MSEL

Materials Science and Engineering Laboratory

METALLURGY

NISTIR 5750
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities
1995

Metallurgy Division

A Surface Evolver calculation of the spreading of a solder drop across a non wetting surface. Surface Evolver software is used to calculate the shape of a molten solder droplet causing a short circuit by bridging the gap between two pads on a printed circuit board. Such calculations help manufacturers understand how the wetting behavior of the solder and the geometry of the pads influence the tendency to form these defects.

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E. N. Pugh, Chief
S. C. Hardy, Deputy

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

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Arati Prabhakar, Director

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INTRODUCTION

METALLURGY DIVISION (855)

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This report describes the technical activities of the Metallurgy Division in 1995. The format this year is different from that in preceding years in that the descriptions of the Division's projects are grouped under major Materials Science and Engineering Laboratory (MSEL) program rather than under specific Groups. This approach emphasizes the collaborative nature of our activities, which cross both Group and National Institute of Standards and Technology (NIST) Division boundaries, and also the degree to which the Division's interests have become focussed.

It also should be noted that the Division underwent considerable reorganization in 1995. The 1995 Organization Chart shows five Groups, reduced from the seven in preceding years. Two Groups, Electrochemical Processing and Magnetic Materials have remained essentially unchanged programmatically. However, the former modified its name from Electrodeposition to more accurately reflect its activities, and the latter was strengthened significantly by the transfer of W.F. Egelhoff from the Chemical Science and Technology Laboratory. In addition to his international reputation in the area of surface science, Dr. Egelhoff brings an unique facility for the synthesis and characterization of nanoscale multilayers, thus further augmenting the Group's thrust in this technologically important area.

The Metallurgical Processing Group has retained its name and function but underwent a modest downsizing, several staff members transferring to a new unit, the Materials Structure and Characterization Group. The Metallurgical Processing Group continues to focus on solidification and powder synthesis, but the solder activities, a major Division thrust, are now centered in the new Group, which also incorporates the Microscopy Facility. The fifth Group, Materials Performance, was formed by combining the former Corrosion and Mechanical Properties and Performance Groups. The latter restructuring resulted from the decision to concentrate our efforts on corrosion to its effects on mechanical behavior, namely on stress-corrosion cracking, hydrogen embrittlement and corrosion-fatigue.

The two other former groups, Metallurgical Sensing and Modeling and High Temperature Materials Chemistry, have been eliminated and the staff re-distributed within MSEL. This action was driven largely by programmatic decisions. It was decided that the primary responsibility for sensor development within MSEL will reside in our Boulder Division (Materials Reliability), although the Metallurgy Division should maintain sensing capability to support its processing projects. Thus some members of the former Metallurgical Sensing and Modeling Group transferred to Boulder and others were distributed among the Metallurgical Processing and other Metallurgy Division Groups. In the case of the High Temperature Materials Chemistry Group, its program had evolved in recent years to the vapor deposition of thin films, and consequently it was considered more effective to transfer the Group to the Ceramics Division which had several projects in this area.

These reorganizations have caused a small downsizing but have led to increased focus. It will be seen that our major projects in 1995 lay in materials processing, electronic packaging and interconnections, and nanostructured materials, the latter concentrating heavily on multilayers. In each of these, the Division has continued to emphasize its traditional strengths in measurement science and materials characterization, and has maintained strong collaborations with U.S. industry. The Division also has maintained strong projects in the traditional NIST areas of standards and data that are essential to the nation's technological infrastructure.

Selected fiscal year 1995 (FY95) Accomplishments are listed below. Additional accomplishments and more extensive descriptions are given in the project summaries.

- In work with the Consortium on Casting of Aerospace Alloys, a "multicomponent-phase diagram subroutine" of code was prepared that gives the liquidus temperature, solid concentrations, liquidus slopes and the liquid and solid enthalpies as functions of the liquid concentrations for use in various kinetic models of alloy solidification. This subroutine employs a thermodynamic database that describes the free energies of the relevant phases.

- In the technology transfer phase of the industrial consortium on Metal Powder Atomization, our industrial partners were provided with a list of the sensors, data acquisition and control hardware, and software necessary to implement NIST technology for intelligent control of the atomization process.

- Developed a non-destructive method for determining the yield strength of high strength, low alloy (HSLA) sheet steel from the measurement of Barkhausen Noise and other magnetic properties. This work is part of an on-going American Iron and Steel Institute-Department of Energy (AISI-DoE) Advanced Process Control Program.

- As part of a National Center for Manufacturing Sciences (NCMS) Consortium on Alternatives to Lead-Base Solders, melting point/freezing ranges were measured for promising lead-free alloys produced at NIST.

- In another NCMS organized consortium (on Printed Wiring Boards), predictive models were developed for the rate and extent of reactive wetting of solder during area-of-spread and wetting balance solderability tests. Complicating factors in these models are solute diffusion and the Marangoni flow from temperature gradients expected under these conditions.

- Awarded a patent for Magnetic Nanocomposite Refrigerants.

- Organized and hosted the third NIST Workshop on Nanomaterials that was attended by 83 representatives from industry, universities and government agencies.

- Research in the Division's Nanostructured Materials Program in FY95 led to the achievement of the highest levels of the giant magnetoresistance (GMR) effect ever recorded in multilayer samples having the commercial spin valve configuration.

- In the National Association of Corrosion Engineers (NACE)-NIST Corrosion Data Program, five material advisory expert systems from the CHEM·COR series addressing the handling and storage of chemical process industry corrosives were released for public distribution in FY95.

- The tomographic scanning acoustic microscope technique developed at NIST was used successfully to obtain the distribution of residual stresses in spot-welded steel samples provided by the Ford Motor company. The technique also was applied to a welded steel pipe provided by DuPont.

- The Division moved a step nearer to the development of a traceable U.S. hardness standard by completing the construction of a computer controlled high precision dead-weight Rockwell hardness tester. Work has begun with U.S. hardness block manufactures to develop test blocks for the preparation of Standard Reference Materials (SRMs).

INTELLIGENT PROCESSING OF MATERIALS

Program Overview

Intelligent processing of materials (IPM) is the conversion of materials to value-added products using model-based control of processing variables. Information for real-time process control is provided by on-line sensors that 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 that 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 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 that has its roots in sensor development for nondestructive evaluation of materials. Unique MSEL capabilities are being used to measure thermophysical properties at elevated temperatures; these data are combined with model enhancements and then incorporated in industrial software for metal casting. In addition, specialized measurement capabilities such as nuclear magnetic resonance and small angle neutron scattering are used to understand microstructure/morphology evolution in ceramics and polymers.

For additional information about any aspect of the IPM Program, please call Tom Siewert, (303) 497-3523, or e-mail to siewert@micf.nist.gov.

Project Title: SOLIDIFICATION PATH FOR CASTING OF MULTICOMPONENT AEROSPACE ALLOYS

Investigators: William J. Boettinger, Ursula R. Kattner, Sam R. Coriell, 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 of castings used to determine soundness. 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 in 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 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 1995:

- A "multicomponent phase diagram subroutine" of code was prepared that gives the liquidus temperature, solid concentrations, liquidus slopes and the liquid and solid enthalpies as functions of the liquid concentrations for use in various kinetic models. This subroutine employs a thermodynamic data base that describes the free energies of the relevant phases.

- Solidification models were developed that treat liquid diffusion at the dendrite tip and solid diffusion in the dendrite interior for an N-component alloy. These effects reduce the degree of microsegregation predicted by the simple Scheil approach and alter how the latent heat is deposited in the mushy zone of a casting.

- Although good results have been demonstrated using a "preliminary" database employing only binary interactions, work on a refined data base involving the ternary subsystems of the Ni-Fe-Cr-Nb system has been performed. DTA melting experiments on Ni-rich alloys in the Ni-Fe-Cr system have permitted the selection of the literature calculation that best fits the data. Similar experiments on the Ni-Cr-Nb system have provided data necessary for developing ternary interaction energies in this system.

Impacts and Technical Highlights:

- NIST prediction of the fraction solid vs. temperature relationship for the alloy, INCONEL 718, has been incorporated into the thermal model of investment castings at Howmet Corporation. Using this model, significantly improved agreement between measured and predicted temperatures has been obtained. Use of this improved modeling capability will increase the manufacturing yield of large structural castings made from this alloy.

Project Title: MODELING OF ALPHA CASE THICKNESS IN TITANIUM CASTINGS

Investigators: W. J. Boettinger, S. R. Coriell, 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 for FY 1995:

- Cooling curves from thermocouples for locations in the metal casting but very close to the shell mold were obtained from a casting produced at Howmet. Measurements of alpha case thickness were made. The casting geometry was a step-wedge with section thickness ranging from 5 mm to 30 mm. This gave a range of cooling rates and hence α-case thicknesses.

- Thermal modeling was performed by Howmet with heat transfer coefficients adjusted to give agreement between measured and modeled temperature-time data.

- A diffusion model was developed to predict penetration of oxygen into the Ti casting. It was tested using the calculated temperature-time data as input. Good agreement was found between the predicted and measured α-case thickness, which ranged from 0.2 mm to 0.8 mm.

Impact and Technical Highlights:

- The diffusion model was implemented into the ProCAST™ code for modeling of castings as part of the NIST Consortium on Casting of Aerospace Alloys.

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

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

Technical Description:

This project seeks to characterize the 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. This work 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 classification system, based on experience of 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 will be developed for the design of single crystal casting processes to avoid the conditions which lead to defect formation.
- More precise criteria will be developed for classifying defects in single crystal castings and thus for identifying the conditions which are responsible for their formation.

Accomplishments for FY 1995:

- Developed model describing the thermal conditions which lead to the formation of defects known as "zebras" on horizontal surfaces of single crystal castings.
- Used X-ray topography to characterize the development of large-scale misalignment patterns in crystal structure during growth of single crystal components.
- Used electron backscattering patterns (EBSP) to measure the small-scale misalignments in single crystal components.

Impacts and Technical Highlights:

- Criteria for prevention of undesirable "zebra" defects were established and made available to manufacturers for their use in design and production of castings.

Project Title: THERMOPHYSICAL DATA FOR CASTINGS

Investigators: Ared Cezairliyan, Tsuyoshi Matsumoto, John McClure, and Louis Phillips

Technical Description:

This project is involved with the accurate determination of selected thermophysical properties of high temperature alloys of technological interest, particularly Ni-base superalloys important for 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 for FY 1995:

- Preliminary measurements of the properties (enthalpy, specific heat, heat of fusion, electrical resistivity, and normal spectral emissivity) of a nickel-base alloy (Mar-M247) were made in both solid and liquid phases, extending to temperatures about 400 K above its melting region. This alloy is used for fabrication of blades in some jet engines.

- Dependence of the solidus temperature on heating rate of the binary alloy 53Nb-47Ti was studied over a ten-fold variation in the heating rate.

- The solidus temperature of the binary alloy 53Nb-47Ti was measured; the result indicates that the solidus curve in the phase diagram for this alloy, as generally accepted in the literature, is likely to be significantly low.

Impacts and Technical Highlights:

- NIST generated thermophysical properties data on selected alloys are used by key U.S. aerospace companies in modeling casting processes.

- Accurate data on solidus and liquidus temperatures have been used to improve the phase diagrams of some high-temperature alloys.

Project Title: METAL POWDER ATOMIZATION

Investigators: Stephen D. Ridder and Frank S. Biancaniello

Technical Description:

Research in this project has focused on measurement and modeling tools for process understanding and control of metal powder produced by atomization. This research has 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 is the NIST Supersonic inert Gas Metal Atomizer (SiGMA). This research apparatus produces up to 30 kg of metal powder with average particle diameters ranging from 5 μm to 50 μm depending on alloy composition and processing conditions. The SiGMA system incorporates a controlled atmosphere chamber with vacuum pumping capabilities to limit powder contamination and enable processing with various gas mixtures. These features also provide an opportunity to apply advanced process control methods to regulate gas and metal flow rates within the atomizer, and control particle size and chemistry.

NIST research groups that have participated in this research effort include the Applied Systems Group of the Manufacturing Systems Integration Division (826.01), the Surface and Particle Metrology Group of the Precision Engineering Division (821.06), the Fluid Flow Group of the Process Measurements Division (836.01) and the Metallurgical Processing Group of the Metallurgy Division (855.14). The NIST scientists collaborated in this work with representatives from Ampal/Metallurg, Crucible Materials Corporation, DOE Office of Industrial Processes, General Electric Company, Martin Marietta Energy Systems, and Pratt & Whitney Aircraft.

The current project activities are focused on the transfer of the process control technology to a commercial atomizer at Crucible Research Center near Pittsburgh. This work involves software and hardware conversion to move the control system from a Macintosh computer environment to a PC computer running Labview for Windows. This change of software and operating platforms has greatly simplified the hardware requirements and relinquished software maintenance to a third party vendor, thus making the system more user-friendly to encourage wide-spread use. The resulting control system is easily portable to other computer systems, other atomizers, and other industrial processes.

Technical Objectives:

- Develop diagnostic tools for measuring process conditions of controlled atmosphere, gas atomizers. 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 powder product characteristics of controlled atmosphere, gas atomizers. This includes Computational Fluid Dynamics (CFD) of supersonic jets, process simulators, statistical analysis of process performance, and calculations of composition and phase stability within the resulting powder product.
- 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 gas atomizers.
- 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 1995:

- Installed and operated new process controller on SiGMA system suitable for transfer of expert system and control software to an industrial gas atomizer.
- Provided industry with list of the sensors, data acquisition and control hardware, and software necessary to implement the NIST developed technology.

- 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:

- CFD code developed by NIST under this project has been used by industry to redesign gas jet geometry and operating parameters to substantially improve efficiency of powder production process. The estimated cost savings are in the millions of dollars.

- Technology Transfer workshops held for industry representatives at NIST disseminated key information concerning NIST-developed sensors, analyses of the atomization process, particle-size measurement systems, and methods for automated process control.

Project Title: MAGNETICS FOR STEEL PROCESSING

Investigators: L. J. Swartzendruber, D. E. Mathews, L. H. Bennett, R. D. McMichael, G. E. Hicho

Technical Description:

In this project we are investigating the use of magnetic properties to characterize the mechanical properties of steel. Magnetic properties can be measured non-destructively, whereas direct mechanical measurements must be done destructively.

Technical Objectives:

- 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.

- Collect experimental data on the magnetic and mechanical properties of a wide variety of commercial sheet steels and on low carbon steel as a function of thermomechanical treatment.

- Relate the magnetic properties of the steel to the microstructure using phenomenological and micromagnetic models.

Anticipated Outcome:

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

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

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

Accomplishments for FY 1995:

- Determined useful multi-parameter models for relating yield strength to magnetic properties in high strength-low alloy (HSLA) sheet steel.

- Determined useful multi-parameter models for relating yield strength to magnetic properties in ultra low carbon (ULC) and low carbon (LC) sheet steel.

Impacts and Technical Highlights:

- The mechanical and magnetic properties of a series of HSLA and ULC sheet steels, produced on a continuous annealing line simulator under a wide range of processing conditions, were determined in detail. These results were used to determine phenomenological models that used the magnetic properties to predict the mechanical properties. Models were found which, using only the magnetic properties, were better predictors of yield strength and ultimate tensile strength than the processing conditions.

- Portions of this project were carried out in cooperation with LTV Steel Company and Weirton Steel Corporation, who provided the sheet steel samples, and with the Industrial Materials Institute in Canada, who provided ultrasonically determined structure parameters for evaluation. Results have possible applications for measurement of mechanical properties on-line in a steel mill and for rapid determination of steel uniformity, thus improving quality and reducing production and testing costs.

HIGH-T_c SUPERCONDUCTIVITY

Program Overview

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

The primary thrusts of the program are as follows:

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

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

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

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

Project Title: MAGNETIC MEASUREMENTS OF SUPERCONDUCTORS

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

Technical Description:

Working with other scientists fron NIST, universities, and industry, high temperature superconducting materials are prepared and their microstructure and magnetic properties determined. Measurements 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.

Technical Objectives:

- Develop and improve magnetic measurements of superconductors.
- Develop relationships between ac and dc susceptibilities and the impurities and phase distributions present in the material.
- Study the flux pinning properties of high temperature superconductors as a function of their microstructure and processing variables.
- Explore superconducting properties of new materials and of materials prepared under varying processing conditions.
- Provide support to the Ceramics Division in the determination of phase diagrams of materials important for the production of high temperature superconductors.

Anticipated Outcome:

- Improvements in the ability of manufacturers and researchers to interpret magnetic measurements in high-temperature superconductors.
- Increased critical current densities by improvements in flux pinning.
- Better quality control in the production of high-temperature superconductors.

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

Accomplishments for FY 1995:

- Determined that small field gradients have a large effect when measuring the Meissner fraction of high temperature superconductors with large pinning.

- Developed a method for measuring Meissner fraction in the presence of field gradients.

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. An instrument, developed by NIST in cooperation with the Institute of Solid State Physics in Russia, for observing the flux distribution in superconductors has been commercialized by a US Company. Phase diagrams developed using the aid of magnetic measurements are currently being used for scientific investigation and industrial development.

- After the discovery of high temperature superconductors, a so-called "positive Meissner effect" was observed in many laboratories. Such an effect is often observed in SQUID magnetometers. Rather than being a real effect, we have shown how such an observation is an artifact of the measurement process. We have also shown how the true Meissner fraction in a sample can be properly measured. Such measurements are important for assessing the flux pinning properties high-temperature superconductors.

ELECTRONICS PACKAGING AND INTERCONNECTION

Program Overview

The U.S. microelectronics industry, valued at over $300 billion in 1995, is confronted with technological changes at an unprecedented pace and urgency. This is due partially to increased consumer expectations, rapid product evolutions, and heightened international competition. In response to these pressures, the U.S. semiconductor and module interconnection industries, representing combined sales of over $54 billion in 1995, have taken the landmark steps of developing technology roadmaps. These two roadmaps, entitled *The National Technology Roadmap for Semiconductors* and *The National Technology Roadmap for Electronic Interconnects*, produced by the Semiconductor Industry Association and the Institute for Interconnecting and Packaging Electronic Circuits, respectively, identify roadblocks and performance characteristics for the manufacture of globally competitive products. Significant portions of these roadmaps address the packaging and interconnection of semiconductor devices, a technology which now amounts to over one-third the delivered cost of integrated circuits.

To assist this strategic and rapidly growing U.S. industry, the NIST Materials Science and Engineering Laboratory has embarked on a new program in electronics packaging and interconnection that addresses industry's most pressing challenges surrounding the utilization of advanced materials and material processes. With a specific mission to develop and deliver to 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 used 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.

This strategy is the outgrowth of two industry-led workshops conducted at NIST. The first, conducted in 1990, set the course for the Laboratory's emerging plans in packaging, interconnection and assembly. The second, conducted in early 1994, identified a series of cross-cutting barriers, critical technical challenges and opportunities for NIST in materials science and engineering deemed most needed by the U.S. microelectronics industry. The results of this workshop are contained in NISTIR 5520, *Metrology and Data for Microelectronics Packaging and Interconnection.*

Now in its second full year of funding, the program has in place a portfolio of projects involving the talents and operations of metallurgists, polymer scientists, and materials reliability specialists. During this period, the MSEL program has developed numerous single-company and consortia-based collaborations that involve over twenty-three U.S. companies; fifteen universities; four other government agencies or laboratories; and eight consortia, standards bodies and associations. These collaborations have resulted in forty-six technical publications, sixteen of which were published in 1995, and numerous individual accomplishments that directly impact industry's research and development needs.

For further information on any aspect of the Electronic Packaging and Interconnection Program, please contact Michael A. Schen @ 301-975-6741 or e-mail requests to michael.schen@nist.gov.

Project Title: ALTERNATIVES TO LEAD-BASED 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, Motorola, MicroFab Technologies, 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 range of 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.

- Develop models of solder wetting and spreading.

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 in order to avoid environmental problems and their performance established before legislation passed that restricts lead use.

- Guidelines for alloy testing established for development of solder alloys that eliminate 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 1995:

- Measured melting point/freezing ranges in special candidate alloys produced at NIST to identify promising lead-free solders.

- Established validity of linearised equations for melting/freezing ranges for solders containing up to eight elements.

- Identified composition limits in ternary and quaternary alloys that meet melting/freezing range requirements.

- Established quantification method for mechanical failure testing and performed related failure analyses.

- Fabricated solder alloys for assembly of test circuit boards.

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 required for manufacturing.

- Solidification experiments were used to determine non-equilibrium phase formation in the solder during cooling from the liquid state.

- 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, automotive applications.

Project Title: ELECTROCHEMICAL CHARACTERIZATION OF SOLDERS

Investigators: Gery Stafford and Sandra 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.

Technical Objectives:

- Determine the electrochemical method (galvanostatic or potentiostatic transient) which provides the most accurate and reliable description of the aged copper surface.

- Determine the reduction potentials for Cu_2O and CuO in an electrolyte comprising 1.0 mol/L $LiClO_4$ and 0.1 mol/L LiOH.

- Electrochemically evaluate the surface oxide of copper after exposure to four different accelerated aging procedures. Analysis should provide information about the oxidation state and amount of oxide present on the surface.

Anticipated Outcome:

- Determine the electrochemical signature for each of the aging procedures and provide comparisons based on both the oxidation state and quantity of surface oxide present.

Accomplishments for FY 1995:

- In 1.0 mol/L $LiClO_4$ and 0.1 mol/L LiOH, the reduction potentials of Cu_2O and CuO are approximately 0.25V apart. However, the reduction potentials for each species varies with oxide thickness, presumably due to the semi-conducting nature of the oxide. Galvanostatic experiments, involving very small currents, yield the most reliable and reproducible reduction transients.

- 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.

Impact and Technical Highlights:

- Electrochemical reduction is an excellent technique for determining the oxidation state of copper, both qualitatively and quantitatively. 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, D. Josell, U. R. Kattner 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 (1) to evaluate components for solderability before committing them to the production line and (2) evaluate solder joint-designs for their manufacturability and reliability. For this purpose, measurements and modeling of wetting balance solderability tests, area-of-spread solderability tests, and solder wetting phenomena are underway.

Technical Objectives:

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

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

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

Anticipated Outcome:

- Improved solderability test methods will be developed which will 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 limits on visual inspection of the solder joint make rework of improperly soldered connections difficult or impossible.

Accomplishments for FY 1995:

- NIST research on wetting-balance solderability tests identified conditions that provide high sensitivity to wettability factors in these tests. These results were incorporated by the Institute for Interconnecting and Packaging Electronic Circuits (IPC) Wetting Balance Test Group into the IPC Joint Industry Standard document on Solderability Tests for Component Leads, Terminations, Lugs, Terminals, and Wires.

- A workshop on Concurrent Design of Advanced Interconnection Technology, held at Amelia Island, FL, Nov. 14-16, 1994 was organized in collaboration with Sandia National Laboratories to obtain views of the microelectronics industry on needed measurement programs in this area.

- Predictive models were developed for the rate and extent of reactive wetting of solder during area-of-spread and wetting balance solderability tests. Complicating factors in these models are solute diffusion and the Marangoni flow from temperature gradients expected under these conditions.

Impacts and Technical Highlights:

- NIST wetting balance measurements have shown how intermetallic formation and oxidation can affect solderability. These results are being used to design improved solderability tests, particularly accelerated tests. Since industry finds that the presently used wetting balance tests are not sufficiently reliable, improved tests are needed. A NIST-designed accelerated test using steam-aging techniques to allow more rapid testing is under consideration by IPC.

- Collaboration of NIST researchers with an industrial consortium on printed wiring boards led by the National Center for Manufacturing Sciences (NCMS) contributed to new solderability tests being developed by this consortium. Members of the consortium are AT&T, IBM, Hamilton Standard, Sandia National Laboratories, and Texas Instruments.

Project Title: SOLDER JET PRINTING FOR MICROELECTRONICS APPLICATIONS

Investigators: Frank W. Gayle, Carol A. Handwerker, Leonard C. Smith, and Maureen E. Williams

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 ink 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 under a range of deposition conditions and substrates, including joints fabricated through reflow.

Anticipated Outcome:

- Materials compatibility issues of next generation devices for creating solder patterns will be developed.
- Solder joint formation after reflow for solder deposited using jet technology will be better understood.

Accomplishments for FY 1995:

- Interfacial reactions associated with containment of Pb/Sn solders in stainless steel reservoirs have been identified and possible solutions to the concomitant contamination have been investigated.

- Problems associated with oxidizing atmospheres during jetting have been identified. Improvements in jetting and in deposit morphology using controlled atmospheres were characterized.

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

- Evaluation of physical and mechanical property data for In-Sn alloys which are necessary for ultrasonic jetting devices. These data were supplied to MicroFab in the form of a computer program which provides data for any composition and temperature.

Impacts and Technical Highlights:

- Our identification of solder oxidation problems contributed to MicroFab's decision to add nitrogen co-flow to the solder jet apparatus. This control of oxygen is necessary for MicroFab's milestones both for operation of the solder jet and for formation of reliable solder joints.

- Wettability problems which we have identified with respect to copper substrates have led to investigations of means (such as imidazole coatings) to enhance wettability. This is essential for MicroFab's milestone to form reliable solder joints with solder jet technology.

- Research at the Metallurgy Division has identified temperature regimes which are suitable for deposition onto Au-coated silicon. This is essential for MicroFab's milestone to form reliable solder joints with solder jet technology.

- 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.

- Electrochemical reduction is an excellent technique for determining the oxidation state of copper, both qualitatively and quantitatively. These measurements can be performed directly on printed wiring boards and component leads and may eventually be used to assess solderability.

Project Title: **SOLDER INTERCONNECT ENGINEERING**

Investigators: James A. Warren, Carol A. Handwerker, W. Craig Carter, Daniel Josell, and Andrew Roosen

Technical Description:

NIST scientists are working with scientists and engineers from industry and academia to develop an integrated design system for solder interconnects. Collaborating with NIST are: AT&T, AMP, Inc., Digital Equipment Corporation, Edison Welding Institute, Ford Motor Company, General Motors/Delco, Rockwell International Science Center, Marquette University, University of Colorado - Boulder, State University of New York - Binghamton, Susquehanna University, University of Greenwich, University of Technology - Loughborough, University of Wisconsin-Madison, Lehigh University and Sandia National Laboratories. Research is focussed on developing algorithms for modeling solder spreading in specific solder joint geometries and on using the resulting joint shapes as input to models of solder assembly processes and mechanical reliability.

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 working group 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:

- The design system will be able to evaluate an interconnect design for its susceptibility to defect formation during soldering and for its reliability under conditions of thermo-mechanical fatigue.

- Integrated solder joint design tools will allow packaging designers greater flexibility in the engineering of the next generation of interconnections with higher interconnect densities.

Accomplishments for FY 1995:

- NIST-sponsored workshop with representatives from industry, academia, and national laboratories identified the needs for modeling specific solder joint geometries and established the Solder Interconnect Engineering working group.

- Developed model for wetting balance test geometries and provided quantitative shape predictions for comparison with observed shapes.

- Developed model for predicting the susceptibility of adjacent pads to solder joint bridging.

- Prepared review of the use of the Surface Evolver program for calculation of solder joint shapes. This manuscript will be part of a Working Group-organized issue of the ASME Electronic Packaging journal.

- Established Internet/WWW site for information exchange and the format for providing models to the public.

- Complete shape calculations for a wide range of gull-wing device geometries and provide the information via WWW.

- Hold two additional working group meetings to promote collaboration and to increase the amount of information on the Web site exchange.

- Collaborate with AT&T, Lehigh University, and other group members in comparing model predictions with experimentally determined geometries.

Impacts and Technical Highlights:

- Scientists and engineers from industry, government, and academia established a collaborative program for the development of software modeling tools for improving solder interconnect geometries with the goals of higher manufacturing yields and higher reliability.

- The critical need for such modeling tools was identified by the Semiconductor Industries Association in its 1992 SIA Technology Roadmap for advanced packaging systems.

Project Title: STRESS MEASUREMENT IN ELECTRONIC PACKAGING

Investigator: Ewa Drescher-Krasicka

Technical Description:

The accumulation of stress in electronic chips can be caused by condensation of moisture in plastics and thermal fatigue during usage or by unavoidable mismatch of the coefficients of thermal expansion between silicon (die) metals (wires) and polymer filler. The growing and accumulating stress creates cracks and delaminations in electronic packages. Failure of one chip in an electronic device frequently causes failure of the device. While modern technology has many techniques for detecting mechanical defects in electronic components, the present urgent requirement of the electronic industry is to detect the area of material which can fail due to concentration of residual stresses. The scanning acoustic microscopy was successfully implemented for detection of popcorn cracks, and delamination in electronic chips. Routine tests are available for control on line for hidden mechanical defects. There is a need for a fast and reliable technique for detecting undesirable accumulations of residual stresses in electronic and microelectronic components.

Technical Objectives:

- Develop test methods needed to detect residual stresses in electronic components.
- Develop test methodology to achieve a known state and level of residual stresses in multilayered chips.
- Apply Scanning Acoustic Imaging of Stress (SAIS) for stress detection in electronic chips without mechanical defects.
- Apply SAIS for detection of residual stresses in electronic components containing cracks.

Anticipated Outcome:

- Detection of the areas containing high residual stresses in electronic chips.
- Recommendation guidelines for the software change in the "Sonix" scanning acoustic microscope in order to separate the two different acoustic modes, shear and longitudinal. The acoustic images obtained by use of a single mode for scanning can be compared with the existing theoretical calculation of the distribution of stress.

- Recommendation guidelines for calibration of the stress value obtained from acoustic images with the residual stress calculations by finite elements modeling (FEM) analysis and analytical solutions.

Accomplishments for FY 1995:

- SAIS technique was successfully applied for detection of residual stresses in electronic components containing "popcorn" and under surface cracks. Surface acoustic waves of high frequency create the acoustic microscope images of the distribution of residual stresses beneath the surface.

- Longitudinal and shear waves were used to detect the cracked areas and the stressed area around the die.

- Semiconductor devices (power switches), subjected to different numbers of fatigue cycles, were prepared for imaging of the increasing residual stresses up to mechanical failure.

Impacts and Technical Highlights:

- NIST prepares in cooperation with Sonix Inc. software and procedure guidelines for routine control of residual stress in electronic components. (CRADA).

- U. S. Patent 5406849 issued 04/18/95. Ewa Drescher-Krasicka and John A. Simmons Method and Apparatus for Detecting Guided Leaky Waves in Acoustic Microscopy.

NANOSTRUCTURED MATERIALS

Program Overview

Nanostructured Materials are a new class of materials which provide one of the greatest potentials for improving performance and extended capabilities of products in a number of industrial sectors, including the aerospace, tooling, automotive, recording, cosmetics, electric motor, duplication, and refrigeration industries. Encompassed by this class of materials are multilayers, nanocrystalline materials and nanocomposites. Their uniqueness is due partially to the very large percentage of atoms at interfaces and partially to quantum confinement effects.

One critical need for their implementation is their characterization and measurement science which are the focuses of the NIST program. For many properties, it is not known whether the exciting novel behavior found in these new materials is due to new physics or to a logical extension of large-size behavior to small dimensions. Examples include the deformation and fracture behavior (wherein it is not known whether dislocations even exist in these materials), optical characteristics (wherein uncertainties exist in whether the properties are due to interface or quantum mechanical effects), magnetic properties (wherein it is not known what magnetic domains even look like in nanostructured materials or how they move in response to a magnetic field), and thermal properties (wherein the propagation of phonons through interfaces is poorly understood). Consequently, implementation of this new type of material into marketable products is significantly delayed. NIST is providing the measurement science to answer these critical unknowns. Important needs also include the identification of preparation methods for industrial-size quantities of material, extension of the capabilities of conventional measurement tools to the nanometer-size scale, and the development of consolidation methods that still retain the nanometer grain size of the initial nanocrystalline powders. For multilayers, understanding the development of epitaxy and control of both composition and interdiffusion at the interface are of critical importance.

By experimentally addressing these issues, by bringing together the industrial and scientific communities through the organization of workshops and conferences in the area, and by the development and preparation of appropriate standards, NIST acts to accelerate the utilization of these materials by the industrial sector. In addition, collaborations established in the area with Xerox, General Motors, Nanophase Technologies, Pratt and Whitney, Caterpillar, Lockheed-Martin, Hewlett Packard, IBM, Seagate, and Motorola Corporations, for example, enable NIST to leverage its activities with the much larger, but complementary, capabilities of other organizations.

For additional information about the Nanostructured Materials Program, please call R. D. Shull, (301)975-6035, e-mail: shull@enh.nist.gov.

Project Title: **MAGNETIC PROPERTIES OF NANOMATERIALS**

Investigators: R. D. Shull, R. D. McMichael, L. H. Bennett, D. E. Mathews, R. V. Drew, H. J. Brown, L. J. Swartzendruber, U. Atzmony, J. W. Weissmueller

Technical Description:

The project aims at promoting the understanding of microstructure - property relations for materials with a nanometer-scale granular microstructure. The motivation for this is threefold: first, enable the creation of materials with optimized properties; second, allow the determination of intrinsic interface properties from measurement of the macroscopic averages of the materials properties; third, provide a better understanding of magnetic interactions in materials near defects like grain boundaries.

Technical Objectives:

- From measurements of the magnetization of nanocrystalline Y-Fe alloys and from Mössbauer data for the material, characterize the magnetic order of the Fe-rich grain boundary segregation layers.

- From measurements of the temperature dependence of the magnetization and high temperature x-ray measurements determine whether the alpha/gamma allotropic transformation of Fe in Fe-Cu alloys was reduced by a factor of 2 when nanometer grain sizes were formed by ball milling.

- Attempt to produce a fine dispersion of nanometer-sized magnetic "compounds" in an oxide by a sol gel process.

- From magnetization and Mössbauer measurements, determine whether nanocrystalline Fe prepared by vapor condensation and by ball milling was magnetically any different than bulk Fe.

- Determine the excess free volume of the grain boundaries in a high density (i.e. nearly pore-free) nanocrystalline solid by a combination of macroscopic density and small-angle scattering measurements.

- Determine whether there was an enhancement of the magnetocaloric effect at a ferromagnetic/antiferromagnetic transition, as occurs in $Fe_{2-x}(Hf_{.83}Ta_{.17})$ alloys.

- Determine whether there was an enhanced magnetocaloric effect in Dy-Al-Fe garnet nanocomposites.

- Interest industry to apply magnetic nanocomposites as refrigerants in magnetic refrigerators.

- Determine the magnetic microstructure in a nanocrystalline ferromagnet by small-angle neutron scattering combined with magnetization data.

- Establish a relation between the intrinsic interface property interface stress on the one hand and measurable characteristics of the microstructure and of the internal stress distribution on the other hand.

- Optimize the processing of gamma-Fe_2O_3/polymer nanocomposites for application as advanced toner materials for high quality color copiers and printers.

- Organize symposia and workshops in the area to acquaint industry with the new observations being made on nanocrystalline materials.

Anticipated Outcome:

- Improved understanding of the intrinsic interface characteristics (magnetic order, thermodynamic properties) in fine-scaled granular solids.

- Answer to the question: how (if at all) is the grain boundary atomic structure at very fine grain sizes different from that in coarse-grained polycrystals.

- An understanding of the role grain boundaries play in determining the magnetic moments of ferromagnetic elements.

- Evidence of the enhanced magnetocaloric effects in magnetic nanocomposites predicted by NIST several years ago.

- Development of a method for preparing magnetic compounds in a nanocomposite.

- Answer to the questions: - What are the relative importance of different microstructural elements / defects (variations of the crystal lattice orientation; interfaces; strain fields; nonmagnetic inclusions) on the magnetic microstructure? - Is one of the above microstructural elements related to the coercivity (a technically important parameter) of the samples?

- Identification of suitable, i.e. relevant and measurable, characteristics of the microstructure.

Accomplishments for FY 1995:

- Showed that Fe-rich grain boundary segregation layers can order ferromagnetically.
- Performed scattering, magnetization and density measurements on electrodeposited nanocrystalline Ni.
- Scattering data will yield a new upper limit on grain boundary excess free volume in a nanocrystalline metal. Preliminary evaluation indicates good agreement of scattering data with a micromagnetic model developed at NIST.
- Prepared a fine dispersion of nanometer-sized ferromagnetic Fe_2P in silica by a sol gel process.
- Found an unusually high, 250 K, blocking temperature in a superparamagnetic Fe_2P/silica nanocomposite.
- Found an unusual time dependence in the magnetization of nanocrystalline Fe prepared by ball milling.
- Showed the ease with which contamination can occur in nanocrystalline Fe-Cu alloys, thereby causing erroneous conclusions by investigators in the field.
- Found an enhanced magnetocaloric effect in Dy-Fe-Al garnet nanocomposites.
- Showed how the averages of the internal elastic stress can be related to measurable stereological quantities, the moments of the interface orientation distribution function.
- Awarded an Small Business Innovation Research (SBIR) to a company for designing and constructing a magnetic refrigerator to show reasonable cooling at room temperature.
- Organized 3 symposia on Nanocrystalline Materials and one symposium on sol gel materials.
- Awarded a patent for Magnetic Nanocomposite Refrigerants.

Impacts and Technical Highlights:

- Xerox Corporation has started a pilot plant for the preparation of large quantities of gamma-Fe_2O_3/polymer nanocomposites for potential use in high quality copiers.
- Combined work has resulted in over 20 invited talks in FY95 to industry, universities, and professional societies.

Project Title: MECHANICAL PROPERTIES OF NANOMATERIALS

Investigators: T. J. Foecke, D. Josell, M. R. Stoudt

Technical Description:

Applications of nanoscale multilayers in the near term include use as protective coatings, whether for thermal barriers, ultrahard coatings, or for wear resistance needs. Future uses might include structural applications. Within the next decade, physical vapor deposition (PVD) structures are expected to amount to as much as 2% of the estimated $25B high performance coatings industry in the U.S., as the dependence on environmentally hazardous electroplated hard chrome decreases. Nanolayered composites are being commercially applied presently, and advances in fabrication technology will allow the exceptional physical properties of this class of materials to be more extensively utilized in high performance and high value-added applications. Potential users of this technology include users of critical, high performance coatings: automotive engines, aircraft engines, cutting and machine tools, and ceramic parts manufacturing.

Technical Objectives:

- Examine the fundamental mechanisms that operate within nanolayered materials that result in such extraordinary properties as ultrahardness, wear resistance, and high thermal conductivity.

- Evaluate how these properties can be controlled using the ability to prescribe the makeup of the microstructure at nearly the atomic level.

- Develop a fundamental understanding of the relationships between materials, structure, and properties.

Anticipated Outcome:

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

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

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

Accomplishments for FY95:

- Organized and hosted the third NIST Workshop on Nanomaterials which was attended by 83 representatives from industry, universities, and government agencies.

- Four papers on this work were completed and submitted for publication.

- Two invited and one contributed presentation were made on this work.

Impacts and Technical Highlights:

- NIST, the National Science Foundation Institute for Mechanics and Materials, and Lawrence Livermore National Laboratory sponsored a workshop entitled "Nanoscale Layered Materials for Protection Coating and Structural Applications" which was attended by 83 representatives from industry, universities, and government agencies.

Project Title: ELECTROCHEMICAL PROCESSING OF NANOSTRUCTURED MATERIALS

Investigator: Thomas P. Moffat

Technical Description:

A variety of nanostructured materials may be synthesized by electrochemical deposition. Our effort currently is 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. In particular, the role of inorganic and organic adsorbates on the evolution of thin film microstructure and morphology are of particular interest.

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:

- Produce well defined model strained-layer superlattices for property measurements by several other researchers. These materials exhibit technically interesting magnetic and mechanical properties.

- The commercial success of electroplating technologies stems largely from the remarkable influence of electrolyte additives on the physical properties of the deposited films. Our SPM studies promise to provide considerable insight into the way these adsorbates influence microstructural evolution.

- Provide a means for inexpensive, low capital cost, patterned electrodeposition of metals.

Accomplishments for FY1995:

- Heteroepitaxial deposition of nickel on copper is being 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 we have produced a series of multilayers on Cu(100) with modulation wavelengths ranging from 2.6 nm to 70 nm oriented exclusively in the <100>. These films typically cover an area of ~2.5 cm^2 and are ~2.4 μm to 4.7 μm thick. The mechanical and magnetic properties of these materials are being examined by several other researchers at NIST. In contrast to the specular films grown on Cu(100), growth on Cu(111) and Cu(110); leads to roughened surfaces. The origin of this instability along with the potential use of surfactants to control interfacial morphology is being explored.

- Developed the capability for studying the structure and dynamics of the deposition/dissolution of Cu and Ni with atomic resolution in-situ scanning tunneling microscopy.

- In a collaboration with H. Yang of IGEN Inc. we have demonstrated the use of self-assembling monolayers (SAM) as masks for pattern plating. Micrometer level features were easily and inexpensively synthesized by contact printing of the SAM's followed by electrodeposition in a typical chemical laboratory environment. Looking to the future we would be able to use this mask technology for pattern electrodeposition in the nanometer regime using SPM, electron beam technology or possibility contact printing.

Impacts and Technical Highlights:

- In-situ scanning tunneling microscopy (STM) is being used to investigate a variety of technologically relevant phenomena (e.g., electrodeposition, corrosion, and etching), that occur at electrode/electrolyte interfaces. The microscope is capable of imaging surface structure and associated real time dynamics over a scale ranging from the atomic to the micrometer 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 chloride ion is a ubiquitous species in most commercial copper electroplating processes, our initial studies have been concerned with copper in acid chloride electrolytes. STM reveals 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.

Project Title: SPUTTERED MULTILAYER NANOSCALE MAGNETIC FILMS

Investigators: W. F. Egelhoff, P .J. Chen (guest researcher), T. Ha, J. H. Judy (U. Minn.), R. D. Gomez (U. Md.), H. D. Chopra (U. Md.), R. D. K. Misra (guest researcher), J. Nir (guest researcher)

Technical Description:

The objective of this program is to provide assistance to U.S. companies in the magnetic data-storage industry so they can operate successfully in the increasingly competitive world market. We have constructed an elaborate new facility to allow us to reach this objective. No facility of such complexity exists anywhere else in the world. Our work should provide U.S. companies with a significant competitive edge.

Technical Objectives:

- The objective of this program is to provide assistance in manufacturing process development to U.S. companies in the magnetic data-storage industry.

- Particular emphasis is placed on helping companies which have received Advanced Technology Program (ATP) grants.

- Our role is to investigate the science underlying the manufacturing process, something these companies cannot afford to do on their own.

Anticipated Outcome:

- The focus of our research for the past year has been to improve upon a new class of magnetic thin films which exhibit a property known as the giant magnetoresistance (GMR) effect.
- Thin-film GMR materials have great commercial potential in ultrahigh density data-storage products.
- It is likely that within a decade, GMR-based data-storage products will account for billions of dollars in annual sales for U.S. companies.
- Our research is anticipated to help make U.S. companies the world leaders in this field.
- In the year to come, we expect to continue to play a leading role in the development of improved manufacturing processes for GMR films.
- Our supply of novel, innovative, and unprecedented ideas for improving these materials shows no sign of diminishing.

Accomplishments for FY 1995:

- The past year was the first full year of operation for the Magnetic Engineering Research Facility (MERF) which was constructed for the competence program.
- The MERF is the most elaborately instrumented thin-film deposition facility in the world.
- MERF is designed to simulate the magnetic thin-film manufacturing environment and at the same time permit *in situ* application of the most advanced structural and magnetic diagnostic techniques.
- Research at MERF in the past year has achieved highest levels of the GMR effect ever recorded in samples of the type (spin valves) best suited to commercial products.
- The key variables for these record-setting results have been identified (e.g., low water vapor pressure during deposition, deposition of GMR films at cryogenic temperatures, and careful substrate cleaning).
- We have found that the response of the GMR film to an external magnetic field can be substantially increased if surfactant atoms such as Pb or In are added during deposition to produce the smoothest possible film.

Impacts and Technical Highlights:

- The information on our record-setting results has been transferred to our collaborators at IBM, Seagate, Hewlett-Packard, Nonvolatile Electronics, and Read-Rite.

- These collaborators are attempting to implement our findings in their production equipment.

- As far as we can determine, the foreign competitors of these U.S. companies are unaware of these recent developments. The U.S. companies thus have a headstart.

- During the coming year we will continue these collaborations and initiate a new one, with Motorola, which will develop a GMR-based version of Dynamic Random Access Memory (DRAMs). We have Advanced Research Project Agency (ARPA) funding to support this collaboration.

Project Title: ELECTROMAGNETIC PROPERTIES OF MULTILAYER NANOSCALE FILMS

Investigators: R. D. McMichael, L. H. Bennett, H. J. Brown, R. Drew, D. Mathews, R. Shull

Technical Description:

In this project, a variety of electrical and magnetic techniques are developed for and applied to the characterization of multilayered nanoscale films, especially magnetic multilayers. An emphasis has been on the identification of processes involved in degradation of the multilayer structures at moderately elevated temperatures.

Technical Objectives:

- Develop electrical and magnetic test methods and magnetic imaging techniques needed to characterize multilayer nanoscale films.

- Develop computational micromagnetic modeling techniques for comparison with experimental results.

- Collect data on the effects of thermally induced damage to nanoscale film structures, and compare with model results.

- Organize workshops to bring the micromagnetic modeling community to consensus on recommended modeling procedures.

Anticipated Outcome:

- Identification of mechanisms for thermal degradation of nanoscale multilayers.

- Reliable nondestructive imaging techniques and electrical and magnetic characterization methods for nanoscale multilayers.

- Improved accuracy and speed in computational micromagnetic modeling.

Accomplishments for FY 1995:

- NIST/Center for Theoretical Computational Materials Science (CTCMS) sponsored workshop on micromagnetic modeling with representatives from academia and industry identified opportunities and challenges in micromagnetic modeling.

- Used superconducting quantum interference device (SQUID) magnetometry, magnetoresistance and ferromagnetic resonance techniques to identify the "weak link" in annealed spin valves. Changes based on this information resulted in improved thermal stability of these structures.

- Identified magnetization reversal mechanisms and magnetic domain structures by optical and scanning probe techniques.

- Wrote computer programs for micromagnetic modeling. This code may become the basis for a public distribution.

Impacts and Technical Highlights:

- Measurement of the temperature stability of the thin films used in multilayers provides critical data about how well the layers will hold up running at elevated temperatures in devices.

DENTAL AND MEDICAL MATERIALS

Program Overview

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 major focus sectors in this program are: (1) Development of dental restorative composites of greater durability and wear resistance, and (2) Development of metallic mercury-free alternative to dental amalgams.

Dental restorative composites are heterogeneous materials having three essential phases: (1) a polymeric matrix which comprises the continuous phase, (2) fillers of various types, sizes, shapes and morphologies which constitute the disperse phase and (3) an interfacial phase that, in varying degree, bonds the continuous and disperse phases into a unitary material rather than a simple admixture. The polymeric matrix of a dental composite is formed by free radical polymerization, under ambient conditions, of a resin which is one or more vinyl monomers, usually of the methacrylate class. Polymerization is started either by the formation of initiating radicals from chemical reduction-oxidation (redox) reactions or by photochemical redox reactions. While all three phases are important in determining the properties of the composites, this program focuses primarily on the interfacial and polymer matrix phases. Since the polymerization shrinkage that occurs in the matrix phases is one of the most commonly cited deficiencies of dental restorative composites, resources are allocated to develop high conversion, low shrinkage polymeric materials for use in dental resin and composite applications. Although only a minor component of these composites, the interfacial phase that develops from the interaction of the silane coupling agent with the polymer matrix and the siliceous filler exerts a profound effect on the properties of the composites. Because these composites are used in an aggressive, aqueous environment that constantly challenges the vulnerable silane mediated polymer-glass bond, understanding of this critical interfacial phase is being acquired so that strategies can be developed for its improvement.

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 the objective of this focus. The approach chosen is based on three main premises: (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.

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 and their projects and accomplishments are reported in a separate publication. 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.

For additional information about the Dental and Medical Materials Program, please contact Francis Wang at 301-975-6726 or e-mail requests to francis.wang@nist.gov.

Project Title: METALLIC MERCURY-FREE ALTERNATIVE TO DENTAL AMALGAMS

Investigators: Gery R. Stafford, Christian E. Johnson, David R. Kelley, Yoshi Oshida* and Moshe P. Dariel**

* Guest Scientist, Indiana University School of Dentistry
** Guest Scientist, Ben Gurion University, Beer-Sheva, Israel

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 the objective of this program. The approach chosen by the Electrochemical Processing Group is based on three main premises: (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.

Technical Objectives:

- Determine, develop and quantitatively characterize the basic processes involved in the room temperature in-situ consolidation of suitable amalgam-free alternative restorative materials.

- Develop a chemical or electrochemical process that reproducibility generates a restorative material with consistent properties.

- Develop test methods to obtain mechanical properties for comparison of alternative restorative materials to dental mercury amalgams.

- Optimize the composition of the alternative restorative material by studying the effects of various strengthening mechanisms on both the properties and processing parameters.

Anticipated Outcome:

- The silver-based compacts have the advantage of being mercury-free and, thereby, provide an alternative in an anticipated situation where the use of mercury-containing restoratives will be curtailed.

- The development of a successful technology that meets the requirements and displays advantages with respect to the various alternatives will be transferred to industrial and marketing partners.

Accomplishments for FY1995:

- In-situ silver-tin intermetallic compound formation was achieved by immersion deposition of pure silver onto very high surface area tin particles.

- The presence of intermetallic compounds increased the hardness of the silver based restorative composite, but adversely affected the propensity to undergo consolidation and consequently yielded low transverse rupture strengths.

- An in-depth reassessment was made to shift the main thrust of the program toward optimizing unalloyed silver metal, condensed at low but realistic consolidation pressure, before addressing the issue of strengthening the restorative material.

- Acid-assisted consolidation continues to be a critical factor in the development of the metallic substitute for amalgams.

- Developed a process to precipitate silver powder (0.3μm-3.0 μm) via two solution technique as a result of Sn^{2+} to Sn^{4+} oxidation.

- Determined that the annealing (450 °C - 2 hours) of silver powders prior to consolidation resulted in doubling the transverse rupture strength of consolidated samples compared to unannealed powders.

- 75-80% of the theoretical density for silver was obtained on precipitated silver compacts using acid-assisted hand consolidation with normal dental tools.

- Current state of the art for precipitated silver compacts, prepared by acid-assisted hand consolidation with normal dental tools, have transverse rupture strengths of 110MPa-140 MPa compared to 115 MPa for a dental amalgam.

- The range of rupture strengths indicate sensitivity to the consolidation technique which includes different personnel, consolidation pressure, and tip sizes and geometries of hand condensers.

Impacts and Technical Highlights:

- With the contention that feasibility has been demonstrated for the development of a silver-based, mercury-free restorative material, NIST has transferred the current state of the art to our CRADA partner, Dentsply/Caulk, for in-house evaluation.

Project Title: COATING THICKNESS CALIBRATION STANDARDS

Investigators: Carlos R. Beauchamp, Hilary B. Gates, David R. Kelly, and Jasper L. Mullen

Technical Description:

These standards consist of pre-configured sets of coupons of fine grained copper with thickness ranging from 2.5 μm to 2 mm, that 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 and are used by the organic and inorganic coating industry for the non-destructive measurement of non-magnetic coatings over magnetic substrates.

Technical Objectives:

- Total re-evaluation of the production and certification of the Coating Thickness Calibration Standards to accommodate advances made on the precision of the instruments employed in the laboratory and in the field.
- Bring the project in compliance with the present policy for reporting uncertainties associated with measurements executed at NIST.
- Manufacture and certification of replacement Primary Calibration Standards employed in the production process.

Anticipated Outcome:

- Increased efficiency during the production and certification stages of the Standard Reference Materials (SRM).
- Reduced uncertainties due to changes made in the fabrication and certification stages of the standard.
- Replacement of the Primary Standards used during the certification process.

Accomplishments for FY 1995:

- The effects of thickness, composition, surface roughness and mechanical working of the substrate material on the uncertainties associated with the thickness measurement have been evaluated during the material selection for the production of these standards.

- Stricter control over the cell geometry and electrode configuration during the electrodeposition stages now yield uniform standards with less than 1% deviation from the average thickness values.

- Proper match of probe sensitivity to the thickness measured, increased resolution during probe positioning, coupled with the elimination of false triggering, reduced degrees of freedom during human operations, and a different mathematical model to fit the probe responses during the calibration modes have reduced the uncertainties of the certification process.

- The Primary Standards have been fabricated and await the certification of their thickness.

Impacts and Technical Highlights:

- The first major revision of the Coating Thickness Calibration Standards SRM's 1357 through 1364a nears its completion and is expected to reach customers before the fiscal year 1996 is over and remaining stock of previous standards is consumed.

- Uncertainties associated with the standard have been minimized allowing greater leeway during the selection of the measurement spot by the end user.

- To better serve the customers of the standard, the thickness distribution offered within the pre–configured sets is being tailored in response to a survey presented to the users of the standard, inquiring about the thickness ranges of their present applications and the uncertainties associated with their measurement protocol.

Project Title: CORROSIVITY OF HALON REPLACEMENT FLUIDS

Investigators: Mark R. Stoudt, Richard E. Ricker, James L. Fink, and James F. Dante

Technical Description:

It has been determined that one of the most commonly used fire suppressants, halon, has sufficient ozone depleting potential that its production and use should be restricted. Non-ozone depleting alternatives to halon could be sufficiently corrosive to cause corrosion failure of fire suppressant storage and distribution systems. The objective of this project is to assist industry's transition from halon to other non-ozone depleting fire suppressants by evaluating the corrosivity of chemical compounds that could be used for fire suppression in place of halon.

Technical Objectives:

- To evaluate the propensity for failure of typical storage system alloys as a result of exposure to any one of the replacement candidate chemicals during service or after deployment.

- To determine the corrosion rates over a broad range of conditions as well as evaluate the susceptibility the alloys to stress corrosion cracking, pitting and crevice corrosion.

Anticipated Outcome:

- If a suitable replacement can be identified for halon 1301, it will significantly reduce the rate of stratospheric ozone depletion without compromising public safety.

- Identification of chemical compounds that should not be developed further as a replacement for halon will save industry and government agencies considerable time and money by allowing them to focus on usable alternatives.

Accomplishments for FY 1995:

- Completed long term testing in each of the halon alternatives selected for further evaluation.

- Performed in-depth statistical analyses of the data from immersion experiments and incorporated those into a predictive model. The model can be used to estimate the corrosion rates in these chemicals.

- Two papers reporting the results of this work were completed and submitted for publication.

Impacts and Technical Highlights:

- Since the corrosion behaviors of these chemicals are not well known, the data and predictive models produced by these evaluations will be of considerable value to the Air Force, the Federal Aviation Administration (FAA), the Envirmental Protection Agency (EPA) and to aircraft system designers as halon 1301 is scheduled to be completely removed from service in the next few years.

- Several compounds which were being considered for development as an alternative to halon have been eliminated from consideration allowing industry to focus on viable candidates, thereby reducing their costs for developing halon alternatives.

Project Title: PRESSURIZED GAS CONTAINERS

Investigator: John H. Smith

Technical Description:

Many industries use metallic cylinders for the storage and shipping of a wide range of different types of gasses from hazardous gasses to breathing air. Occasionally, these cylinders fail releasing their contents and injuring personnel. The purpose of this project is to evaluate the structural integrity and safety of metal and composite high pressure gas cylinders and to develop improved design standards and testing procedures for the cylinders.

Technical Objectives:

- Develop the technical basis for safety regulations governing the design, manufacture, and testing of new, advanced high pressure gas cylinders.

- Develop test methods and fracture resistance acceptance criteria for high strength steel cylinders.

- Evaluate sustained load cracking in aluminum cylinders.

- Develop design standards and test methods for full composite cylinders.

Anticipated Outcomes:

- Improved safety and reduced liability costs associated with the storage and shipping of industrial gasses.

- Improved public safety and increased use of compressed gasses by the general public including compressed natural gas for public and private transportation.

Accomplishments for FY 1995:

- Interactions with the compressed gas industry through the Compressed Gas Association have resulted in the U.S. technical standards governing the design and manufacture of high pressure steel cylinders being significantly updated and consolidated.

- Through participation in the Cylinder Design committee of the International Organization for Standardization (ISO) a new standard for the design and construction of high strength steel cylinders has been adopted.

- The technical basis was developed to support changes in the present standards for the manufacture of aluminum cylinders to prevent failures that have occurred due to sustained-load-cracking.

- Summary report and a failure analysis report on sustained-load-cracking in aluminum cylinders has been prepared.

- A draft of revised technical standards for the design and construction of steel cylinders.

Impacts and Technical Highlights:

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

- The adaptation of a single ISO worldwide standard for high strength steel cylinders permits the U.S. manufacturers to produce steel cylinders for worldwide use.

Project Title: TEARING RESISTANCE OF AL ALLOYS

Investigators: Roland deWit and Richard J. Fields

Technical Description:

Sudden catastrophic failure of an aging aircraft may result from the linking up of corrosion and fatigue damage at rivet holes along the fuselage skin lap joint as occurred in a Boeing 737 operated by Aloha Airlines in 1988. To help the commercial aircraft and airline industries avoid these failures, a project was designed which would quantify the crack propagation process and mechanics of crack linkup at multiple site damage (MSD) for thin-sheet aluminum alloys which are prototypic of those used in airplane fuselage design.

Technical Objectives:

- The objective of this research project is to assist the aircraft industry by providing data for evaluating the possibility of sudden catastrophic failure of aging aircraft with short fatigue cracks emanating from rivet holes.

Anticipated Outcome:

- The major outcomes are expected to be the development of accurate calculations of the residual strength of an aluminum sheet containing multiple cracks and multiple site damage.

- The improved understanding of the cause of sudden catastrophic failure of aircraft that will result from this research will enable the commercial aircraft industry and the airline industry to design, operate, and maintain aircraft in a safer more cost effective manner.

Accomplishments for FY 1995:

- The experimental work was completed during the fiscal year.
- Three papers on this work were prepared and submitted for publication.

Impacts and Technical Highlights:

- The FAA Center of Excellence in Computational Modeling of Aircraft Structures at Georgia Institute of Technology is using the results of this work to develop a methodology to predict the residual strength of structures containing widespread fatigue damage.

Project Title: FULL-THICKNESS CLAD-BEAM FRACTURE TOUGHNESS TESTS

Investigators: Roland deWit and Richard J. Fields

Technical Description:

Currently, industry predicts the fracture resistance of large structures and pressure vessels from measurements made on small samples with relatively large or deep flaws. Currently, industry uses scaling theories, that are controversial and mostly untested, to account for the difference in size between the sample and the structure. The goal of this project is to use NIST's unique 52MN (12-million lbf) testing facility to provide industry with test data on the fracture resistance of a pressure vessel wall with small flaws representative of actual service conditions. To accomplish this objective, samples 225mm wide (8.8in), 225mm thick (8.8in) and >1.2m (> 47in) long were cut from a nuclear reactor pressure vessel wall and realistic (shallow) flaws were created in the samples. Then, the fracture toughness was determined for the samples with these well characterized flaws. The results of these measurements will be used to evaluate new and existing scaling theories.

Technical Objectives:

- This research project is designed to assist the nuclear industry by providing a data base for evaluating controversial scaling theories for fracture toughness to assess the life of existing reactor pressure vessels.

Anticipated Outcomes:

- This research will enable industry to more accurately predict if a given flaw will cause catastrophic failure of pressure vessels such as nuclear and chemical reactors and this will result in improvements in the reliability and safety of nuclear reactors as well as any component where full-size testing is difficult or expensive.

Accomplishments for FY 1995:

- The tests were completed and the results reported to Oak Ridge National Laboratory for subsequent analysis.

Impacts and Technical Highlights:

- Oak Ridge National Laboratory is analyzing the results of these tests to enable the safer and more cost-efficient operation of nuclear reactors, but the impact will be beyond this industry to include any industry or government agency concerned with the performance of large structures where full-scale testing is impractical.

METALS PROCESSING

Program Overview

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, grains, 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 already have been achieved, notably in interactions with the aerospace, powder metallurgy, and electronic industries. For example, cooperative research and development projects with industry (Crucible Compaction Metals, Fluxtrol Manufacturing) have resulted in significant improvements in process control for steel and superalloy powders. Moreover, 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, thin films, intermetallic alloys and electronic materials. Frequent interactions and collaborations with industrial producers are maintained, including participation in industrially-oriented consortia and the American Society for Testing and Materials (ASTM). Standard reference materials, such as the homogeneous standard reference materials currently being produced by rapid solidification, which are unobtainable by other means, are another important output of the current work.

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 this purpose, 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), Department of Defense (DoD)/ARPA (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 goal is to help U.S. industry apply measurements and predictive modeling to produce improved materials at reduced cost.

For additional information about the Metals Processing Program, contact John R. Manning at 301-975-6157 or e-mail to manning @enh.nist.gov.

Project Title: MICROHARDNESS STANDARDS

Investigators: Christian E. Johnson, David R. Kelley, Ed Escalante, Sandra Claggett, and Perry Sharpless

Technical Description:

These Standard Reference Materials (SRM) are intended primarily for use in calibrating Knoop and Vickers-type microhardness testers and are certified for average hardness values at 0.245 N, 0.490 N, 0.980 N, 2.94 N, 4.90 N, 9.80 N (25 g-f, 50 g-f, 100 g-f, 300 g-f, 500 g-f, 1000 g-f). This project involves the production/certification of copper and nickel standards as well as the development of a new gold standard.

Technical Objectives:

- Develop an electrodeposited gold microhardness standard having uniform hardness through the coating thickness.

- Develop a gold electrolyte from which resulting electrodeposits would have microhardness values in the range on 70 kg/mm^2-90 kg/mm^2.

- Production of 50 SRM 1906 Nickel Microhardness Standards having uniform nominal hardness of 600 kg/mm^2.

- Production of 25 SRM 1894 Copper Microhardness Standards having uniform nominal hardness of 125 kg/mm^2.

Anticipated Outcome:

- Technical feasibility will be demonstrated for the development of an electrodeposited gold microhardness standard having uniform hardness.

- Production of 50 SRM 1906 Nickel Microhardness Standards.

- Production of 25 SRM 1894 Copper Microhardness Standards.

Accomplishments for FY1995:

- Electrodeposited pure gold (24K) was found to have non-uniform microhardness value through a thickness of 100 µm due to an increase in grain size with thickness.

- A hard gold electrolyte was modified to obtain microhardness values in a range of 70 kg/mm^2-90 kg/mm^2 on resultant electrodeposits.

- Uniform microhardness values were obtained through the 100 µm thickness of electrodeposits obtained from the modified hard gold electrolyte due to the uniform microstructure of small equi-axed grains.

- Production of 50 SRM 1906 Nickel Microhardness Standards.

- Production of 25 SRM 1894 Copper Microhardness Standards.

Impacts and Technical Highlights:

- Feasibility was demonstrated that a (100 µm-125 µm) uniform gold electrodeposit could be developed as a microhardness standard.

- These SRM would satisfy a need in industry for a standard to verify microhardness testers at nominal hardness of 90 kg/mm^2, 125kg/mm^2, and 600 kg/mm^2.

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, F. W. Gayle and J. S. Adams

Technical Description:

Advanced powder metallurgy processing methods are being evaluated to determine the effect of process measurements and control on high performance materials. The current studies in this area are an outgrowth of a highly successful NIST/Industry Consortium project where intelligent processing (measuring and modeling) techniques were applied to production of rapidly-solidified alloy powder by controlled atomization. Homogeneity advantages provided by rapid solidification are being used to produce improved standard reference materials. Effects of rapid solidification on powder size, grain size, and properties of nitrogenated stainless steel are being evaluated and kinetic and thermodynamic models are being investigated to explain, predict, and optimize these rapid solidification effects. The measurements and process models being developed will provide guidelines for designers of industrial processes. Property measurements include hardness, ultimate tensile strength, yield strength, elongation, pitting potential, resistance to crevice corrosion, and stress corrosion cracking.

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.

- 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 intermetallic phases.

- Investigate possible synergism between ultra fine grain size and solute strengthening effects in providing improved mechanical and corrosion properties. Develop an improved method of measuring these grain sizes.

- Apply powder metallurgy models to aid in studies of powder consolidation by hot isostatic pressing (HIP) at temperatures greatly reduced from those normally used.

Anticipated Outcome:

- Availability of more homogeneous standard reference materials.

- Development of improved measurements and process models for producing alloys with greatly improved properties.

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

- Development of guidelines for achieving powder consolidation by hot isostatic pressing at much lower temperatures than those normally used, thus improving alloy properties and reducing costs.

Accomplishments for FY95:

- Developed methods for producing iron-and nickel-based standard reference materials with greatly improved homogeneity.

- Applied process models, kinetic measurements, and phase diagram data to establish methods for predicting nitrogen solubility and phase stability in atomized and consolidated stainless steel alloys. Validated predictive models by producing stainless steel alloys with enhanced phase stability at high nitrogen contents.

- Combined nanostructure research with atomization to produce nitrogenated stainless steel test samples with grain sizes as small as 72 nm, as determined by x-ray diffraction, and very high tensile strength, estimated at 2137 Mpa (310 ksi) from Vickers hardness measurements. Developed method for correcting systematic errors in x-ray diffraction measurements of grain sizes in these materials.

- As an outgrowth of a project on powder consolidation done in cooperation with the auto industry, showed that use of nanostructure steels allowed HIP consolidation at 700 °C, a 400 °C-500 °C reduction from current industrial powder metallurgy practice.

Impacts and Technical Highlights:

- Melt practice developed at NIST for atomization applications is currently being incorporated by industrial producers into their metals processing.

- Nitrogenated stainless steel test specimens produced at NIST by atomization and HIP consolidation showed excellent properties for use as extremity and body armor in a test report by Sandia National Laboratories. Similar material is currently being evaluated by the U.S. Army for tank and armored personnel carrier armor.

Project Title: SOLIDIFICATION MODELING

Investigators: Sam 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 effect of fluid flow on the breakdown of the crystal-melt interface from planar to nonplanar cellular shapes, as well as 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 for application to microgravity directional solidification research including: the initial and final transients of a finite length sample, the effect of diffusion in the product liquid phase on monotectic composite solidification, and the effect of fluid flow on the formation of macrosteps and associated solute nonuniformities in crystals with anisotropic growth kinetics.

- Perform numerical analysis of melt convection due to thermal and solute gradients for the growth of lead bromide-silver bromide crystals.

- 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 the absence of melt convection during solidification. These include experiments on directional solidification of bismuth-tin alloys at the University of Florida and the growth of monotectic composite alloys of aluminum-indium at the University of Alabama in Birmingham.

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

- Supercomputer calculations of a single dendrite will permit construction of improved approximate models for equiaxed castings that will lead to improved mechanical properties.

Accomplishments for FY 1995:

- The concentration profiles obtained in the initial and final transients were simulated allowing a more accurate prediction of microsegregation. Diffusion in the product liquid phase of a monotectic composite has little effect on the composite spacing. For crystals growing by the motion of steps, a shear flow in the direction of the step motion destabilizes the interface and causes solute inhomogeneities.

- Growth conditions to avoid melt convection due to temperature and solute gradients were computed for lead bromide-silver bromide crystals.

- Simulations of alloy dendritic growth using the phase-field, diffuse interface, numerical technique showed the effect of recalescence (reheating due to latent heat release) on the microsegregation pattern.

Impacts and Technical Highlights:

- Knowledge gained from experiments and analysis of faceted Bi-Sn alloys will provide increased understanding of faceted materials in general, such as semiconductors.

- Numerical calculations of the onset of fluid flow during the growth of lead bromide-silver bromide crystals have provided guidance to the Westinghouse Science and Technology Center on the best processing conditions for this non-linear optical material.

- Many international scientific groups have begun to utilize the phase field methods developed at NIST for the simulations of dendritic growth (Nature 375 (1995) 103).

Project Title: MODELING AND SIMULATION OF MATERIALS PROCESSING

Investigators: J. A. Simmons, L. A. Bendersky, W. J. Boettinger, J. W. Cahn, J. R. Manning and J. A. Warren

Technical Description:

The focus of this work is to identify areas of process technology where quantitative modeling can lead to the production of improved materials at reduced cost. The project was initiated to foster interactions between mathematical modeling and materials science with emphasis on the development of new mathematical techniques. It involves extensive interactions with mathematicians and computational scientists outside of NIST funded by the ARPA Computational and Applied Mathematics Program and includes, as an important aspect, the organization of workshops and symposia to enhance the dialog between mathematicians and material scientists. With the advent of large scale parallel processing, computational modeling of material microstructure development as a function of processing conditions offers a promising method of predicting process results. Because understanding of materials science is becoming more quantitative and computational capabilities are becoming more profound, 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 alloy solidification, solid-state diffusive transformations, and incorporation of stress effects. This work is now being redirected to focus on thin film technology.

Technical Objectives:

- To involve mathematicians in the development of new mathematics needed to treat practical materials science problems.

- To model effects arising from complex conditions that arise in real materials processes, including ordering processes, thin films, and facetted surfaces, where discontinuities abound.

Anticipated Outcome:

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

- Improved interactions will be developed between the materials and mathematics communities to facilitate cooperative efforts even beyond the direct topics treated in this project.

Accomplishments for FY1995:

- Developed geometric measure theory important for understanding the effect of anisotropic surface energies on interface facetting during both solidification and grain growth. Microstructural facetting strongly influences interface properties.

- Developed and applied new Master Equation techniques to model ordering processes in alloys. Time sequences were obtained showing the growth of ordered domains, which are crucial in the development of alloy properties.

Impacts and Technical Highlights:

- Mathematical models and comparisons with experiment developed in the current work have verified assumptions being incorporated by the ARPA-funded industrial Investment Casting Cooperative Arrangement into commercial software for casting design and the NIST-sponsored Consortium on Casting of Aerospace Alloys in models for processing aerospace materials.

- A symposium on "Mathematics of Thermodynamically Driven Microstructural Evolution", Oct. 29-Nov 1, 1995, TMS/ASM Materials Week Meeting, Cleveland, Ohio was organized as part of this project, bringing together mathematicians and material scientists to focus on this topic. The symposium was jointly sponsored by ASM International, the Society of Industrial and Applied Mathematics (SIAM), The Minerals, Metals and Materials Society (TMS), Advanced Research Projects Agency (ARPA), and NIST.

Project Title: ELECTRODEPOSITION OF ALLOYS

Investigators: Gery Stafford, Carlos Beauchamp, Ed Escalante

Technical Description:

Electrogalvanized steel utilizes a variety of coatings, ranging from simple elements to rather sophisticated alloys. Coating thickness and composition standards have failed to keep pace; consequently there is little accountability among most sheet manufacturers both in the U.S. and abroad. Our objective is to develop the electrochemical expertise that will enable us to produce coating thickness (certified as mass/area) and composition standards of electrogalvanized coatings, starting with Zn and Sn and eventually the zinc-based alloys. These will be used by the steel industry to calibrate on-line x-ray fluorescence instruments for process control of continuous strip plating.

Technical Objectives:

- Develop prototype large panel thickness standards of Zn and Sn on 1010 steel. Coatings should consist of randomly oriented grains which are 5 μm-10 μm in size. Deposit thickness should be on the order of 1 μm to 10 μm.

- Identify the zinc alloys that are currently being used by the electrogalvanizing community. Determine composition, structure and level of uncertainty required by the industry.

Anticipated Outcome:

- Develop SRM prototypes for zinc and tin on steel. In addition, we expect to add to the science base for the electrodeposition of these metals.

Accomplishments for FY 1995:

- Demonstrated the electrodeposition of zinc on 1010 steel. Deposits of highest quality were obtained using a zinc sulfate electrolyte (pH=1.5) and a current density of 50 mA/cm^2.

- Succeeded in producing 10 cm x 15 cm coupons of zinc on 1010 steel. Target thicknesses of 3 μm to 8 μm were obtained with thickness variation within a coupon limited to 1.5%. Orientation was an acceptable 55% pyramidal.

- Identified two zinc alloys which will be the focus of future work. Zn-Fe (15-25 atomic % Fe) has good corrosion resistance and weldability. Zn-Ni (10-16 atomic % Ni) is by far the most popular, having exceptional hardness, good corrosion resistance and formability.

Impacts and Technical Highlights:

- We have demonstrated that zinc and tin can be electrodeposited on a 10 cm x 15 cm coupon of 1010 steel within the thickness and variability limits put forth by the continuous strip plating industry.

METALS DATA AND CHARACTERIZATION

Program Overview

Basic data describing the properties of metals and advanced materials based on metals form an important component of the technological infrastructure which NIST supports. Information of this kind is essential to the understanding of the behavior of metals in different conditions and to the effective design of structures and devices containing metals. The measurement base is an MSEL mission strategic thrust.

This program is focussed on the development of techniques to measure various properties of metallic materials and the acquisition of data of technological significance. It includes activities involving measurements of mechanical, magnetic, and optical properties which impact a number of different technology sectors. For instance, in collaboration with the Copper Development Association, the mechanical behavior of lead-free plumbing solders is being investigated to provide the comprehensive and reliable database needed to establish internal working pressure standards for drinking water pipe joints. In contrast with these traditional measurements involving a widely used material, high speed optical techniques are being developed in another project to measure selected thermophysical properties of solid and liquid materials at high temperatures. The goals of this work are to generate accurate bench-mark data on selected key materials and to develop new high-temperature thermophysical standards. In other 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. The goal of this work is to develop lightweight materials for automotive applications. In the area of characterization, a technique is being developed which visualizes stress fields in opaque materials using the acoustic microscope. Work in the past year has included collaborations with DuPont, Libby Owens Ford, and Sonix.

For additional information about any aspect of the Metals Data and Characterization Program, please call Steve Hardy, 301-975-6159, email - shardy@micf.nist.gov

Project Title: CORROSION FATIGUE OF DUPLEX STAINLESS STEELS

Investigators: Mark. R. Stoudt, Richard E. Ricker

Technical Description:

Large duplex stainless steel cylinders called "suction roll shells" are used in the processing of pulp to make paper. The service life of a typical suction roll shell is 5 to 8 years and it costs several hundred thousand dollars to replace one. Working with the only U.S. manufacturer of suction roll shells, Sandusky International, NIST is designing and conducting experiments which should lead to the development of a more fundamental understanding of the causes of these failures and metallurgical changes that could lead to an increase in the service life of these components.

Technical Objectives:

- Develop electrochemical measurement techniques and evaluate the electrochemical properties of the alloys used in the construction of suction roll shells in typical pulp and paper process environments and the influence of chemical composition and microstructure on corrosion.

- Develop experimental techniques for the measurement of corrosion fatigue crack propagation at low velocities in the pulp and paper process environment.

- Develop experimental techniques for studying corrosion fatigue crack initiation in pulp and paper process environments.

- Apply these experimental techniques to develop a fundamental understanding of the relationships between microstructure and performance of duplex stainless steels in the pulp and paper process environment.

Anticipated Outcome:

- Sandusky International will use the experimental methods, database and knowledge gained from their cooperation with NIST in this CRADA to develop suction roll shells with longer service life significantly reducing the cost of producing paper and strengthening their competitive position on the international market.

- Chemical species brought into the process environment from recycled paper may be contributing to reducing the service life of suction roll shells. As a result, development of alloys more resistant to this failure may result in promoting recycling of paper products.

- Reducing the cost of paper products and increased recycling will increase the use of paper products and influence many industries beyond the pulp and paper industry and their equipment manufacturers.

- The knowledge and the experimental techniques developed for this industry and environment can be used to help other industries where corrosion fatigue limits the service life of their components.

Accomplishments for FY 1995:

- Designed and began fabrication of a system for measuring corrosion fatigue crack propagation in a simulated pulp and paper process environment.

- Working with Sandusky International, designed a series of experiments for evaluation of the influence of microstructure on corrosion fatigue crack propagation.

- Conducted preliminary evaluation of the electrochemical behavior of the duplex stainless steel currently used for the construction of suction roll shells.

Project Title: MECHANICAL BEHAVIOR OF PLUMBING SOLDERS

Investigators: Roger Clough, Richard Fields, Donald Harne, George Hicho, and David Pitchure

Technical Description:

In 1993, the Copper Development Association (CDA) approached NIST with a problem. Lead-free solders will be required for plumbing in new building construction, but no one knows if these solders will perform as well as traditional lead-containing alloys. Because there is little data on the fracture and creep resistance of joints soldered with these new alloys, the maximum safe operating pressure for plumbing systems using these new solders cannot be determined and put into building procedures and codes. Working with CDA, NIST designed and is conducting tests that will provide the information required to establish safe plumbing codes for these new solders.

Technical Objectives:

- To determine the shear strength at room temperature and creep properties in shear at mildly elevated temperatures (38 °C - 139 °C) in air of copper tubing whose ends are joined by sleeves using lead-free solders.

- To assist industry, through the CDA, in establishing pressure ratings for the new lead-free solder joints in copper tube planning.

- To develop a better understanding of the mechanisms of delayed shear fracture of copper/solder joints.

Anticipated Outcome:

- Allow cost savings to the multi-billion dollar building construction industry by using maximum safe pressure ratings for the new solders.

- Prevent environmental degradation by enabling the use of lead-free solders.

- Development of new copper pipe plumbing codes.

Accomplishments for FY95:

- Creep equipment has been purchased, installed, and instrumented for computerized test monitoring.

- Approximately half of the proposed tests have been completed and analysis of these fractures indicates that the percentage of the joint wetted and joined with solder is a critical variable in determining actual performance of joints.

- Three test methods have been implemented and are being evaluated to determine:
  - Pre-test acoustic power transmission
  - Pre-test sample load compliance
  - Post-mortem optical fractography

Impacts and Technical Highlights:

- Millions of plumbing joints are made each year in the construction of houses, office buildings and high-rise apartments. In terms of shear volume of solder, the building industry's use of solder is substantial compared to that of the electronics industry. Cost savings can be appreciable if the required introduction of the new lead-free solders does not require the use of more expensive soldering techniques or the use of more expensive plumbing materials by the multi-billion dollar building construction industry. These test results will permit this industry and its regulators to optimize performance and safety.

Project Title: LIGHTWEIGHT MATERIALS FOR AUTOMOTIVE APPLICATIONS

Principal Investigators: Richard J. Fields, Roger B. Clough, and Anna C. Fraker

Technical Description:

Reducing the weight of automobiles will reduce fuel consumption and pollution without degrading performance. Many automotive components which are made of steel, could be replaced by components made from less dense metals or metal matrix composites. The purpose of this investigation is to examine the properties of these materials that determine their performance during fabrication and service. Currently, this project consists of two activities: one focused on understanding the performance of particle reinforced aluminum (PRA) metal matrix composites during press-and-sinter processing and another that examines the performance of magnesium and magnesium matrix composites in the corrosive automotive environment. These were selected to address the technical barriers inhibiting the selection and use of these two materials: the cost of producing PRA composites and the corrosion of components made from magnesium.

Technical Objectives:

- Develop models for the press-and-sinter and powder forge processes for metal matrix composites.
- Validate powder forging models for commercial aluminum matrix composites.
- Assist industry in applying these models to accelerate die design and optimize preforming/forging conditions.
- Provide reproducible electrochemical corrosion testing procedures for active metals which will yield results representative of automotive service.
- Provide mechanistic information for understanding electrochemical behavior in magnesium alloys which will enable the development of more corrosion resistant alloys by industry.

Anticipated Outcome:

- This work will enable the automotive industry to produce automobiles which are lighter and more fuel efficient without sacrificing performance.
- This work will help U.S. producers of metal matrix composites and Mg alloys by removing the technical barrier inhibiting the use of their products by automobile manufacturers.

Accomplishments for FY 1995:

- Unique powder test equipment procured and setup.
- Collaborations with USCAR, ALCOA, and Cambridge University established.
- Macromechanical model for MMC compaction proposed.
- Established a reproducible electrochemical test method.
- Four papers reporting the results of this work were prepared and submitted for publication.

Impacts and Technical Highlights:

- Provided data evaluations demonstrating the effectiveness of a protective coating on a magnesium alloy to industry. Luke Engineering and Mfg. Co. is using these results to help them identify applications for their products.

Project Title: THERMOPHYSICAL PROPERTIES

Investigators: Ared Cezairliyan, Tsuyoshi Matsumoto, John McClure, Louis Phillips and Daniel Josell

Technical Description:

This project is involved with the development of new techniques for the accurate measurements, 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 bench-mark data on selected key materials.

- Development of high-temperature thermophysical standards.

Accomplishments for FY 1995:

- Completed development of a novel high-speed laser polarimetry technique for measurements of normal spectral emissivity of metals and alloys at high temperatures (up to the melting region) in millisecond-resolution pulse-heating experiments.

- Validated operation of the new technique by performing simultaneous measurements of normal spectral emissivity (at 633 nm) with the new laser polarimetry technique and a conventional method involving spectral radiometry.

- Obtained accurate data on normal spectral emissivity (at 633 nm) of molybdenum and tungsten in the temperature range 2000 K - 2800 K.

- Introduced significant improvements (automation) in the millisecond-resolution system by incorporating a new computer-controlled, fast-acting, high-current, solid-state switch.

- Modified existing laser pulse thermal diffusivity measurement system to permit delivery of very short pulses (50 ns). Conducted preliminary experiments on pure molybdenum and thin multilayered specimens composed of molybdenum and alumina in order to assess operational characteristics of the system. Prepared computer programs to process the experimental data and analyze the thermal diffusivity results.

Impacts and Technical Highlights:

- NIST developed 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.

- 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.

Project Title: TITANIUM ALLOYS FOR IMPLANTS

Investigator: Anna C. Fraker

Technical Description:

Biomedical implant reliability depends largely on the corrosion, wear, and fatigue resistance of the materials used to make the implant. This project examines the techniques used to characterize the influence of composition, microstructures, processing, and surface preparation on the performance and durability of metallic biomaterials and the application of these techniques to the characterization of new alloys for use in implants. This project also participates in standards development for medical materials and devices.

Technical Objectives:

- Determine the electrochemical behavior of materials developed for aerospace and defense applications to determine the suitability of these materials for use as surgical implants.

- Prepare a database that would eventually go to the Food and Drug Administration (FDA) on all aspects of these materials, including heat treatment, mechanical properties, corrosion behavior, biocompatibility and other properties.

- Interact with Howard University and George Washington University in studies of coupled metals in dentistry and materials used in the spine and neurosurgery, respectively.

- Participate and provide data for the development of standards for beta titanium alloys and for updating existing standards.

Anticipated Outcome:

- Technology transfer of an aerospace alloy to health use by alloy modification and characterization. Data on biocompatibility, mechanical properties, corrosion and suitability for surgical implants is being provided to the Food and Drug Administration (FDA).

- Participation in the ASTM F-4 Committee on Medical and Surgical Materials and Devices to develop standards and specifications for titanium alloys that are being considered for use as surgical implant materials.

Accomplishments for FY 1995:

- Data showing electrochemical behavior and the importance of microstructures in a beta titanium alloy (Ti21SRx) were provided to the consortium for inclusion with data from mechanical tests and biocompatibility tests that eventually will go to the FDA.

- Provided information on corrosion testing of implant materials to the FDA for use in standard test method development in the International Organization for Standardization (ISO).

- Summarized mechanical properties, microstructures, effects of heat treating and corrosion behavior of titanium alloys for surgical implant use.

- Reported on corrosion studies of dental casting alloys coupled with titanium.

- Prepared a review of implant materials and the use of ASTM standards.

Impacts and Technical Highlights:

- The results of this work are being used by a biomedical implant consortium consisting of titanium alloy producers and biomedical implant producers to evaluate alloys for use in implants and, eventually, will be included in the submission of the FDA.

Project Title: MAGNETIC MATERIALS

Investigators: R. D. McMichael, L. H. Bennett, M. J. Donahue, D. E. Mathews, H. J. Brown

Technical Description:

A wide variety of complementary techniques are brought together to study the details of magnetic structures and magnetization processes in metals. For example, surface magnetic structures can be imaged directly with techniques such as magnetic force microscopy (MFM) and the magnetic indicator film technique (MOIF). Interpretation of such images and extrapolation to interior structures can be accomplished with micromagnetic modeling, which uses bulk magnetic measurements (VSM and Barkhausen signal measurements) as auxiliary inputs.

Technical Objectives:

- Accurately determine microstructural defects affecting both magnetic and material properties.
- Develop an understanding of the role of manufacturing processes on magnetic properties.
- Image dynamic magnetization behavior, e.g., Barkhausen effect and magnetic creep.
- Develop micromagnetic computer codes with experimental verification.
- Perform experiments and computer simulations on the influence of metallic structures on magnetic field distributions surrounding the structures.

Anticipated Outcome:

- Improved performance of commercial magnetic recording devices and sensors.
- More accurate micromagnetic models as a result of better input data and experimental verification.
- Standardization of MFM and MOIF techniques.

- Improved database of magnetic material properties.
- Improved magnetic resonance imaging (MRI).

Accomplishments for FY 1995:

- Successful MOIF and MFM imaging of magnetization processes and structures in thin films and multilayers.
- Development of a 2D micromagnetic code applicable to thin film studies.
- Modeling of metallic parts in large applied fields led to the recognition that part geometry and eddy currents are particularly important factors controlling artifacts in MRI.

Impacts and Technical Highlights:

- MOIF and MFM results provide an initial benchmark against which future work can be compared.

Project Title: INTERMETALLICS

Investigators: Leonid A. Bendersky and Shinji Mitao (guest scientist from NKK, Japan)

Technical Description:

In this research program we initiated the study of factors affecting stability of materials having high density of interfaces. In particular, the study of initiation and development of discontinuous coarsening (DC) in lamellar structures of the technologically important novel TiAl-based alloys (gamma aluminides) was designed. Since the gamma aluminides are designed for structural heat resistant applications (e.g., for aircraft engines), the structural instability can be one of the factors that limits the application temperature. The research program includes both an experimental (TEM, SEM, electron channeling) and theoretical study.

Technical Objectives:

- Understanding the mechanisms of initiation of grain boundary instability in lamellar grains.
- Understanding the mechanisms of growth of highly anisotropic lamellar structures.

- Understanding factors affecting DC, including (a) presence of grain boundary allotriomorphs; (b) orientations of grain boundary with respect to the orientation of lamellae; (c) effect of ternary alloying.

- Modification of Livingston-Cahn kinetic equations for the case of anisotropic lamellae.

Anticipated Outcome:

- Equations of DC growth for the anisotropic case of gamma aluminides.

- Ability to predict the beginning and extent of microstructural changes by DC.

- Optimization of microstructure for minimal DC, and, therefore, for higher application temperatures.

- Fundamental understanding of migration of grain boundaries controlled by grain boundary diffusion.

Accomplishments for FY 1995:

- Continuous and discontinuous coarsening of Ti_3Al (α_2)/TiAl (γ) lamellae was studied for binary Ti-Al alloys containing atomic fractions of Al at 40%, 44% and 47%, at temperatures ranging from 900 °C to 1050 °C. Based on the experimental results, we were able to identify different mechanisms of DC initiation. Kinetics of the coarsening was analyzed using thermodynamic considerations of the chemical and interfacial driving forces derived from non-equilibrium in primary lamellae formed by Widmanstatten-type growth.

- Equations of growth of the DC grains were obtained for the anisotropic case of gamma aluminides by modifying Livingston and Cahn treatment.

- Three types of morphology in DC lamellar structure were identified and described. Appearance of these morphologies was predicted by the developed modified equations.

Project Title: DEVELOPMENT OF TOMOGRAPHIC SCANNING ACOUSTIC MICROSCOPY FOR THIN FILMS, THERMAL COATINGS AND COMPOSITES

Investigator: Ewa Drescher-Krasicka

Technical Description:

Ultrasonic Computer Tomography of Stress is based on comparison of the amplitudes of polarized modes propagating through stressed and unstressed areas in the sample and its interference. Although the theoretical solutions for longitudinal and leaky modes do not exist, such an effort is in progress in cooperation with J. R. Willis from the Department of Mathematics and Theoretical Physics in Cambridge. The first approach to this problem is to apply a surface leaky wave propagating partially in solid material and partially in liquid also known as an interface wave and scan the same areas with the different wave length (different frequencies). It is known that the penetration depth of a surface-leaky wave in solid is on the order of one wave-length. By changing the scanning frequencies one can observe different images of the distribution of stress averaged over the thickness equal to the wave length. Even without the existing solution one can experimentally calibrate the grey scale of image for the layer with known distribution of stress across the thickness and attribute certain shades of color to certain ranges of stress values. The surface leaky wave monitors the sum of the two principal stresses close to the surface if the principal stresses are acting along the surface. At present, the scanning acoustic imaging can not assess the values of residual stresses for an arbitrary orientation of the principal stresses acting in a sample.

Technical Objectives:

- Attempt a development of tomography based on the accumulated effects of the perturbations, with the object providing quantitative assessment of stress in a sample when this varies across the thickness (experimental approach-four point bending-cooperation with the Mechanical Engineering Department of Stanford University).

- For assumed (known) distribution of stresses, calibrate the intensity of images as a function of stress experimentally for the materials tested (aluminum, glass, inconel).

Anticipated Outcome:

- Experimental data of the distribution of stress in the plane of scan obtained by surface waves and comparison with the analytical or FEM model solutions for the few chosen problems (four point bending, diametrally compressed disk, uniaxial compression). The penetration depth of the acoustic waves defines the thickness of the "slice" of the material where the average value of stress is monitored.

Accomplishments for FY 1995:

- Obtained qualitative distribution of residual stresses in the sections of thick cylindrical pipe containing a weld, comparison with the DuPont experimental data from drill-hole technique.

- Obtained distribution of residual stresses in the spot-welded steel samples-solution for Ford Motor.

Impacts and Technical Highlights:

- NIST proved feasibility of ultrasonic technique of assessing the distribution of stress across the thickness of the sample.

- "Ultrasonic Computer Tomography of Stress," Patent Application filed May 1994. (pending).

Project Title: CORROSION DATA PROGRAM

Investigators: Charles P. Sturrock, Carole D. Flanigan, and Bijan 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. 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 over-riding 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 Outcome:

- Reliable materials performance informatics that 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 1995:

- Five materials advisory expert systems from the CHEM•COR series addressing the handling and storage of chemical process industry corrosives were released for public distribution in FY 1995. These expert systems were developed 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.

  CHEM•COR 8: Materials selection and performance for the handling and storage of once-through waters
  CHEM•COR 9: Materials selection and performance for the handling and storage of hydrogen fluoride
  CHEM•COR 10: Materials selection and performance for the handling and storage of dilute sulfuric acid
  CHEM•COR 11: Materials selection and performance for the handling and storage of nitric acid
  CHEM•COR 12: Materials selection and performance for the handling and storage of ammonia

- The first prototype of 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.

- Two software modules from the POWER•COR series addressing corrosion issues in the electric power industry were released for public distribution in FY 1995. These modules were developed with funding and corrosion expertise provided by the Electric Power Research Institute, a consortium of some 600 utility companies.

  POWER•COR 5: Corrosion in wet flue gas desulfurization systems
  POWER•COR 6: Corrosion in service water systems

- A variety of inductive learning methods were evaluated using three typical corrosion databases. 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.

- Two papers were prepared on this work and submitted for publication.

- One invited presentation was made on this work.

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.

Project Title: HARDNESS STANDARDS

Investigators: Richard Ricker, Sam Low, John H. Smith, and David Pitchure

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, the Standards Reference Materials program, the National Voluntary Laboratory Accreditation Program (NVLAP), the Statistical Engineering Division, 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.
- Interface 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 1995:

- Improved the repeatability of the NIST dead-weight standardizing hardness tester by a factor of two or three.

- Established U.S. national Rockwell hardness reference facility consisting of a computer controlled high precision dead-weight Rockwell hardness tester.

- Worked with a U.S. hardness block manufacturer to develop hardness test blocks for the Rockwell C scale which exhibit better hardness uniformity than any test block currently available. These test blocks will be used for the NIST SRM Rockwell standards.

- Acquired and characterized a group of Rockwell diamond indenters which meet the geometrical and performance criteria for NIST standardizing indenters.

- Experimentally determined the effects of indenter velocity and dwell times during the Rockwell C scale test cycle for the SRM test block material.

- Initiated and conducted hardness intercomparisons between NIST and the national hardness standardization laboratories of Japan and China.

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

- Three papers on this work were prepared and submitted for publication.

Impact and Technical Highlights:

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

- Equipment and materials for the production of Rockwell C scale standard reference materials were obtained, tested, and evaluated. Production of the Rockwell C scale SRM will begin in the next fiscal year and these hardness calibration blocks will be available.

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Pitner, W. R., Hussey, C. L., and Stafford, G. R., "Electrodeposition of Nickel-Aluminum Alloys from the Aluminum Chloride-1-Methyl-3-Ethylimidizolium Chloride room-Temperature Molten Salt,: J. Electrochem. Soc., (in press).

Stafford, G. R., Bendersky, L. A., and Janowski, G. M., "The Structure of Electrodeposited Al-Ti Alloys," Proc. of Symposium on Defect Structure, Morphology, and Properties of Vapor and Electrodeposits, TMS Fall Meeting, October (1994).

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

Stafford, G. R., Bendersky, L. A., Janovski, G. M., "The Structure of Electrodeposited Al-Ti Alloys," Proceeding of the Symposium on Defect Structure, Morphology and Properties of Deposites, TMS (1995) p. 231.

Thermophysical Properties

Cezairliyan, A., Krishnan, S., and McClure, J. L., “Simultaneous Measurement of Normal Spectral Emissivity by Spectral Radiometry and Laser Polarimetry at High Temperatures in Pulse-Heating Experiments: Application to Molybdenum and Tungsten,” Int. J. Thermophysics.

Josell, D., Warren, J. A., and Cezairliyan, A., “Analysis for Determining Thermal Diffusivity from Thermal Pulse Experiments,” J. Appl. Phys. 78, 6867 (1995).

Kaschnitz, E., and Cezairliyan, A., “Radiance Temperatures at 1500 nm of Niobium and Molbdenum at Their Melting Points,” Int. J. Thermophysics, (in press).

Titanium Alloys for Implants

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., American Society for Testing and Materials, West Conshohocken, PA (in press).

Chohayeb, A. A., Fraker, A. C., Eichmiller, F. C., 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 STP 1272, S. A. Brown and J. E. Lemons, eds., American Society for Testing and Materials, West Conshohocken, PA, (in press).

Magnetic Materials

Bennett, L. H., McMichael, R. D., Swartzendruber, L. J., Hua, S., Lashmore, D. L., Shapiro, A. J., Gornakov, V. S., Dedukh, L., Nikitenko, V. I., "Dynamics of Domain Structure in Magnetic Multilayers," IEEE Trans. MAG, 31, 4088 (1995).

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., "Magneto-optical indicator film observation of domain structure in magnetic multilayers," Appl. Phys. Letts., 66, 888 (1995).

Intermetallics

Ricker, R. E., "Origins of the Aqueous Corrosion and Stress Corrosion Cracking Behavior of Ductile Nickel Aluminide," Materials Science and Engineering A, A198, pp. 231-238, 1995.

Gayle, F. W., Biancaniello, F. S., "Stacking Faults and Crystallite Size in Mechanically Alloyed Cu-Co," Nanostructured Materials, 6, 1995, pp. 429-432.

Biancaniello, F. S., Gayle, F. W., Suryanarayana, C., Froes, F. H., "Control of BCC and FCC Phase Formation During Mechanical Alloying of Ti-Al-Nb," TMS Conference Proceedings, (in press).

Bendersky, L. A., Biancaniello, F. S., Williams, M., "Evolution of the Two-Phase Microstructure Ll_2 + DO_{22} in Near-Eutectoid $Ni_3(Al,V)$ Alloy," J. of Mater. Res., 9, 3068 (1994).

Bendersky, L. A., Biancaniello, F. S., Williams, M., "Ordering and Microstructural Evolution in Near-Eutectoid $Ni_3(Al,V)$ Alloy," Proceeding of the International Conference on Solid-Solid Phase Transformations in Inorganic Materials, TMS (1994), p. 899.

Mitao, S., Bendersky, L. A., "Coarsening Behavior of Lamellar Structure in Ti (40-47) Al Intermetallic Alloys," Proceeding of International Symposium on Gamma Titanium Alumindes, TMS (1995).

Rowe, R. G., Bendersky, L. A., "The Mechanical Properties and Microstructure of 0 + $B8_2$ Titanium Aluminide Alloys," to be published in TMS Proceeding (1995).

Bendersky, L. A., Stalick, J. K., Portier, R., Waterstrat, R. M., "Crystallographic Structures and Phase Transformations, in ZrPd," accepted for publication in J. of Alloys and Compounds.

Development of Tomographic Scanning Acoustic Microscopy for Thin Films

Drescher-Krasicka, E., "Scanning Acoustic Imaging of Stress in Solids," Proceedings of 29th Annual Conference of the Microbeam Analysis Society, Breckenridge, CO, 453-455, (1995).

Thermal Coatings and Composites

Josell, D., Wang, Z. L., "Determining Interfacial Free Energies from Creep Experiments on Silver-Iron Multilayers," Materials Research Society Symposium Proceedings 356, Thin Films: Stresses and Mechanical Properties V, Eds. S. P. Baker et al, 357 (1995).

Corrosion Data Program

Begley, E. F. and Sturrock, C. P., "Matching Information Technologies with the Objectives of Materials Data Users," Computerization and Networking of Materials Databases, Vol. 4, C. P. Sturrock and E. F. Begley, eds., American Society for Testing and Materials, Philadelphia, PA, 1995.

Hardness Standards

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, Germany, 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, Germany, 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.

Other Publications

Wheeler, N. S., "Microstructural Characterization of Cobalt-Tungsten Coated Graphite Fibers," J. of Research, NIST, 100, 6, (in press)

Wheeler, N. S., and Lashmore D. S., "Electrodeposited Cobalt-Tungsten as a Diffusion Barrier Between Graphite Fibers and Nickel Composites," J. of Composites Technology and Research, 17, 301-307 (1995).

Barnett, J. T., Clough, R. B., and Kedem, B., "Power Considerations in Acoustic Emission," J. of the Acoustic Soc. of America, Vol. 98, pp. 2070-2081, 1995.

Choi, C.S., Starke, W., Barker, J., and Fields, R. J.,"Neutron Diffraction Study of Austempered Ductile Iron," accepted for publication in Metall. Trans., 1995.

Feng, Z., Zhu, Y. Y., Zacharia, T., Fields, R. J., Brand, P. C., Prask, H. J., and Blackburn, J. M., "Modeling and Validation of Residual Stress Distribution in a HSLA-100 Disk," accepted for publication in Proceedings of the Fourth International Conference on Trends in Welding Research, ASM International, Metals Park, Ohio, 1995.

Fraker, A. C., "Medical and Dental Metals and Alloys," Corrosion Tests and Standards - Application and Interpretation, Robert Baboian, ed., American Society for Testing and Materials, West Conshohocken, PA, (1995) pp. 705-715.

Hicho, G. E., Brand, P. C., and Prask, H. J., "Determination of the Residual Stresses Near the Ends of Skip Welds Using Neutron Diffraction and X-Ray Diffraction Procedures," NISTIR 5671, Report to the Federal Railroad Adm., June 1995.

Clough, R. B. and Simmons, J. A., "Acoustic Emission," Encyclopedia of Advanced Materials, D. Bloor, R. Brook, M. Flemings, and S. Mahajan, eds., Pergamon Press, (invited article), 1994.

Cockman, J. R., Fields, R. J., Delph, T. J., and Harlow, D. G., "Spatial Statistics of Creep Cavities," J. of Modeling and Simulation in Materials Science, Vol. 3, pp. 187-200, 1995.

Ledbetter, H., Dunn, M., Kim, S., and Fields, R. J., "Void Shapes in Sintered Titanium," Rev. of Progress in Quantitative NDE, 14, pp. 1633-1639, 1995.

Ricker, R. E., "Can Corrosion Testing Make the Transition from Comparison to Prediction?" Journal of Metals, 47, 9, pp. 32-35, 1995.

Handwerker, C. A., Wallace, J. S., Foecke, T., Kattner, U. R., Blendell, J. E., "In-Situ Formation of Composite Microstructures," Journal of Microscopy, 179 (3) 286-296 (1995).

Handwerker, C. A., Foecke, T. J., Wallace, J. S., Kattner, U. R., Jiggets, R. D., "Formation of Alumina-Chromia-Chromium Composites by a Partial Reduction Reaction," Materials Science and Engineering, A195, 89-100 (1995).

Wang, Z. L., Shapiro, A. J., " Microstructure and Reconstruction of $LaAlO_3$ {110} surfaces," Proceedings of Spring MRS Conference, vol. 3.5 5, (1995) pp. 175-180.

Wang, Z. L., Shapiro, A. J., " Delocalization Effect in Energy Filtering Electron Images of Surfaces and Interfaces," Proceedings of Microscopy and Microanalysis, vol. 1995, pp. 264-65.

Wang, Z. L., Shapiro, A. J., "Steps Structure in 5x5 Reconstruction of Annealed $LaA10_3$ {100}," Proceedings of Microscopy and Microanalysis, vol. 1995, pp. 346-347.

INVITED TALKS

Electrochemical Processing Group

Johnson, C. E.

1. "Coating of Al-SiC Composite Thermal Management Components," Lanxide Electronic Components LP, Newark, DE, December 1994.

2. "Alternative Dental Restorative", NIDR Dental Student Research Conference, NIST, March 1995.

Moffat, T. P.

1. "Electrodeposition of Alloys," 2nd Annual Joint AESF/NIST Workshop on Electrodeposition, NIST, November 11, 1994.

2. "Electrodeposition of Cu-Ni Multilayers," Symposium on Nanostructured Materials in Electrochemistry, 187th Electrochemical Society Meeting, Reno, NV, May 24, 1995.

3. "Electrochemical Synthesis of Multilayer Materials," Workshop on Nanoscale Layered Materials for Protective Coating and Structural Applications, NSF-NIST, August 14, 1995.

4. "On the Passive State of Chromium Alloys," NACE Baltimore/Washington Section, October 20, 1994.

Stafford, G, R.

1. "Aluminum Alloy Deposition from Chloroaluminate Electrolytes," Gordon Research Conference on Molten Salts and Liquid Metals, Plymouth State College, Plymouth, NH, August 6-11, 1995.

2. "The Electrodeposition of Al-Ti Alloys from Chloroaluminate Electrolytes," Stevens Institute of Technology, Hoboken, NJ, November 16, 1994.

3. L. A. Bendersky and G. M. Janowski, "The Structure of Electrodeposited Al-Ti Alloys," Proc. of Symposium on Defect Structure, Morphology, and Properties of Vapor and Electrodeposits, TMS Fall Meeting, Rosemont, Il, October 2-6, 1994.

Magnetic Materials Group

Bennett, L. H.

1. "Magnetic Refrigeration," G. W. University, February 8, 1995.

Egelhoff, W. F.

1. "Surface and Interfacial Roughness in GMR Multilayers by STM," National Storage Industry Consortium Quarterly Workshop, Minneapolis, October 7, 1994.

2. "Recent Studies of GMR Multilayers at NIST," Surface Science Lunch Group Seminar, NIST, December 12, 1994

3. "Recent Studies of GMR Multilayers at NIST," Metallurgy Division Seminar, NIST, January 5, 1995.

4. "GMR Exceeding 21% in Simple Spin Valves," National Storage Industry Consortium Quarterly Workshop, San Diego, January 12, 1995.

5. "Low Temperature Epitaxy in Metal on Metal Systems," Workshop on Fundamental Processes in Epitaxy, Bonn, Germany, February 14, 1995.

6. "Surfactant Assisted Growth of GMR Spin Valves," National Storage Industry Consortium Quarterly Workshop, San Jose, March 25, 1995.

7. "Surface Segregation and Interdiffusion in Metal/Metal Systems," American Chemical Society National Meeting, Anaheim, April 2, 1995.

8. "Structure, Morphology, and GMR of Symmetric Spin Valves," Materials Research Society, San Francisco, April 20, 1995

9. "Thermal Degradation of Spin Valve Components," Materials Research Society, San Francisco, April 18, 1995

10. "Low Temperature Growth of GMR Spin Valves," (poster) Materials Research Society, San Francisco, April 20, 1995.

11. "The Magnetic Engineering Research Facility," ARPA/NRL/ONR Workshop on Magnetic Materials, Arlington, May 16, 1995.

12. "The Structure and Properties of GMR Multilayers," Read-Rite Corporate Research Labs, Fremont, CA, May 31, 1995

13. "Optimizing the Performance of GMR Multilayers," National Storage Industry Consortium Annual Review, Monterey, June 1, 1995.

14. "Optimized Growth of GMR Multilayers," (poster) 2nd International Conference on Magnetic Multilayers, Cambridge, England, September 14, 1995.

15. "Recent Studies of GMR Multilayers at NIST," National Storage Industry Consortium Quarterly Workshop, Pittsburgh, September 21, 1995.

McMichael, R. D.

1. "FMR studies of annealed spin-valve component films," NSIC quarterly meeting, Oct 6-7, 1994, University of Minnesota, Minneapolis. (On unbiased Co and permalloy films.)

2. "The magnetocaloric effect in bulk and nanocomposite materials," University of Maryland, Baltimore Campus Physics Dept., Oct 12, 1994.

3. "Death of a spin valve II" NSIC quarterly meeting, Jan 12-13, 1995 Center for Magnetic Recording Research, UCSD. (On FeMn biased films and NiO biased symmetric spin valves)

4. "Spin valves from beyond the grave," NSIC quarterly meeting, March 22, 1995, IBM Homestead site, San Jose CA. (On thermally stable NiO-biased symmetric Cu/Co spin valves.)

5. "Ferromagnetic Resonance: How it works and what it does" Televised PL-EEEL magnetics seminar March 29, 1995.

Shull, R. D.

1. "The Nobel Experience", Convocation speaker, Mountaineering Youth Science Camp, Elkins, W. Virginia, August 10, 1995.

2. "Superparamagnetism in nanocomposite Magnetic Materials - (An Abbreviated Incomplete Overview)", EEEL/PL/MSEL Teleconference Seminar speaker, NIST, August 1, 1995.

3. "Superconductivity, Physics and Chemistry", Maryland State Governor's Academy for Mathematics, Science and Technology, NIST, July 12, 1995.

4. "Nanocomposite Materials", University of Minnesota Mechanical Engineering Department Seminar Speaker, Minneapolis, MN, May 18, 1995.

5. "The Enhanced Magnetocaloric Effect of Magnetic Nanocomposites", Materials and Molecular Chemistry seminar, Columbia University, April 18, 1995.

6. "Nanostructured Materials", Freshman seminar, Chemistry Dept., Columbia University, April 18, 1995.

7. "A Quick View of the 1994 Nobel Prize Ceremonies," Oak Ridge National Laboratory, April 10, 1995.

8. "Magnetic Applications of Nanocomposite Materials", Condensed Matter Physics seminar, Carnegie Mellon University, April 6, 1995.

9. "The 1994 Nobel Prize Ceremonies," Carnegie Mellon University, April 5, 1995.

10. "A Week of Experiences at the 1994 Nobel Prize Ceremonies," The Philosophical Society of Washington Meeting, Washington, DC, Feb. 3, 1995.

11. "The 1994 Nobel Ceremony: An Inside View," NIST Sigma Xi Chapter Meeting, NIST, Jan. 26, 1995.

12. "Nanocrystals for Magnetic Refrigeration," Workshop on Future Energy Applications of Nanostructured Materials, MIT Dec. 2, 1994.

13. "Magnetic Applications of Nanocomposite Materials," Materials Research Society Fall Meeting, Boston, Nov. 30, 1994.

14. "Nano-Composite Magnetic Refrigerants," Workshop on Nanoparticulates 94, Business Opportunities, Technologies, Markets, and Applications, Monterey, Nov. 15, 1994.

15. "NIST Activities in Nano-Structured Materials," Workshop on Nanoparticulates 94, Business Opportunities, Technologies, Markets, and Applications, Monterey, Nov. 14, 1994.

16. "Nanostructured Materials," National Educator's Workshop, NIST, Nov. 11, 1994.

17. "Magnetic Nanocomposites: Magnets of the Future?," ASM International Washington D.C. Chapter Meeting, Nov. 1, 1994.

18. "NIST Nanostructured Materials Activities," Dept. of Materials Science Seminar, Berlin Technical University, Berlin, Oct. 14, 1994.

19. "Magnetic Nanocomposites," First Colloquium Speaker for the Institute on Nanostructured Materials, Univ. of Saarlandes, Saarbruecken, Germany, Oct. 12, 1994.

20. "Magnetic Nanocomposites," Dept. of Materials Science Seminar Speaker, Univ. of

Darmstadt, Darmstadt, Germany, Oct. 11, 1994.

21. "Magnetic Nanocomposites," Robert Bosch GmbH, Stuttgart, Germany, Oct. 10, 1994.

22. "The Enhanced Magnetocaloric Effect of Magnetic Nanocomposites," Second International Conference on Nanostructured Materials, Stuttgart, Germany, Oct. 7, 1994.

Swartzendruber, L. J.

1. "Magnetic Inspection," The American Society of Nondestructive Testing meeting in Atlanta, Fall 94.

2. "The Relationship of Magnetic and Mechanical Properties," American Iron and Steel Institute Meeting held in Cleveland, May 1995.

3. "Magnetic Particle Standards," The American Society of Nondestructive Testing, Greater Philadelphia Chapter, October 1995.

Weissmueller, J.

1. "Atomic Structure and Thermodynamic Functions of Nanocrystalline Solids"

2. "Materials Science Seminar, University of Wisconsin-Madison, February 16, 1995

3. "Interdepartmental Seminar on Nanostructured Materials", Massachusetts Institute of Technology, February 22, 1995

4. "Materials Science Seminar", Harvard University, February 23, 1995

5. "Materials Science Seminar", Argonne National Laboratory, March 29, 1995

6. "Materials Research Lab Seminar", University of Illinois in Urbana/Champaign, March 30, 1995

7. "Atomic Short-Range Order in Nanocrystalline Solids from Atomic Distribution Functions," 9th International Conference on Liquid and Amoprhous Metals, Chicago, August 27 - September 1, 1995

Materials Performance Group

deWit, Roland

1. "Aluminum Tearing," The National Educator's Workshop, Nov. 8, 1994.

2. An hour long lecture was given at the Training Course for Engineers and Standards Experts from the Newly Independent States (NIS), Rio Marriott, Gaithersburg, MD, March 8, 1995.

3. Co-authored a talk with Prof. R. W. Armstrong entitled "The Quantized Burgers Circuit: From Beginning to Present," presented at the Johannes M. Burgers Centenary Symposium, Univ. of Maryland, College Park, May 4, 1995.

4. "Fracture Testing of Large-Scale Thin Sheet Aluminum Alloy," 27th National Symposium on Fatigue Fracture Mechanics, Williamsburg, VA, June 26-29, 1995.

Fields, Richard

1. "Modeling the Consolidation of Particle Reinforced Aluminum," USCAR Meeting, Detroit, Oct. 10, 1994.

2. "Powder Consolidation Research at NIST," Workshop on Dynamic Magnetic Consolidation, IAP Research Inc., Dayton, Oct. 26, 1994.

3. "Large-Scale Mechanical Testing," National Educators Workshop, NIST, Gaithersburg, November 8, 1994.

4. "Lead-free Solders for Plumbing Applications," Inst. for Mechanics of Materials Workshop on Lead-free Solders, Chicago, July 25, 1995.

Foecke, Tim

1. "The Microscopy of Fracture," National Educators Workshop, Washington, DC, November 8, 1994.

2. "Processing and Mechanical Properties of Nanoscale Laminate Composites," NSF Institute for Mechanics and Materials, UCSD, LaJolla, CA, November 18, 1994.

3. "TEM Observations of Plastic Deformation and Fracture in Nanoscale Multilayered Materials," Workshop on Nanoscale Layered Materials for Protective Coating and Structural Applications, Gaithersburg, MD, August 15, 1995.

Smith, John H.

1. "The Development of National Hardness References Scales in the U.S.," ASM Materials Week, Rosemont, IL, Oct. 4, 1994.

2. "Advances in Hardness References Standard Scales," The XIII IMEKO World Congress - From Measurement to Innovation, Turin, Italy, September 5-0, 1994.

3. "Safety Considerations for New Cylinder Technology," U.S. DOT Symposium on Carbon-Fiber Composite Cylinders, May 26, 1994, Washington, DC.

Sturrock, C. P.

1. "Corrosion Engineering Informatics," Katholieke Universiteit Leuven (Belgium), Dept. of Metallurgy & Materials Engineering, March 7, 1995.

<u>Materials Structure & Characterizaiton Group</u>

Bendersky, L. A.

1. "Intermetallics," Wright Patterson Air Force Base in Dayton, OH, Sept. 18, 1995.

Drescher-Krasicka, E.

1. "Another Approach to Acoustic Elasticity," Seventh International Symposium on Nondestructive Characterization of Materials, Prague, Czech Republic, June 19, 1995.

2. "Acoustic Microscopy Application for Thin films Testing," 29th Annual Meeting of the Microbeam Analysis Society, Breckenridge, CO, August 6, 1995.

Gayle, F. W.

1. "Precipitation Hardening in the Wright Flyer Crankcase," Materials Science Seminar Series Newark, NJ, March 1, 1995.

Handwerker, C. A.

1. "Thermodynamics and Kinetics of Anisotropic Interfaces," Materials Research Society Fall Meeting, Boston, MA, November 29, 1994.

2. "Stability of Interfaces," Rutgers University, Brunswick, NJ, December 9, 1994.

3. "Interfaces Shapes and the Forces that Mold Them," Drexel University, Philadelphia,

PA, March 6, 1995.

4. "Critical Issues in Grain Growth in Ceramics, University of Pennsylvania, Philadelphia, PA, March 14, 1995.

5. "Interface Shapes and the Forces that Mold Them," Lehigh University, Bethlehem, PA, March 21, 1995.

6. "Critical Issues in Reactive Wetting," American Ceramic Society Meeting, Cincinnati, OH, May 1, 1995.

7. "Solder Joint Modeling," NIST Workshop on Solder Interconnect Design, June 12, 1995.

8. "Modeling in Si_3N_4 Grain Growth," International Conference on Sintering, Pennsylvania State University, State College, PA, September 26, 1995.

9. "Grain Growth in Facetted Systems," Pennsylvania State University, State College, PA, September 26, 1995,

Shapiro, A. J.

1. "Electron Microscopy," The Seventh Annual International Scientific Meeting," in Monterey, CA, March 31, 1995.

Warren, J. A.

1. "Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," Materials Research Society Fall Meeting, Boston, MA, December 1994.

2. "Phase-Field Models of Alloy Solidification," City College, New York City, NY, April 26, 1995.

3. "Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," Case Western Reserve University, Cleveland, OH, November 1994.

4. "Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," Columbia University, New York, January 1995.

5. "Wavelength Selection During Directional Solidification of a Binary Alloy," American Physical Society Meeting, Pittsburgh, PA, March 1995.

6. Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," American Physical Society Meeting, Pittsburgh, PA, March 1995.

7. Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," University of Wisconsin, Madison, WI, August 1995.

8. "Prediction of Dendritic Microsegregation Patterns Using a Diffuse Interface Phase-Field Model," London, United Kingdom, September 1995.

<u>Metallurgical Processing</u>

F. S. Biancaniello

1. "Control of BCC and FCC Phase Formation During Mechanical Alloying of Ti-Al-Nb," International Conference, Los Vegas, Nevada, Synthesis/Processing of Lightweight Metallic Materials, TMS, Feb. 1995.

2. "Processing and Characterization of Nitrogenerated Stainless Steel for Body and Extremity Applications," Symposium at Aberdeen Proving Grounds, Body Armor Insert Replacement Program in Progress Review/Covert Armor Discussions, March 8, 1995.

3. "Dramatically Improved Corrosion Behavior of 304L Stainless Steel Through Nitrogen Atomization," International Conference on Powder Metallurgy in Aerospace, Defense and Demanding Application, May 8-10, 1995.

4. "Minimization of Nitride Formation During Mechanical Alloying of Ti-Al-Nb, International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials, Quebec, Canada, July 1995.

W. J. Boettinger

1. "A Phase-Field Diffuse Interface Solidification Model for Binary Alloys," Materials Week, Rosemont, IL, October 4, 1994.

2. "Reactive Wetting of Solder," Virginia Polytechnic and State University, Blacksburg, VA, October 21, 1994.

3. "Multicomponent Alloy Solidification," Semi-Annual Meeting, Consortium on Casting of Aerospace Alloys, American Foundryman's Society, Des Plaines, IL, Nov. 7, 1994.

4. "Alpha-case Formation in Ti Castings," Semi-Annual Meeting, Consortium on Casting of Aerospace Alloys, American Foundryman's Society, Des Plaines, IL, Nov. 7, 1994.

5. "Reactive Wetting of Solder," Dept. of Materials Science and Engineering, Iowa State University, Ames, IA, November 29, 1994.

6. "A Phase-Field Diffuse Interface Solidification Model for Binary Alloys," Department of Mechanical Engineering, University of Iowa, December 1, 1994.

7. "The Future of Phase Diagrams for prediction of Fraction Solid vs. Temperature Relationships, ProCAST™ User's Group Meeting, Annapolis, MD, March 30, 1995.

8. "Dendritic Growth: From Fundamental Studies to Superalloys", AT&T Distinguished Lecturer: Materials Science Seminar Series, University of Wisconsin, Madison, WI, April 6, 1995.

9. "Multicomponent Alloy Solidification," Annual Meeting, Consortium on Casting of Aerospace Alloys, NIST, May 4, 1995,

10. "Alpha-case Formation in Ti Castings," Annual Meeting, Consortium on Casting of Aerospace Alloys, NIST, May 4, 1995.

11. "A Phase-Field Diffuse Interface Solidification Model for Binary Alloys," Department of Materials Science and Metallurgy, Cambridge University, September 21, 1995.

S. R. Coriell

1. "The Effect of Shear Flow and Anisotropic Kinetics on the Morphological Stability of a Binary Alloy: Self-Stabilization of Step Trains, International Conference on Crystal Growth XI, Netherlands, June 18-23, 1995.

S. D. Ridder

1. "Control of Particle Size During Inert Gas Atomization," International Conference on Powder Metallurgy in Aerospace, Defense and Demanding Applications, Anaheim, CA, May 1995.

J. A. Simmons

1. "On the Superposition of Probabilities I; Partition Lattices and the Moebius Formalism," International Workshop on the Theory and Applications of the Cluster Variation and Path Probability Methods, Mexico, June 1995.

CONFERENCES AND WORKSHOPS
FY 1995

AESF/NIST Coatings Workshop on Functional Chromium Coatings from Trivalent Electrolytes
November 1994
C. E. Johnson
25 attendees

A jointly sponsored NIST/AESF Workshop on "Recent Developments in Electrodeposition" was held at NIST in November 1994 for government and industry representatives from the MD, VA, PA, DE and DC areas.

International Organization for Standardization (ISO) Meeting
July 1995
C. E. Johnson
20 attendees

A week-long meeting of ISO TC 107 - Metallic and Inorganic Coatings - was held at NIST in July 1995 preparing international specification as they pertain to the coatings industry. Representatives from the U.S., United Kingdom, Germany, France, South Africa, Sweden , Switzerland and Australia were in attendance.

ISO Subcommittees TC164/SC1 and TC164/SC3
March 27-31, 1995
J. H. Smith
30 attendees

The ISO Subcommittees TC164/SC1 on Tensile Testing and TC164/SC3 on Hardness Testing were held at NIST.

National Educators' Workshop: Update '94
November 7-9, 1994
A. C. Fraker
130 attendees

To strengthen materials science and engineering (MS&E) education by bringing together individuals from academia, industry technical societies and government to present demonstrations of experiments and to discuss issues of MS&E.

Workshop on Nanoscale Layered Materials for Protective Coating and Structural Applications
August 14-15, 1995
T. [illegible]. Foecke
86 attendees

Intended to bring together leading researchers in this emerging field for an exchange of information, and to introduce this class of materials to industrial representatives for potential use in engineering applications.

Nanostructured Materials
August 27-September 1, 1995
R. D. Shull
200 attendees

A symposium held at the Fourth International Conference on Advanced Materials (ICAM IV), Cancun, Mexico for the purpose of reviewing the present status in the field.

Structure and Properties of Nanophase Materials
February 13-14, 1995
R. D. Shull
150 attendees

A symposium held at the TMS Annual Meeting, Las Vegas, NV, for bringing together researchers in the field of nanostructured materials to assess the present understanding of the structure/property relationships.

Sol Gel and Aerogel Materials
February 13-14, 1995
R. D. Shull
40 attendees

A symposium held at the TMS Annual Meeting, Las Vegas, NV for bringing together the various researchers in the area of sol gel materials so as to initiate further collaboration between research activities on different materials types.

µMAG Micromagnetic Modeling Workshop
April 18, 1995
R. D. McMichael
90 attendees

The inaugural workshop of the micromagnetic modeling activity group, (µMAG) sponsored by the NIST Center for Theoretical and Computational Materials Science to discuss applications and future directions in micromagnetic modeling.

Workshop on Design of Advanced Interconnection Technology
November 13-16, 1994
C. A. Handwerker, J. R. Manning
60 attendees

To obtain views of the microelectronics industry on needed measurement programs in this area.

Mathematics of Thermodynamically Driven Microstructural Evolution Symposium
October 29-November 1, 1995
J.A. Simmons
100 attendees

Organized to bring together mathematicians and material scientist to focus on this topic. The symposium was jointly sponsored by ASM International, the Society of Industrial and Applied Mathematics (SEAM), The Minerals, Metals and Materials Society (TMS), Advanced Research Projects Agency (ARPA) and NIST.

Solder Interconnect Design Workshop
June 1995
James Warren
30 attendees

A workshop was held at NIST (Center for Theoretical and Computational Materials Science) in July 1995 to determine what tools/methods are most promising for the modeling of solder interconnects.

Mathematics of Thermodynamically Driven Microstrucutral Evolution
October 29-November 1, 1995
J. R. Manning
60 attendees

A symposium on Mathematics of Thermodynamically Driven Microstructural Evolution, TMS/ASM Materials Week Meeting, Cleveland, Ohio was organized as part of this project, bringing together mathematicians and material scientists to focus on this topic. The symposium was jointly sponsored by ASM International, the Society of Industrial and Applied Mathematics (SIAM), the Mining, Metals and Materials Society (TMS), Advanced Research Projects Agency (ARPA), and NIST.

PATENTS
FY 1995

Granted
Nanocomposite Material for Magnetic Refrigeration and Superparamagnetic Systems Using the Same
L. H. Bennett, R. D. Shull, L. J. Swartzendruber (Metallurgy)
Patent No. 5,381,664

A Process for the Controlled Preparation of a Composite of Ultrafine Magnetic Particles Homogeneously Dispersed in a Dielectric Matrix
J. J. Ritter (Ceramics), R. D. Shull (Metallurgy)
Patent No. 5,316,699

Methods and Electrolyte Composition for Electrodeposited Chromium Coatings - The Process
C. E. Johnson (Metallurgy), two others (non-NIST)
Patent No. 5,415,763

Pending

Composite Solder
R. B. Clough, three others (non-NIST)
Disclosure Filed

Steel Hardness Measurements System and Method of Using Same
G. E. Hicho, L. J. Swartzendruber (Metallurgy)
Application No. 08/503,263 (10/12/95)

Methods and Electrolyte Composition for Electrodeposited Chromium Coatings - The Article
C. E. Johnson (Metallurgy), two others (non-NIST)
Application No. 08/411,191 (2/95)

Chemical Precipitation of Metal Powders by a Two Solution Technique
C. E. Johnson (Metallurgy)
Disclosure Filed

EXTERNAL RECOGNITION
FY 1995

Roger B. Clough	Requested to write article on "Acoustic Emission" for Encyclopedia of Advanced Materials (Pergamon Press)
Timothy J. Foecke	Member of the NSF Institute for Mechanics and Materials (UCSD) Young Investigators Advisory Committee
	Member of the Editorial Board, ASM Metals Handbook Volume 19: Fatigue and Fracture
Sam R. Low	Certificate of Appreciation, ASTM Committee E-28 on Mechanical Testing
E. Neville Pugh	NACE Fellow
	Academic Program Review Team, Iowa State University
Richard E. Ricker	Member of the Board of Review for the Journal Metallurgical and Materials Transactions A, published by ASM International and The Minerals, Metals and Materials Society (TMS), Warrendale, PA
	Elected Chairman of the Joint ASM International and TMS Committee on Corrosion and Environmental Effects, 1994-96.
	Represented NIST, Hemispheric Trade and Commerce Forum, Summit of the Americas (1995)
	Represented NIST, Frontiers of Engineering, Summit of the Americas (1995)
Robert D. Shull	Elected Secretary/Treasurer of the International Committee on Nanostructured Materials (1991); re-elected, 1994.
	Elected Vice Chairman of the TMS Chemistry and Physics of Materials Committee (1992); Chairman (1994).
	Associate Editor, Journal of Nanostructured Materials (1992-1994).

	Selected as Chairman of the Third International Conference on Nanostructured Materials (1994).
	President Elect, NIST Chapter of Sigma Xi (1994).
	Appointed to the Committee of Visitors to review the NSF Program on Fluid, Particulate, and Hydraulic Systems (1995).
John H. Smith	Member of the Technical and Safety Standards Council (TASS) of the Compressed Gas Association
Mark R. Stoudt	Elected Treasurer of the Washington, DC Chapter of ASM International (1995)
Charles P. Sturrock	Chairman, Session on New Aspects of Data Content, ASTM Fifth International Symposium on Computerization and Networking of Materials Property Data (1995)
	Appointed to ASM Materials Information Committee (1995)
	Materials Technology Institute sponsorship for collaborative research at Katholieke Universiteit Leuven (Belgium), Department of Metallurgy and Materials Engineering (1993-95)

TECHNICAL/PROFESSIONAL COMMITTEE LEADERSHIP ACTIVITIES

Aluminum Association
- S. D. Ridder, Standards Committee

American Association for the Advancement of Science
- T. P. Moffat

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

American Ceramic Society
- C. A. Handwerker, Sosman Award Committee
  Basic Science Division, Chair-Elect

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

American Institute of Mining, Metallurgical and Petroleum Engineers
- The Metallurgical Society
- W. J. Boettinger, Solidification Committee
- C. A. Handwerker, Electronic Packaging and Intramural Materials Committee
- R. E. Ricker, Vice Chairman, Committee on Corrosion and Environmental Effects
- S. D. Ridder, Solidification Committee
- S. D. Ridder, Powder Metallurgy Committee
- R. D. Shull, The Chemistry and Physics of Materials Committee
- R. D. Shull, Alloy Phases Committee
- R. D. Shull, Acta Metallurgica and Hume Rothery Award Committee
- R. D. Shull, EMPMD Division Council
- R. D. Shull, SMD Division Council

American Physical Society
- R. deWit
- A. Cezairliyan

American Powder Metallurgy Institute
- S. D. Ridder

American Society for Metals
S. D. Ridder, Processing Committee

American Society of Mechanical Engineers
R. deWit
A. Cezairliyan, Thermophysical Properties Committee
R. B. Clough, Materials Characterization 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 Information Committee
C. P. Sturrock

Technical Divisions Board
E. N. Pugh

Technology Transfer Committee
E. N. Pugh, Chairman

Washington, DC Chapter
M. R. Stoudt, Treasurer
R. J. Fields
G. E. Hicho

ASNT

E7:04:	Acoustic Emission J. A. Simmons

ASTM

A6:	Magnetic Properties L. H. Bennett L. J. Swartzendruber

B2:	Nonferrous Metals and Alloys S. D. Ridder
B5:	Copper and Copper Alloys L. J. Swartzendruber
B7:	Light Metals R. D. Shull W. J. Boettinger
B8:	Metallic and Inorganic Coatings D. R. Kelley E. Escalante
B8.01:	Ancillary Matters E. Escalante, Vice-Chairman
B8.10:	General Test Methods C. E. Johnson
B8.10.03:	Microhardness Testing C. E. Johnson, Liaison to EO4
B9:	Metal Powders and Metal Powder Products J. R. Manning
D30:	High Modulus Fibers and Their Composites M. R. Stoudt
DS-56D	Metal and Alloys in the Unified Numbering System G. E. Hicho
E4: E4.05:	Metallography Microhardness C. E. Johnson L. J. Swartzendruber
E7:	Nondestructive Testing L. Mordfin, Executive Subcommittee L. J. Swartzendruber L. H. Bennett

E7:04:	Acoustic Emission J. A. Simmons R. B. Clough
E7.10:04:	Infrared NDT Methods L. Mordfin, Chairman
E8:	Fatigue and Fracture R. deWit R. J. Fields G. E. Hicho S. R. Low J. H. Smith J. A. Simmons
E8.02:	Fractography & Associated Microstructure R. J. Fields G. E. Hicho
E8:04:	Environmental Cracking R. E. Ricker
E8.08:	Elastic-Plastic G. E. Hicho
E28:	Mechanical Testing L. Mordfin, Executive Subcommittee 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 Test Program S. R. Low
E28.94:	USA Committee for ISO TC 164 L. Mordfin
E28.94.06:	Terminology and Symbols L. Mordfin, Chairman
E37:	Thermal Measurements A. Cezairliyan
E49:	Computerization of Materials and Chemical Property Data C. P. Sturrock, Chairman
F4:	Medical and Surgical Materials and Devices A. C. Fraker
F16:	Fasteners S. R. Low
G1:	Corrosion of Metals E. Escalante R. E. Ricker M. R. Stoudt
G1.06:	SCC and Corrosion Fatigue R. E. Ricker
G1.10:	Corrosion of Metals in Soil E. Escalante
G1.10.01:	Measurement of pH of Soil E. Escalante
G1.10.02:	Measurement of Soil Resistivity E. Escalante

G1.11: Electrochemical Measurements in Corrosion
R. E. Ricker

G1.14: Corrosion of Steel in Concrete
E. Escalante

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
J. H. Smith, Technical and Safety Standards Council (TASS)

Electrochemical Society
T. P. Moffat, Chairman of Washington Local Chapter
Secretary/Treasurer of Electrodeposition Division
Member at Large of the Electrodeposition Division

High Temperature Science Journal
A. Cezairliyan, Editorial Board Member

IEEE Committee on Metric Policy
L. H. Bennett, Magnetic Society Representative

Intermay 96 Program Committee
L. H. Bennett

International Advisory Committee for International Conference on Rapidly Quenched Metals
W. J. Boettinger, Member

International Advistory Committee and Program Committee for the International Conference on Nanostructured Materials
R. D. Shull, Recording Secretary/Treasurer

International Commission on Standardizing Thermophysical Properties Measurement Methods
A. Cezairliyan, Member

International Conference on Sintering, 1995
C. A. Handwerker, Organizing Committee

International Journal of Thermophysics
A. Cezairliyan, Founding Editor-in-Chief

International Organization for Standardization
TC58.SC: 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

TC107.03: Electrodeposited Coatings and Related Finishes
C. E. Johnson, Delegate

TC135: Nondestructive Testing
L. Mordfin, Delegate

International Organizing Committee for International Workshop on Magnetic Properties of Fine Particles and their Relevance to Materials Science
R. D. Shull, Member

International Organizing Committee for μMAG Workshop
R. D. McMichael, Chairman
M. J. Donahue, Steering Committee

International Organizing Committee of the Subsecond Thermophysics Workshops
A. Cezairliyan, Member

International Organizing Committee of the European Thermophysical Properties Conferences
A. Cezairliyan

International Organizing Committee of the Seventh Symposium on Magnetic Layers
W. F. Egelhoff

International Thermal Expansion Conferences
A. Cezairliyan, Governing Board

International Thermophysics Congress
A. Cezairliyan, Chairman

Iowa State University Academic Program Review Team
E. N. Pugh

IPC Subcommittee on Solderability
Alternate Final Finishes Task Group
Component and Wire Solderability
Specification Task Group
Steam Aging Task Group
Wetting Balance Task Group
C. A. Handwerker

Journal of the American Ceramic Society
C. A. Handwerker, Associate Editor

Journal of High Temperatures - High Pressures
A. Cezairliyan, Editorial Board Member

Journal of Crystal Growth
S. R. Coriell, Assoc. Editor

Journal of Nanostructured Materials
R. D. Shull, Associate Editor

Maryland Institute of Metals
R. deWit

Materials Research Society
T. P. Moffat

Materials Science and Engineering
L. H. Bennett, Associate Editor

NACE Awards Committee
E. N. Pugh

NACE International
M. R. Stoudt

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

NACE Research Committee
E. N. Pugh, Past Chairman and Chairman of Research in Progress Symposium Sub-Committee

National Aeronautics and Space Administration
Materials Science Discipline Working Group
R. J. Schaefer

NSF Institute for Mechanics and Materials Young Investigator Advisory Committee
T. Foecke

Office of Energy-Related Inventions, National Institute of Standards and Technology
L. H. Bennett, Reviewer of Invention Disclosures

Sigma Xi
R. deWit
T. P. Moffat
R. J. Fields
R. D. Shull

Society for Experimental Mechanics
R. deWit

Society for Natural Philosophy
R. deWit

Society of Automotive Engineers/ASTM
Unified Numbering System for Metals and Alloys
L. H. Bennett, NIST Representative

The Minerals, Metals & Materials Society (TMS) of the AIME
R. deWit
T. P. Moffat
R. E. Ricker
M. R. Stoudt

Washington Academy of Sciences
R. deWit

INDUSTRIAL/ACADEMIC INTERACTIONS

INDUSTRY

1. Advanced Ceramics Research, Inc.

   R. Fields provided Dr. E. Danko of Advanced Ceramics Research, Inc. with correlations between the Charpy Impact Energy and the fracture toughness (K_{Ic}) of steel, titanium alloys, and aluminum alloys.

2. Aerojet General Inc.

   John Smith collaborated with Aerojet on the development of carbon fiber reinforced composite cylinders to be used for the transportation high pressure gasses.

3. ALCOA

   Richard Fields and Robb Thomson collaborated with J. Hirth, H. Zbib, and L. Levine (Washington State Univ.) and Owen Richmond and Hasso Weiland of Alcoa to elucidate the role of dislocation patterning in determining the internal variables used to describe metals during metal forming operations.

4. American Iron and Steel Institute (AISI)

   G. E. Hicho and L. J. Swartzendruber 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.

5. Amorphous Technology International (ATI)

   R. E. Ricker has been working with ATI to develop an experimental program which will help ATI evaluate the corrosion resistance of their amorphous alloys.

6. Ampal/Metallurg, Crucible Materials Corp., General Electric Co., Martin Marietta Energy Systems, Alcoa, and Pratt & Whitney Aircraft.

   The NIST SiGMA system has been the focal point of this pioneering work in advanced sensor development and Expert System based control. Scientists from the participating companies, in collaboration with NIST scientists, have developed modeling tools, advanced sensors, and process control strategies.

7. Atotech (M & T Harshaw)

Christian E. Johnson has completed a CRADA for joint research on development of trivalent chromium as a replacement for toxic hexavalent chromium electrolytes with the submission of a final report.

8. Ball Grid Array Consortium - National Center for Manufacturing Sciences

F. W. Gayle and C. A. Handwerker are working with Eastman Kodak, Motorola, View Engineering, Delco Electronics, MicroFab Technologies, Northern Telecom, AMP, Indium Corp., Amkor, Universal Instruments, Texas Instruments, and the National Center for Manufacturing Sciences to organize a consortium with the goal of applying solder jet technology to ball grid array assembly techniques for microelectronics applications.

9. Biomedical Implant Consortium

A. C. Fraker participated in a consortium of Ti alloy producers and implant manufacturers set up by the Air Force Materials Lab, as part of their technology transfer program, to characterize and establish a data base on an alloy, Ti21SRx, a modification of the Ti21S alloy that was developed for the aero-space plane. The Ti21SRx alloy is being tested for its suitability for use in surgical implants, and it will be available to implant producing companies.

10. BOC Gases

John Smith has been working with BOC Gases to study the use of ultrasonic testing to inspect high pressure steel and aluminum gas cylinders.

11. Bureau of Mines, Albany Research Center

F. W. Gayle and F. S. Biancaniello are working with Cynthia Dogan and James Rawers of the Bureau of Mines on processing of nanostructured ferrous alloys.

12. 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 is working with CGA to help them develop specifications and standards for the construction and inspection of pressurized gas cylinders.

13. COMSAT

R. Fields assisted T. Kirkendall, formerly of COMSAT, and Dr. Mark S. Fan, formerly of Unisys's Goddard Operations, by providing fatigue data for copper wired and helping to determine the reliability of electronic motors for space craft applications.

14. 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.

15. Copper Development Association (CDA)

R. B. Clough and R. J. Fields are working with members of the CDA determining the mechanical properties of new lead-free solders used in piping applications.

16. Creswell Scientific Products and Services

L. J. Swartzendruber is working with Creswell Scientific Products and Services to develop test methods whereby the properties of steel samples may be depth profiled using surface magnetic methods.

17. Crucible Compaction Metals

Crucible Compaction Metals and NIST have signed a Cooperative Research and Development Agreement concerning improved nitrogenated alloys produced by atomization, including investigations of approaches patented by NIST (F. Biancaniello, S. Ridder, and G. Janowski).

18. 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.

19. Digital Equipment Corporation

Collaboration with G. Freeman of Digital Equipment Corporation to develop solutions in solder interconnect design problems. Using the software Surface Evolver we are able to address many issues of interest to the electronic packaging industry.

20. 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.

21. Elkhart Products, Inc.

R. Fields provided Karen Crago of Elkhart Products Inc., Elkhart, Indiana with mechanical properties of certain copper alloys to assist them in predicting the manufacturability of particular parts.

22. Faratech

Christian E. Johnson is engaged in informal cooperation to further development and commercialization of trivalent chromium electrolytes as replacement for toxic hexavalent electrolytes.

23. FDA and Telectronics Pacing Systems, Inc.

Christian E. Johnson is engaged in informal cooperation with FDA to determine the failure mode of composite electroplated flex solder tabs in recalled pulse generators (pacemakers).

24. Federal Railroad Administration

G. E. Hicho has had interactions with the railroad industry while he was performing research with the research divisions of the Federal Railroad Administration. A numerous number of reports were published that pertained to the safety of future tank cars that would carry hazardous materials.

25. FIBA Inc.

J. Smith is working with FIBA to develop and evaluate new methods for the use of ultrasonic testing to inspect high pressure steel and aluminum gas cylinders.

26. Fluxtrol Manufacturing

A Cooperative Research and Development Agreement 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.

27. General Electric

L. A. Bendersky is collaborating with the General Electric Company, Corporate Research and Development Center (Dr. R. G. Rowe) in studies of the microstructures and properties of new Ti-Al-Nb alloys. This research program is supported by Naval Air Warfare Center, Aircraft Division.

28. 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.

29. General Motors (GM)

R. D. Shull and R. D. McMichael of the Magnetic Materials Group have teamed up with Jan Herbst and Carl Fuerst of GM in a collaborative effort (CRADA CN-695) to study the magnetocaloric properties of rare earth transition metal alloys.

30. GMW Associates

L. J. Swartzendruber and L. H. Bennett are collaborating GMW (CRADA CN-701) in the development and testing of a new laboratory electromagnet capable of very rapid changes in magnetic field.

31. 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.

32. IAP Research Inc.

R. Fields and Z. Livne (Nuclear Research Center, Negev, Israel) collaborated with Dr. Bhana Cheluri of IAP Research Inc. to identify the benefits of dynamic magnetic consolidation for nanoscale powders. Canned powders of various types were prepared at NIST. They were dynamically consolidated magnetically and returned to NIST for characterization.

33. 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.

34. Industrial Quality Inc. (IQI)

J. Smith is working with IQI to assist them in the development of an ultrasonic test system to be used to detect sustained load cracking (SLC) in aluminum cylinders. This work is being funded by an SBIR grant from NIST.

35. Instituto di Metrologia "G. Colonetti" (IMGC), Turin, Italy

S. Low and J. Smith measured the hardness of standardized hardness test blocks from Instituto di Metrologia "G. Colonetti" as part of an international intercomparison to establish worldwide Rockwell hardness standards.

36. 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.

37. Lanxide Electronic Components, L.P.

Christian E. Johnson provided research and development of a process technology for deposition of metallic coating on Al Alloy-SiC composites using formulated metallo-organic compounds to promote solderability to thermal management devices.

38. Luke Engineering & Manufacturing Co.

A. C. Fraker worked with C. Jurey of Luke Engineering & Mfg. Co. to evaluate the effectiveness, in reducing corrosion, of a Germany patented Magoxid Coat© on magnesium alloys and prepared a letter report on the study.

39. 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.

40. Matec Instruments Inc.

J. Smith is collaborating with Matec to assist in the development of an ultrasonic test system to be used to detect sustained load cracking (SLC) in aluminum cylinders. This work is being funded by an SBIR grant from NIST.

41. Materialprufungsamt (Dortmund, Germany)

Sam Low and John Smith collaborated with Materialprufungsamt (MPA) as part of an international intercomparison to establish worldwide Rockwell hardness standards.

42. Materials Technology Institute of the Chemical Process Industries, Inc. (MTI)

MTI is an industrial consortia of about 40 member companies who are concerned with materials used in plants which produce chemicals. With MTI sponsorship, the Corrosion Group and the NACE research associates in the NACE-NIST Corrosion Data Program work with representatives of MTI member companies and their consultants to develop expert systems for selection of materials for the storage and handling of hazardous chemicals.

43. Metal Powders, Inc.

R. Fields provided Richard and Donald Clark of Metals Powders, Inc. with information on the ductility of different pure irons as a function of temperature below room temperature.

44. Metcal

Christian E. Johnson is engaged in the investigation of new, air stable iron plating electrolyte for coating multiple soldering iron tip geometries.

45. MicroFab Corporation

R. Fields interacted with scientist at MicroFab Corp. and developed for them a computer program that predicts the bulk modulus and sound velocity of molten Pb/In solders as a function of temperature and composition. They need this information as part of an ATP award to extend inkjet technology to soldering on printed circuit boards.

46. 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.

47. Nanophase Technologies Corporation

R. D. Shull is collaborating with Dr. John Parker at Nanophase Technologies on the preparation of composite materials with nanometer-scale dimensions. Magnetic methods are being used to characterize the materials, including the determination of whether true composite morphologies are achieved.

48. National Center for Manufacturing Sciences (NCMS)

A broad-based collaboration has been established between the National Center for Manufacturing Sciences (NCMS) and NIST (C. A. Handwerker, R. J. Fields, and U. R. Kattner) to evaluate environmentally safe alternatives for lead-based solder alloys used for electrical/electronic assembly interconnections. In addition to NIST, members include AT&T, Rockwell International, Texas Instruments, Ford, GM-Hughes, GM-Delco, MicroFab Technologies, United Technologies Corporation, RPI, and Sandia National Laboratories.

49. 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 consortium project on Alternatives for Lead-Based Solders to aid in practical testing of new lead-free solder alloys.

50. National Research Laboratory of Metrology (NRLM), Tsukuba, Japan

Sam Low collaborated with NRML as part of an international intercomparison to establish worldwide Rockwell hardness standards.

51. National Storage Industry Consortium (NSIC)

W. F. Egelhoff and R. D. McMichael are cooperating with the NSIC heads group to improve spin valve materials for magnetoresistive high density recording heads. NIST is using ferromagnetic resonce, magnetoresistance and superconducting quantum interference device (SQUID) magnetometry to evaluate magnetic behavior and thermal stability of films proudced by NSIC companies and films produced by NIST.

52. Naval Research Laboratory (NRL)

A. C. Fraker collaborated with M. A. Imam of the Naval Research Laboratory (NRL) in a study to compare and evaluate mechanical and electrochemical properties of titanium alloys for use as surgical implant materials.

53. 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, White Oak, MD on developing wavelet techniques for improved detection of acoustic emission signals from very small cracks (< 20 microns in size).

54. NOAA

Christian E. Johnson held a preliminary meeting to discuss solutions to plating and corrosion problems associated with Dew Point Sensors that are placed at airports throughout the U.S.

55. Norris Cylinder Corporation

John Smith worked with a NIST guest worker sponsored by the technical staff at Norris Cylinder Corporation. The purpose was to revise to develop improved specifications for the design and construction of pressurized gas containers.

56. Norton Corporation

Christian E. Johnson held a preliminary discussion to provide support for composite and wear resistant coatings for complex shape mandrels used in the production of critical dimension mirrors.

57. Oak Ridge National Laboratory (ORNL)

R. deWit, R. J. Fields, D. E. Harne, and D. J. Pitchure collaborated with ORNL to develop data on the fracture toughness of nuclear pressure vessels by testing full thickness bend bars extracted from an unused vessel.

58. PCC Airfoils, Inc.

W. J. Boettinger has provided PCC with calculations of the amount of different crystal phases present in commercial alloys with a range of compositions. PCC has provided NIST with superalloy samples with varying concentrations of sulfur for evaluation as a reference material.

59. 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. W. J. Boettinger has provided Pratt & Whitney with a model describing the reaction between titanium castings and the mold. This work is part of the activities of the Consortium on Casting of Aerospace Alloys.

60. 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.

61. Pulse Electronics, Inc.

Christian E. Johnson developed specifications for Zn coating, electrodeposited or mechanically plated, hardened steel fasteners used to attach electronic control boxes to railway cars.

62. Sandia National Laboratories

Sandia National Laboratories (F. Yost, P. Vianco) and NIST (W. J. Boettinger and C. A. Handwerker) are jointly investigating fundamental reaction mechanisms that influence solderability mechanisms and solderability tests. NIST has supplied Sandia with bulk samples of the intermetallic compounds Cu_6Sn_5 and Cu_3Sn that form in solder interconnects. NIST is the only available source of these alloys, which play key roles in the wettability and mechanical properties of these interconnects. Sandia plans to use these samples in its mechanical measurements and other solder interconnect studies. NIST also has supplied Sandia with thermo-dynamic data for phase stability in solder-substrate systems and with data on the wettability of these intermetallic compounds and their oxides by solders.

63. Sandusky International, Inc.

M. R. Stoudt is collaborating with Sandusky International to characterize and evaluate corrosion fatigue related failure in duplex stainless steels used in pulp paper industry.

64. Science Applications International Corporation (SAIC)

A. C. Fraker collaborates 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.

65. Solder Jet Consortium and MicroFab Technologies, Plano, TX

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, Texas Instruments, and MicroFAB) to measure contamination and wettability of jetted solders.

66. Sonix, Inc.

E. Drescher-Krasicka has a CRADA with Sonix, Inc., Springfield, VA on residual stress detection on electronic packaging.

67. 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.

68. U.S. Council for Automotive Research (USCAR--Ford, Chrysler, and GM)

Richard Fields and Roger Clough of the Materials Performance Group worked with John Allison (Ford and USCAR), Bill Jandeska (GM), Gene Lynn (Chrysler), E. Hyland and P. Wang of Alcoa, and M. F. Ashby, N. Fleck, and A. Cocks of

Cambridge Univ. Micromechanical Modeling Center to develop a modeling program to address the needs of USCAR's initiative to develop low cost processing technologies for consolidation of particle reinforced aluminum for automotive applications.

69. 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 compress gas cylinders.

70. 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.

71. Westinghouse Science and Technology Center

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; this material has nonlinear optical applications.

72. 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.

73. Wilson Instruments

S. Low and J. Smith 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.

74. Xerox Corporation

R. D. Shull is collaborating with R. F. Ziolo, Xerox (CRADA CN 784) to evaluate the polymeric method for creation of magnetic nanocomposites useful in magnetic regrigeration magnetic fluid.

INDUSTRY/UNIVERSITY

1. ALCOA, Washington State University

   Richard, 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. BIRL/Northwestern University and Chicago Faucet Co.

   Christian E. Johnson is investigating the viability of replacing Hex Cr with Tri Cr.

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 the 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 the 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, General Electric Co., GM-Delco, Norfolk Southern, Rensselaer Polytechnic Institute, Stratonics, Inc, and the University of Idaho have interacted in this program.

UNIVERSITIES

1. ACCESS e. V., Aachen, Germany

   Cellular growth during the directional solidification of binary alloys is being studied by G. 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. The properties are needed for modeling casting of these materials.

3. California Institute of Technlogy

   R. D. Shull is collaborating with B. Fultz to magnetically probe and characterize ball-milled nanocomposite ferromagnetic applications.

4. Cambridge University, Cambridge, UK

   R. Fields of the Materials Performance Group is working with M. Ashby of Cambridge University to develop and validate powder forging models for metal matrix composites.

5. Centre d'Etudes Nucleaires de Grenoble

   Collaborative research between J. J. Favier and S. R. Coriell is investigating the initial transient during the directional solidification of tin alloys; the Seebeck voltage is used to determine the interface temperature which is being compared with theoretical predictions.

6. Chonnam University

   A collaborative effort is underway between C. A. Handwerker of NIST and Prof. D. J. Lee of Chonnam University, Kwangju, Korea to study interface properties in solder joint.

7. City College of New York

Ongoing communication with H. Cummins at City College of New York on directional solidification experiments, recent confirmation of Warren and Langer's theory. More intensive studies continue to determine the concentration profile in a solidifying system.

8. Darmstadt University

R. D. Shull is collaborating with H. Hahn to investigate the possibilities for preparing magnetic nanocomposites via vapor-condensation routes.

9. Ecole Polytechnique Federale de Lausanne (Switzerland)

R. J. Schaefer is working with Ch.-A. Gandin of EPFL to develop models for the shape of dendritic metal crystals growing in temperture gradients.

10. George Washington University, Washington, DC

G. E. Hicho is Associate Professorial Lecturer in Engineering in the Civil, Mechanical, and Environmental Engineering Department.

11. George Washington University, Washington, DC

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.

12. Georgia Institute of Technology, Atlanta, GA

R. deWit and R. J. Fields collaborate with Professor Satya Atluri and his colleagues to further the understanding of fracture behavior of thin-sheet structural aluminum alloy panels. They have analyzed our data with their elastic-plastic finite element alternating method (EPFEAM) with the T^*-integral fracture parameter as a crack growth criteria. The results have been published in a three-part report "The Elastic-Plastic Finite Element Alternating Method (EPFEAM) and the Prediction of Fracture under WFD Conditions in Aircraft Structures," by L. Wang, F. W. Brush, and S. N. Atluri, FAA Center of Excellence for Computational Modeling of Aircraft Structures, Georgia Institute of Technology, August 1995.

13. Howard University, Dental School, Washington, DC

A. C. Fraker worked with Dr. A. A. Chohayeb, Dental School, Howard University, studying effects of coupled dissimilar metals on corrosion behavior in dental implants and restorations.

14. Italian Metrology Institute

A. Cezairliyan is a participant to 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.

15. Johns Hopkins University, Baltimore, MD

Tim Foecke is collaborating with Professor Tim Weihs to study the mechanical properties and thermal stability of Nb/Nb_5Si_3 microlaminates for the Air Force's Office of Scientific Research.

16. Johns Hopkins University, Baltimore, MD

Tim Foecke is collaborating with Professor Pete Searson to study imaging techniques for studying nanowire structures.

17. 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.

18. Massachusetts Institute of Technology

As part of the activities of the Consortium on Casting of Aerospace Alloys, R. J. Schaefer has collaborated with Professor Julian Szekely of MIT on modeling of fluid flow and dendritic growth during directional solidification of aerospace alloys.

19. National Research Laboratory of Metrology (Japan)

A. Cezairliyan is a participant in the cooperative research in thermophysics between NIST and NRLM of Japan.

20. Norfolk State University, School of Technology, Norfolk, VA

A. C. Fraker worked with J. A. Jacobs, School of Technology, Norfolk State University, to produce a NIST hosted workshop that included experiments for use in the classroom, for college and university science and engineering teachers. Miniworkshops were provided in 16 different NIST laboratories.

21. Northwestern University, Evanston, IL

Tim Foecke is collaborating with Professor Scott Barnett in a National Science Foundation funded study of the mechanical properties of W/Al_2O_3 nanolaminates.

22. Ohio State University, Columbus, OH

Tim Foecke is collaborating with Professor Peter Anderson in an investigation into dislocation generation and motion within nanolayered metallic structures.

23. Osaka Prefecture University

R. D. Shull is collaborating with T. Yamamoto to prepare and magnetically characterize vapor condensed magnetic nanocomposites for applications as recording heads and magnetic refrigerants.

24. Paris VI University, School of Chemistry

L. A. Bendersky is collaborating with Prof. R. Portier of the CECM/CNRS and University of Paris VI in a cooperative study of phase transformations and structures of the Ar-Pd-Ru and quasicrystalline Al-Fe-Cu systems using methods of high-resolution transmission electron microscopy.

25. Purdue University

A. Cezairliyan is a consultant to the Thermophysical Properties Research Center - CINDAS at Purdue University.

26. Russian Academy of Sciences

A cooperative program with Professor Dr. V. I. Nikitenko of the Russian Academy of Sciences and A. Shapiro, F. W. Gayle and D. Kaiser of NIST is underway to examine the dynamics of twin boundary migration in high temperature superconductors.

27. Smithsonian Institution

C. A. Handwerker is collaborating with P. Vandiver of the Smithsonian Institution's Conservation Analytical Laboratory in an examination of ancient materials technologies.

28. Universite Libre de Bruxelles

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 soldification processes. The effect of Soret diffusion on the onset of morphological stability during the directional soldification of a binary alloy has been calculated.

29. 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.

30. University of Cambridge

E. Drescher-Krasicka is collaborating with Professor John Willis, Department of Applied Mathematics & Theoretical Physics at the University of Cambridge on developing the amplitude approach to acoustic elasticity for stress imaging.

31. 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.

32. University of Frankfurt

A collaboration among S. Klein of the University of Frankfurt and P. Vandiver of the Smithsonian Institute and C. A. Handwerker of NIST is underway to investigate ancient methods of bronze production in the Middle East.

33. 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.

34. University of Illinois

Professor Jonathan Dantzig of the University of Illinois is working the NIST Consortium on Casting of Aerospace Alloys to develop methods to optimize the design of castings.

35. University of Iowa

Professor C. Beckerman and W. J. Boettinger are collaborating on the modelling of freckle formation in multicomponent superalloys.

36. University of Maryland, Mathematics Department and Institute for Systems Research, College Park, MD

R. B. Clough, Materials Performance Group, is collaborating with Prof. Benjamin Kedem of the Mathematics Department at the University of Maryland. They are using advanced signal processing methods in the development of smart acoustic emission sensors for industrial and aerospace applications.

37. University of Michigan, Ann Arbor, MI

Tim Foecke is collaborating with Professor Gary Was in the preparation of an invited review paper on fracture and deformation of microlaminates for the journal Thin Solid Films.

38. University of Minnesota, Minneapolis, MN

Tim Foecke is collaborating with Professor William Gerberich in a study of dislocation/crack interactions.

39. The University of Mississippi

Gery R. Stafford is collaborating with Charles Hussey on electrodeposition of alloys.

40. University of Notre Dame, Notre Dame, IN

R. E. Ricker has been working with the University of Notre Dame to study oxide film growth under anodic polarization and the control and use of this to create two dimensional nanostructures.

41. University of Southhampton (UK)

W. J. Boettinger is collaborating with Dr. Adam Wheeler, Mathematics Department, University of Bristol on phase-field methods to treat solidification problems. The alloy theory developed previously was extended to treat eutectic solidification.

42. University of Surrey

S. D. Ridder and F. S. Biancaniello collaborated wtih Professor Panos Tsakiropoulos to study EHD atomized sub-micron particle of Mg-rare earth alloys.

43. University of Wisconsin at Madison

W. J. Boettinger and U. R. Kattner are working with Professor Y. Austin Chang at 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.

44. Vanderbilt University

R. D. Shull is collaborating with C. Lukehart to prepare and characterize magnetic nanocomposites prepared by sol-gel routes for magnetic recording applications.

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855.10 ELECTROCHEM. PROCESSING

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Moffat, T.P.
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Sharpless, P.N.

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855.11 MAGNETIC MATERIALS

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Donahue, M.J.
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Egelhoff, W.F.
Ha, M.
Mathews, D.E.
McMichael, R.D.
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855.12 MATERIALS PERFORMANCE

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DeWit, R.
Fields, R.J.
Flanigan, C.D.
Foecke, T.J.
Fraker, A.C.
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Hicho, G.E.
Low, S.R.
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855.13 MATLS. STRUCT. & CHARACT.

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855.14 METALLURG. PROCESSING

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Biancaniello, F.S.
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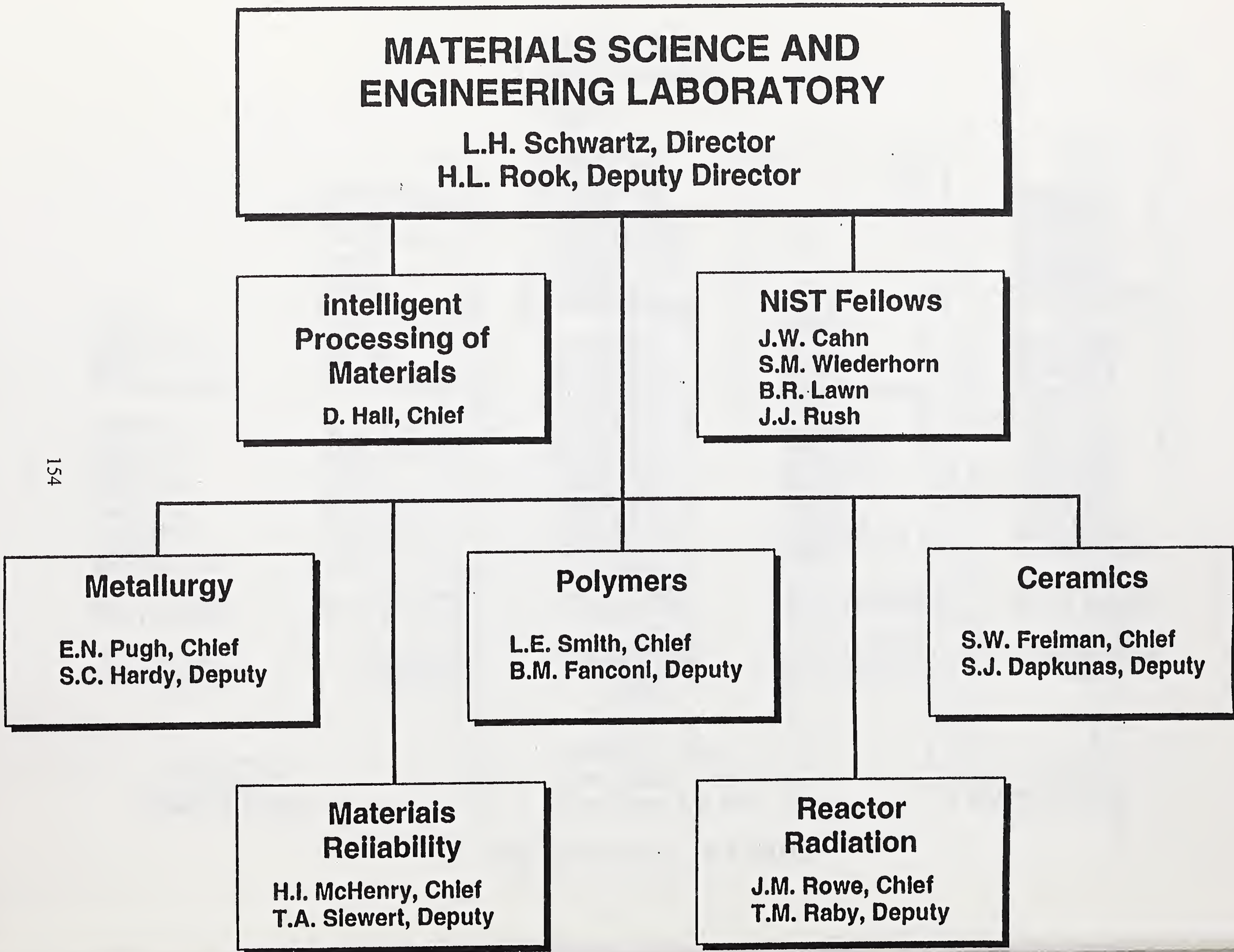
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National Institute of Standards and Technology

Organizational Chart