Microhardness
C. E. Johnson

E7: Nondestructive Testing
L. Mordfin, Executive Subcommittee

E7.04: Acoustic Emission
R. B. Clough
J. A. Simmons

E7.91: USA Committee for ISO TC 135
L. Mordfin, Chairman

E7.10.04: Infrared NDT Methods
L. Mordfin, Chairman

E8: Fatigue and Fracture
R. deWit
S. R. Low
R. J. Fields
G. E. Hicho
J. H. Smith
J. A. Simmons

E8.02: Fractography & Associated Microstructure
G. E. Hicho
R. J. Fields

E8.04: Environmental Cracking
R. E. Ricker

E8.08: Elastic-Plastic
G. E. Hicho

E28: Mechanical Testing
L. Mordfin, Executive Subcommittee
R. J. Fields
S. R. Low

E28.04: Uniaxial Testing
S. R. Low

E28.06: Indentation Hardness Testing
S. R. Low
J. H. Smith

E28.06.03: Macro-Rockwell Hardness
S. R. Low, Chairman

E28.06.07: Traceable Hardness Standards
S. R. Low, Chairman
J. H. Smith

E28.12: Accreditation of Mechanical Testing Labs
S. R. Low

E28.13: Residual Stress Measurement
L. Mordfin, Chairman

E28.90.02: Proficiency Testing
S. R. Low

E28.94: USA Committee for ISO TC 164
L. Mordfin

E28.94.06: Terminology and Symbols for Mechanical Testing
L. Mordfin, Chairman

E37: Thermal Measurements
A. Cezairliyan

E49: Computerization of Materials and Chemical Property Data
C. P. Sturrock

F4: Medical and Surgical Materials and Devices
A. C. Fraker

F16: Fasteners
S. R. Low

G1: Corrosion of Metals
R. E. Ricker
M. R. Stoudt

G1.06: SCC and Corrosion Fatigue
R. E. Ricker
M. R. Stoudt

G1.11: Electrochemical Measurements in Corrosion
R. E. Ricker

Blue Ribbon Panel for Presidential Faculty Fellows
R. J. Fields, Member

Committee on Data for Science and Technology of the International Council of Scientific Unions - CODATA
C. P. Sturrock

Compressed Gas Association
J. H. Smith, Cylinder Specification Committee
Technical and Safety Standards Council (TASS)

Electrochemical Society
T. P. Moffat, Chairman of the National Capital Section - Local Chapter
Secretary-Treasurer of the Electrodeposition Division

Fifth International Conference on the Fundamentals of Fracture
T. Foecke, Co-chair, Local Organizing Committee

Gordon Research Conferences
G. R. Stafford, Vice-Chairman of Conference on Electrodeposition
C. A. Handwerker, Vice-Chairwoman of Conference on Solid State Studies in Ceramics

High Temperature Science Journal
A. Cezairliyan, Editorial Board Member

IEEE Magnetics Society, DC Metro Area Chapter
R. D. McMichael, Vice President

Institute for Mechanics and Materials Summer School on Mechanics and Materials
T. Foecke, Chair, Organizing Committee

International Advisory Committee for the 14th World Conference on Non-destructive Testing
R. B. Clough

International Advisory Committee for the International Conference on Nanostructured Materials
R.D. Shull, Secretary/Treasurer

International Committee on Nanostructured Materials
R. D. Shull, Vice Chairman

International Commission on Standardizing Thermophysical Properties Measurement Methods
A. Cezairliyan, Member

International Community for Composites Engineering
E. Drescher-Krasicka, International Advisory Committee
Program Organizing Committee

International Conference on Internal Friction and Ultrasonic Attenuation
E. Drescher-Krasicka, Session Chair

International Conference on Nanostructured Materials, Third
R. D. Shull, Principal Organizer

International Journal of Thermophysics
A. Cezairliyan, Co-founding Editor-in-Chief

International Organizing Committee for the Third International Conference on Nanostructured Materials
R.D. Shull, Chairman

International Organization for Standardization

TC58.SC3: Cylinder Design
J. H. Smith, Delegate

TC107: Metallic and Other Non-Organic Coatings
C. E. Johnson, Delegate

TC107.02: Methods of Inspection and Co-Ordination of Test Methods
C. E. Johnson, Delegate

TC107.02: Working Group 1- Microhardness
C. E. Johnson, Chairman

TC107.03: Electrodeposited Coatings and Related Finishes
C. E. Johnson, Delegate

TC135: Nondestructive Testing
L. Mordfin, Delegate

TC135.03: Acoustical NDT Methods
L. Mordfin, Secretary

TC164.03 Hardness Testing
S. R. Low, Delegate

International Organizing Committee of the Subsecond Thermophysics Workshops
A. Cezairliyan, Member

International Organizing Committee of the European Thermophysical Properties Conferences
A. Cezairliyan

International Symposium on Molten Salts, 10th
G. R. Stafford, Co-organizer

International Thermal Expansion Conferences
A. Cezairliyan, Governing Board

International Thermophysics Congress
A. Cezairliyan, Chairman

IPC Subcommittee on Solderability
C. A. Handwerker, Alternate Final Finishes Task Group
Component and Wire Solderability Specification Task Group
Steam Aging Task Group
Wetting Balance Task Group

Journal of the American Ceramic Society
C. A. Handwerker, Associate Editor

Journal of Crystal Growth
S. R. Coriell, Associate Editor

Journal of High Temperatures - Pressures
A. Cezairliyan, Editorial Board Member

Journal of Nanostructured Materials
R. D. Shull, Special Guest Editor

Langmuir, ACS Surface Science Journal
W. F. Egelhoff, Editorial Board

Materials Research Society
R. D. Shull, Organizer of Symposium on Properties of Nanostructured Materials

Metallurgical and Materials Transactions Board of Review
F. W. Gayle
R. E. Ricker
G. R. Stafford

Journal of Nanostructured Materials
R.D. Shull, Associate Editor

Micromagnetic Modeling Activity Group (μMAG).
R. D. Michael, Chair

MIL Handbook 17 on Metal Matrix Composites
A. C. Fraker

NACE International

T.3U: Computers in Corrosion Control
C. P. Sturrock

T.3U2: Artificial Intelligence Applications in Corrosion
C. P. Sturrock

NACE-NIST Corrosion Data Program
Joint Coordination Committee
R. E. Ricker
C. P. Sturrock

NSF Institute for Mechanics and Materials
T. Foecke, Young Investigator Advisory Committee

Physical Review Letters
W. F. Egelhoff, Divisional Associate Editor

Sigma Xi
R. E. Ricker, Nominating Committee
R.D. Shull, President Elect: NIST Chapter

INDUSTRIAL/ACADEMIC INTERACTIONS

INDUSTRY

1. Aerojet General Inc.

   John Smith collaborated with Aerojet on the development of carbon-fiber-reinforced composite cylinders to be used for the transportation of high pressure gasses.

2.. Allied Signal, Inc.

   W. J. Boettinger has collaborated with scientists at Allied Signal in the modeling of porosity formation in castings, providing them with data on the solidification characteristics of aluminum alloys.

3. American Iron and Steel Institute (AISI)

   G. E. Hicho, L. J. Swartzendruber, E. N. Pugh and R. E. Ricker are currently interacting with AISI under a CRADA (CN-987) to develop on-line methods for the determination of the mechanical properties of sheet steel using magnetic methods.

4. Amorphous Technology International (ATI)

   ATI approached the NIST Materials Performance Group for help with the evaluation of the corrosion resistance of the bulk amorphous alloys that it is developing. To protect ATI's intellectual property rights to any subsequent alloy which may be developed as a result of this collaboration, a CRADA between NIST and ATI was suggested. NIST and ATI researchers prepared a CRADA for this purpose which is being evaluated by ATI's management.

5. AT&T

   L. Bendersky collaborated with Dr. T. Sigrist on structural determination of compounds in the $BaO:Fe_2O_3:TiO_2$ system.

6. Bethlehem Steel

   Tim Foecke is cooperating with Harold Reesmsnyder at the Bethlehem Steel Homer Laboratories on a project underwritten by the Society of Naval Architects and Marine Engineers and The Discovery Channel looking into the metallurgical aspects of the sinking of the RMS Titanic.

7. BOC Gases

John Smith has been working with BOC Gases to evaluate new methods for the use of ultrasonic testing to inspect high pressure steel and aluminum gas cylinders.

8. BP and Praxair, Inc.

E. Drescher-Krasicka has been working with BP and Praxair to detect and map residual stresses in ceramic membranes under an ATP intramural program.

9. Consortium on Plastic Encapsulated Packaging

E. Drescher-Krasicka is working with Sandia National Labs on the calibration of the Scanning Acoustic Imaging of Stress technique by use of plastic encapsulated chips with built-in stress sensors. This program involves close work with DARPA.

10. Compressed Gas Association (CGA)

The Compressed Gas Association is an industry association responsible for the development of safety standards for the transportation and use of pressurized gases. John Smith has continued collaboration with CGA to develop specifications and standards for the construction and inspection of pressurized gas cylinders.

11. Containerless Research Inc.

A. Cezairliyan is collaborating with the scientists at Containerless Research, Inc. in Chicago to develop and verify a unique optical technique for measuring normal spectral emissivity of high temperature materials.

12. Copper Development Association (CDA)

R. B. Clough is working with members of the CDA determining the mechanical properties of new lead-free solders used in piping applications.

13. Crucible Compaction Metals

Crucible Compaction Metals and NIST have signed a CRADA concerning improved nitrogenated alloys produced by atomization, including investigations of approaches patented by NIST (F. Biancaniello, S. Ridder, and G. Janowski).

14. Crucible Materials Corporation

The NIST SiGMA facility for producing metal powder by atomization processes has been the focal point of pioneering work in advanced sensor development and expert system based control. Scientists from Crucible Materials Corporation interacting with NIST in this work are applying these developments to provide improved process control in one of Crucible's industrial atomizers.

15. Crucible Research Center (Crucible)

Crucible approached NIST's Materials Performance Group for help with electrochemical measurement of the corrosion resistance of the new alloys they are developing. A NIST scientist, Dr. R. E. Ricker, visited their laboratory to review their experimental technique and to help them understand the results. Further collaborations have been proposed for FY97.

16. David L. Ellis Co., Inc.

S. Low and D. Pitchure conducted hardness tests on D. L. Ellis test blocks to assist in the company's effort to improve the hardness uniformity of their test blocks.

17. Dentsply

Gery R. Stafford, Christian E. Johnson and David R. Kelley are engaged in a CRADA for joint research on development of dental restoratives as replacements for silver amalgams.

18. DuPont

E. Drescher-Krasicka has a long term cooperation with DuPont studying the stress tomography in thick sections of Inconel pipes containing welds. This research involves close work with Dr. Henry Prask of the Reactor Radiation Division who provides neutron diffraction data needed for calibration of stress maps obtained for different cross sections of the welds.

19. Faratech

Christian Johnson is engaged in informal cooperation to further develop and commercialize trivalent chromium electrolytes as a replacement for toxic hexavalent electrolytes.

20. Fluxtrol Manufacturing

A CRADA has been signed for NIST scientists (F. Biancaniello, L. Swartzendruber and S. Ridder) to collaborate with Fluxtrol Manufacturing, Inc. in investigating improved materials for magnetic confinement and energy conservation.

21. Electric Power Research Institute (EPRI)

With EPRI sponsorship, the NACE-NIST Corrosion Data Program works with representatives of EPRI member companies and their consultants to develop computer programs which will assist electric utility engineers in avoiding critical equipment failures. This program focuses on selected applications in power plant condensers, steam generators, flue gas desulfurization systems and service waters.

22. Exotec, Inc.

Christian Johnson is collaborating with Sidney Wiser to investigate the feasibility of using a laser scanner to determine crystallographic morphology of Zn and Zn alloy coatings.

23. Federal Railroad Administration

G. E. Hicho continues to interact with scientists and engineers in the railroad industry based on past collaborative research with the Federal Railroad Administration on safety of future tank cars for carrying hazardous materials.

24. FDA and Telectronics Pacing Systems, Inc.

Christian Johnson is cooperating with the FDA to determine the failure mode in composite electroplated flex solder tabs in recalled pulse generators (pacemakers).

25. FIBA Inc.

J. Smith collaborated with FIBA to develop and evaluate new methods for the use of ultrasonic testing to inspect high pressure steel and aluminum gas cylinders.

26. General Electric Aircraft Engines

R.J. Schaefer is working with GE Aircraft Engines and with members of the Analytical Chemistry Division on a program to study the measurement of low levels of sulfur in superalloys.

27. High Temperature Fatigue Resistant Solder Consortium, National Center for Manufacturing Sciences (NCMS)

NIST is working to develop solders needed by the automotive and avionics industries for high temperature applications through a collaboration with members of the High Temperature Fatigue Resistant Solder Consortium, organized through the National Center for Manufacturing Sciences (NCMS). F.W. Gayle, Ursula Kattner, J. Adams, and L. Smith are working with other members, including Ford, Delco, Allied Signal, Johnson Manufacturing, Indium Corp., Heraeus Cermalloy, Edison Welding Institute, US Navy EMPF, RPI, and Ames Laboratories.

28. Hochtechnologie- Materialien (HTM AG), Biel, Switzerland

S. Low and D. Pitchure collaborated with HTM AG by conducting hardness measurements on test blocks which were produced by HIPing metal powders. This collaboration was to determine the feasibility of producing test blocks by consolidating metal powders.

29. Howmet, Battelle, and EPRI

Collaborative efforts have been established by D. Josell with Howmet, Battelle, and EPRI to determine the thermal transport properties of thermal barrier coatings at temperatures relevant to industrial applications. Results obtained on industry-provided material have been provided to industrial partners.

30. Howmet Corporation

Members of the Metallurgical Processing Group are collaborating with scientists at Howmet Corporation on several aspects of the casting of aerospace alloys. NIST scientists develop theoretical models of the casting process and carry out measurements of alloy microstructure and properties, while Howmet makes special instrumented castings, analyzes heat flow, and provides carefully selected samples for evaluation by NIST.

31. Industrial Materials Institute (IMI) of Canada

L. J. Swartzendruber and G. E. Hicho are cooperating with the IMI in the development of methods for on-line determination of the mechanical properties of sheet steel. IMI is using laser ultrasonics and NIST is using magnetic measurements sharing a common set of test materials and data.

32. Instituto di Metrologia "G. Colonetti" (IMGC), Turin, Italy

S. Low and D. Pitchure collaborated with IMGC by having them participate in a NIST organized Rockwell hardness intercomparison to examine differences in the standardizing hardness testers maintained at the national hardness standardization laboratories around the world. NIST also measured the hardness of standardized hardness test blocks from IMGC as part of a bilateral intercomparison between the national hardness standardization laboratories of Italy and the U.S.

33. Instituto Nacional de Metrologia, Normalizacão e Qualidade Industrial (INMETRO), Rio de Janeiro, Brazil

Sam Low collaborated with INMETRO by visiting their standardization laboratories, and providing advice and assistance in developing a national hardness standardization laboratory in Brazil.

34. International Organization for Standardization (ISO)

Through the ISO technical committee (TC 58) on Gas Cylinders, an extensive research and testing program was carried out to develop tests to evaluate the fracture resistance of high strength steel cylinders and to develop fracture resistance acceptance criteria. This program involved testing of high strength steel cylinders by all of the cylinder manufacturers in the world. Cylinder manufacturers from the United States, United Kingdom, France, Germany, Austria, Sweden, and Japan conducted nearly 1000 flawed burst tests as part of this program. The tests were carried out according to the plan developed by NIST. The test results were compiled and analyzed by John Smith of NIST.

35. Johnson Wax, S.C. Johnson & Sons, Inc.

In 1993, S. W. Tait of Johnson Wax approached the Materials Performance Group for advice on the detection and measurement of corrosion inside aerosol containers as a result of chemical attack by the product inside the container. When Quantum Magnetics approached the Materials Performance Group in 1996 for potential applications of its new technology, the Materials Performance Group contacted S. W. Tait of Johnson Wax and set up a collaboration between Johnson Wax and Quantum Magnetics to evaluate Quantum Magnetics measurement technique as a means for the detection and evaluation of corrosion in sealed aerosol containers.

36. Libby-Owens-Ford

E. Drescher-Krasicka is working with the Libby-Owens-Ford Company to establish scanning acoustic techniques for localization and evaluation of the distribution of residual stresses in glass laminates.

37. Louis Vuitton Moët Hennessy, Inc. (LVMH)

LVMH makes cosmetics in the U.S. and was interested in finding out if hydrostatic compaction was superior to die compaction for cosmetic powder compacts. Working with Francis Massie of LVMH, R. J. Fields was able to show that die compaction was the better approach to strong, yet usable compacts for the powders of interest to LVMH. Hydrostatically compacted powders resulted in brittle compacts that spontaneously fractured into many pieces on removal from the isostatic pressing container.

38. Luxfer USA Inc.

J. Smith conducted an evaluation of eddy current test methods for detecting cracks in aluminum cylinders due to sustained load cracking (SLC) for Luxfer.

39. Materialprüfungsamt Nordrhein-Westfalen (MPA NRW), Dortmund, Germany

S. Low and D. Pitchure collaborated with MPA NRW by having them participate in a NIST organized Rockwell hardness intercomparison to examine differences in the standardizing hardness testers maintained at the national hardness standardization laboratories around the world. NIST also collaborated with MPA NRW by assisting in the planning of a new international intercomparison to establish worldwide standardization of Rockwell hardness.

40. Material Resources International

F. S. Biancaniello is collaborating with Dr. R. Smith of Materials Resources International on advanced metals processing techniques.

41. Motorola/DARPA MRAM Project

W. F. Egelhoff is a partner with Motorola in a DARPA-funded program to develop non-volatile random access memory chips based on the giant magnetoresistance effect.

42. NACE International (formerly National Association of Corrosion Engineers)

NACE is a professional society whose membership is concerned with the avoidance of corrosion failures. NACE and NIST joined together in 1982 to create the NACE-NIST Corrosion Data program. The objective of this program is to use the latest advances in information science to provide industry with convenient and reliable information on corrosion and corrosion prevention. For this program, NACE provides a full time Research Associate who works with NIST scientists to collect, evaluate, and develop computer databases, expert systems and hybrid systems for the dissemination of information on materials performance and corrosion failure avoidance. NACE distributes the computer software developed by this program to industry.

43. NASA - Kennedy Space Center

Christian Johnson is collaborating and providing support for NASA on various issues of electroplating, specifically the coating of laser positioning mirrors.

44. National Center for Manufacturing Sciences (NCMS), Lead-Free Solder Consortium

A broad-based collaboration has been established between the National Center for Manufacturing Sciences (NCMS) and NIST (C. A. Handwerker, U. R. Kattner, R. J. Fields, and L. C. Smith) to evaluate environmentally safe alternatives for lead-based solder alloys used for electrical/electronic assembly interconnections. In addition to NIST, members include Lucent Technologies, Rockwell International, Texas Instruments, Ford, GM-Hughes, GM-Delco, United Technologies Corporation, RPI, EMPF and Sandia National Laboratories.

45. National Center for Manufacturing Sciences (NCMS)

F. Biancaniello and S. D. Ridder of the NIST Metals Processing Laboratory provided a series of special alloy samples to industrial members of the NCMS Lead-Free Solder Consortium to aid in practical testing of new lead-free solder alloys.

46. NKK

L. Bendersky is involved in a cooperative research program with NKK, Japan (Dr. S. Mitao) to study the microstructural stability of Gamma aluminides materials having a high density of interfaces.

47. National Institute of Metrology (NIM), Beijing, China

S. Low and D. Pitchure collaborated with NIM by having them participate in a NIST organized Rockwell hardness intercomparison to examine differences in the standardizing hardness testers maintained at the national hardness standardization laboratories around the world.

48. National Research Laboratory of Metrology (NRLM), Tsukuba, Japan

S. Low and D. Pitchure collaborated with NRLM by having them participate in a NIST organized Rockwell hardness intercomparison to examine differences in the standardizing hardness testers maintained at the national hardness standardization laboratories around the world. NIST also conducted an intercomparison of the performance of NIST and NRLM Rockwell diamond indenters in an effort to determine the influence of the diamond shape on hardness measurements.

49. National Storage Industry Consortium (NSIC)

Scientist in the Magnetic Materials Group are collaborating with this ATP-funded consortium to develop giant magnetoresistive materials for read-heads in a new generation of ultrahigh density hard-disk drives.

50. Naval Surface Warfare Center

R. B. Clough is collaborating with Patricia Carter of the Nonlinear Dynamics and Wavelets Group at the Naval Surface Warfare Center (now located in Dahlgren, VA) on developing wavelet techniques for improved detection of acoustic emission signals from very small cracks (< 20 microns in size).

51. Nonvolatile Electronics, Inc.

W. Egelhoff is collaborating with this Minneapolis-based company to develop improved giant magnetoresistive (GMR) materials. This company is presently the only company in the world with a GMR-based product on the market (a sensor in automotive antilock brake systems)

52. Norris Cylinder Corporation

John Smith worked with a NIST guest worker sponsored by the technical staff at Norris Cylinder Corporation. The purpose was to develop improved specifications for the design and construction of pressurized gas containers.

53. Northrop Grumman STC

S. R. Coriell is collaborating with N. B. Singh and colleagues on the modelling of convection and interface stability during the directional solidification of lead bromide doped with silver bromide, a material with nonlinear optical applications.

54. Norton Corporation

Christian Johnson provided support for Norton Corporation in the application of composite and wear resistant coatings for complex shaped mandrels used in the production of critical dimension mirrors.

55. Pratt & Whitney

R. J. Schaefer is collaborating with scientists at Pratt & Whitney to develop a classification scheme for defects in single crystal castings and to identify the processes responsible for generation of the defects.

56. Praxair Inc.

John Smith worked with Praxair to develop tests to evaluate the fracture resistance of high strength steel cylinders and to develop fracture resistance acceptance criteria. Results of this work are to be published in the proceedings of an ASME conference.

57. Quantum Magnetics, Inc.

Through the NIST SBIR Program, Quantum Magnetics developed a magnetic technique for detecting and/or measuring active corrosion in closed systems where it is hidden from view or detection by other techniques. Quantum Magnetics approached the Materials Performance Group for their advice on potential commercial applications. The Materials Performance Group suggested that Quantum Magnetics and Johnson Wax work together to evaluate this technique as a possible technique for the detection and evaluation of corrosion in sealed aerosol containers. The Materials Performance Group contacted both parties and established this collaboration

58. Sandusky International, Inc.

M. R. Stoudt and R. E. Ricker are collaborating with Sandusky International to characterize and evaluate corrosion fatigue related failure in duplex stainless steels used in the pulp paper industry.

59. Science Applications International Corporation (SAIC)

A. C. Fraker collaborated with Dr. Richard Wulleart in two areas: (1) a database that provides information on corrosion of materials and effects of environments on corrosion in high-level waste storage tanks and (2) materials used for conductive ceramic or metal electrodes and other aspects of electrochemistry involved in the electrolysis of water.

60. Solder Jet Consortium and MicroFab Technologies

F.W. Gayle and C.A. Handwerker are working with MicroFab Technologies in their development of solder-jet printer devices. This program involves close work with the Solder Jet Consortium (Motorola, Delco, AMP, Universal Instruments, Eastman Kodak, and MicroFab) to measure contamination and wettability of jetted solders.

61. Sonix, Inc.

E. Drescher-Krasicka has a CRADA with Sonix, Inc., Springfield, Va., on residual stress detection on electronic packaging. This research will lead to routine measurements of stresses in plastic encapsulated packages.

62. Texas Instruments

E. Drescher-Krasicka is working with Tom Moore of Texas Instruments, Dallas, Texas, to measure stresses in plastic packages for microelectronics.

63. Top Die Casting Company

R. J. Schaefer and R. D. Jiggetts have worked with Top Die Casting Company in studies of the distribution and connectivity of porosity in aluminum die castings

64. UES, Inc.

W. J. Boettinger of the Metallurgical Processing Group has provided UES with models for microstructure development, which are being incorporated in their ProCAST™ software for modeling of metal casting. The enhanced software will be available to members of the Consortium on Casting of Aerospace Alloys.

65. U.S. Automobile Manufacturing Partnership (USAMP)

USAMP's project on low cost technology for producing particle reinforced aluminum automobile components is directed at reducing the curb side weight of the next generation of vehicles. The participants include all U.S. auto manufacturers, their suppliers of powder metallurgical parts, machine tools manufacturers, and several national laboratories and universities. R. J. Fields and R. B. Clough of NIST work with Cambridge University to provide validated process modeling for this project. The processes currently being considered are press-and-sinter (including transient liquid sintering) and powder forging, both for particle reinforced powders.

66. U.S. Department of Transportation (DOT)

John Smith is working with the DOT under an interagency contract to develop new technology and safety standards for compressed gas cylinders. The work involves evaluation of new materials for the construction of compressed gas cylinders and new test methods for evaluating the structural integrity of compressed gas cylinders.

67. U.S. Hardness Industry

S. Low is acting as liaison between the National Voluntary Laboratory Accreditation Program (NVLAP) at NIST and the primary U.S. companies that sell and/or manufacture hardness calibration test blocks. NVLAP is seeking assistance from the hardness industry in the development of the requirements for accrediting laboratories that conduct calibrations of hardness test blocks, indenters, and hardness test machines.

68. Weirton and LTV Steel Companies

G. E. Hicho is working with Weirton and LTV Steel research metallurgists at Weirton, WV and Cleveland, OH, respectively, on the on-line measurement of the mechanical properties of fast moving sheet metals.

69. Wilson Instruments

R. Fields and S. Low collaborated with Wilson Instruments to develop improved and more uniform hardness test blocks. This project is a cooperative project between Wilson Instruments and the Massachusetts Institute of Technology (MIT) and is being conducted under the Manufacturing Leaders Program at MIT.

70. Wyman-Gordon, Inc.

Wyman-Gordon is trying to produce extremely large superalloy disks for land-based turbines using a new, incremental forging technique. T. Foecke and R. J. Fields are collaborating with Dr. M. Morra of Wyman-Gordon to identify the precipitate size and distribution associated with superior, low-cycle fatigue resistance.

71. Xerox Corporation

R.D. Shull is collaborating with R.F. Ziolo, Xerox Webster Research Laboratory, to evaluate magnetic ferrofluids prepared by Xerox corporation.

INDUSTRY/UNIVERSITY

1. ALCOA, Washington State University

   Richard Fields, Roland deWit, and Robb Thomson are collaborating with ALCOA and Washington State University to develop a better fundamental understanding of the relationships between microstructure and performance during deformation processing.

2. Automotive Sheet Metal Forming Consortium

   Environmental Research Institute of Michigan (ERIM) is managing a consortium consisting of Chrysler, Ford, General Motors, the Budd Co., ALCOA, US Steel, Northwestern University, Ohio State University, University of Michigan, Sandia National Laboratory, and Lawrence Livermore National Laboratory. The goal of this consortium is to accurately predict springback in automotive stampings as part of the ATP focused program, Motor Vehicle Manufacturing Technology. R. J. Fields, R. deWit, and R. Thomson are working with H. Zbib, J. Hirth, and L. Levine and this consortium to provide additional understanding and prediction capability at the microstructural level, and to assist in the development of finite element codes for predicting springback. From this interaction, the NIST participants hope to gain insight into industrial needs in sheet metal formability and measurement of formability.

3. Metalogic, Materials Technology Institute (MTI), Katholieke Universiteit, Leuven, Belgium (KUL)

   C. P. Sturrock and researchers at KUL/Metalogic are collaborating to develop an expert system that predicts the performance of stainless steels in aqueous environments commonly found in industry. Advanced information technologies such as Bayesian inference networks, fuzzy logic and case-based reasoning are being combined with mathematical models of corrosion phenomena to provide as assessment of the overall suitability of candidate alloys and the likelihood and severity of various corrosion morphologies. This research is being sponsored by MTI.

4 Micromagnetic Modeling Activity Group (μMAG)

   With the sponsorship of NIST Center for Theoretical and Computational Materials Science, R. D. McMichael, L. H. Bennett, and M. J. Donahue, along with Jim Blue (CAML) and John Oti (EEEL) have initiated a Micromagnetic Modeling Activity Group (μMAG). With participants from industry and from academic institutions, μMAG has one project to pose and solve standard problems in micromagnetics and another project to produce portable publicly available micromagnetic computer code.

5. NIST Metals Processing Laboratory

Facilities and personnel in the NIST Metals Processing Laboratory (F. S. Biancaniello, R. D. Jiggetts, S. D. Ridder, R. J. Schaefer) aid in preparation of special samples for various materials characterization studies by the scientific and technological community. University and industry scientists can assist in sample processing for independent or collaborative research projects involving alloy development, rapid solidification and particulate consolidation. During the past year, investigators from Crucible Materials Corp., Fluxtrol Manufacturing, Ford, NCMS, Sandia National Laboratories, GM-Delco, Rennselaer Polytechnic Institute, Stratonics, Inc, and the University of Idaho have interacted in this program.

6. Solder Design Working Group

Jim Warren and Carol Handwerker head the Solder Interconnect Design Team, a group of researchers from industry, academia, and government labs which works through informal collaboration to identify and encourage the development of software for the modeling of the manufacturing and reliability of solder interconnects. The consortium is composed of NIST, DEC, Ford, Edison Welding Inst., Lucent Technologies, Motorola, Heraeus, AMP, Rockwell, Delco, U. of Colorado, Susquehanna U., U. Colorado, U. Mass., U Wisc., U. Loughborough, Greenwich U, Lehigh U., Marquettue, RPI, and SUNY Binghamton.

UNIVERSITIES

1. ACCESS e. V., Aachen, Germany

   Cellular growth during the initial transient in directional solidification of transparent materials is being studied by Gerhard Zimmermann of ACCESS. S. R. Coriell and colleagues are carrying out linear stability calculations and modelling of the directional solidification process.

2. Auburn University

   A. Cezairliyan of the Metallurgical Processing Group is working with the Space Power Institute at Auburn University to measure important thermophysical properties of nickel-based superalloys. These properties are needed for modeling of the casting of these materials.

3. BIRL/Northwestern University and Chicago Faucet Co..

   Christian Johnson is working with Northwestern University in studies of the viability of replacing hexavalent Cr electrolytes with environmentally superior trivalent Cr electrolytes.

4. Bologna University

   J. Bonevich is collaborating with Prof. G. Pozzi on the modeling of holographic observations in superconducting materials.

5. California Institute of Technology

   R. D. Shull is collatorating with B. Fultz to investigate ball-milled nanocrystalline compounds for soft magnetic properties.

6. Cambridge University, Cambridge, UK

   R. Fields and R. Clough of the Materials Performance Group are working with M. Ashby and N. Fleck of Cambridge University to develop and validate powder forging models for metal matrix composites.

7. Carnegie Mellon University

   J. Bonevich is collaborating with J. Dooley to image magnetic contrast in magnetic sensor materials such as Terfenol.

8. Darmstadt University, Germany

R.D. Shull is collaborating with H. Hahn to investigate the preparation of magnetic nanocomposites via vapor condensation routes.

9. Ecole Polytechnique Federale Lausanne, Switzerland

Research completed during 1996 by W. J. Boettinger in collaboration with M. Rappaz permits the modelling of equiaxed dendritic solidification for multicomponent alloys.

10. George Washington University

R.D. Shull is collaborating with L.H. Bennett and M. Pardavi-Horvath to investigate the magneto-optic properties of magnetic nanocomposites. R. D. McMichael is also collaborating with E. DellaTorre on micromagnetics.

11. George Washington University

G. E. Hicho is Associate Professorial Lecturer in Engineering in the Civil, Mechanical, and Environmental Engineering Department.

12. George Washington University

A. C. Fraker participated in meetings with George Washington University set up by C. M. Gilmore to establish studies in the biomaterials field, including materials used in aneurysm clips and spinal devices.

13. Georgia Institute of Technology

R.D. Shull is collaborating with Z.L. Wang in the study of magnetic nanotubes.

14. Howard University

A. C. Fraker worked with Dr. A. A. Chohayeb, Dental School, Howard University, studying the effects of coupled dissimilar metals on corrosion behavior in dental implants and restorations.

15. Iowa State University and Ames Lab

F. S. Biancaniello and S. D. Ridder have collaborated with Professor Iver Anderson of Iowa State University on powder handling techniques and atomization research.

16. Italian Metrology Institute

A. Cezairliyan is a participant in the US-Italy Joint Project on Metrological Research and is collaborating with the scientists at the Italian Metrology Institute on a project related to thermophysical properties of high-temperature materials.

17. The Johns Hopkins University

Tim Foecke is interacting with Professor Tim Weihs of the Department of Materials Science and Engineering on the thermomechanical behavior of Nb/Nb5Si3 microlaminated materials for the Air Force.

18. The Johns Hopkins University

J. Bonevich is collaborating with Prof. P. Searson on the characterization of Cu/Ni/Co multilayer nanowires formed by electroplating.

19. Katholieke Universiteit, Leuven, Belgium

C. P. Sturrock is collaborating with Professor W. F. Bogaerts to advance the state-of-the-art in applications of artificial intelligence to problems in corrosion science and engineering.

20.. National Research Laboratory of Metrology (Japan)

A. Cezairliyan is a participant in cooperative research in thermophysics between NIST and NRLM of Japan.

21. Northwestern University, Evanston, IL

Tim Foecke is interacting with Prof. Scott Barnett of the Department of Materials Science and Engineering on the mechanical behavior of NbNiMo single crystal nanolaminates.

22. Norwegian University of Science and Technology

Gery R. Stafford is collaborating with G. Martin Haarberg on the electro-deposition of Al-refractory metal alloys.

23. Ohio State University, Columbus, OH

Tim Foecke is interacting with Professor Peter Anderson of the Department of Materials Science and Engineering on modeling the mechanical behavior of nanolaminated materials.

24. Purdue University

A. Cezairliyan is a consultant to the Thermophysical Properties Research Center - CINDAS at Purdue University.

25. Southampton University (UK)

W. J. Boettinger is collaborating with Dr. Adam Wheeler, Southampton Univ., U.K., on phase-field methods to treat solidification problems. The alloy theory developed previously was applied to solute trapping in alloys at high solidification rates.

26. Technion, Israel

L. Bendersky collaborated with Prof. Brandon and I. Levin on the crystallography of metastable polymorphs in alumina.

27. Universite Libre de Bruxelles, Belgium

S. R. Coriell and colleagues are collaborating with Dr. S. Van Vaerenbergh and Professor J. C. Legros on the effect of Soret diffusion on various alloy solidification processes. The effect of the temperature dependence of diffusion on the onset of morphological stability during the directional soldification of a binary alloy has been calculated.

28. University of Arizona

W. J. Boettinger is working with the University of Arizona to develop models describing fluid flow, macrosegregation, and porosity during the solidification of metal castings.

29. University of California, Berkeley

E. Drescher-Krasicka is working with Prof. Claudia Ostertag in the Department of Engineering of the University of California, Berkeley. They are presently working on the evaluation and distribution of residual stresses in samples containing welds which cracked as a result of a recent California earthquake.

30. University of Florida

A collaborative effort between Professor Abbaschian (University of Florida) and S. R. Coriell on the in-situ monitoring of crystal growth processes using the MEPHISTO furnace has continued. Bismuth alloys have been solidified; the microstructures and in-situ measurements will be compared with numerical calculations.

31. University of Florida

F. S. Biancaniello and S. D. Ridder are collaborating with Professor M. E. Kaufman and graduate student T.M. Adams on a fundamental study to assess the equilibria between metastable ordered phases (MOP) and stable faceted phases (SFP) in faceted/nonfaceted systems.

32. University of Idaho

F.S. Biancaniello and F. W. Gayle are collaborating with F. Froes and C. Suryanarayana of the University of Idaho in the development of processing techniques to minimize contamination during mechanical alloying.

33. University of Michigan

Tim Foecke is interacting with Professor Gary Was of the Department of Materials Science and Engineering on an invited review article for the journal Thin Solid Films on the mechanical properties of microlaminates.

34. University of Minnesota

NIST scientists are collaborating with Jack Judy of the Magnetic Information Storage Center in the Electrical Engineering Department to develop improved giant magnetoresistive materials.

35. University of Mississippi

Gery R. Stafford is collaborating with Charles Hussey on the electrodeposition of aluminum alloys using room temperature molten salt electrolytes.

36. University of Missouri, Rolla

Tim Foecke is cooperating with Prof. Leighly, Department of Metallurgy, on a project underwritten by the Society of Naval Architects and Marine Engineers and The Discovery Channel looking into the metallurgical aspects of the sinking of the RMS Titanic.

37. University of Notre Dame

Profs. A. E. Miller and S. Bandyopadhyay of the University of Notre Dame were investigating nanomaterial synthesis techniques for the fabrication of quantum dot arrays. Because the NIST Materials Performance Group had experience with electrochemical techniques for the measurement and control of anodic processes on aluminum alloys, these Professors approached this Group for help with developing their electrochemical technique. This resulted in a collaboration which included a guest worker from the University working at NIST (prior to FY96) the results of which were compiled, evaluated, and published in FY96.

38. University of Wisconsin at Madison

W. J. Boettinger is working with the University of Wisconsin to develop models describing multicomponent phase equilibria in superalloy systems. The models are used to predict solidification behavior and microstructural development in complex industrial alloys.

39. Vanderbilt University

R.D. Shull is collaborating with C. Lukehardt in the preparation and characterization of sol gel routes to magnetic nanocomposites.

STANDARD REFERENCE MATERIALS

In Production

SRM #1358	Cu & Cr Coating on Steel
SRM #1362a	Cu & Cr Coating on Steel
SRM #1363a	Cu & Cr Coating on Steel
SRM #1364a	Cu & Cr Coating on Steel
SRM #2810	Rockwell C Scale Hardness - Low Range
SRM #2811	Rockwell C Scale Hardness - Mid Range
SRM #2812	Rockwell C Scale Hardness - High Range

Under Development

SRM #772	Absolute Magnetic Moment Standard
(no number assigned)	Gold Microhardness Standard
SRM #c1150b	White Cast Iron
SRM #1267	Stainless Steel, AISI 446
SRM # 2809	Rockwell Diamond Indenter
SRM #2814	Rockwell B Scale Hardness - Low Range
SRM #2815	Rockwell B Scale Hardness - Mid Range
SRM #2816	Rockwell B Scale Hardness - High Range

METALLURGY DIVISION

Administrative Office
M.L. Slusser
N.E. Wilkerson

Division Chief: C.A. Handwerker
Deputy Chief: R.J. Schaefer
Secretary: L.J. Toms

Laboratory Scientist
J. W. Cahn

855.10 ELECTROCHEM. PROCESSING

GL: G.R. Stafford
Sec: S.L. Tobery

Beauchamp, C.R.
Claggett, S.A.
Gates, H.G.
Johnson, C.E.
Kelley, D.R.
Moffat, T.P.
Mullen, J. L.
Sharpless, P.N.

Guest Researchers
Escalante, E.
Waterstrat, R.M.
Wheeler, N.S.
Yang, H.

855.11 MAGNETIC MATERIALS

GL: R.D. Shull
Sec: M.E. Pickett

Brown, H.J.
Donahue, M.J.
Drew, R.V.
Egelhoff, W.F.
Mathews, D.E.
McMichael, R.D.
Swartzendruber, L.J.
Washington, P.L.

Guest Researchers
Bennett, L.H.
Chen, P.
Chopra, H.D.
Gornakor, V.S.
Nikitenko, V.I.
Pardavi-Horvath, M.
Satija, I.I.
Schindler, A.I.
Tsory, E.
Weissmueller, I.J.
Wang, K.
Research Associates

Creswell, J.S.
Judy, J.H.
Koehler, T.R.
Contractor
Gomez, R.

855.12 MATERIALS PERFORMANCE

GL: E.N. Pugh
Sec: J.V. Teagle

Clough, R.B.
deWit, R.
Fields, R.J.
Flanigan, C.D.
Foecke, T.J.
Fraker, A.C.
Harne, D.E.
Hicho, G.E.
Low, S.R.
Pitchure, D.J.
Ricker, R.E.
Smith, J.H.
Stoudt, M.R.
Sturrock, C.P.

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Bertocci, U.
Brady, C.H.
Cammarata, R.G.
Interrante, C.G.
Mallen, A.J.
Levine, L.E.
Mordfin, L.
Shives, T.R.
Stern. A.
Weihs, T.P.
Wullaert, C.A.
Research Associate
Mashayekhi, B.
Consultant
Liggett, W.S

855.13 MATLS. STRUCT. & CHARACT.

GL:Gayle, F.W.
Sec: P.E. McCubbin

Adams, J.S.
Bendersky, L.A.
Bonevich, J.E.
Josell, D.
Krasicka, E.
Shapiro, A.J.
Smith, L.C.
Warren, J.A.

Guest Researchers
Klein, S.
Mitao, S.
Moon, K.W.
Rosen, M.
Wang, Z.L.
Yoo, Y.

855.14 METALLURG. PROCESSING

GL: J.R. Manning
Sec: J.A. Lepine

Biancaniello, F.S.
Boettinger, W.J.
Boyer, P.A.
Cezairliyan, A.
Coriell, S.R.
Hardy, S.C.
Jiggetts, R.D.
Kattner, U.R.
Parke, R.L.
Ridder, S.D.
Schaefer, R.J.
Simmons, J.A.
Williams. M.E.

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Banerjee, D.
Basak, D.
Bolcavage, A.
Matsumoto, T.
Munitz, A.
Roytburd, A.L.
Consultant
Sekerka, R.F.
Contractor
McClure, J.L.

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B.M. Fanconi, Deputy

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S.J. Dapkunas, Deputy

Materials Reliability

H.I. McHenry, Chief
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Reactor Radiation

J.M. Rowe, Chief
T.M. Raby, Deputy

National Institute of Standards and Technology

Organizational Chart