IMSE

Institute for Materials Science and Engineering

METALLURGY

NAS-NRC
Assessment Panel
February 1–2, 1990

NISTIR 89-4151
U.S. Department of Commerce
National Institute of Standards
and Technology

Technical Activities 1989

The Supersonic inert Gas Metal Atomization (SiGMA) technique produces ultra-fine (<45μm median particle diameter), rapidly solidified metal powder via high energy gas atomization. Research at the National Institute of Standards and Technology on SiGMA has focused on providing this system the ability to monitor and control particle size during atomization. The novel process controller currently under development for SiGMA includes a real-time particle size measurement sensor and multiple level I/O command interfaces. The modular nature of both the hardware and software design will be adaptable to other particulate producing equipment. Gas and liquid flow imaging, gas flow modeling and process control will be discussed as well as real-time particle size measurement, and computer control strategies.

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J.H. Smith, Deputy Chief

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ABSTRACT

This report summarizes the FY 1989 activities of the Metallurgy Division of the National Institute of Standards and Technology (NIST). These activities center upon the structure-processing-properties relations of metals and alloys, on methods of measurement, and on the generation and evaluation of critical materials data. Efforts comprise studies of metals processing and process sensors; advanced materials - including metal matrix composites, intermetallic alloys and superconductors; corrosion and electrodeposition; mechanical properties; magnetic materials; and high temperature reactions.

The work described also includes two cooperative programs with professional societies (the Alloy Phase Diagram Program with ASM International, and the Corrosion Data Program with the National Association of Corrosion Engineers); two with trade associations (the Temperature Sensor Program with the Aluminum Association, and the Steel Sensor Program with the American Iron and Steel Institute); and several with industry including the Powder Atomization Consortium with two companies.

The scientific publications, committee participation, and other professional interactions of the 77 full-time and part-time permanent members of the Metallurgy Division and its 36 guest researchers are identified.

TABLE OF CONTENTS

OVERVIEW

METALLURGY DIVISION (450)

E. Neville Pugh, Chief
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June Toms, Secretary

The basic programs in the Metallurgy Division fall into the traditional NIST areas of measurement science, materials characterization, and data and SRMs, but many have undergone significant changes over the past few years. In response to NIST'S clear mandate to assist in increasing the nation's industrial competitiveness, our emphasis in measurement science continues to evolve towards processing, specifically to process sensors and, in some instances, to process modeling and automated process control. The focus on processing has also led to a more direct interaction with industry, as evidenced by the consortium for powder atomization and other joint programs. In materials characterization, the thrust is towards new, advanced materials, including metal matrix composites, intermetallic alloys, and nanocrystalline magnetic materials, as well as high T_c superconductors. The Corrosion Data Program (with NACE) has replaced the successful Alloy Phase Diagram Program (with ASM International) as our largest data activity. It will be seen in the following summary that there is considerable cross cutting between the main program areas, and that many programs involve strong interactions between the seven groups which constitute the Division.

The major programs are conveniently grouped into three categories:

Metals Processing and Sensors

Powder processing represents a major current thrust, with programs involving both powder atomization and consolidation. The former centers on the Metallurgical Processing Group's high pressure inert gas atomizer, and is a cooperative effort with two other NIST Centers (Chemical Engineering and Manufacturing Engineering) and three companies, via a consortium. The program is directed towards the measurement and real-time control of powder size, such control being important for efficient consolidation and to obtain desired properties, particularly in RSP applications. During this second year of the scheduled three year program, work has continued on the use of a Fraunhofer diffraction technique for in-situ measurement of particle size during the atomization of Type 304 steel. The studies have shown that this laser technique can successfully measure the particle size distribution, and the system is now being modified to improve the response time. Progress has also been made in the development of an artificial intelligence based controller which utilizes adaptive learning procedures. Studies of the atomization process have also yielded new insight, indicating that droplet formation is initiated from a thin cylindrical sheet of molten metal that forms below the liquid delivery tube; thinning of this sheet leads to ligament and droplet formation.

Powder consolidation studies have been focused on hot isostatic pressing of Ti-Al intermetallics in a program carried out in the Advanced Sensing and

Metallurgical Processing Groups. The goal of this DARPA supported program is to develop intelligent processing systems for fabricating difficult materials such as titanium aluminides into near net shape components. The immediate NIST role is the development of sensors for monitoring densification in real time and, during the past year, continued progress has been made in adapting the eddy current technique for this application. The results are being compared with the predictions of process modeling by Division staff and by M. Ashby, Cambridge University, who is collaborating in the program. Collaboration is also continuing with computer scientists from the BDM Corporation, who are making significant progress in developing the controller for the system. During the year, NIST hosted the Second International Conference on Hot Isostatic Pressing, and organized several workshops to inform industry representatives of progress in the DARPA program.

Other sensors are being studied in the Advanced Sensing Group. Work is continuing on the use of the eddy current sensor, mentioned in connection with the HIP program, to measure the temperature of aluminum during fabrication processing. This joint project with the Aluminum Association had made substantial progress in the past year. It has been extended from cylindrical and square sections to more complex shapes such as I-beams and channels, and is now being applied to sheet and plate. In addition, two plant tests were successfully performed. Work has also continued on ultrasonic sensors being developed with the American Iron and Steel Institute. Earlier laboratory tests demonstrated the feasibility of a method for measuring the internal temperature of solid steel, and ongoing studies are aimed at using the approach to determine the position of the liquid-solid interface in partially solidified metal, information which would be valuable to the measurement of shell thickness during continuous casting. The approach has been successfully applied to aluminum, used in preliminary tests to avoid the high temperatures associated with steel, and is currently being extended to aluminum-silicon alloys to investigate the effect of the mushy zone associated with alloys. An exploratory study is also being conducted for the General Electric Company to determine the feasibility of ultrasonic sensing of shell thickness during skull melting of a nickel-based superalloy.

The High Temperature Materials Chemistry Group is continuing its development of thermochemical computer models for liquid iron and steel processing. Such models are particularly timely in view of recent renewed interest by industry in developing radically new direct reduction steelmaking processes. The modeling research is supported by solution thermochemical data obtained using an unique high pressure, high temperature mass spectrometer facility which allows measurements to be made at temperatures, pressures and compositions representative of steel processing conditions.

<u>Characterization of Advanced Materials</u>

Metal matrix composites (MMC), represent an important emerging technology in which we have mounted a significant effort, cutting across several groups. The bulk of the work so far has dealt with continuous fibers and, in the Metallurgical Processing Group, has focussed on the fiber-matrix interface, which controls the properties of these high performance composites. The factors controlling interfacial reactions in composites are being identified through transmission electron microscope studies of Al-SiC composites using a range of SiC phases and morphologies as reinforcing agents. In particular, reactions at Al-SiC interfaces were used as models to evaluate interface roughness and facetting generated by diffusion, phase connectivity, and orientation relationships between nucleating phases and the SiC substrate. These results suggest that composites containing a reaction product susceptible to corrosion can be made more corrosion-resistant if the reaction product forms as isolated precipitates rather than completely coating the reinforcing phase.

The Electrodeposition Group is continuing its novel approach to composites, in which electrodeposition is employed to coat the fibers, either to produce barrier or other intermediate layers or to directly electroform the matrix. Development of a cell for high rate deposition on single fibers has continued and significant increases in the rate of mass transport, and hence deposition rate, have been achieved. In the last year, the cell was used successfully in a feasibility study for Textron Specialty Materials in which carbon fiber was plated with a complex multilayer of copper, nickel and an alloy diffusion layer. Work on the direct electrosynthesis of TiAl is also progressing satisfactorily, the focus is now on increasing the titanium concentration in the electrolyte to increase the deposition rates.

Intermetallic alloys represent another major activity in this program area. It was seen that HIP studies and alloy electrodeposition are being carried out on Ti-Al alloys, which are potentially important for high temperature structural applications. A major DARPA supported program on ternary alloys based on this system is in progress in the Metallurgical Processing Group. Although many of the early promising property reports for intermetallics were obtained with single phase materials, further improvements in properties are anticipated using multiphase structures. This requires the selection of new compositions and heat treatment schedules. For this purpose, activities are focussed on a determination and/or evaluation of important alloy phase diagrams like Ti-Al-Nb and a determination of the phase transitions available to manipulate intermetallic microstructure. In this system two new phases have been studied, an ω-type phase and an ordered orthorhombic variant of Ti_3Al. This research has required improved competence in displacive and chemical ordering transitions as well as crystallographic group theory. The use of rapid solidification to form unique precursor intermetallic materials for subsequent heat treatment is also being pursued. Ni_2AlTi alloys have been solidified into the B2 structure rather than the equilibrium $L2_1$ using pulsed laser surface melting.

Intermetallic compounds are also being studied by the Corrosion Group. Over the past year, this work focussed on the aqueous corrosion and environmental induced embrittlement or stress corrosion cracking behavior of nickel aluminide alloys being developed by Oak Ridge National Laboratory. The Corrosion Group has characterized the corrosion and pitting behavior of these alloys in solutions of acidic, neutral and alkaline pH and demonstrated that these alloys are embrittled by environmental conditions which promote hydrogen absorption. In addition to this work on nickel aluminide, work was initiated on iron aluminide which will continue into the next year.

Activity in high T_c superconductors was maintained at a high level this year. Using their newly modernized magnetic materials laboratory, the Magnetic Materials Group employed a variety of techniques to characterize bulk and thin film samples produced in MSEL and elsewhere. In addition to producing laser ablated thin films of YBCO and other compounds, the High Temperature Materials Chemistry Group continued its investigation of the mechanism of the deposition process by probing the plume. A particularly interesting development this year was the preparation of "detwinned" single crystals of YBCO by the Metallurgical Processing Group in cooperation with the Ceramics Division. Magnetization measurements of these and of twinned monocrystals is contributing significantly towards our understanding of the critical phenomenon of flux pinning. For example, it was shown that the large anisotropy for the magnetic field, H, parallel and perpendicular to the c axis is not the result of pinning by the twin boundaries.

Other advanced magnetic materials were studied by the Magnetic Materials Group. Their measurements on Ni/Cu compositionally-modulated thin films produced by the Electrodeposition Group with their improved electroplating procedure exhibited (1) high magnetizations and (2) smaller temperature dependencies of the magnetization than any reported previously by any technique. These results demonstrate that electrodepositing nanometer thick layers of the highest quality is feasible. Thin films of a magnetic and a nonmagnetic constituent with a nanometer-sized granular morphology have been produced both by sputtering and by a sol-gel method. The sol-gel process provides monolithic pieces with permit modification of the form of the magnetic species after preparation. These nanocomposites were characterized by Mossbauer spectroscopy, x-ray diffraction, magnetization measurements, transmission electron microscopy, and field-emission scanning-electron microscopy.

<u>Data and SRM Programs</u>

The Alloy Phase Diagram Program on binary alloys, conducted cooperatively with ASM International, is drawing to a successful conclusion. Development and loading of the interactive database containing numerical and graphical data for nearly 1,600 binary systems has been completed and the database transferred to ASM for dissemination. In addition to providing the Editor of the Bulletin of Alloy Phase Diagrams, the Division's remaining activity in the binary program is the evaluation of the iron binaries. The main thrust of our program is now in ternary diagrams, both in evaluation of certain critical systems and in developing a ternary graphics package, and in providing support for other in-house programs such as those described above on MMC and intermetallic alloys.

The Corrosion Data Center, formed in cooperation with NACE in response to a critical need for a centralized, computerized database of corrosion data, continued to expand in 1989. Industry support for the development of focussed programs addressing critical needs has grown. Emphasis has been placed on knowledge-based expert systems which use artificial intelligence concepts to aid corrosion scientists and engineers in selection of materials for applications in corrosive environments. Programs are under developments which examine the storage and handling of hazardous chemicals, downhole equipment for oil and gas production and equipment used in electric power generation under sponsorship of the Materials Testing Institute of the Chemical Process Industries, the Division of Scientific and Industrial Research of New Zealand and the Electric Power Research Institute respectively.

Another data program is being conducted by the Corrosion Group for the Nuclear Regulatory Commission (NRC). The objective of this program is to assist the NRC in evaluating the ability of metallic containers to safely store high level nuclear waste for extended periods without failing due to corrosion. For this program, the Corrosion Group evaluates the scientific principles and the corrosion measurements that the Department of Energy (DOE) is utilizing in their modelling of the service lifetime of high level waste containers by reviewing DOE's technical reports and by conducting experiments to evaluate the validity of the results and conclusions in the reports. The results of these reviews are then stored in a computer data base which can then be used by the NRC in evaluating the high level waste storage techniques which will be proposed by DOE.

As a result of a large oil spill resulting from the rupture of an electric resistance welded (ERW) pipeline, an extensive review of ERW pipeline failures was carried out at the request of Congress to determine if older ERW pipelines are particularly susceptible to failure. It was concluded that the relatively small number of failures in ERW pipelines does not warrant special tests and standards except in critical locations.

The Division continues to maintain a strong program in Standard Reference Materials. In the Electrodeposition Group, production of coating thickness standards proceeded at a high level and research continued on the development of a lead-tin standard for the electronics industry. An intercomparison of test blocks used to calibrate Rockwell hardness test machines was carried out in collaboration with ASTM and hardness block manufacturers. This intercomparison showed that the tests blocks available in the United States for calibrating the Rockwell C scale do not agree within the limits required by the ASTM Standard. Further, a limited intercomparison showed that the U.S. test blocks do not agree with Rockwell C scale test blocks that were calibrated on the metrologically correct hardness standardizing machine at the national metrology laboratory in Italy (IMGC). Therefore, the U.S. test blocks are not compatible with the presently used international hardness scale. This identifies the need to establish a traceable national hardness standard in the U.S.

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Boettinger, William J.	o Relation of alloy microstructures to processing conditions o High temperature alloys/intermetallics o Rapid solidification
Burton, Benjamin P.	o Thermodynamic modeling of alloy phase diagrams o Experiments on metal-oxide phase equilibria o Order-disorder and phase separation in alloy systems
Coriell, Sam R.	o Modeling of solidification processes o Interface stability o Convection and alloy segregation during solidification
deWit, Roland	o Fracture mechanics o Dislocation theory o Stereology
Fields, Richard J.	o Mechanical properties o High temperature materials o Quantitative metallography
Gayle, Frank W.	o Aluminum metallurgy; intermetallics o Transmission electron microscopy o Structure/property relationships
Handwerker, Carol A.	o Interface studies o Metal matrix composites o Diffusion-induced grain boundary migration

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Ricker, Richard E.	o Stress corrosion cracking (SCC) o Corrosion fatigue o Hydrogen embrittlement o Corrosion and SCC of advanced materials o Corrosion and SCC of aircraft alloys

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Beauchamp, Carlos R.	o Computer simulation of electrodeposition process on moving fibers o Electrochemical measurements of kinetic parameters o Composition modulated alloy deposition
Claggett, Sandra W.	o Scanning electron microscopy o Metallographic specimen preparation o General electroplating
Johnson, Christian E.	o Ultra-black coatings o Electroless deposition process o Metallic glass alloy deposition o Microhardness SRM research o Chromium deposition o Pulsed alloy deposition
Kelley, David R.	o Microhardness SRM development o Dye penetrant SRM development o Precious metal electrodeposition o Plating on aluminum
Lashmore, David S.	o Electrochemical mechanisms of coating processes o Pulsed alloy deposition o Composition modulated alloy deposition o Properties and structure of electrodeposited coatings o Amorphous alloys o Transmission electron microscopy o Metal matrix composites
Mullen, Jasper L.	o Development of automated hardness testing o Electrochemical measurements for determining metal corrosion o Analytical spectroscopy
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Magnetic Materials Group

Bennett, Lawrence, H.	o Magnetic measurements o Magnetic thin films o High T_c superconductors o Topology of local environments o Alloy phase stability
Brown, Henrietta J.	o Magnetization measurements
Shull, Robert D.	o Nanocomposites o Magnetic susceptibility o Mossbauer effect o X-ray and neutron diffraction o Scanning electron microscopy
Swartzendruber, Lydon J.	o Magnetic susceptibility o Magnetic methods, NDE o Gamma-ray resonance spectroscopy o Barkhausen effect

High Temperature Materials Chemistry

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Hastie, John W.	o High temperature chemistry of inorganic materials o Phase equilibria thermochemistry and solution models o High temperature-pressure mass spectrometry o Chemistry of combustion
Plante, Ernest R.	o Thermodynamics o Mass spectrometry
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Mechanical Properties of Metals

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Shapiro, Alexander	o Scanning electron microscopy o X-ray microanalysis o Image analysis o Transmission electron microscopy
Shives, T. Robert	o Hardness test methods o Mechanical properties o Failure analysis
Smith, John H.	o Mechanical properties of materials o Fracture of materials o Structural integrity analysis

METALLURGICAL PROCESSING

John R. Manning

Metals processing technology has made such rapid advances in the past several years that it is now thought reasonable for designers of high performance products to ask for alloy properties that previously had been regarded as unobtainable. Material producers frequently can tailor alloy properties by altering compositions and microstructures to fit particular applications. Overall, there is a strong new emphasis on understanding how to control compositional distributions and microstructural features of alloys and then on understanding how these metallurgical aspects influence the alloy properties. The present NIST work on metallurgical processing supports this move toward understanding and control of material properties by emphasizing the development of new measurement techniques, especially those which can provide real-time information about metallurgical processes as they occur, and by working on predictive models that show how processing conditions influence microstructure and alloy properties.

As part of our increased emphasis on interaction with industry, projects on intelligent processing for (1) production and (2) consolidation of alloy powders are underway in collaboration with industrial companies. In the atomization work, the concern in industry for control of the sizes of atomized powder particles has led to a cooperative program in which industrial scientists are working with NIST scientists to develop real-time measurement and control techniques for atomization of steel. In the consolidation area, work is being done with the assistance of industrial companies and DARPA on measurement and control of densification rates during hot isostatic pressing.

During the past few years, NIST rapid solidification investigations have shown how the non-equilibrium conditions and improved homogeneity produced by strong undercoolings and subsequent rapid freezing of alloys can greatly improve final products. Efforts in this area at NIST are now being directed toward specific applications. For example, investigations are being made of the effect of rapid solidification on intermetallic alloys, which are expected to have important aerospace applications. A new effort has been started to measure properties of the intermetallic alloys that occur in solder joints. These alloys cannot be prepared in bulk amounts by conventional techniques but can be produced by rapid solidification. Understanding the influence of phase diagrams on the selection of processing paths is important for each of these efforts.

A similar generic approach of developing measurement techniques and predictive models is being applied to other material applications. Particular areas of work discussed in this report include interactions at interfaces in metal matrix composites, properties of new aluminum alloys, performance of steel at high temperatures, production of single-crystal high T_c superconductors, and alloy coarsening measurements.

Much of this work on applications to specific materials is in response to requests by industry groups or other agencies. For example, funding is supplied by an industrial consortium for the atomization work, by DARPA for work on intermetallics, by the Harry Diamond Laboratory for the solder investigations, by ONR for work on composites, by NASA for the work on directional solidification and alloy coarsening, and by the Nuclear Regulatory Commission for work on crack arrest processes in reactor steels. Cooperation continues with the national steel program mandated by Congress for development of new steel technology and with national efforts on high T_c superconductors and metal matrix composites.

FY 89 Significant Accomplishments

- High speed cinematography at 10,000 frames per second was used to study droplet formation in our Supersonic Inert Gas Metal Atomizer during the atomization of stainless steel to form fine steel powder. Examination of these frames showed that droplet formation initiates from a hollow sheet formed at the end of the liquid delivery tube. The liquid sheet formation and the droplet recirculations that are observed are new and unexpected results of this flow visualization study. These results provide important information for automated control of this system.

- Rapid solidification and hot isostatic pressing techniques have been applied to produce for the first time bulk samples of the difficult-to-prepare intermetallic alloy Cu_6Sn_5. This alloy is a brittle phase that forms in solder joints where a solder connection is made to copper. This result makes it possible to measure many properties of this material, which at present are not well known.

- A thermomechanical method was invented for detwinning single crystals of $YBa_2Cu_3O_{6+x}$ high T_c superconductor. This accomplishment allows measurements to be made that distinguish for the first time orientation dependences along the two basal principal axes of this material.

- The elevated temperature deformation properties of structural steel have been analyzed to provide a base for predicting the behavior of structures exposed to fire. The data have been summarized by a set of equations which predict the elastic, plastic and creep strain which will occur in steel exposed for various lengths of time to temperatures up to 650°C. These equations have been expressed as transient deformation mechanism maps which describe at a glance the strain behavior as a function of stress and temperature.

- Diffusion across interfaces in composite materials can introduce large local stresses when lattice parameters depend on composition. These stresses lead to interface roughening which strongly affects composite properties. The critical self-stress conditions for transition from the interface roughening instability to recrystallization were determined using model experiments in the Mo-Ni-Fe system.

Powder Processing
S. D. Ridder, F. S. Biancaniello, R.J. Schaefer

Two industry/government consortia projects on powder processing, one on powder production and the other on powder consolidation, have provided the major thrust of the powder metallurgy work at NIST during the past year. The former is a cooperative effort of the NIST Metallurgy Division with industrial researchers and two other NIST centers, the Center for Chemical Engineering (CCE) and the Center for Manufacturing Engineering (CME), on automated processing and particle size control of rapidly solidified stainless steel powders produced by high pressure inert gas atomization. The latter is a cooperative effort of the NIST Metallurgy Division, BDM Corporation and DARPA, with advice provided by a dozen other industrial companies involved in hot isostatic pressing (HIP), to develop sensors, software and metallurgical information to apply intelligent processing and control to HIP densification procedures.

These projects are examples of the new emphasis at NIST on applications of intelligent processing, which require the development of sensors that measure processes in situ, while the material is actually being produced. The output of these sensors and comparison with previous results then can be used to design processing conditions that provide improved products and, in some circumstances, even modify the process while it is occurring, thus reducing the number of rejected parts.

<u>Atomization -- Real-Time Powder Particle Size Measurement and Control</u> - The NIST/Industrial consortium on automated processing for atomization has completed two years of a scheduled three year multi-disciplinary study on automation utilizing the Metallurgical Processing Group's supersonic inert gas metal atomizer (SiGMA). For purposes of controlling alloy properties or for improved powder handling, as in injection molding, there frequently is need for fine powder of uniform size. This project is directed toward the measurement and control of powder sizes during the atomization process. NIST scientists have collaborated in this work with industrial representatives from Crucible Materials Corp., General Electric Co., and Hoeganaes Corp.

Current activities include the incorporation of an in-situ particle size measurement sensor in an advanced process controller to provide rapid feedback and control of stainless steel atomization. Fluid mechanics experts from CCE are participating in the analysis and modeling of gas and liquid flows. The particle measurement sensor, being developed in cooperation with CCE fuel spray diagnostic experts, is a Fraunhofer diffraction instrument specially modified for rapid data transmission. CME scientists are involved in the design of an Artificial Intelligence based adaptive controller that will process sensor data and output appropriate actuator signals to maintain or change the particle size distribution as directed by a response function "learned" during previous programming sessions. This system will adapt to new sensor input data patterns by continuously updating the response function during each atomization session.

Fluid flow studies aid by providing understanding of how to control the atomization process. In this process, a liquid stream is broken up by high pressure inert gas into very fine droplets which then rapidly solidify into very fine powder particles. The fluid flow studies include detailed velocity and pressure profiles of the gas jets used to produce the ultra-fine (less than 45 μm diameter) powder in the SiGMA facility and also high speed movies of atomization of 304 stainless steel. Careful frame-by-frame examination of these movies has shown that droplet formation is initiated from a hollow liquid sheet that forms at the bottom of the liquid delivery tube. Ligaments and droplets form as the sheet thins and holes-through, as illustrated in Figure 1. The initial droplets often recirculate for several cycles in the vicinity of the liquid delivery tube before they are entrained in the supersonic gas flow. The liquid sheet formation and recirculation are new and unexpected results of this flow visualization study.

Hot Isostatic Pressing -- Real-Time Monitoring of Densification - Densification of metal powders by hot isostatic pressing (HIP) has been measured in detail at temperatures up to 1373 K and pressures up to 170 MPa. The density is measured in real time, using sensors developed by the Advanced Sensing Group, and the results are compared to the predictions of a process model developed by Prof. M. Ashby of Cambridge University. A controller system for the HIP has been developed by computer scientists from the BDM Corporation, working on the project under a Cooperative Agreement. The goal of this DARPA-sponsored project is to increase the efficiency of fabricating difficult materials such as titanium aluminides into near net shape components.

Titanium aluminides are a particularly challenging case for application of the process model, because they often undergo a phase transformation during heating to the HIP temperature. A recent set of measurements showed that this transformation, which occurs at about 1050 K, correlates with an unexpected temporary increase in the powder densification rate. Measurements with other materials have also shown deviations from the process model, pointing the way to its refinement. In the case of copper an unexpectedly high rate of densification at low temperatures was identified by Prof. Ashby as low temperature creep, and he has added this effect to the process model. A variety of other powder materials, including high-temperature superconductors, steels and Cu-Sn alloys, have been consolidated by HIP without detailed analysis of the densification kinetics.

An International Conference on HIP was organized and held at NIST on June 7-9, 1989. With an attendance of over 100, the conference covered the latest advances in HIP science and technology, and the proceedings will be published by the American Society for Metals.

Microstructure and the Properties of Steel
R. J. Fields, R. deWit, R. D. Jiggetts, and S. R. Low III

Three Dimensional Microstructure - The importance of steel in its varied applications arises from its mechanical behavior, formability, and other performance-related characteristics such as fire resistance, surface finish, or paintability. These properties are determined by microstructures that result from processing. Intelligent processing of steel requires a continuous sensing of microstructure and using that information to control the forging, rolling, or drawing. This sensing is inferential and is based on correlations with actual measurements of microstructure. Such measurements are made on a two dimensional cut through the three dimensional microstructure. In this research, methods have been pursued for deducing the three dimensional microstructure (to which sensors respond) from the two dimensional section. A computer code was developed during the past year for simulating the 3-D grain structure of materials. The code permits grains of various sizes and shapes to fill space. A projected image of this packing can be displayed and 2-D planar cuts through this structure can then be made. These cuts simulate metallographic sections which are traditionally used to determine grain size in real materials. In the computer, the generating 3-D grain size and shape are known. This code is being used to evaluate various stereological techniques for deducing the 3-D microstructure from 2-D metallographic sections.

Toughening Steel - Many steels are used today under conditions that would result in brittle fracture (i.e., cleavage) if cracking started. There is a need to identify the microstructural bases for toughness in cleavage and the sources of ductility that lead to more plastic behavior. To identify what physical processes are responsible for steel behavior, detailed microstructural and theoretical analyses of the recently completed NIST/NRC wide-plate crack-arrest tests on nuclear pressure vessel steels were made. These investigations indicate that the high arrest toughnesses exhibited by these steels are associated with the formation of late breaking ligaments (i.e., escarpments formed between non-coplanar cracks). These ligaments are similar to the microscopic ledges which are visible on all cleavage surfaces and are responsible for the typical river pattern of cleavage. The present research points to these ledges as the source of dislocation activity triggering the brittle-to-ductile transition. Hence, processing routes which encourage the formation of these microstructural features should produce more fracture resistant steel.

Fire Resistance of Steel - With the increasing use of steel skeletons in high rise buildings, it is becoming more important to be able to assess or predict the damage sustained by such a building during a fire. An analysis of the problem needs to be based on knowledge of the stresses and temperatures involved and constitutive equations to determine the resulting strains. To provide a constitutive data base for predicting the behavior of structures exposed to fires, the elevated temperature deformation properties of structural steel have been collected, evaluated, and analyzed in recent work at NIST. The data have been summarized by a set of equations which predict the elastic, plastic, and creep strains which will occur in steel exposed for various lengths of time to temperatures up to 650 C. These equations have been evaluated for specific times and expressed as transient deformation mechanism maps, such as shown in Figure 2, which describe at a glance the strain behavior as a function of stress and temperature.

Physical Metallurgy and Processing of Intermetallics and Other Advanced Alloys

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Recent scientific and technological developments have produced new processing methods and new classes of alloys with significantly improved properties. Examples are new high temperature capabilities resulting from rapid solidification and improved intermetallic alloys and composites. These advances depended on the production of more nearly optimum alloy microstructures. The focus of the present work is on development of techniques to predict and control these microstructures.

To this end, analysis of solidification and solid state transformation kinetics is combined in the present work with a program on alloy thermodynamics and phase diagrams to determine how changes in processing conditions affect microstructures. A significant part of this research is directed toward the examination of non-equilibrium processing paths involving precursor metastable phases produced by rapid solidification or solid state quenching. These paths can be used to produce stable multi-phase structures after heat treatment. Such microstructures often are unobtainable by conventional means. Intermetallic and Al-base alloys and their associated phase diagrams are being examined. All parts of this task require an increased understanding of the metallurgy of ordered phases.

<u>Solubility Extension and Disordering of Intermetallic Compounds by Rapid Solidification</u> - The formation of ductile second phase particles in intermetallics has the potential to increase the toughness of these materials. Since equilibrium phase diagrams suggest only limited opportunities for precipitation from supersaturated intermetallic phases by conventional heat treatment, the possibility of extending the range of solubility of intermetallics by rapid solidification is being examined. Experimental research on the microstructure of rapidly solidified alloys in the NiAl-NiTi system is being conducted in collaboration with Professor Mike Aziz of Harvard using pico-second pulse surface melting. This technique produces solidification rates of 1-5 m/s. Results on alloys with composition Ni_2TiAl show that extremely fine $L2_1$ domains are produced by this technique, suggesting that the solidification process suppressed the formation of the highly ordered $L2_1$ structure and produced the less ordered B2 phase, which can also have a large range of solubility. To be certain that a nonequilibrium solidification product was obtained, it was necessary to establish that the $L2_1$ phase is in fact the equilibrium phase at the solidus. This was confirmed using high temperature X-ray diffraction of Ni_2TiAl by measuring the long range order parameter as a function of temperature. Once the precise solidification velocity of this transition from $L2_1$ to B2 is determined using the laser melting technique, a comparison to a recently completed "Theory for the Trapping of Disorder and Solute in Intermetallic Phases by Rapid Solidification" will be performed.

<u>Investigation of Solid-State Phase Transitions of Quenched BCC and B2 Alloys in the Ti-Al-Nb Intermetallic System</u> - Titanium aluminides (Ti_3Al and TiAl) with ~10 at% Nb additions have received considerable attention as potential low density, high strength and creep resistant materials. However, the phase equilibria in this ternary system are poorly understood. Because the presence of BCC-based phases in these alloys seems to play an important role in the deformation, in-depth studies of the BCC-based phase fields are being conducted. A series of alloys surrounding the composition Ti_2AlNb have been examined by TEM to determine the structure of phases present in arc cast and heat treated samples. A broad composition range has been identified where the Nb stabilized BCC-Ti or β high temperature phase field is ordered into the B2 structure. On quenching or slow cooling, a composition range has been

identified in which this high temperature B2 phase transforms to an ordered ω phase. An associated equilibrium $B8_2$ phase field has also been found. The details of this transformation have been analyzed as a simultaneous chemical and displacive ordering transition. The poor toughness of this phase has been confirmed by high load microhardness testing. Alloys containing an orthorhombic phase based on the stoichiometry Ti_2AlNb exhibit significant toughness and a microstructure consisting of a complex morphology of intersecting plates of the orthorhombic phase. This phase is an orthorhombic distortion of the α_2-Ti_3Al phase caused by ordering of the Ti and Nb on the Ti sites. The formation of this plate structure is governed by the minimization of elastic energy when the B2 phase transforms to the orthorhombic in a coherent fashion on cooling. The role of this plate structure on the toughness is being examined.

Theoretical studies of coupled displacive/chemical order-disorder transitions have been conducted to support of the experimental work on Ti-Al-Nb alloys. A technique was developed for combining a Landau theory treatment of displacive instabilities with a cluster variation method (CVM) treatment of chemical ordering. A simplified two dimensional model has been constructed and it demonstrates many of the phenomena observed. For example, in Ti_4Al_3Nb the observed ω-type collapse which occurs in the chemically ordered B2 phase was accompanied by a subsequent chemical rearrangement which involved a disordering step. In the model calculation, one sees that an increase in Q, the order parameter associated with ω-collapse, leads to a decrease in T_c, the critical temperature for the order/disorder transition. This occurs in the model, as in the real system, because the distortion associated with Q increases the frustration of chemical ordering. That is, the Q distortion leads to a geometry in which it is more difficult to arrange all pairs, triangles, etc. of near-neighbor atoms in their most energetically favorable configurations. A CVM treatment of the chemical ordering is required to get realistic models of frustrated systems because they are the systems in which lower order approximations such as Bragg-Williams fail. This approach should be very fruitful for studying a wide variety of martensitic transitions in ordered alloys.

Aluminide Phase Diagrams - Ternary phase diagrams, which are important for a systematic approach to alloy development, are being calculated for the Nb-Al-Si and Ti-Al-Nb systems. For this purpose, a new thermodynamic description for the Nb-Al system was formulated which takes into account the crystallography of the Nb_3Al and Nb_2Al phases; i.e., it treats the two phases as ordered intermetallic compounds which consist of at least two sublattices. The deviations from stoichiometry of these two phases are modelled as the formation of substitutional solutions on each of these sublattices. Also a thermodynamic description for the Nb-Si system was developed and was used together with the newly developed description of the Nb-Al system and a description of the Al-Si system from the literature to predict the ternary Nb-Al-Si system. This system is of interest for potential high temperature oxidation resistance due to alumina or silica formation. From experimental ternary measurements in the literature, only equilibria involving solid phases or the (Al,Si)-rich liquid are known. Also the existence of two ternary phases is known. One ternary phase, $Nb_5(Al,Si)_3$, is modelled as a solid solution of βNb_5Si_3 and a metastable phase Nb_5Al_3, while $Nb(Al,Si)_2$ is modelled as a true ternary compound with the stoichiometry $Nb_3Al_2Si_4$. The most uncertain aspects of the calculated phase equilibria in the ternary system are the Gibbs energies of formation of Nb_5Al_3 and $Nb_3Al_2Si_4$. Calculations were carried out for various values to demonstrate their influence on the liquidus surface. The results from these calculations were presented at the CALPHAD XVIII Conference and are shown in Fig. 3. Critical experiments can now be planned to remove this uncertainty. Calculation of the Ti-Al-Nb ternary system is also in progress.

ASM/NIST Phase Diagram Program - Ten re-evaluations of binary alloy systems have been submitted to ASM. This includes a updated literature survey and an assessment of the quality of the data. The Nb–Al, Ta–Al, Fe–Al, Ga–Te, Fe–Li, Fe–Na, Fe–K, Fe–Rb, Fe–Pb and Fe–Zn binaries have been evaluated. In the case of Fe–Al, the order-disorder in the Fe-rich region has been modeled with CVM calculations that are based on the tetrahedron approximation. Several of these systems are important for aluminide alloy development.

Aluminum-base Quasi-Crystal Investigations - The Al-Cu-Fe ternary system is technologically important (e.g., aluminum bronzes, Fe-Al high temperature intermetallics, etc.) as well as scientifically interesting. As an example of the latter, the most perfectly ordered quasicrystal to date appears to be an equilibrium phase at approximately $Al_{64}Cu_{25}Fe_{11}$. However, very little is known regarding a large region of the ternary phase diagram. The (Al > 50at%, Fe < 25at%) region is being investigated experimentally to establish primary phase fields, lines of two-fold saturation with the liquid, phase equilibria in various isothermal sections, and ranges of solubility for each phase. The topology of the liquidus surface has been mapped, and tie lines between several phases have been located. Location of this liquidus surface should provide the information required to grow large quasi-crystals from the melt.

Using rapid solidification, polycrystalline aggregates with overall icosahedral symmetry were found in AlMnFeSi alloys. The orientation relationship between crystals is such that icosahedral motifs in all the crystals are parallel. Although the cubic axes undergo five-fold rotation about irrational $<1,\tau,0>$ axes, only five orientations occur among hundreds of crystals. This is a special orientation relationship, but there is neither a coincidence or twin lattice ($S=\Sigma=\infty$). The concept of twinning and special grain boundaries was reexamined, and a new definition of special orientations (hyper twins) based on the reduction of the number of arithmetically independent lattice vectors was proposed. It includes both old and new special orientations and can be easily interpreted in terms of quasilattices.

Strengthening Phases in Aluminum-Lithium Alloys - Martin-Marietta Laboratories is collaborating in a study of the physical metallurgy and microstructure of a new class of weldable, ultra-high strength aluminum-lithium-copper alloys. These alloys, originally targeted for the Advanced Launch System, have twice the yield strength of the present alloy (2219) of the Space Shuttle External Tank and hold promise for a quantum jump in structural alloys for aerospace and other applications. The research has been directed at determining the microstructural basis for the high strength and toughness and other physical, chemical, and mechanical properties of these alloys. The system has been found to contain many strengthening precipitate phases which form during aging of solution heat treated and quenched alloys, ranging from a metastable equilibrium structure of Al, GP zones and $\delta'(Al_3Li)$ in the naturally-aged condition to a combination of three or four strengthening phases in the peak strength condition: T_1 (Al_2CuLi), S' (Al_2CuMg), θ' (Al_2Cu), and a previously unidentified new phase.

Processing and Properties of Special Materials
J. R. Manning, F. S. Biancaniello, W. J. Boettinger, S. R. Coriell, R. J. Fields, F. W. Gayle, S. R. Low III, and R. J. Schaefer

A guideline in the NIST work on metallurgical processing is the idea that the best way to maximize alloy performance is to understand how alloy properties depend on microstructure and composition distributions and then to devise methods to reliably control and reproduce these features. With this idea in mind, projects on a number of technologically important materials for predictive modeling of the effect of processing conditions on microstructure and then of the effect of microstructural distributions on properties are being pursued. Investigation on steels, intermetallics, and aluminum alloys were described in the previous two sections of this report. In addition, work was initiated on solder joint materials during the past year, and work of this type on electronic materials and high T_c superconductors is continuing. Accomplishments in these work areas are described here.

Materials Properties of Solder Joint Materials - The prediction of in-service performance of solder joints requires a complex blend of mechanical, thermal and electric design with data on the properties of the constituent phases or mixtures of phases which occur in joints. The joint microstructures are sensitive to the materials being joined, the composition of the solder and the soldering conditions as well as the post-joining thermal environment. A project has been initiated at NIST to determine property data of various intermetallic phases, such as Cu_6Sn_5, found in joints and to determine the plastic yield surface for Pb-Sn eutectic solder with selected microstructures. This information is needed for predictive models that are being prepared at other cooperating laboratories as part of an industry/government national effort initiated because of the increasing importance of surface mount solder joints in microelectronics.
A processing method for the preparation of bulk (greater than 1 cm^3) intermetallic phases has been developed using melt spinning of the desired composition, such as Cu_6Sn_5, to reduce microsegregation of the solidified material. Powder ground from the ribbons was consolidated by hot isostatic pressing (HIP) to produce fine grained single phase intermetallic samples. Previous attempts carried out elsewhere to produce bulk samples of this alloy by slow cooling have been unsuccessful because of alloy segregation that occurs before the peritectic temperature is reached. The presently produced alloy samples achieved by rapid solidification and subsequent consolidation of powder constitute the first bulk samples of this difficult-to-prepare Cu_6Sn_5 intermetallic.

Hot indentation testing of Cu_6Sn_5 has been completed in the temperature range 20-220°C. Results confirm that the intermetallic compound is extremely hard at 20°C. However, it shows a dramatic reduction in hardness (i.e., flow strength) with temperature. Many indentation impressions exhibit cracking from their corners and measurement of the crack length can be used to derive fracture toughness values when the elastic modulus is known.

Testing of 63Sn-37Pb high purity solder samples was begun after the bulk microstructure of the test specimens was shown to resemble that of solder from certain areas of selected circuit board joints. Superplasticity and serrated yielding, as well as normal behavior, have been observed at various strain rates and temperatures.

<u>Directional Solidification of Alloys and Semiconductor Materials</u> - Control of solute segregation during solidification will allow preparation of materials with improved properties. At rapid solidification velocities, it is possible to produce partitionless solidification where in essence the different types of atoms do not have time to partition themselves into equilibrium phases and compositions. At slower solidification velocities, however, typical of directional solidification of semiconductors for use as electronic materials and in continuous casting of steel, fluid flows and instabilities at the solidification interface can strongly affect the microstructures and composition distributions produced in the resulting solid. To aid in prediction and control of these effects, convection and interface stability during alloy solidification have been analyzed and modelled. This work was done in collaboration with G.B. McFadden, L.N. Brush, and B.T. Murray of the NIST Center for Applied Mathematics, R.F. Sekerka of Carnegie-Mellon University, and A.A. Wheeler of the University of Bristol.

Electric pulsing is a technique often used in model experiments to mark interface positions at successive times during solidification. In the present work, a numerical method has been developed to study the free boundary problem for the motion of a planar crystal-melt interface during directional solidification of a binary alloy in a time-dependent electric current. The model includes the Thomson effect, the Peltier effect, Joule heating and electromigration of solute in the coupled set of equations governing heat flow in the crystal and the melt as well as solute diffusion in the liquid. Quantitative predictions were made for tin-bismuth and germanium-gallium on the effect of an electric pulse on solute distribution in the solidified materials.

For processing conditions in which interface instabilities occur, the question of wavelength selection for these instabilities is crucial in defining the distance scales of the resulting microsegregations and microstructures. These features in turn determine alloy properties. For the case in which a heavier solute is rejected at the solidifying interface, our calculations show that destabilization is enhanced as the variation of density with solute concentration is reduced. For neutrally dense solute there is a long wave-length instability and the destabilization is very sensitive to the ratio of crystal and melt thermal conductivities. Calculations also were performed for crystals having anisotropic thermal conductivities. For low growth velocities, the onset of instability occurs for perturbations having a wave vector that lies along a principal crystallographic axis.

High T_c Superconductors - Thermomechanical Detwinning - An application of the influence of stress on the thermodynamics of ordered phases has led to a method to detwin an important high T_c superconductor material. To date, most investigations of the high temperature superconductor $YBa_2Cu_3O_{6+x}$ have been performed on twinned "single crystal" and polycrystalline ceramic samples. These materials contain two twin variants, in which the a and b axes of the orthorhombic unit cell are interchanged. Consequently, physical characterization studies have not been able to resolve a-b anisotropy or study the effect of twin boundaries on magnetic and electrical properties. These phenomena will be critical to the performance of the material, particularly in electronic devices which are fabricated on single crystals or thin films.

In collaboration with the NIST Ceramics Division a thermomechanical process has been developed for the complete removal of twins from $YBa_2Cu_3O_{6+x}$ single crystals. In the process, a crystallographically-oriented compressive stress is applied to a single crystal at elevated temperature. By a ferroelastic-like transformation, the a and b axes of the unfavored twin variant unit cell are switched, leading to migration of the twin boundaries and ultimately a complete predominance of the favored twin variant. This is an excellent example of symmetry breaking (making the energy of the two twin domains unequal) due to an applied field, which is stress in this case. Collaborations are ongoing with Louisiana State University and Iowa State University for structural analysis of the untwinned crystal at cryogenic temperatures, and with the Magnetic Materials Group (L. Swartzendruber, L. Bennett) regarding magnetic anisotropy and the effect of twins on magnetic properties.

Composites and Interface Reactions
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Metal matrix composites are prime examples of complex engineering systems where materials are not in thermodynamic equilibrium during initial fabrication, during production of components, or in use. This presents a major challenge for the predictive modeling and control of composite interfaces and the mechanical properties of the resulting material. In current NIST work on composites, properties of both fiber-matrix and particulate-matrix composites are being investigated. In addition, tests are being made of theories of alloy coarsening and its influence on dispersion-strengthened alloys. In all of these projects, the objective is to determine the effects that reactions in these multi-phase materials have on their microstructures and, hence, on material properties.

Thermodynamics and Kinetics of Reactions at Composite Interfaces - Because of the practical consequences of interface reactions in composites, principles governing these processes need to be understood and their importance in controlling composite properties evaluated. Three general topics are being examined in the current work, both theoretically and experimentally: (1) stress effects on the stability of interfaces during interdiffusion and phase transformations; (2) the application of bulk and interface thermodynamics to the design of interpenetrating phase composites; and (3) principles governing reactions in the Al-SiC system, a model system for commercial metal-matrix composites.

Stresses created by diffusion can perturb originally planar solid-liquid interfaces when solute diffusion leads to stresses in the diffusion zone. A range of microstructures, from gross interface roughening with a sinusoidal interface to grain size refinement to new grains formed by recrystallization, can be created depending on the magnitudes of the stresses, the diffusion conditions in the liquid, and the temperature. The role of these stresses in causing microstructural instabilities was established by measuring the wavelength of the instability in the Mo-Ni-Fe system. The wavelength was found to decrease as the stress decreased, with the wavelength going to infinity as the stress approached zero.

The critical self-stress conditions for the transition from the interface roughening instability to recrystallization were determined using model experiments in the Mo-Ni-Fe system. The transition between these two interface instabilities was characterized in terms of the magnitude of the self-stress caused by diffusion. These processes of interface breakdown by interface roughening or recrystallization have the potential for creating materials with fine particle sizes unattainable by more conventional techniques and for producing surface alloyed layers, as well as the potential for causing disintegration of reinforcing phases in composite systems.

Use of composites in high performance applications requires a composite with good strength, damage tolerance, and creep resistance. In a study of Al_2O_3-Cr_2O_3-Cr metal composites, a fiber-reinforced composite is being designed with controlled interface facetting and distribution of phases to enhance toughness by control of the fiber-matrix properties. The design approach is based on three main ideas: (a) well-dispersed ductile particles can increase the toughness of the matrix; (b) ductile particles at the fiber-matrix interface can change the fiber debonding conditions, particularly at high temperatures; and (c) the grain size within the fiber should be large for good creep resistance, yet the fiber-matrix interface should remain smooth so that sliding of the fiber can occur during crack propagation.

Composites have been fabricated of Al_2O_3 fibers surrounded by a matrix of an Al_2O_3-Cr_2O_3 solid solution phase and a Cr-Al solid solution phase. The Cr-Al solid solution phase, which can be ductile at high temperatures, forms by a decomposition reaction both as particles dispersed in the matrix and as polycrystalline coatings on the fibers by annealing Al_2O_3-Cr_2O_3 solid solutions at low oxygen partial pressures. As the oxygen partial pressure is reduced, an Al_2O_3-Cr_2O_3 solid solution decomposes into an Al_2O_3-Cr_2O_3 solid solution with a lower Cr concentration and a Cr-Al metal solid solution, where the compositions and the volume fractions of the equilibrium solid solutions depend on the original composition of the mixed oxide, the oxygen partial pressure and the reduction kinetics. Exploiting these features of the Al-Cr-O phase diagram allows the fiber/matrix interfacial debonding to be tailored within large limits (30-750 MPa).

Transmission electron microscopy (TEM) measurements were made of reactions at Al-SiC interfaces to evaluate interface roughness and facetting generated by diffusion, phase connectivity, and orientation relationships between nucleating phases and the SiC substrate. The crystalline phase and the grain morphology of the reinforcing SiC were found to affect strongly the interface facetting and connectivity of phases in the Al-SiC system. In particular, isolated Al_4C_3 precipitates formed on (0001) SiC planes of 4H-SiC while Al_4C_3 completely covered SiC planes of other orientations. These results suggest that composites containing a reaction product susceptible to corrosion can be made more corrosion-resistant if the reaction product forms as isolated precipitates rather than completely coating the reinforcing phase.

Composites Produced by Liquid Phase Sintering - A new liquid phase reaction sintering plus HIPing process has been perfected for the production of metal matrix/ceramic particulate composites starting with ceramic powders and elemental metal powders. This process has been applied with success to produce Ti_3Al strengthened with 50 μm diameter ZrO_2 spheres. Additions of 10 and 20 volume percent ZrO_2 nearly doubles the flow resistance. Heat treatment and high resolution TEM studies are being conducted on this new material with the aim of optimizing the strength/toughness trade-off. Application of this new process to other metal/ceramic systems is being pursued.

Coarsening of Solid-Liquid Alloy Mixtures - The measured coarsening rates of particles in solid-liquid mixtures is much larger than predicted by theory. This discrepancy is often attributed to the presence of convection in the liquid, which accelerates mass transport, or to errors in the thermophysical property values used in the theory. However, equilibrium measurements are possible only in the high volume fraction solid regime where the development of a skeletal structure stabilizes the particles against sedimentation driven by the density difference between the solid and liquid phases, and theory is only approximate at these values of volume fraction solid. Thus, an alternative explanation is that the difference between the measured and predicted coarsening rates may simply be due to the inadequacy of theory in the high volume fraction solid regime.

To investigate the volume fraction and convection effects on discrepancies between theory and experiment, a sample rotation technique has been used to study the coarsening of Sn-Pb solid-liquid mixtures over the entire range of volume fraction solid. In this technique, disc-shaped samples are rotated in the vertical plane so that the particles describe circular orbits in low volume fraction solid samples. At higher volume fraction solid, collisions occur which interfere with the free motion of particles. However, the separation of the sample into a particle-free liquid layer and a high volume fraction solid layer is prevented and an approximately uniform distribution of particles is obtained. Measurements of the coarsening process in these rotation experiments showed the coarsening temporal exponent is 3, the same value as in the stationary experiments except for very low volume fraction solid samples. At low and intermediate values of volume fraction solid, the measured rate constants are higher than the values extrapolated from the static experiments. However, at high values of volume fraction solid, the measured rate constants for stationary and rotating samples are the same. This suggests that the skeletal structures are very stable and the coarsening rates of the constituent particles are not perturbed by convection.

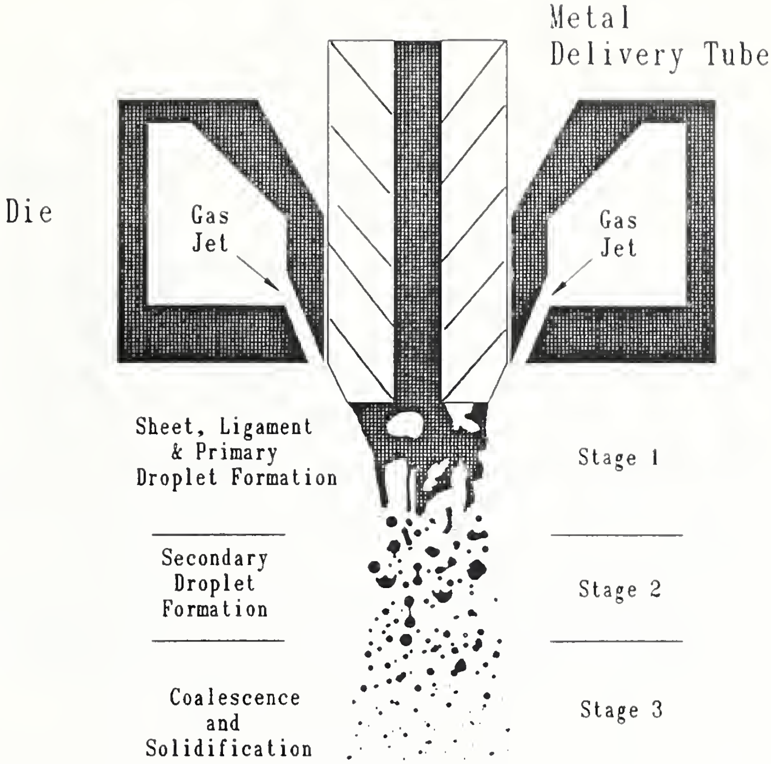


Figure 1. Diagram of high speed movie frame showing break-up of liquid metal stream in supersonic inert gas metal atomizer. The liquid metal, shown as dark material in this diagram, exits from nozzle at bottom of metal delivery tube, shown as hatched area, in middle of diagram. The metal instead of falling directly downward flows to the edge of the nozzle where it again starts downward, but now as a hollow cylindrical sheet or shell. High pressure gas exiting from the atomization die disrupts this sheet to form ligaments and then very fine droplets from the liquid.

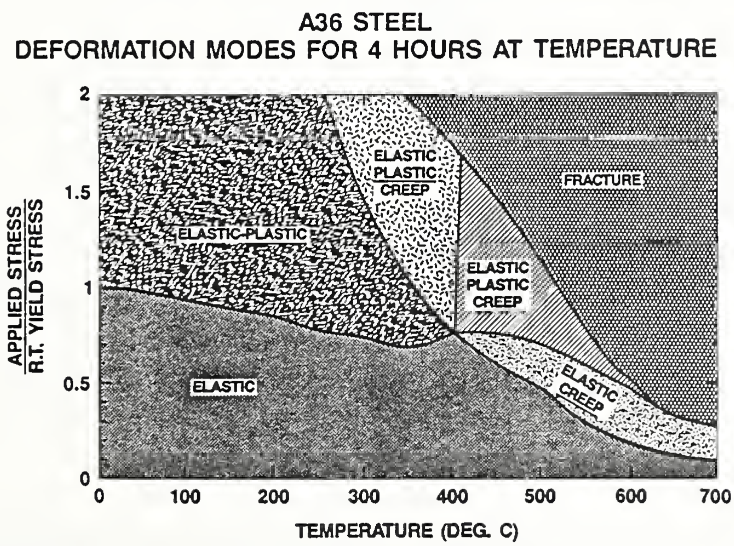


Figure 2. A transient deformation mechanism map for structural steel (A36) showing the quantity and type of strain occurring in four hours for a wide range of temperatures and stresses.

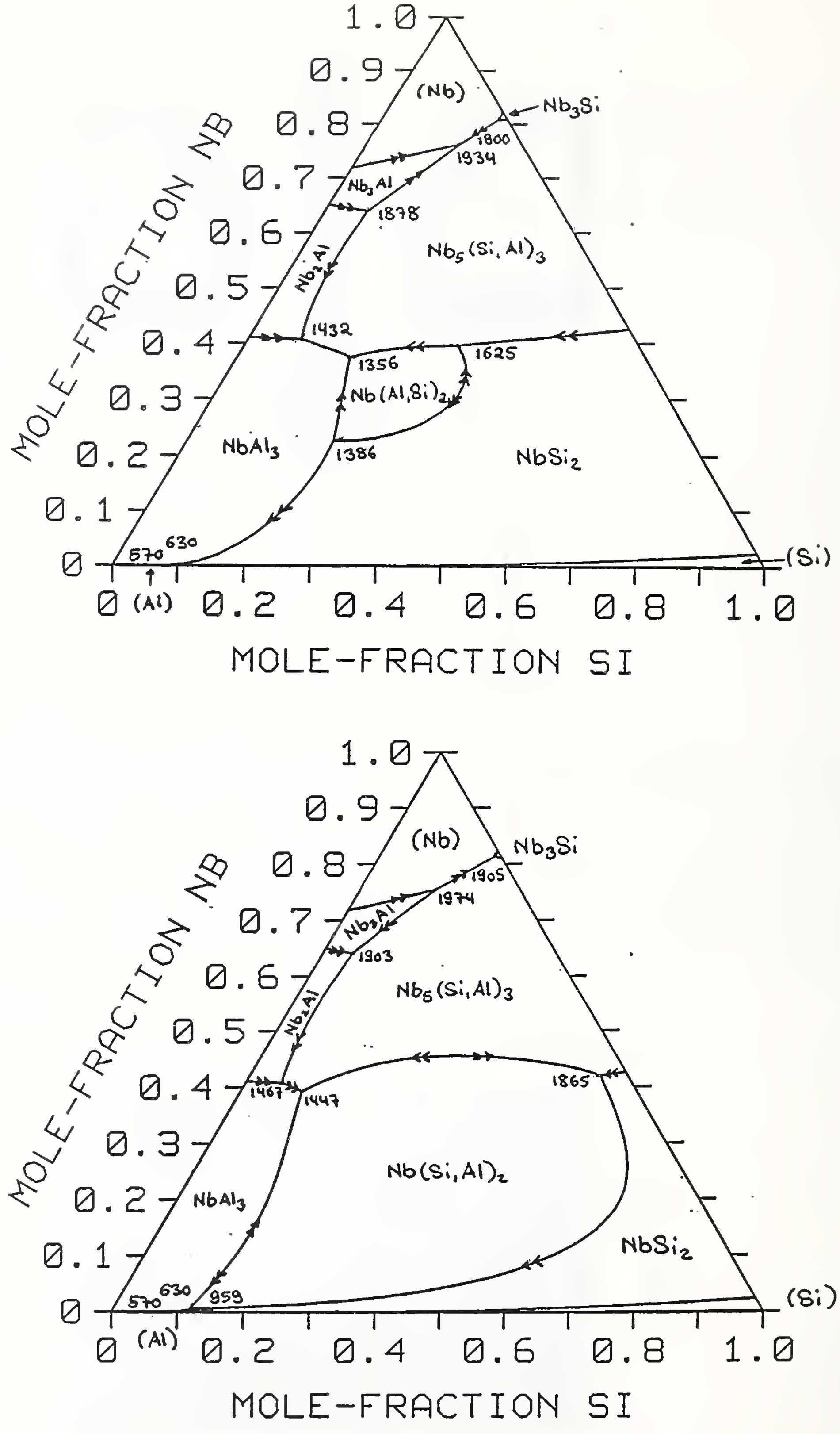


Figure 3. Calculated liquidus surfaces for the Nb-Al-Si ternary system. Both are consistent with measured binaries but differ in the thermodyamic assumptions made about the ternary phase $Nb(Al,Si)_2$ and the $Nb_5(Si-Al)$ phase. In composition ranges where the diagrams differ, experimental investigation can be efficiently planned to determine the correct diagram.

ADVANCED SENSING

H. Thomas Yolken

The mission of the Advanced Sensing Group is to enhance the measurement science of nondestructive evaluation sensing methods and to apply them to the needs of U. S. industry and the U. S. materials science and engineering community. These needs fall usually into two classes: noninvasive techniques of sensing for process control, and methods for in situ observation of materials phenomena for process modeling.

A major new thrust of the Group is in support of the Institute for Materials Science & Engineering/Office of Nondestructive Evaluation concept of intelligent processing of materials. This concept couples on-line NDE sensors for monitoring of microstructure or properties of the materials with process models and utilizes computer integration with expert systems and artificial intelligence. To develop this concept, a systems approach is required with an interdisciplinary team of researchers.

In cooperation with the Ceramics Division, the Group is developing sensors and data for high Tc superconductors for both process modeling and process control. To apply intelligent processing techniques to the hot isostatic pressing of intermetallic alloys. A project jointly funded by DARPA and NIST is underway. This project couples researchers in the Metallurgy Division's Advanced Sensing and Processing Groups with BDM Corporation and the University of Cambridge (England) to develop process sensing methods, validated process models, and new control concepts incorporating artificial intelligence methods. In another project funded jointly by DARPA and NIST, NIST researchers in materials science are working with research mathematicians at Georgia Institute of Technology and the University of California at Los Angeles to model microstructure evolution using nonlinear methods emerging from the mathematics community.

In a collaborative project with General Electric (Aircraft Engines Division) under DARPA/GE support the Advanced Sensing Group is exploring microstructure sensing during Rapid Solidification Plasma Deposition (RSPD). The ongoing collaborative programs with the American Iron and Steel Institute (AISI) and Aluminum Association have continued. The AISI/NIST program on internal temperature sensing is now evaluating the limitations of imaging or locating a liquid-solid interface in alloys that form a mushy zone. The ultrasonic technique used is an extension of the earlier developed method for measuring the internal temperature field in solid bodies. In related research, a study was carried out in cooperation with GE's Aircraft Engines Business Group to determine the feasibility of ultrasonic sensing of shell thickness during skull melting of nickel-based super alloys.

The collaborative program with the Aluminum Association is exploring the use of electromagnetic (eddy current) methods to determine the temperature of aluminum during extrusion processing. A technique utilizing two flat coils has been successfully developed and tested in an extrusion mill on such shapes as channels and I beams. This effort will be extended next year to include efforts on strip and plate.

Cooperative programs are underway with General Electric, BDM, Cambridge University (England), and Georgia Institute of Technology and the Johns Hopkins University. An industry/academia consortium of researchers interested in hot isostatic pressing continues, and negotiations are currently underway to initiate new cooperative efforts with the University of Virginia, General Dynamics, the Army's Watervliet Arsenal, and the NIST Fracture and Deformation Division.

FY 89 Significant Accomplishments

- The feasibility of using an ultrasonic technique and an eddy current technique to monitor, in-situ, the processing of high T_c ceramic superconductors at elevated temperature was demonstrated.

- An eddy current method for sensing metal temperature during extrusion has been extended to measure complex shapes such as I-beams and channels. The method, which utilizes a pair of coils configured for through transmission, has been successfully tested in plant trials.

- Modeling of the mechanical properties of Ti-Al during HIPing has been successfully carried out and applied to an improved model of the HIP process.

- A new analytical technique has been developed that allows electrical conductivity profiling based on eddy current measurements. This technique will be useful in locating the solid-liquid interface in partially solidified conducting materials

- The initial feasibility of utilizing ultrasonic and eddy current sensors to monitor the processing of Ti-Al intermetallic compounds has been demonstrated.

Modeling of Microstructure Evolution in Intermetallic Alloys
J. W. Cahn, John A. Simmons, Shui-Nee Chow*, J. K. Hale*, Ryoichi Kikuchi**, S. Osher**

* Guest Scientist - Georgia Institute of Technology
** Guest Scientist - UCLA

The ability to predict metastable phases and the kinetics of microstructure morphology occurring during solid-solid transformations is imperfectly understood for multicomponent alloy systems. This NIST/DARPA sponsored project addresses the application of emerging methods from the mathematical theory of dynamical systems to predict the evolution of microstructure in advanced intermetallic alloys with both partial ordering and segregation. This project will develop new techniques permitting the rigorous solution in symbolic form of the cluster variation formulation for partially ordered multicomponent alloys to obtain metastable state phase diagrams and extend these techniques to the path probability method for developing a partial differential equation system to serve as a state-of-the-art basis for modeling microstructure evolution in intermetallic alloys. New mathematical algorithms from the dynamical theory of nonlinear partial differential equations and computational algebraic topology will be applied to both the qualitative and quantitative solution of equations modeling alloy microstructure evolution. Global stress and local fluctuation effects will be incorporated into the modeling equations. Nonlinear image analysis techniques will be developed to characterize microstructure evolution. Experiments will be designed to critically evaluate theoretical models and their predictions.

A one dimensional difference-differential equation system modeling ordering on planes in a binary alloy system is being analyzed by current dynamical system methods. Using the Bragg-Williams approximation, where the ordering is expressed in terms of the concentration of each of a number of constituents on a set of sublattices, each of which is the translation by the superlattice of one of the elements in a unit cell, the generalization of the Cahn-Hilliard equation describing segregation to include ordering of multiple constituents takes the form:

$$\frac{\partial c_{i\alpha}}{\partial t} = D_{i\alpha j\beta}\nabla^2 c_{j\beta} - K_{i\alpha j\beta}\nabla^4 c_{j\beta} + f_{i\alpha}(c) \quad \begin{cases} i,j = 1,\ldots,\#\text{constituents} \\ \alpha,\beta = 1,\ldots,\#\text{sublattices} \end{cases}$$

where

$$\sum_{\alpha} f_{i\alpha}(c) = 0.$$

This equation has been provided to the mathematical community and interactions are being established with the University of Utah, the University of Minnesota as well as Hiroshima University in Japan where extensive modeling of reaction-diffusion type equations is taking place.

The extension of the Bragg-Williams approximation to include more precise ordering parameters and the derivation of the parameters $D_{i\alpha j\beta}$ and $K_{i\alpha j\beta}$ are being undertaken in collaboration with Professor Kikuchi who has published a paper describing the classical cluster variation approach to ternary systems as well as the path probability method using only nearest neighbor pair interactions.

A new method for doing the cluster variation expansion to obtain the parameters $f_{i\alpha}$ and their extensions to higher ordering parameters is being investigated. For this purpose a collaborative program is being instituted with the Energy Mineral and Research Center at the University of North Dakota where the calculation of metastable phase transitions as well as the related evolution equation parameters is being investigated.

Analysis of microstructures obtained will be carried out in collaboration with Professor Osher, whose specialty in analyzing discontinuities in nonlinear partial differential equations is also of importance in an associated DARPA/NIST program which addresses mathematical problems associated with the free boundary problem in solidification.

In Situ Fragmentation of Weibull Fibers in a Metal Matrix Composite
R. B. Clough and A. Manor*

* Guest Scientist - RAFAEL, Haifa, Israel

One of the barriers to developing improved statistical theories of fiber fragmentation during elongation of a fibrous composite has been a lack of accurate real-time data on the strengths and lengths of the individual segments. A load-drop test method was developed which can provide the strengths in situ, during elongation. This is the only method available at the present time for such a measurement. The segment lengths during the test were determined, to a high degree of accuracy ($\pm$ 150 μm), by using acoustic emission for location of the fracture sites. These measurements were combined and strength versus length curves have been determined for two processing conditions. These results show (as do individual fibers not incorporated in a composite) a Weibull distribution, strength decreases with increasing length. This is observed in nearly all materials, and it suggests that the shear lag theory, which was not formulated for Weibull fibers, and predicts precisely the opposite effect, needs to be reformulated for Weibull materials. This problem is being investigated in collaboration with E.Fuller of the Ceramics Division.

Sensing and Process Modeling of Hot Isostatic Pressing of TiAl
A. H. Kahn, R. B. Clough, and M. Mester*

* Research Associate - The Aluminum Association

Traditional control concepts for hot isostatic pressing are based upon sensing and maintaining temperature and pressure on a predetermined path. Trial and error is used to identify the "best" path. Recent advances in modeling the HIPing process at the University of Cambridge are being coupled with research on novel sensors for measuring density, temperature gradients, microstructural coarsening and microcracking to explore the feasibility of implementing an Intelligent Processing of Materials control concept. The program jointly funded by DARPA and NIST teams NIST researchers with the BDM Corporation, and the University of Cambridge (England) to test the IPM concept on the consolidation of intermetallic composites.

The eddy current sensor system devised for measuring sample cross-section area during hot isostatic pressing has been modified to allow observation of titanium aluminide powders sealed in titanium canisters. The high resistivity of titanium and the higher temperatures than used in previous work on copper samples required extending the operating frequencies to over 1 MHz.

In order to interpret the sensing results to obtain density measurements during the processing, it has been necessary to make assumptions about the length change of the HIP sample, usually that the relative change of length is proportional to the relative change of diameter. A direct in situ measurement of length would be preferable. The design of an electromagnetic sensor to detect changes in length during processing is now being carried out.

It has been observed in HIPing copper in cylindrical canisters that there is significantly greater shrinkage in the radial direction than in the longitudinal. This problem of anisotropic densification has been solved using a new triaxial micromechanical theory of plastic flow/densification of porous materials. The Ashby one-dimensional model was used to establish the dilatational energy in the theory. The theory was applied to plastic flow in a solid canister, with which the compact must maintain strain compatibility. In certain situations, such as when the powder is soft relative to the can material, or if the compact density is low, the mechanical properties of the can predominate, so that there is almost no axial shrinkage of the can (see Fig. 1). The theory gives quite good agreement with experimental results on copper and titanium aluminide.

Materials Characterization During Inductively Coupled Plasma Deposition of Titanium Aluminides
W. Johnson and A. H. Kahn

This DARPA/NRL funded project teams NIST with General Electric, Drexel University, and the University of Virginia to explore process control concepts for Inductively Coupled Plasma Deposition (ICPD) of intermetallic compounds. Work at NIST is directed towards exploring ultrasonic and eddy current techniques for determining the crystalline phases present in the deposited material. Ultrasonic longitudinal velocity measurements have been performed at room temperature on Ti-Al samples with aluminum content ranging from 0 at% to 50 at% (spanning the α, $\alpha+\alpha_2$, α_2, and $\alpha_2+\partial$ phase regions). The velocity shows a large (21%) monotonic increase as the aluminum content is increased over this range. Eddy current measurements indicate that the resistivity increases by a factor of ~4 over the same composition range. This work will extend these measurements to elevated temperatures, using a newly constructed vacuum furnace.

Sensors and HIP Processing of High-T_c Superconductors
E. Drescher-Krasicka, W. Johnson, R. J. Schaefer, F. Biancaniello, and A. H. Kahn

This project, has focused on both materials characterization and processing of high-T_c superconductors. It has explored the feasibility of implementing sensors based on ultrasonic and eddy current techniques for monitoring the evolution of material parameters during high-temperature processing, with $Ba_2YCu_3O_{7-x}$ serving as a model system. It has, also, sought to determine the prospects for producing fully dense metal-encased Bi-Sr-Ca-Cu-O superconductors through hot isostatic pressing (HIPing).

The physical basis for the ultrasonic, on-line high temperature sensor rests on a large measured change in velocity, corresponding to the structural transition of $Ba_2YCu_3O_{7-x}$ from tetragonal to orthorhombic symmetry (and "vice versa"). The feasibility of monitoring in situ the effects of the processing time and temperature on mechanical and structural properties of high temperature $Ba_2YCu_3O_{7-x}$ ceramic superconductors was demonstrated. A lithium niobate ultrasonic transducer was utilized for the ultrasonic velocity measurements at elevated temperature. This material has a Curie temperature near 1200°C. However, when in a reducing atmosphere, the transducer resistivity decreases rapidly above 400°C. This, and the bond technique limitations were overcome in the subsequent high temperature experiments, and the orthorhombic/tetragonal and tetragonal/orthorhombic phase transitions in Ba-Y-Cu-O samples were monitored during high temperature processing. Simultaneous attenuation and velocity measurements as a function of the time of processing and the temperature were monitored by the ultrasonic sensor placed at the surface of the ceramic samples, demonstrating the potential for in-process materials characterization using ultrasonics. The ordered orthorhombic phase was found to have a higher velocity than the disordered tetragonal phase of the processed sample.

The eddy current technique has been deveeloped for monitoring oxygen content during processing of $Ba_2YCu_3O_{7-x}$. The superconducting properties of $Ba_2YCu_3O_{7-x}$ depend critically on the value of x, and this is, in turn, determined by the details of powder processing and sintering. The physical basis for an eddy current sensor is the dependence of the normal state resistivity on carriers which are contributed by oxygen. A sensor has been designed which consists of a resonant circuit, with a coil surrounding the sample and a capacitor connected in parallel, having a resonant frequency near one megahertz. This configuration is suitable for studying small pill-shaped samples which are now commonly used in research on high-T_c ceramics. Figure 2 shows room temperature measurements of the sensor impedance as a function of frequency with the coil empty and with each of two model silicon samples inserted. The resistivities calculated from the values of the impedance at resonance agree within 5% with four-point measurements. At elevated temperatures, the impedance with a $Ba_2YCu_3O_{7-x}$ sample inserted have been monitored as the atmosphere around the sample is varied (Figure 3). The expected changes in impedance associated with absorption and loss of oxygen in the sample are easily detected. The simplicity of the device would allow it to be incorporated into most existing furnaces used for processing high-T_c ceramics.

Research on the HIPing of Bi-Sr-Ca-Cu-O has succeeded in producing fully dense superconducting material using powder with a T_c near 80K. Experiments with several HIP can materials have led to the conclusion that loss of oxygen is the primary concern at HIPing temperatures near 500°C. While gold and stainless steel cans allow the ceramic to remain superconducting, silver is inappropriate, due to the high diffusivity of oxygen in this metal.

Steel Sensors

F. A. Mauer, D. Pitchure, S. J. Norton, W. L. Johnson, and V. Grinberg*

* Guest Scientist - Atomic Energy Commission, Israel

This ongoing program is directed at satisfying steel industry needs for sensing internal temperature and measuring shell thickness during continuous casting and subsequent thermo-mechanical processing of steel. Previous work at NIST has demonstrated the feasibility of an ultrasonic approach for reconstructing temperature profiles in a 6 x 6 inch section of stainless steel. An ultrasonic method has also been demonstrated for locating the solid/liquid interface in a 6 x 6 inch strand with a liquid core using aluminum as an analog material in order to avoid the high temperatures required to simulate a steel strand.

The original experiments for reconstructing the solid/liquid boundary were carried out using 1100 aluminum, which is the commercially pure grade. Additional complications arise when the method is applied to alloys of steel or aluminum because of the formation of a so called mushy zone and the changes in composition of the solid and the melt that occur during solidification. The mushy zone which forms as a molten alloy cools through a two phase region in the phase diagram is characterized by dendrites growing normal to the solid/liquid interface. These lead to severe scattering of the ultrasonic wave. The additional attenuation that results may prevent the observation of the ultrasonic time of flight on which this method for locating the solid/liquid interface depends. Even if the time of flight can be measured, it may be difficult to interpret the results because of the effect of compositional changes on the velocity.

To evaluate the limitations imposed on the ultrasonic method for reconstructing the boundary of the liquid core in aluminum alloys, reference data for longitudinal velocity versus temperature are now being obtained on a series of binary alloys in the system Al-Si at temperatures below the liquidus. Results recently obtained on aluminum containing 2 at % Si are shown in Fig. 4. In making these measurements it was observed that the amplitude of the longitudinal wave was reduced to approximately 19% of its original value above the solidus temperature (577°C). With signal averaging, the wave arrival can probably still be observed, but it remains to be seen whether the compositional changes that occur as solidification progresses will introduce sufficient uncertainty in the reference data to prevent us from obtaining a quantitative estimate of the interface location. A final judgement cannot be made until measurements have also been completed above the liquidus temperature. Preparations are under way for measurements of the longitudinal velocity on molten aluminum alloys.

An exploratory study has been carried out for the General Electric Company Aircraft Engines Business Group to determine the feasibility of ultrasonic sensing of shell thickness during skull melting of the nickel-based superalloy Rene-95. The ultrasonic properties of two samples were studied. One was a cast ingot characterized by coarse grain structure, texture, and compositional inhomogeneity. The other, which was prepared by powder metallurgy, was fine-grained, homogeneous, and free from texture. The attenuation in each of these materials was evaluated and used to construct a diagram indicating the maximum distance over which signals should be observable by the pulse-echo and the through-transmission method. Reference data for longitudinal velocity versus temperature were obtained for each material at temperatures up to 1200°C. Based on the experimental results, recommendations were made for additional work coordinated with General Electric in order to provide insight into engineering problems connected with developing a practical sensor for plant use.

<u>Eddy Current Sensing - Temperature Measurement in Aluminum Extrusion Processing</u>
A. H. Kahn and M. L. Mester*

* Research Associate - The Aluminum Association

Two plant tests were performed using the sensor design completed in the previously. This design consists of a driven coil and a receiver coil configured for through-transmission observation of temperature in flat sections of aluminum products, in this case the web of an extruded I-beam. The tests were conducted at the R. D. Werner Co. plant in Greenville, PA. In addition to the eddy current test, temperature data were collected from two optical pyrometer systems and from a hand-held thermocouple; all methods collected data concurrently. A typical temperature-time plot showing the complete passage of three extruded billets is shown in Fig 5. The eddy current readings typically fell between the two pyrometer readings. In the transition between billets, the eddy current readings underwent a sharp drop associated with the change of velocity on interruption of the process. This velocity effect was then studied theoretically and tested in the laboratory up to 8000 ft/min.

Currently the research effort is being directed toward temperature profiling in billets and effects on temperature measurement of variation of alloy constituents within a given commercial alloy designation.

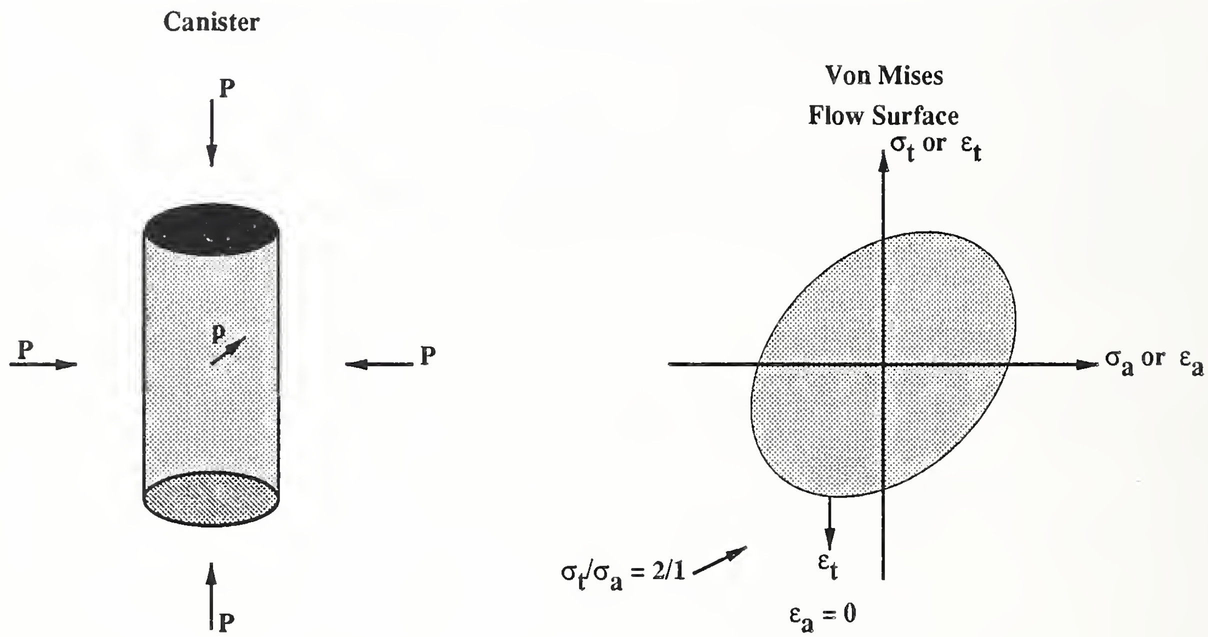
<u>Electrical Conductivity Profiling Based on Eddy Current Measurements</u>
S. J. Norton, A. H. Kahn, and M. L. Mester*

* Reseach Associate - Aluminum Association Research Associate

Conventional eddy current techniques generally assume uniform electrical conductivity in the sample under examination. However, multi-frequency eddy current measurements are sensitive to spatial variations in conductivity and, thus, can be used as the basis of a new approach to conductivity profiling. This spatial sensitivity is a consequence of the frequency-dependence of the AC skin depth of the induced eddy currents.

Based on this principle, a general profiling methodology was developed for reconstructing conductivity profiles in both cylindrical and planar (plate) geometries. A computer algorithm was created which reconstructs radially-varying conductivity profiles in cylindrical conductors. The heart of the algorithm is an iterative nonlinear least squares routine designed to determine the particular profile (suitably parameterized) that most closely reproduced the eddy-current data. The algorithm was successfully tested both on simulated impedance measurements as well as on actual measurements recorded using a dual coil system. A future task is to extend the profiling algorithm developed for cylinders to the problem of depth profiling in plates. It is expected that this technique will find applications in profiling layered structures (e.g., coated or plated products) of both flat and cylindrical geometry. Moreover, because of the significant difference in conductivity between the solid and molten states in many metals, conductivity profiling may provide a means for locating the solid-liquid interface in partially solidified metallic samples.

INFLUENCE OF CANISTER ON HIPING



WALL STRESSES:

σ_t(hoop stress) = (P-p)r/t

σ_a(axial stress) = (P-p)r/2t

$\sigma_t/\sigma_a = 2/1$

where:

r = radius of can

t = wall thickness

P = applied pressure

p = internal pressure

ASSOCIATED FLOW RULE: The plastic strain vector is normal to the flow surface.

RESULT: The plastic strain in the axial direction is ZERO.

Fig. 1 Influence of canister on HIPing

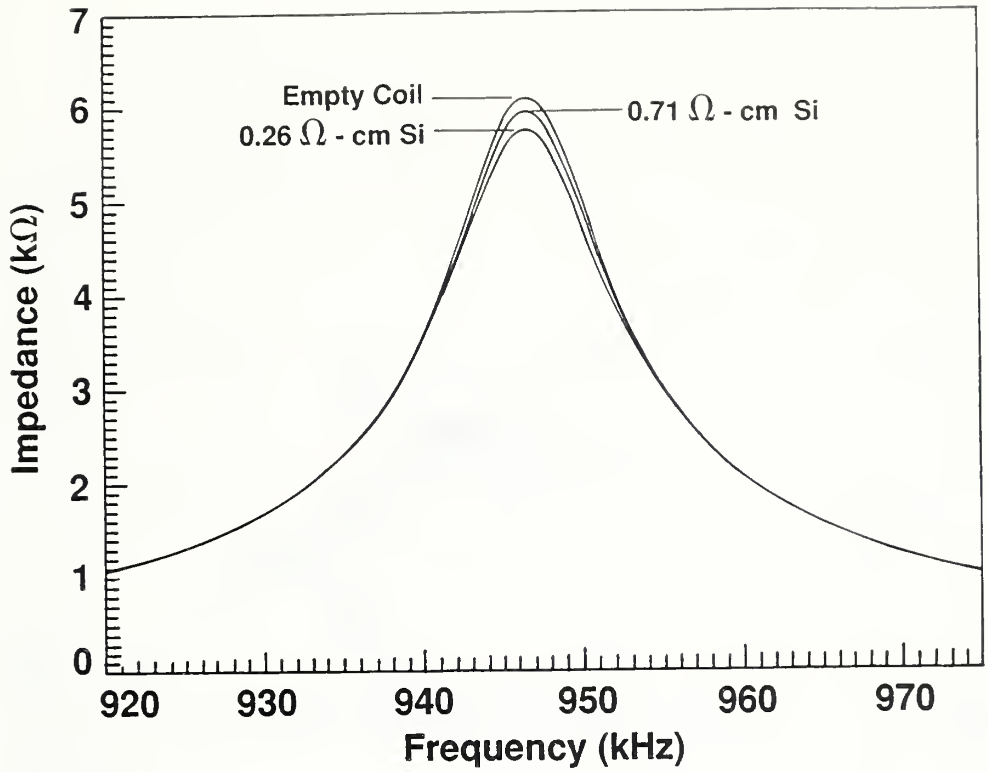


Fig. 2: Eddy current sensor impedance as a function of frequency near resonance with no sample inside the coil and with two silicon samples having resistivities of 0.26 Ω-cm and 0.71 Ω-cm.

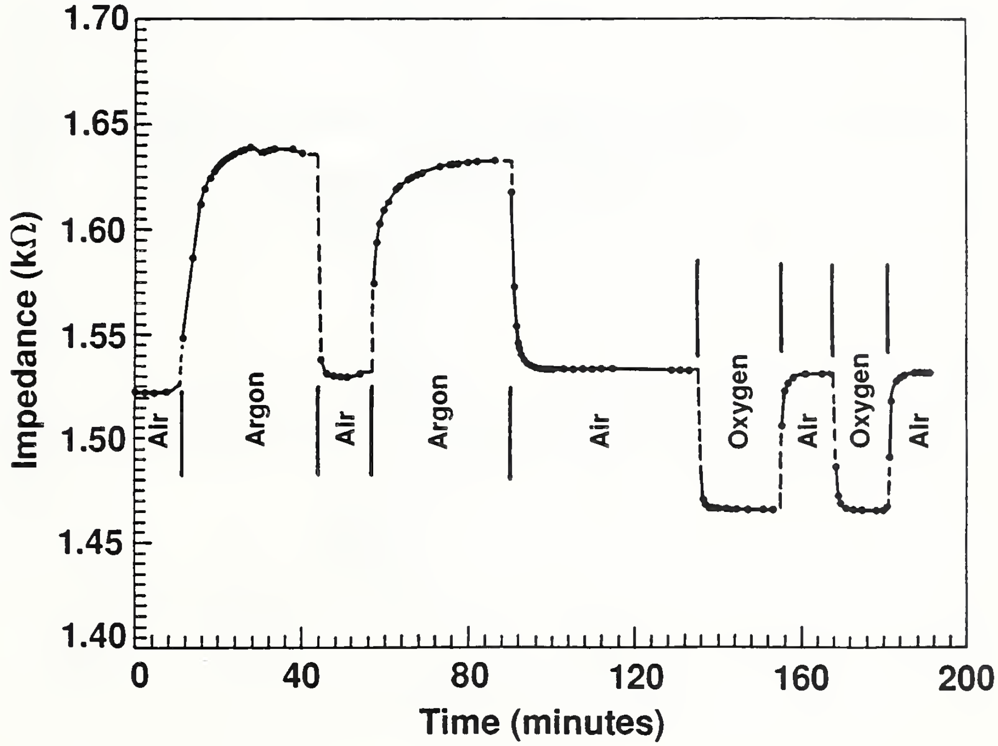


Fig. 3: Sensor impedance at 790°C with a $Ba_2YCu_3O_{7-x}$ sample, as the atmosphere is cycled between air, argon, and oxygen.

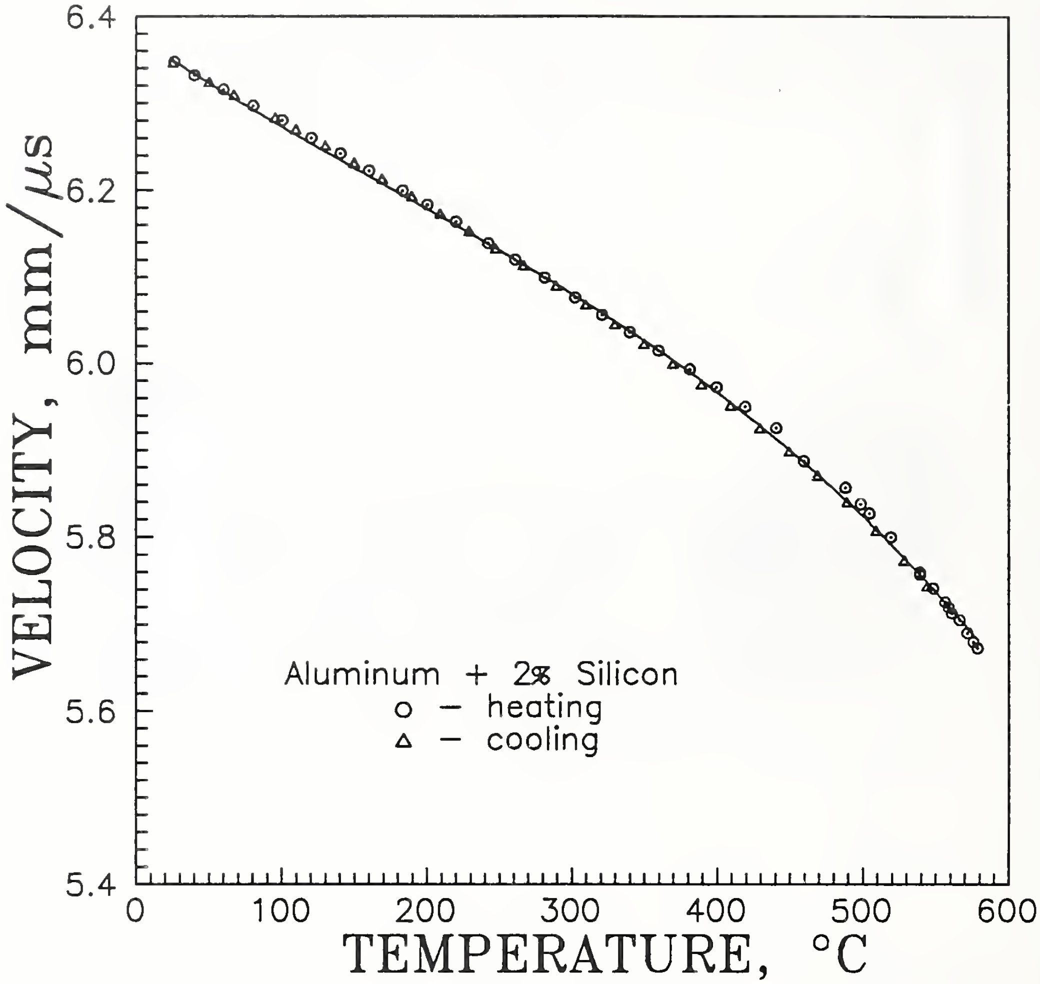


Fig. 4 Longitudinal velocity versus temperature for aluminum containing 2 at. % silicon.

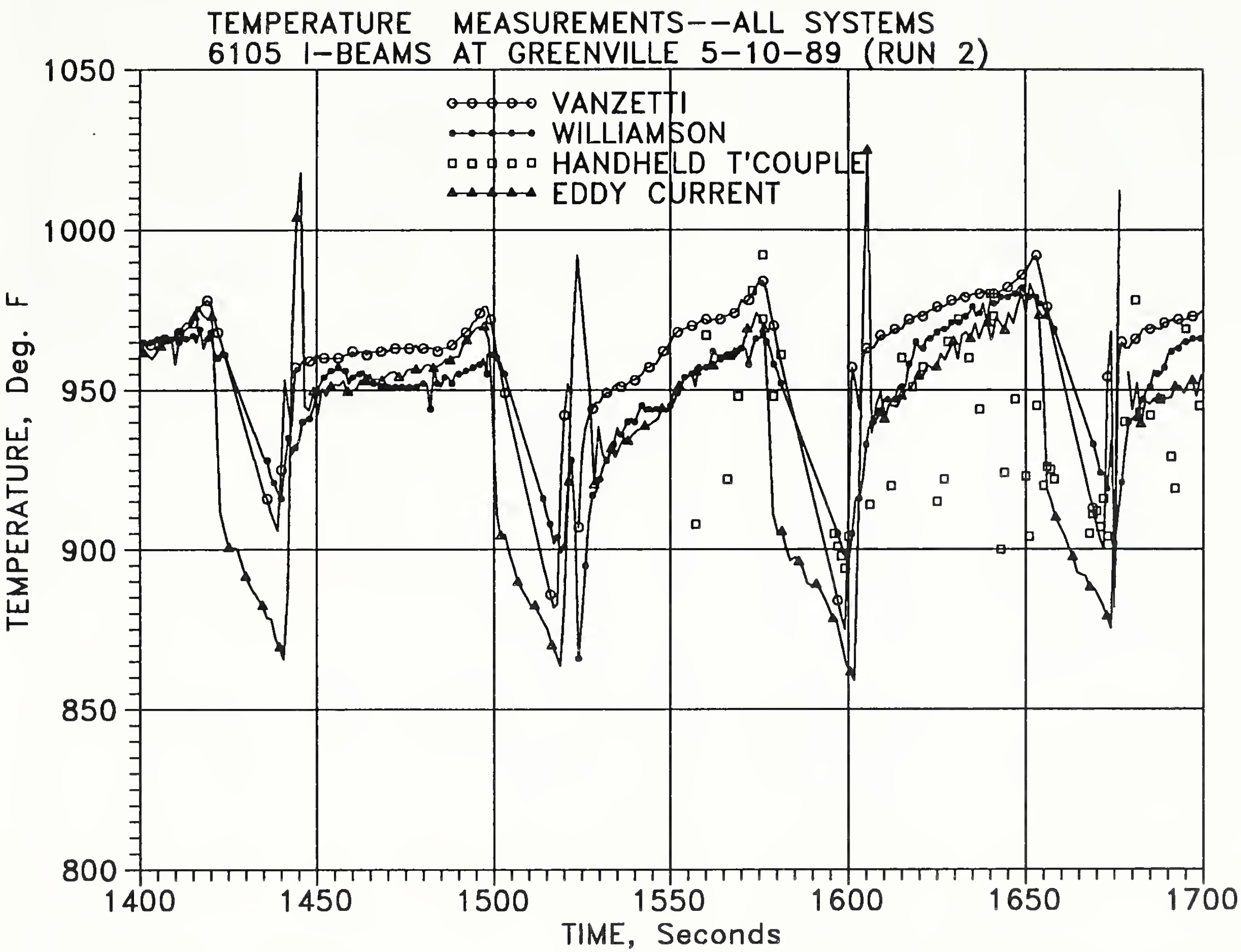


Fig. 5 Measured temperature vs. time during extrusion processing. Eddy current sensor data are indicated by triangles; Vanzetti and Williamson curves are data from commercial optical pyrometers; squares indicate data from a hand-held thermocouple.

CORROSION Richard E. Ricker

Corrosion is the primary cause of shortened service life of engineering components, and frequently causes catastrophic failures involving loss of life and property. Corrosion adversely effects industrial competitiveness by reducing plant efficiencies and by reducing the usable lifetime or quality of industrial products. The cost of corrosion to the U.S. economy is estimated to be close to $200 billion per year but this figure ignores the long term impact of corrosion on industrial competitiveness. The key factors in reducing these losses are (1) better utilization of existing knowledge, (2) better understanding of corrosion mechanisms (leading to better corrosion prevention technology), (3) the development of better corrosion measurement science (enabling more accurate estimation of usable lifetimes) and (4) the development of new advanced materials (aiding industry to develop new technologically superior products).

To promote the better utilization of existing knowledge, the corrosion group, through the Corrosion Data Program, a joint activity between the National Association of Corrosion Engineers (NACE) and NIST, serves as a central source of reliable, evaluated corrosion data. The commercially available software from the program has been enthusiastically received by users, and work is continuing on evaluated corrosion data and the development of expert systems for use in materials selection and corrosion control.

The corrosion group has several programs aimed at improving our understanding of corrosion mechanisms. During the past year, the corrosion group made progress in understanding the mechanisms of stress corrosion cracking, pitting corrosion and underground corrosion. Studies of the mechanism of transgranular stress corrosion cracking in pure copper point toward localized dissolution as the cause of cracking in this material. Our understanding of pitting corrosion was advanced through a study of the mechanism of pitting corrosion of an intermetallic compound (nickel aluminide) in a sodium chloride solution and in an environment that does not contain halide ions. The role of transport in underground corrosion was studied through the development of computer model for mass transport in a particulate medium. This model should assist the interpretation of electrochemical measurements conducted in soils as well as the prediction of the actual corrosion rates of structures undergound.

The corrosion group is studying corrosion measurement methods and the application of these to the prediction of useable lifetimes as part of its program to assist the Nuclear Regulatory Commission (NRC) in assessing the performance of different container designs for the disposal of high level nuclear waste. In addition to laboratory studies aimed at developing better methods for predicting service lifetimes, this program includes the evaluation of published information on container alloy corrosion and assessment of the experimental work performed by the Department of Energy (DOE) as part of the program to safely dispose of nuclear waste.

To assist the development of advanced materials, the corrosion group initiated new programs in FY 89 on corrosion in composites and on the resistance of high strength low alloy (HSLA) steels to cracking. In addition to these new programs, work on the corrosion of ductile nickel aluminide in cooperation with Oak Ridge National Laboratory was continued. Ductile nickel aluminide is being considered for a number of low and high temperature applications and the results of the research performed at NIST were shared with Oak Ridge and their industrial partners at a technology transfer workshop attended by potential producers, fabricators, and users of these alloys.

FY 89 Significant Accomplishments

- The NACE-NIST Corrosion Data Center expanded industry support for the development of focused programs addressing critical needs. Emphasis continued on knowledge-based expert systems using artificial intelligence concepts to aid corrosion scientists and engineers in selection of materials to assure optimum corrosion resistance. Programs are based on rules derived from the experiences of established experts and critical reviews of published data compiled in databases. Current programs focus on storage and handling of hazardous chemicals, downhole equipment in sour oil and gas production and equipment used in electric power generation.

- The pitting behavior of ductile nickel aluminide was found to be controlled by nickel in a sodium chloride solution but, pitting of nickel aluminide was found in acidic solutions where pitting of nickel is not observed. This pitting was attributed to a passive-to-active transition at occluded sites which occurs as a result of Al dissolution. Studies are underway to verify this hypothesis.

- The stress corrosion cracking behavior of ductile nickel aluminide, first reported by this group last year, was shown to be due to hydrogen absorption and embrittlement of this $L1_2$ compound.

- An experimental study of stress corrosion cracking of copper indicated that the formation of small pits in the oxide film, rather than fracture of an intact oxide film, was responsible for initiating crack propagation.

- Corrosion Group expertise in test methods for evaluating localized corrosion phenomena is being used to provide critical data for interpretation and evaluation of DOE-sponsored studies on corrosion of nuclear waste packaging materials.

Corrosion Data Center

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The Corrosion Data Center was established in 1985 as a joint program with the National Association of Corrosion Engineers (NACE). The Center serves as a focal point for the development of personal computer programs to facilitate widespread distribution of evaluated corrosion performance data for engineering materials exposed to a wide range of industrial environments.

Activities in 1989 centered on:

(1) Completion of prototype database structure designed to permit user development of compatible databases using standardized format.

(2) Database compilation from industrial data sources and completion of arrangements to access additional data focusing on exposures to hazardous chemicals.

(3) Maintaining leadership role in development of consensus standards for corrosion data formats through ASTM and NACE.

(4) Completion of initial expert system modules to guide material selection for storage and handling of hazardous chemicals (sponsored by the Materials Technology Institute of the Chemical Process Industries) and downhole equipment in sour gas and oil production environments (sponsored by the New Zealand Department of Scientific and Industrial Research and NACE).

(5) Initiation of a new multi-year program sponsored by the Electric Power Research Institute to develop computer software to guide electric utilities in such diverse areas as corrosion control in condenser systems, corrosion related fracture and intergranular corrosion in steam generators, flue gas desulfurization systems and microbiological corrosion in service water systems.

(6) Completion of a United Nations funded program to develop a corrosion database structure to support expansion of the industrial infrastructure in the Peoples Republic of China.

(7) Supporting a cooperative program to develop a personal computer program to assess corrosion thermodynamic data by computation of potential-pH stability diagrams. The program will utilize extensive NIST evaluated thermodynamic data compilations.

The Corrosion Data Center continues to evolve as a focal point for addressing the complex issues in gathering and dissemination evaluated corrosion data with industry financial support and aid in assuring consensus on the relevance and applicability of the data.

Corrosion Mechanisms and Measurement Methods

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* Guest Scientist - John Hopkins University
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Mechanisms of Stress Corrosion Cracking - The mechanism of environmentally induced transgranular cleavage-like fracture in pure copper has been the subject of a joint program with The Johns Hopkins University which concluded this year. In this study, the relationship between oxide formation and transgranular stress corrosion cracking (T-SCC) was studied. The results show that a thin oxide film must be present for cracking to occur, both in nitrite and in acetate solutions. The kinetics of the formation of the oxide film was followed ellipsometrically and it was found that the morphology of the film changes rapidly with time, and that the initial adherent film breaks down with the formation of a loose, thick tarnish layer. Although other workers found that only the initial film was present at the crack tip, this study indicated that cracking occurs at potentials and time periods that coincide with the breakdown of the initial adherent film. When this film breaks down micro-pits form which resemble the pores which form during dealloying of brass. As a result, it was concluded that these pits act to initiate fracture in the same manner as the pores that form in brass and that the observations are consistent with the film induced cleavage mechanism for T-SCC.

Diffusional transport in porous media - In a large number of cases, corrosion occurs in environments, such as soils and concrete, which are sufficiently porous so as to contain an aggressive electrolyte, but which prevent material transport by convective motion. Very often, the rate of corrosion, as well as its morphology, is dependent on the transport of oxygen through the porous medium to the metal surface. It was therefore interesting to find out methods for the measurement of the rate of oxygen transport in porous media.

The model system chosen was sand of different sizes, and the apparent diffusion coefficient was measured by applying a voltage step and measuring the ensuing current transient. It was found that the current does not decay linearly with the reciprocal of the square root of time, as it happens in aqueous solutions, but that the slope of the decay curve continually decreases.

In order to understand the experimental results, a computer model of the sand/solution/metal system was developed and the current transient simulated, computing the complicated transport of the electroactive species by the finite differences method. Although the computer model was only a simplified approximation of the real system, the calculated curves were in good agreement with the experimental results. The computer simulations also have implications in the interpretation of a. c. impedance measurements in porous media.

Effect of varying resistivity and diffusivity of the environment on the localization of the corrosion process - One of the parameters which are important in determining the severity of the damage caused by corrosion, is the degree of localization of the attack. Numerous studies all over the world have been carried out to determine the conditions favoring pitting of corrosion-resistant materials, but little is known on which circumstances favor uniform attack and how the surface roughness varies with time. Experiments, conducted as part of the NRC program, found, that, when the corrosion rate is slowed down by increasing the hindrance to electric charge and electroactive species transport, the roughness of the corroded surface increases more rapidly than when the corrosion rate is faster. For the moment, the measurement of the surface roughness has been carried out with rather unsophisticated means and the results need extensive work before they can be considered reliable. However, if such trends are confirmed, they would be of great practical significance and further work on this topic is planned.

Advanced Materials

R. E. Ricker, U. Bertocci, E. Escalante, J. L. Fink, P. V. Madsen* and M. R. Stoudt

* Guest Scientist - Danmarks Ingenior Akademi, Denmark

Corrosion of Ductile Nickel Aluminide - Nickel aluminide (Ni_3Al) is an intermetallic compound that forms when ordering occurs in a nickel-aluminum alloy forming a $L1_2$ superlattice. Nickel aluminide can be made ductile by microalloying with boron (200 ppm) and by keeping the aluminum content slightly sub-stoichiometric. Ductile nickel aluminide (Ni_3Al+200 ppm B) has many properties that make it attractive for high and low temperature applications. However, the published information on the corrosion of Ni_3Al alloys is limited. Therefore, an investigation into the aqueous corrosion behavior of a ductile Ni_3Al alloy was undertaken.

From a fundamental point of view, a study of the corrosion behavior of a compound which is made from two metals which form passive films in different environments could contribute significantly to our understanding of passivity. The chemical potentials of the elements in this ordered alloy will be different from those of the pure elemental phases or the disordered solid solution but, for a first approximation, thermodynamic considerations predict three different regions of electrochemical behavior. In the low pH solutions, neither Ni nor Al is expected to form a protective layer, although Al should exhibit passivity in lower pH solutions than Ni. In neutral solutions, passive films composed of both Ni and Al oxides or hydroxides may form but the resistance of Ni and Al films to localized attack are very different and this was examined. At high pH, Ni is usually passive, but Al usually dissolves rapidly with the formation of the aluminate ion AlO_2^-. As a result, dealloying might be expected in this environment.

The results of the electrochemical experiments indicate that this material may be considered as a substitute for pure nickel, if other properties, such as its high strength or low density, make it attractive. The presence of aluminum does not seem to confer additional corrosion resistance where this metal should have a favorable influence. The passive film on Ni_3Al seems to be marginally less stable than that on pure nickel. Since the resistance of

Ni and Ni alloys to pitting is significantly better than that of Al and its alloys, it is noteworthy that substantial amounts of Al in the material have no influence on the pitting potential, which is hardly distinguishable from that of pure Ni.

A rather interesting and unexpected finding was the observation of pitting in a strongly acidic solution such as 0.5 M H_2SO_4. Since local acidification cannot be invoked as the cause of stable pit growth, the most reasonable explanation is that an ohmic drop inside the pit drives the potential into the active region. The voltage drop, particularly at the beginning, cannot be too large, and this agrees with the fact that the pitting range is close to the potential at which the active-to-passive transition occurs. Pitting does not occur in analogous circumstances on pure nickel. As a result, it was hypothesized that Al dissolution may cause hydrogen bubble formation, which in turn increases the voltage drop inside the pits. Experiments are planned to test this hypothesis.

<u>Stress-Corrosion Cracking of Ductile Nickel Aluminide</u> - The stress-corrosion cracking resistance of a ductile nickel aluminide alloy was evaluated by conducting slow strain rate tests in environments with different pH's. These tests were conducted under freely corroding conditions or at fixed potentials with respect to a reference electrode. The results indicated that under conditions where hydrogen absorption from the solution could occur, the fracture mode switched from ductile transgranular to brittle intergranular. Since hydrogen embrittlement was suspected, samples were pre-exposed to an environment that caused intergranular failure followed by testing in air and intergranular failure resulted. When these pre-corroded samples were were stored in a vacuum to remove any absorbed hydrogen, complete recovery of ductile behavior was observed. As a result, it was concluded that hydrogen absorption and embrittlement was responsible for the observed environmentally induced intergranular fracture. No evidence of any other environmentally induced fracture mechanism was observed but experiments are continuing.

<u>Corrosion Issues in Nuclear Waste Containment</u>
C. G. Interrante, U. Bertocci, E. Escalante, A. Fraker, S. A. Harrison, W. Liggett*, E. Plante, E. N. Pugh, R. E. Ricker and M. R. Stoudt

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This project has been ongoing since 1985, and an in depth expertise on associated materials problems and environmental effects has been developed. Corrosion issues are addressed regarding materials used in High Level Nuclear Waste Storage, and technical support is provided to the Nuclear Regulatory Commission (NRC). NRC regulations for this nuclear waste storage require that nuclear waste shall be substantially complete for a period of 300 to 1000 years and that thereafter no more than one part in 10^5 of the inventory of radionuclides present at 1000 years after closure may be released annually from the engineered barrier system of a geologic repository.

Approximately ninety percent of the waste will be spent fuel (Most UO_2 spent fuel is in Zircaloy with three percent in stainless steel cladding.), and approximately ten percent will be embedded in glass. There are potential problems and many concerns relating to properties of the glass, the UO_2 fuel, the metal canister material and metal cladding. Three of the major areas of this project include the following.

(1) Critical evaluation of scientific research data, measurement techniques and mechanisms used in modeling for the NIST/NRC data base. The data base has over 1088 entries. Some representative reviews for this past year included papers on the six materials currently being considered for the waste canister. These materials are CDA 102 oxygen-free copper, CDA 613 7% aluminum bronze, CDA 715 copper-nickel, AISI 304L and 316L stainless steels and the high-nickel austenitic alloy, 825. Phase stability, leaching of glass, stress corrosion susceptibility, corrosive effects of radiation, effects of temperature, environment, etc. have been the subjects of papers that were reviewed and critically evaluated.

(2) General technical assistance that deals with specific requests from the NRC. An example of this assistance was the review of the Site Characterization Plan (SCP) for nuclear waste storage submitted by the Department of Energy (DOE) to the NRC. The part of the plan dealing with the metal canister, glass waste and spent fuel waste was reviewed by six NIST staff members for scientific considerations relating to predicted long term durability. Sixteen comments with associated explanations and recommendations were given. Most of these issues were accepted and dealt with by the DOE.

Also, this past year, work was started for furnishing elements of proof needed for assuring that a waste disposal system involves containment that is substantially complete. This involved description of materials, tests, etc. is being designed to determine if materials, designs, modeling and predictions are adequate.

(3) Laboratory experiments are conducted on basic corrosion processes. There are three experimental programs that provide data pertinent to materials and conditions in the repository. These data can also be used for verification of existing data and for establishing mechanisms of the corrosion processes for use in long term corrosion durability predictions. The three laboratory projects were (1) effects of transport and resistivity on corrosion, (2) evaluation of methods for detection of stress corrosion crack propagation in fracture mechanics specimens and (3) corrosion behavior of Zircaloy nuclear fuel cladding. Also, some work was conducted to complete a project dealing with pitting corrosion of steel. These studies are described under separate headings in this report.

There is a need to have various scientific and technical aspects clarified relating to specific materials and problems that could occur during storage. It is necessary to be able to answer scientific questions before approving the storage of the nuclear waste as proposed by the DOE. In preparation for this, the NRC has requested that interpretative papers be provided on several subjects. Three pertinent topics that are being studied and papers written

1. Mechanisms of Localized Aqueous Corrosion of Copper and Its Alloys

2. Mechanisms of Stress Corrosion Cracking

3. Mechanisms of Internal Corrosion of Spent Fuel Rods

The conduct of this work provides an opportunity for NIST scientists to pursue the basic objectives of the corrosion effort including passivity, localized corrosion, stress corrosion cracking, measurement methods and underground corrosion. Personnel involved in this project serve as a good scientific information resource for the NRC as it makes determinations regarding procedures for safe long term storage of nuclear waste.
Effects of Transport and Resistivity on Corrosion - Studies sponsored by DOE indicate that metals considered for nuclear waste containers will corrode uniformly, leading to a container lifetime of thousands of years. If corrosion is localized, however, penetration of the canister could occur earlier than expected and before the nuclear waste has lost its nuclear toxicity. Generally, the DOE sponsored corrosion research has been conducted under environmental conditions of high oxygen transport and high conductivity.

This study is directed at investigating the rate and form of corrosion (uniform versus localized) that develop under conditions where transport of oxygen and conductivity of the environment are controlled over a wide range, including low conductivity as expected in the Yucca Mountain environment. The results indicate that the corrosion rate of steel is directly related to the rate of oxygen transport over several orders of magnitude (Fig. 1). It was also found that increasing conductivity of the environment by one order of magnitude increases corrosion rate by a factor of two or three. Of greater significance is the result that which shows that as conductivity of the environment decreases (Fig. 2), and corrosion rate decreases (Fig. 3), the degree of localized attack increases.

Corrosion Behavior of Zircaloy Nuclear Fuel Cladding - This project deals with the corrosion behavior of Zircaloy in aqueous media. Zircaloy-2 and -4 are used as nuclear fuel cladding. Both alloys are more than ninety eight percent zirconium and are hafnium free. Zircaloy is a registered trademark of the Westinghouse Electric Corp., Specialty Metals Division, Pittsburgh, PA. Approximately ninety percent of the nuclear waste for storage will be spent fuel, and most of this spent fuel will be clad with Zircaloy. Only three percent will have a stainless steel cladding. The thickness of the Zircaloy cladding is less than 1 mm. The purpose of this project is to provide data that are needed on the nature of the oxide film, general corrosion behavior and susceptibility to localized corrosion. The general corrosion behavior and susceptibility to localized corrosion have been investigated in the initial stages of this work.

Electrochemical measurements using polarization techniques have been made on these zirconium alloys in aqueous media with pH values of 8.3 and 8.5. The ionic concentration of the solution varied from 1XJ13 to 10XJ13, and tests were conducted at temperatures of 22°C and 95°C. Results showed a passive

region for Zircaloy typically ranging from -400 mV to + 800 mV where breakdown occurred. Voltages were measured versus a saturated calomel electrode (SCE). Corrosion rates under the conditions of the tests were negligible, but there were indications of crevice corrosion attack.
Evaluation of Methods for Detection of Stress Corrosion Crack Propagation in Fracture Mechanics Specimens - The purpose of this project is to detect crack extension or crack initiation activity, resulting from stress at flaws or material discontinuities, which occur after long times of up to 4.5 years. This project is directed toward early detection and toward developing procedures for accelerated testing. An acoustic emission technique is used for these measurements. The success of this technique would result in the capability to measure crack growth at a sensitivity which has not been possible previously and would improve predictions of stress corrosion cracking susceptibility and failure. Instrumentation and software were developed for these tests. Tests were conducted on 2.25 Cr 1 Mo steel in deaerated acetic acid solution saturated with hydrogen sulfide. Some aspects of the test apparatus were changed to keep the environment oxygen free. After the test method is developed, other environments and materials will be studied.

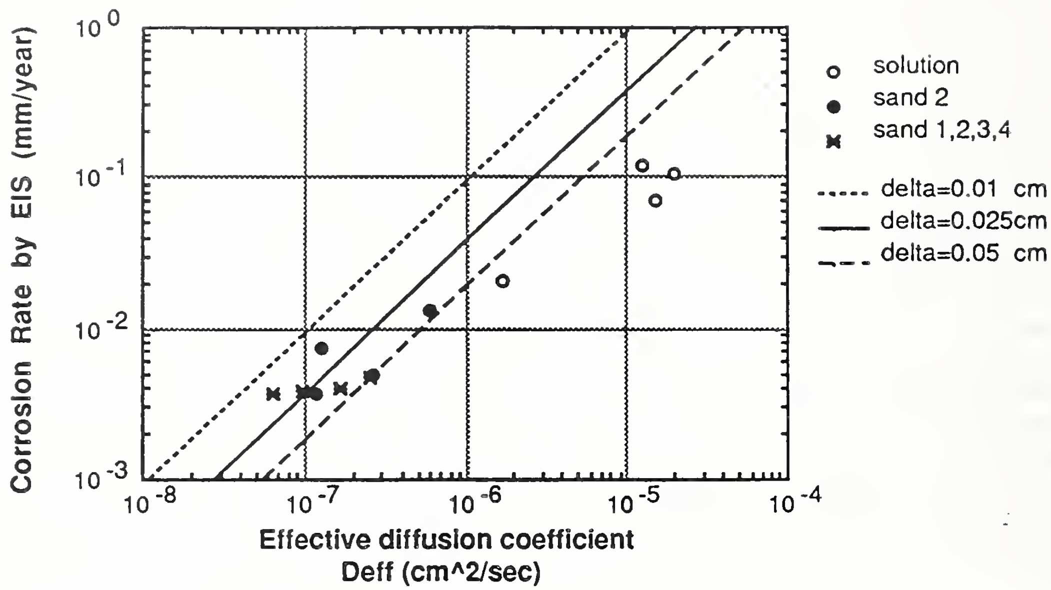


Figure 1. The corrosion rate of steel as a function of oxygen transport in different media.

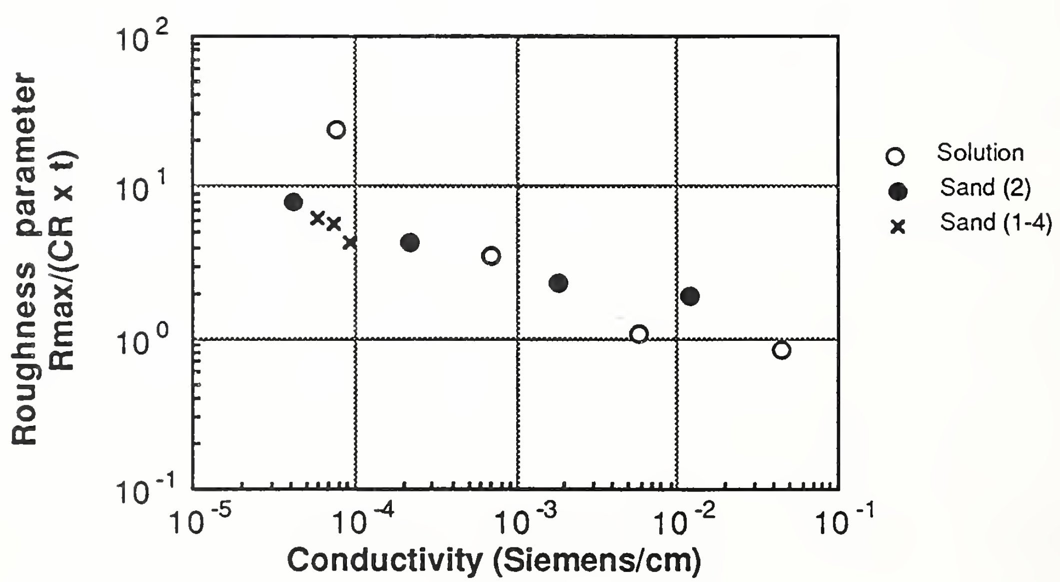


Figure 2. The surface roughness of steel as a function of conductivity in different media.

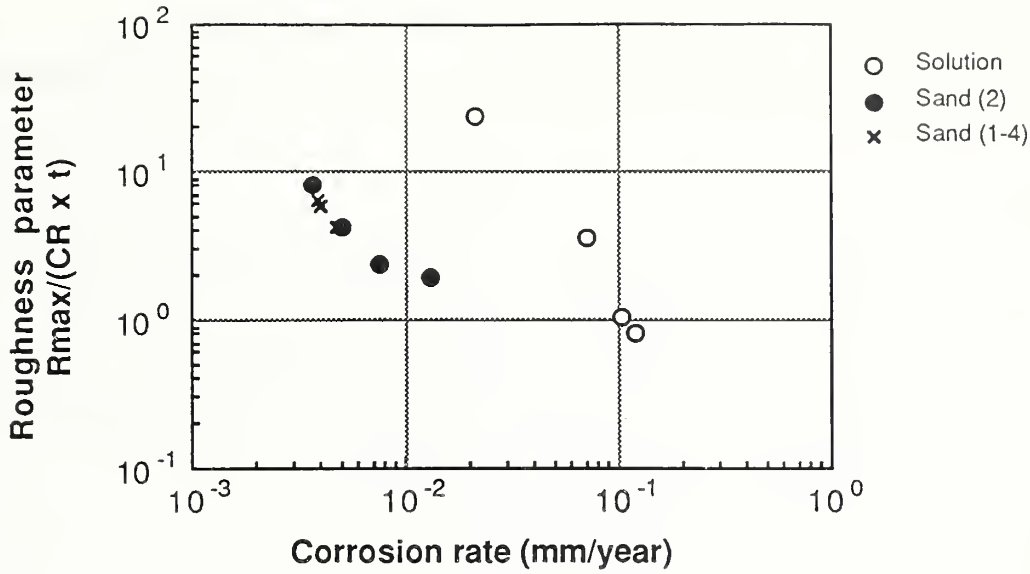


Figure 3. The roughness of the surface as a function of the corrosion rate in different media.

ELECTRODEPOSITION David S. Lashmore

The Electrodeposition Group is responsible for measurements and standards associated with electrodeposited alloys and intermetallics. The objectives of the group are: (1) the determination of the critical mechanistic, materials, and process variables controlling the structure/property relationships of electrodeposited coating and the development of approaches that will result in improved materials and processing controls for industry; (2) the provision of standards such as coating thickness standards, dye penetrant crack standards, tin-lead alloy standards, stage micrometer for SEM calibration and corrosion step test standards; (3) the development of new standards requiring electrodeposition for their fabrication or utilizing the unique properties of electrodeposited alloys; (4) the provision of government expertise to industry, through research associates and to the government agencies, through appropriate contracts and consulting arrangements; (5) to represent the interests of the United States Government on international and domestic standards organizations.

The following are some of the many areas in which alloy coatings are important for the commerce of the United States: (1) Strategic Materials - it has been shown that appropriate alloy coatings can provide a 30% savings of imported raw chromium. Research on new alloys indicate that for many applications, coatings can replace bulk stainless steels. (2) Corrosion - electrodeposited coatings play an important role in corrosion protection. It has been estimated that the cost of corrosion to the U.S. economy is in excess of 200 billion dollars per year. (3) Wear - the cost of wear to the U.S. economy has been estimated at about 50 billion dollars per year. Electrodeposited coatings play an important role in improving wear properties and surface coatings can be optimized for particular wear situations. (4) Electrodeposition Industry - specifically plays an essential role in the United States economy. For example, almost 900K tons of electrogalvanized sheet and strip are produced annually with an estimated value of about $150M per year projected by 1990; approximately 550K tons of metal coated wire and wire products are produced per year; tin plate production is about 8800 tons with a dollar value of $120M; and foil production accounts for about $200M per year (5) Magnetic Materials - Most hard disk drives are produced utilizing electrodeposition technology which thus has an important impact on the United States computer industry. (6) Electronic Materials - all printed circuit boards use electrochemical deposition in many stages of their fabrication including lead frames, electric contacts and through hole plating. (7) Processes - including decorative coatings, electroforming (compact discs), and electronic application (contacts, PC boards, etc.) are so important that without electrodeposited coatings much of our current industry would not be able to function in its present form. (8) Metal and Intermetallic Composites - a study carried out for the Japanese Ministry of International Trade and Industry (MITI) estimated a market of $3.5B/year for metal matrix composites alone by the year 2000. The market for aircraft engines, as given by the U.S. Industrial Outlook 1986, was $17 billion dollars and growing by $1 billion per year. This area of technology is important for the U.S. balance of trade - about $3 billion of U.S. exports are accounted for by this market and together with $96 billion in aerospace exports. High speed deposition of alloys with near atomic control of interface composition and deposition of intermetallics can play an important role in this area of advanced technology.

FY89 Significant Accomplishments

- The bulk of our SRM related computer programs were rewritten and incorporated into one large program. One of the special features is the 30 subroutines written as separate programs to facilitate debugging. All program features are menu accessible so that minimal training is now required for SRM production technicians. Some options available include calibrating, measuring samples, resetting and initializing six pieces of equipment including 2 types of magnetic gauges, a Beta backscatter and an x-ray florescent instrument and complete control and calibration of the robot measuring system.

- Artificial metal superlattices of a very high degree of perfection have been produced with most samples exhibiting at least second order satellites and all samples exhibiting magnetic viscosity, a phenomena not reported for superlattices produced by other processing technologies.

- It has been shown that during the electrodeposition of Al-Mn alloys from chloroaluminates containing manganese chloride, the extent of deviation from equilibrium and the degree of ordering is defined by the concurrent processes of new layer formation and surface diffusion.

- A high speed cell designed for alloy deposition on fibers using a triaxial impinging jet technique has been fabricated and is capable of depositing three separate alloys on monofilaments. This cell allows complex metal and intermetallic composite precursors to be produced to test new concepts in tailored interfacial zones and to test new matrix types. Cost data obtained with the cell suggests that electrochemical techniques can be more than an order of magnitude cheaper than other competing processing technologies.

- Electrochemical deposition of modulated chromium has yielded apparent anisotropic deformation dependent upon modulation wavelength. This deformation is significant at wavelengths of 300-500 nm, but insignificant at 1-3 nm. The microhardness of the modulated chromium was found to be 1500-1800 kg/mm^2 at wavelengths of 1-3 nm compared with a typical microhardness of 1000 kg/mm^2 for the unmodulated material.

- The direct electrodeposition of AlTi has been demonstrated from a chloroaluminate electrolyte containing controlled additions of Ti^{++} at 150 °C. The process is presently limited by the solubility of $Ti[AlCl_4]_2$ in the electrolyte which results in AlTi formation only at very low current densities. Forming intermetallic compounds by electrodeposition may help overcome fabrication and machining problems inherent to the material and presently limiting other processing techniques.

Metal Matrix Composites

C. R. Beauchamp, S. A. Claggett, C. E. Johnson, D. R. Kelley, D. S. Lashmore, J. L. Mullen, G. R. Stafford, E. Rosset* and N. Wheeler**

* Guest Scientist - Swiss Federal Inst. of Tech.
** Guest Scientist - American Iniversity

A three year investigation of the behavior of interfaces in composites is being continued with the American Cyanamid Corporation. A contract for several miles of coated monofilament fiber has just been completed with the Textron division of the AVCO Corporation. This program has yielded new technology concerned with (1) fiber handling and (2) making electrical contact with fibers. It has also yielded cost data comparing electrochemical technology with other processing techniques. It appears that graphite tows can in principle be coated with sufficient material to constitute the matrix at a cost of around $70 per pound including the cost of the fiber. An improved electrolyte for depositing a diffusion barrier layer Studies this next year will focus on (1) high speed direct deposition of AlTi, (2) investigation of the bromo-aluminate system, (3) incorporation of a fused salt cell in the fiber coating system and (4) investigation of titanium aluminide deposition.

Interfaces in Composites

N. Wheeler** and D. S. Lashmore

** Guest Scientist - American University

The emphasis of this year's cooperative effort between NIST and American Cyanamid Company has been mechanical testing, characterization, and optimization of the electrodeposition parameters of polyacrylonitrile (PAN) graphite fibers that have been electrolytically coated with an alloy (diffusion barrier) and then with nickel. The coating, which has been proven to inhibit the interdiffusion of carbon and nickel, was shown by fiber-punchout tests on unannealed samples to have better than twice the adhesion of the fiber/nickel bond. Transmission Electron Microscopy (TEM) of the uncoated fibers in the nickel matrix after annealing for 24 hours at 800 °C shows preferential dissolution of certain regions of the fiber into the nickel, resulting in extreme notching of the fibers. This phenomenon is shown in Figure 1 of a graphite fiber coated with nickel heated at 800 °C for 24 hours. Note the periodic serrations which extend for some distance along the fiber. There is some suggestion that carbon is concentrated at the nickel grain boundaries as shown in Figure 2. As shown in Figure 1, not only does carbon diffuse onto the nickel but the nickel diffuses through graphite. When the same type of fiber is coated with a diffusion barrier and then with nickel and heated to the same temperature then a very different microstructure develops as shown in Figure 3. With this barrier coating applied there is no appreciable degradation of the fiber at 800 °C for 24 hours. The immediate neighborhood of the graphite/interphase zone seems to be full of very fine carbide particles as shown by x-ray diffraction. There also seems to be a very thin (10 nm) zone of very different morphology from the bulk material.

Fiber Plating

E. Rosset* and C. R. Beauchamp

* Guest Scientist - Swiss Federal Inst. of Tech.

A new cell design has been developed based on the electrochemical simulations of current distribution on a high resistivity moving fiber taking in account the mass transport in the solution. Three rows of crossing jets impinge on the fiber (Figure 4) in order to get the high mass transport required for high rate deposition. With a cell fabricated according to the model, it has been shown that the deposition rate is at least 16 times higher (50 μm/min) than what is considered as a high speed electrochemical deposition.

At the request of Textron Specialty Material a 33 μm diameter amorphous carbon fiber has been overplated with a flash (0.1 μm) of a diffusion layer followed by a thick layer of copper (1.5 μm) and a flash of Ni (0.2 μm). An EDAX line scan shown in Figure 5, shows the three layers with a Cu overplate.

Work on optimization of the previously developed fiber plating cell continued. The time consuming process of threading the cell was resolved by the removal of the center glass cylinder that generated the triaxial jets. The main function of the jets was to enhance mass transport, by reducing the diffusion layer thickness and the turbulent flow created by increasing the flow rate at the small cross sectional area of the cell was tested. Current densities ranging from 114 mA/cm^2 up to 3161 mA/cm^2 were achieved without decreasing the quality of nickel deposit into a copper wire. Adhesion was tested using the "bend" method and proved satisfactory for the complete range. In addition, nickel deposition onto 140 μm diameter SCS-2 fiber was successfully accomplished. The implications are that a much simpler high speed cell can be produced.

Micromechanical Modeling and Testing of the Strength of Electrodeposited Coatings on Graphite Fibers

R. Clough, N. Wheeler**, E. Rosset*, D. Lashmore and S. A. Claggett

* Guest Scientist - Swiss Federal Inst. of Tech.

** Guest Scientist - American University

Electrodeposition of coatings on fibers is a preferred technique for altering both interfacial chemistry and strength in metal matrix composites. At the present time, however, the only methods for testing interfacial strength of these coatings are push-in and pull-out type methods. These generate shear stresses at the interface by means of an indenter or external tensile load. They have the disadvantage of creating additional stresses, which are not present at a natural fiber fracture in a composite. An in-situ method has been developed for testing interfacial strength which generates the shear stresses (normally essentially absent on an infinite fiber) from a fiber fracture. The technique has been successful in larger specimens and is being adapted to microtensile specimens.

So far, the stress analysis of the fiber fracture has been obtained using a new approximation technique developed by Professor Arridge of the University of Bristol using distortion energy technique to delineate the plastic flow surface at the fracture. In the accompanying experimental program, a new gripping technique has been developed for the fibers which minimizes bending moments and possible handling damage, and the tensile strengths of electrodeposited nickel coatings has been measured. A pronounced size effect for graphite fibers as a function of fiber diameter has also been shown. At present, the proper experimental parameters to generate fiber fractures without fracturing the coatings is being developed, so that the interface strength can be measured.

Electrochemical Deposition of Aluminum Alloys and Intermetallics
G. R. Stafford

Amorphous, quasicrystalline, metastable crystalline and equilibrium crystalline structures have been observed in binary aluminum-manganese alloys electrodeposited from a chloroaluminate electrolyte containing controlled additions of $MnCl_2$. During electrodeposition, the extent of the deviation from equilibrium and the degree of ordering is defined by the concurrent processes of new layer formation and surface diffusion. Several metastable phases are formed at deposition temperatures of 150-250 °C. Among these are supersaturated fcc-Al, an Al-Mn amorphous phase, and Al_8Mn_5 with a cubic γ-brass structure. The direct formation of quasicrystals and stable intermetallics has been achieved by an increase in deposition temperature in a manner somewhat analogous to that which has been reported for sputter deposition. The icosahedral and decagonal metastable phases as well as stable Al_6Mn and triclinic $Al_{11}Mn_4$ are formed at deposition temperatures approaching 325 °C.

In the aluminum-titanium system, the formation of a broad range of alloy compositions is possible as long as the concentration of Ti^{+2} is sufficient to support the partial current required for titanium deposition. The process is presently limited by the solubility of Ti^{+2} in the electrolyte. Electrodeposition from a chloroaluminate electrolyte containing 120 mM $Ti[AlCl_4]_2$ at 150 °C and at moderate current densities (5-50 mA/cm^2) results in the formation of alloys containing 10-40 wt.% Ti. The structure of these electrodeposits is a mixture of supersaturated Al and an a Al-Ti amorphous phase of unknown composition Figure 6. At very low current densities (100 μA/cm^2), the titanium current efficiency increases significantly and the resultant deposit is entirely crystalline. The x-ray diffraction pattern of the electrodeposit is consistent with that of AlTi in that the (001) and (110) superlattice reflections are well-defined. An SEM micrograph of the AlTi electrodeposit on a copper substrate is shown in Figure 7. The electrodeposition of these alloys from molten salt electrolytes offers several advantages over other solidification techniques. Alloy composition can be rigorously controlled through melt composition and electrode potential; and information regarding nucleation and growth can be obtained from the current transient at the beginning of potentiostatically controlled deposition. Forming intermetallic compounds by electrodeposition may help overcome fabrication and machining problems inherent to the material and presently limiting other processing techniques.

Test for Shear Stress of Various Micron Graphite Fiber Matrix Composites
J. Mullen

The push-pull test for graphite fibers has been shown to be an excellent way to evaluate adhesion of the matrix. The fibers are in three different conditions as shown in Figure 8. The graph reveals that the adhesion of fibers coated with a diffusion barrier layer is much greater than that for fibers coated with unannealed on annealed copper. The unannealed and annealed nickel-coated polycrylonirile graphite fibers should show the greatest difference in adhesion because of the diffusion of the carbon onto the nickel matrix. At room temperature, it is not expected that much diffusion of graphite into the matrix would occur. The large value of the adhesion for fibers coated with the diffusion barrier layer compared with copper suggests that some degree of covalent bonding is being established the barrier layer present.

Wear Resistant Coatings for the Printing Industry
C. E. Johnson, D. R. Kelley, J. Mullen and D. S. Lashmore

A second year study of the wear of electrodeposited coatings on wiper blades used on water-wipe currency printing presses, at the Bureau of Engraving and Printing (BEP) was started at mid-year. The purpose of the study is twofold, (1) to evaluate the most promising coatings, chosen from the results of NIST accelerated laboratory wear tests, under controlled conditions on a water-wipe press and (2) to provide specifications and a procedure for procurement of hard chromium plated W-1, W-2, and W-3 blades by outside vendors subject to the final approval by BEP.

Four types of coatings were proposed for use on wiper blades; a standard hard chromium as a control standard, multilayer chromium which is a modulated deposit, particulate composites of silicon carbide (SiC), boron carbide (B_4C) particles in nickel and chromium matrices, and a flame-sprayed chromium oxide coating. All of the coatings will be applied to each of the three blades (W-1, W-2, and W-3) used in the water-wipe press to evaluate the wear in the environments to which the different blades are subjected.

W-3 blades, which operate in a dry abrasive environment, have been coated with the control standard hard chromium and the flame-sprayed chromium oxide. A plating cell has been designed and fabricated to deposit the particulate composites on the various wiper blades. Specifications for hard chromium plating of wiper blades were drafted and presented to three outside vendors. One vendor is now supplying chromium plated W-1 and W-3 blades for in-situ testing at BEP. An extension of the work on pulse plated duplex chromium coatings on wiper blades has led to an investigation into the deposition of structure or textured modulated chromium coatings. Based on the theory that chromium is deposited as a hydride (FCC or HCP structure) which decomposes with time and temperature to the BCC metallic chromium structure, control of the operating parameters during deposition (such as electrolyte temperature, current density, and pulsing condition) can result in either layers of different preferred orientation of a BCC structure or layers of a BCC structure and a mixed BCC, HCP, and FCC structure. Long term stability of the structure modulated coating is not known, but the texture modulated coatings appear to be stable after six months at ambient temperature. The microlayered chromium deposits have been found to have an apparent anisotropic deformation characteristic which depends on the layer spacing. The characteristic is significant at layer spacing of 300-500 nm, but insignificant at 1-3 nm. The microhardness of the microlayered chromium was found to be 1500-1800 kg/mm^2 at layer spacing of 1-3 nm compared with hard chromium hardness of 1000 kg/mm^2.

<u>SRM Coating Thickness Standards</u>

H. G. Brown, C. R. Beauchamp, D. R. Kelley and D. S. Lashmore

The SRM software programs were rewritten and incorporated into one massive program. One of the special features of this program is that each subroutine (about thirty in all) is written as a separate program, making it much easier to locate and solve any problems. It also has a very extensive menu with thirty-seven options. Some of the options are calibrating, measuring samples and, resetting and initializing six pieces of equipment including the magna, a magnetic tester and the Beta backscatter, an x-ray tester. This program is now completely user friendly.

An extensive comparison study between NIST coating thickness standards and the coating thickness standards of the Community Bureau of Reference (BCR) of the Commission of the European Communities. Three different measurement techniques were used in this study to cross reference each thickness with two different techniques. The result of the study shows that the NIST coating thickness standards are well within our claim of a five percent reproducibility (less than 1.5 percent deviation). The deviations for the BCR samples were reproduced within 1.3 percent when using NIST Standards. It was shown that sputtered BCR samples become rough and develop large pits as they become thicker compared to the NIST electroplated samples which have a more uniform surface texture. Errors may arise when using energy dispersive instruments, as in the case of the 14 mg/cm^2 sample, and incorporating a requirement for the measurement of an infinitely thick standard. Calibration curves that require x-ray measurements depend on surface roughness.

A bright acid copper plating system is being designed and fabricated. This new system will be completely automated with the exception of rotating the panels. Thickness uniformity will be improved due to the system design. Brightner will be added automatically and the temperature, current, and pH of the electrolyte will also be automatically monitored and controlled.

<u>Research on Lead-Tin Standard Reference Material</u>

P. N. Sharpless, D. S. Lashmore and H. Brown

An analysis of the sources and magnitude of errors associated with production of the lead-tin coating thickness SRM was made. New techniques to increase the accuracy of atomic absorption measurements have been developed. These techniques concern the measurement of the Pb-Sn alloy, as well as the copper substrate, which dissolves slightly during coating stripping. Use of a precision density meter and a balance to compute dilution volumes resulted in a five-fold decrease in error over the more common method of using volumetric glassware. Improvements in the measurements of the sample area increased the accuracy of area measurements to 99.99867 %. The focus of the past years efforts was to develop a clear understanding of the accuracies of the various steps in the certification process. Delivery of standards is scheduled for the first quarter of FY90.

Research on Ni-P Alloys
J. Mullen

A new technique to measure the phosphorus content in nickel phosphorus alloys, used to coat magnetic discs has been developed. This technique makes use of spectrophotometry of a dissolved sample. The difference between this technique and previously used techniques is that (1) it is a room temperature process, (2) there are no elements added which may interfere with the phosphorus measurement, and (3) the process is much faster. The accuracy has been verified by comparing gravimetric phosphorus measurement and the atomic absorption nickel measurement.

Research on a Scanning Electron Microscope Calibration Standard
D. R. Kelley

The Electrodeposition Group has a contract with OSRM to electroform, cut, mount and polish 4 SEM panels. Between 350-400 samples are obtained from each panel. New electroforming techniques are proposed to improve thickness and uniformity of the layered structure for this second generation SRM.

Direct Deposition of Intermetallics
C. E. Johnson, D. S. Lashmore, G. R. Stafford

Evidence has been found which suggests that crystalline intermetallic compounds of nickel and phosphorus can be deposited directly from aqueous electrolytes at 75°C. Electrodeposition of these alloys was accomplished at low current densities (3-5 ma/cm^2); whereas, electrodeposition at higher current densities (50-400 ma/cm^2) resulted in amorphous alloys. Preliminary x-ray diffraction investigation has indicated a crystalline structure not yet identified. Transmission electron microscopy studies have indicated that the coatings have a textured crystalline structure with long range order over a very large lattice constant which is consistent with typical nickel phosphide structures. A typical micrograph is shown in Figure 9. Further studies are expected to clearly identify the appropriate compound and enable correlation with the nucleation kinetics.

Artificial Superlattices
D. S. Lashmore, R. Oberle, L. Bennett, L. Swartzendruber

Further progress has been made in the deposition of copper-nickel artificial superlattices under sponsorship of the IBM corporation. Changes in the electrolyte formulation have now resulted in the consistent development of second order satellites as shown in the example in Figure 10. These alloys were deposited onto (100) highly textured polycrystalline annealed copper foil. The x-ray diffraction usually exhibits only the (100) Bragg reflection. The saturation magnetization for these samples is somewhat lower than the best of our Cu-Ni superlattices. The best samples had significant

(111) and (110) texture but only showed a single order satellite. The curvature of the magnetic saturation vs. temperature relationship is related to the degree of alloying at the interface between the copper and nickel. The latest (100) samples show more curvature than the earlier (111) + (100) textured superlattices suggesting that interdiffusion on the (100) plane is responsible for the lower saturation magnetization. This phenomenon is now being studied by conducting the deposition on single crystal substrates of different orientations.

TEM studies of copper nickel bicrystals with thickness large enough so that epitaxy should not occur are shown in Figure 11. Both Moire Fringes as well as periodic but non uniform strain features are apparent. Epitaxy seems to be maintained up to thickness of 5.0 nm consistent with the latest theory of Jesser and Van der Merwe.

A computer model of x-ray diffraction from composition modulated alloys is in the process of being developed and makes use of expressions of the periodic electron density function of which a fast fourier transform is made. The model will eventually allow non uniform strains as well as the effects of interdiffusion to be modelled. Future work under this program will involve Co-Cu and Co-Cr superlattices and reducing nickel thickness to near 0.5 nm.

Figure 1. A transmission electron micrograph of a nickel coated PAN graphite fiber heated to 800 °C for 24 hours showing periodic serrations along the fiber. (300 keV)

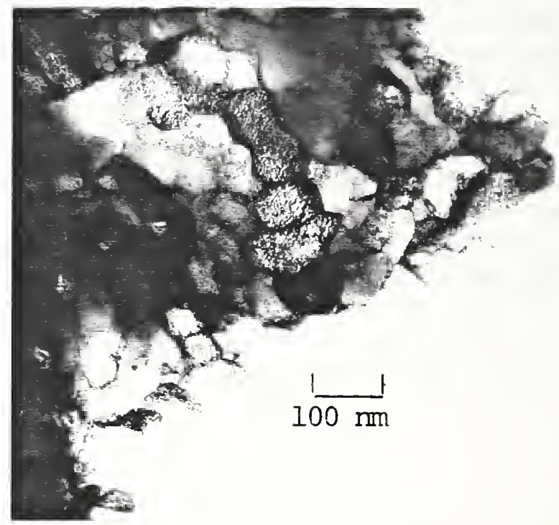


Figure 2. A TEM micrograph of the nickel region suggesting that graphite is concentrated along the nickel boundaries.

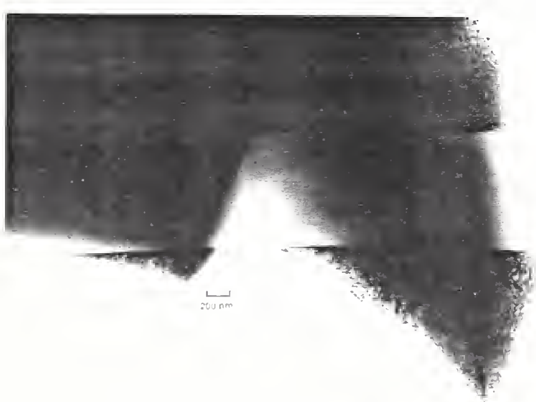


Figure 3. A TEM micrograph of a cobalt tungsten coated PAN graphite fiber subsequently overcoated with nickel then heated to 800 °C for 24 hours. Note that this barrier layer inhibits dissolution of the graphite and that a larger population of very small carbides exist.

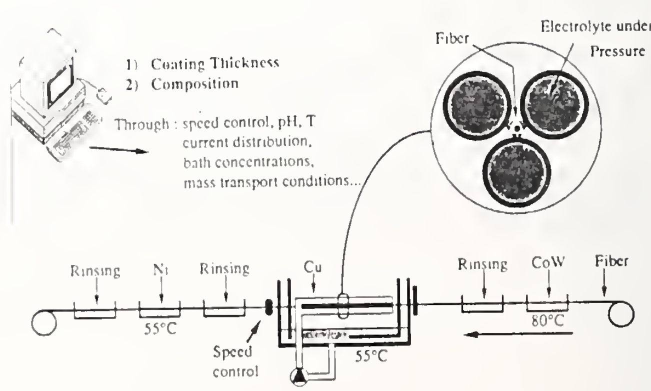


Figure 4. A schematic diagram of a high speed alloy deposition cell designed for coating fibers.

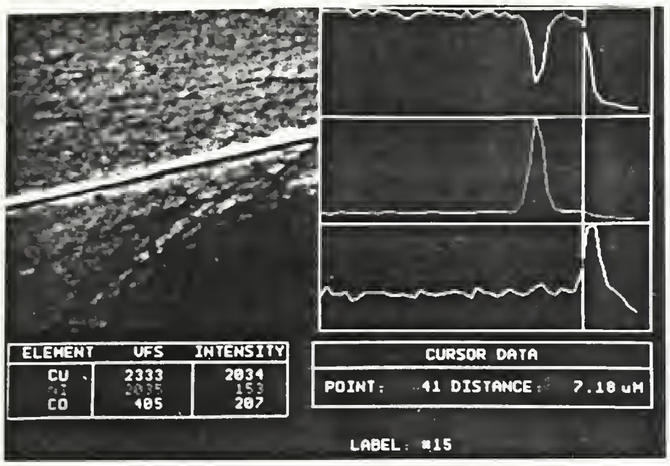
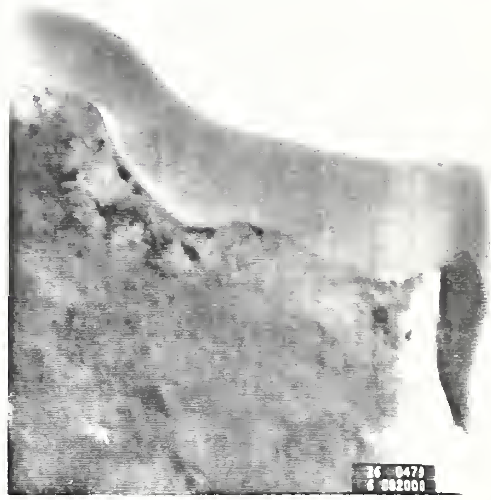


Figure 5. An EDAX line scan of a Co-W-Cu-Ni coated graphite fiber.

Figure 6. A duplex structure of electrodeposited Ti-Al (18% wt. percent Ti) consisting of supersaturated fcc-Al and an aluminum titanium amorphous phase of unknown composition.



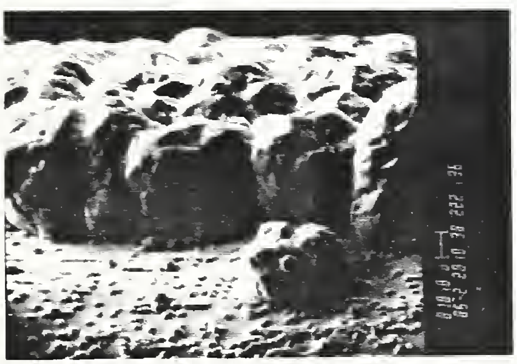
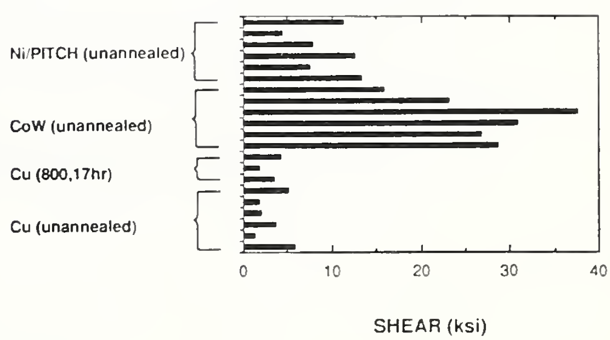


Figure 7. An SEM micrograph of a Ti-Al intermetallic produced electrochemically.

Figure 8. Adhesion measured by a punch out test of Co-W compared with copper both electrochemically produced on 7 μm graphite.



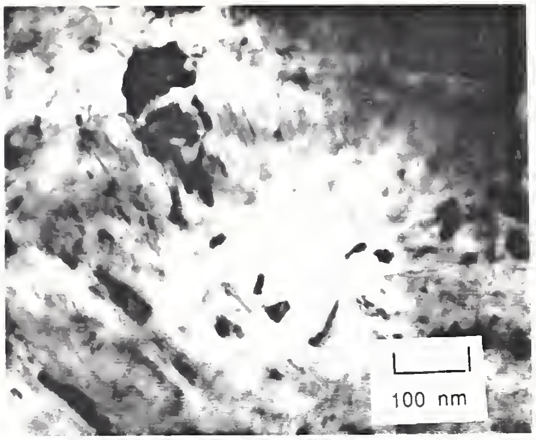


Figure 9. A TEM micrograph of an intermetallic phase of nickel phosphorus produced by direct/electrochemical deposition at room temperature.

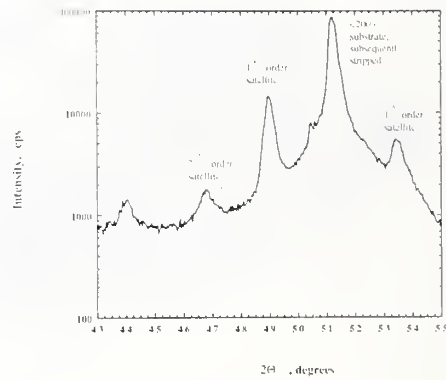


Figure 10. An X-ray pattern of a typical (100) textured superlattice showing second order satellites.

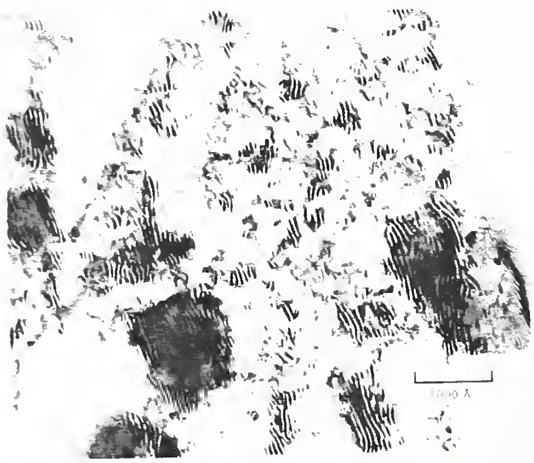


Figure 11. A TEM micrograph of a (100) Cu/Ni bicrystal showing periodic strain features. (300 KeV)

MAGNETIC MATERIALS

Lawrence H. Bennett

The Magnetic Materials Group is concerned with the measurement of the magnetic properties of advanced magnetic and superconducting materials. The objectives are (1) to relate the metallurgical, magnetic and electronic structure to the magnetic and superconducting properties of materials, (2) to develop new and improved magnetic measurement methods, (3) to develop magnetic reference standards, (4) to apply magnetic phenomena to the nondestructive evaluation of materials and structures, and (5) to provide expertise to industry, universities and other government agencies.

Magnetic materials are important to the commerce of the nation. The sales of soft magnetic materials (primarily for information and data storage) amounts to more than $25 billion per year. The sales of hard magnetic materials (primarily for motors) are more than $1 billion per year and have been recently increasing due to the discovery of supermagnetic rare-earth alloys. Magnetic nondestructive evaluation methods are used for quality control everywhere steel is used. Conventional superconductors have many existing commercial uses, including MRI, and the new high-temperature superconductors have important potential applications, both near-term and future.

FY 89 SIGNIFICANT ACCOMPLISHMENTS

o Magnetic viscosity measurements were carried out for a series of Ni/Cu compositionally-modulated alloys (CMA) produced by the Electrodeposition Group. The data vary with temperature and sample, but the peak always occurs at the coercive force. Such time-dependent effects are critical to scientific understanding and to magnetic recording applications.

o Magnetic viscosity measurements on a sputtered Ni/Cu nanocrystalline multilayer sample (made by W. Abdul-Razzag, West Virginia U.) demonstrated that the magnetic aftereffect which we had previously discovered in the electrodeposited multilayers is not an artifact of the preparation process.

o The magnetization of a single crystal of the high T_c superconductor $YBa_2Cu_3O_7$ with a single habit for the twin planes was studied as a function of field and temperature, proving, for the first time, that the effect of twin planes on flux pinning is small but detectable.

o Flux dynamics in a sample of $YBa_2Cu_3O_7$ exhibiting very large pinning (prepared at Nippon Steel by a melt-growth process) was compared with a sample prepared by a ceramic technique. These studies revealed a number of important properties concerning the time behavior of the flux lattice, a property of great concern for practical applications.

o A new phenomenon in high-temperature superconductivity - the suspension of a superconductor below a magnet - was investigated (in collaboration with Hughes Aircraft and NASA/Huntsville) and the first explanation given.

o "A Brief Review of Recent Superconductivity Research at NIST" for the NIST Journal of Research was completed. This review paper portrayed the breadth of the NIST's contributions to the materials science, standardization, and engineering applications of superconductors.

- A highly successful workshop, co-sponsored by NIST and NASA, on Materials Science of High T_c Superconductors was conducted. The proceedings have been published. A follow-on Colloquium will be held at NASA Goddard Space Flight Center in April 1990.

- In cooperation with J. Ritter of the Ceramics Division, the morphology and magnetic properties of bulk nanocomposite made by the sol-gel process (Fe + silica gel) was measured, and a spin-glass effect was discussed.

- Barkhausen noise data from spheroidized carbide in ferrite (NIST SRM 493) were obtained and analyzed. The noise from these samples gave uniform characteristics, showing potential for use as a Barkhausen noise standard.

Compositionally-Modulated-Alloy Thin Films
L. H. Bennett and L. J. Swartzendruber

Compositionally-modulated alloys (CMA) with individual layers in the nanometer range have been produced by the Electrodeposition Group using electrochemical deposition. Magnetic measurements on these Ni/Cu multilayer thin films have demonstrated that electrodepositing nanometer-thick layers of the highest quality is feasible, and that the magnetic measurements constitute a meaningful characterization tool for them. Magnetic viscosity measurements have now been carried out for a series of such films; typical results are shown in Fig. 1. The lineshape in Fig. 1a varies with sample and temperature, but its peak always occurs at the coercive force of the material. The shape of the curve in Fig. 1b, including the peak position, varies with sample. Such time-dependent effects are scientifically useful in understanding the arrangement of magnetic moments in the thin films and they are of some consequence to the application of magnetic multilayers to magnetic recording. This work is partially supported by the IBM corporation.

There has been a large body of studies of the magnetic properties of Ni/Cu superlattices produced from the vapor phase by sputtering or other deposition techniques. It was discovered that there was a relaxation of the magnetic moment upon field changes in the electrodeposited Ni/Cu. Experiments were conducted to determine if the effect might be unique to the deposition process. It was discovered the same magnetic relaxation in sputtered Ni/Cu. Thus the effect does not appear to be an artifact of the preparation process.

High-Temperature Superconductors
L. J. Swartzendruber, L. H. Bennett, and R. D. Shull

Conventional levitation of a magnet above a superconductor occurs because of a "repulsive" force created between the two as the magnetic flux of the magnet is excluded from the superconductor. Superconducting composites of AgO and $YBa_2Cu_3O_{7-x}$ were found to possess a novel property at 77 K of being able to levitate by "attraction" as seen in Fig. 2, with the composite being suspended in space "below" a permanent magnet. Magnetization measurements as

a function of field for several composites of AgO and $YBa_2Cu_3O_{7-x}$ were performed to explain the origin and composition dependence of the novel "attractive" force. These data showed that the ability of the material to be suspended below a magnet is dependent upon the degree of magnetic flux trapping in the superconductor and that the ability to obtain a stable equilibrium position depends on the shape of the hysteresis loop.
A major barrier to commercial development of high T_c superconductors is our lack of understanding of the magnetic flux pinning and its effect on critical currents, including their dynamic characteristics. The effect of twin structures is of considerable interest because of speculation that twin boundaries provide a site for flux pinning. Several models have been proposed which predict that the interaction energy between twin boundaries and flux vortices will be large; hence the twin boundaries will provide strong flux pinning. The magnetization as a function of temperature and applied magnetic field in a single crystal containing only one twin boundary habit was studied. This permitted a magnetic field with a definite orientation with respect to both the crystalline c axis and the twin planes to be applied. It was shown that there is a small but measurable interaction between the flux vortices and the twin boundaries.

The flux dynamics in a polycrystalline sample (made by a melt-growth process at Nippon Steel) that exhibits very large pinning has been compared with a sample prepared by a ceramic technique. Fig. 3 shows the hysteresis loops from these two samples. Magnetization as a function of time shows the characteristic logarithmic behavior for both samples.

Continuing a cooperative program with the Johns Hopkins University Applied Physics Laboratory, two melt-grown single crystals of $YBa_2Cu_3O_{7-x}$ (grown by D. Kaiser, Ceramics Division) in the shape of nearly perfect parallelepipeds were measured by both SQUID magnetometry and by magnetically modulated microwave absorption (MAMMA). The two methods respond to the change in magnetic properties at the superconducting transition temperature, but in different ways, giving complementary information, with the MAMMA technique revealing structural details that are not evident in the magnetometry.

With the exception of Pr and Ce, any rare earth may be substituted for the Y in $YBa_2Cu_3O_{7-\delta}$ with little effect on the superconducting transition temperature. To explore the role of Pr in suppressing the superconductivity, we have performed Mössbauer effect and magnetic susceptibility measurements on a series of $Y_xPr_{1-x}Ba_2(Cu_{0.98}Fe_{0.02})O_{7-\delta}$ compounds were performed. These results indicate that a strong magnetic interaction between the Pr ions and the Cu(1) sites is responsible for the observed changes in superconducting properties.

A three-day workshop on the Materials Science of High T_c Superconductors (sponsored by NIST and NASA Goddard Space Flight Center) was conduced on October 11-13 at NIST. The Workshop featured panel discussions and demonstrations as well as lectures. The most successful feature of the workshop was the interaction between basic science and applications. The proceedings have been published. A follow-on Colloquium will be held at the NASA Goddard Space Flight Center in April 1990.

Nanocomposite Materials

R. D. Shull, L. J. Swartzendruber, and L. H. Bennett

Composite materials (immiscible metals and oxides with a size scale of the order of nanometers) have been shown to possess unusual composition-dependent electronic and magnetic properties. These materials provide a unique opportunity for the atomic engineering of specific material properties. Thin film nanocomposites made by sputtering (in collaboration with K. Moorjani, Applied Physics Laboratory, JHU) still continue to provide one avenue to these engineering possibilities. The role of processing in the tailoring of these materials' magnetic properties was shown. The long range (multidomain) magnetic behavior of Fe_3O_4 was destroyed by the addition of small amounts of Ag (< 3 wt. % Ag) to the nanocomposite when sputtering from a target comprised of a mechanical mixture of the two constituents. However, more than 40 wt % Ag was required to achieve this magnetic state when the two constituents were physically separated in the sputtering target.

In collaboration with J. J. Ritter (Ceramics Division, NIST) bulk nanocomposite materials have been prepared from homogeneous gelled composites of iron and silica containing 5-30 wt. % Fe by low temperature polymerization of aqueous solutions of ferric nitrate, tetraethoxysilane, and ethanol (with an HF catalyst). X-ray diffraction data, characterized by the presence of a diffuse scattering peak at $2\theta \approx 24°$, and the absence of Bragg scattering from the iron-containing regions, indicated that these bulk materials are nanometer-sized regions of iron compounds embedded in a silica gel matrix. Scanning electron microscopy observations show that this matrix has many interconnected pores; the pore size is related to the size of the Fe-containing particles. The room-temperature paramagnetism of these materials, as well as the small size of the iron-containing regions, was indicated by the appearance of only a high intensity central doublet in the ^{57}Fe Mössbauer spectra. The Mössbauer effect data demonstrated that the form of the iron can be changed by a subsequent treatment in an atmosphere of ammonia or hydrogen at elevated temperatures: for a 10 wt. % Fe sample treated with ammonia, only a central doublet was observed but with a much larger quadrupole splitting and isomer shift. Both of these subsequently treated materials became superparamagnetic at room temperature. In addition, magnetic susceptibility measurements indicated that the hydrogen treated material becomes a spin glass at low temperatures (Fig. 4).

A new concept of obtaining an enhanced magnetocaloric effect by using nanocomposites was analyzed and a patent application filed.

Magnetic NDE

L. J. Swartzendruber and H. Ettedgui*

* Guest Scientist - Nuclear Research Center - Negev, Israel

Activity during the current year has centered on Barkhausen noise experiments, computer modelling, and magnetic particle test rings. The Barkhausen noise method is capable of determining applied or residual stresses, whether in tension or compression. Other applications which have been proposed include measurement of grain size and hardness. For these measurements to be useful, proper calibration is required. This calibration requires both a knowledge of how the noise characteristics change with stress for the particular alloy microstructure being measured, and a knowledge of how the measurement instrument responds to the changes in noise characteristics. In order to characterize the noise from a sample, we have developed instrumentation which obtains a complete cycle of noise in digitized form. This information can then be analyzed by digital techniques to obtain all the noise properties of interest. Fig. 5 shows results obtained on a sample of spheroidized carbide in ferrite (NIST standard reference material 493), which gives a moderately strong noise output. The figure shows the noise pulse magnitude as a function of applied field, the integral of the noise, and a fit to the noise based on the assumption that the noise amplitude output as a function of applied field has a Lorentzian shape. The fact that the Lorentzian shape fits so closely makes this material a possible standard for calibrating Barkhausen instruments.

A simple, almost exactly solvable, model which simulates the dynamics of a single magnetic domain wall crossing a single impurity site in a ferromagnetic material, was solved numerically, and chaos found for some ranges of parameters. The dynamics are complex and very sensitively dependent on the physical parameters: the frequency and strength of the applied field, the strength of the pinning, the reversible permeability, the effective mass of the domain wall, the wall viscosity, and the amount of energy retained by the wall when it breaks free of the pinning site. A period quintupling was discovered. Phase space portraits, return maps and total energy spectra were used to display the results.

Cooperative work on standards with ASTM committee E07 and committee K of the SAE has continued during the current year. The magnetic particle inspection standard E709 is being revised and updated based, in part, on work performed on this project in the past. A new ring standard for use in magnetic particle inspection is being designed. During the coming year it is planned to produce several of the new rings and perform round-robin tests in cooperation with these committees.

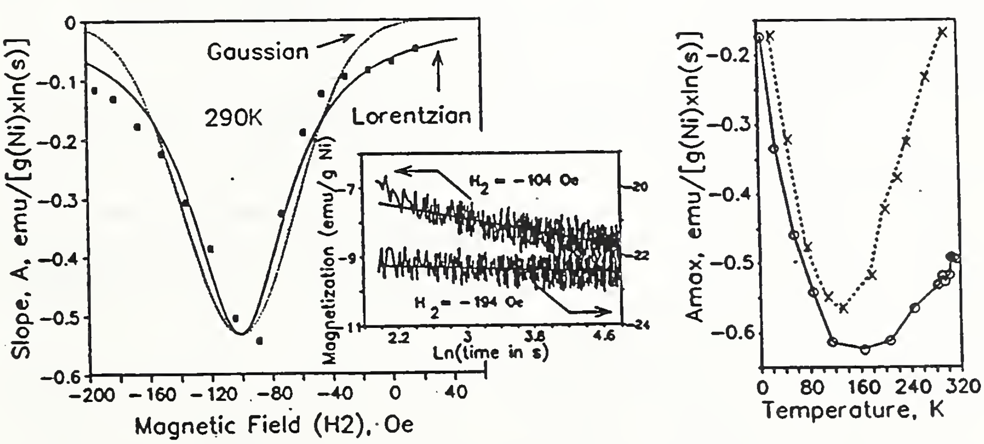


Fig. 1 (a) Inset: The decay of magnetization (magnetic viscosity) of a Cu/Ni multilayer thin film, as a function of the logarithm of time after a sudden change of applied field from 2500 Oe to field H_2. Main figure: The slope, from data similar to that shown in the inset, as a function of the field H_2. (b) The maximum value of the slope of the decay from figures such as Fig. 3a, plotted as a function of temperature.

Fig. 2. A superconductor (the small black disk) which has just been raised from liquid nitrogen is shown suspended below a magnet. This novel effect was explained in terms of magnetic flux trapping.

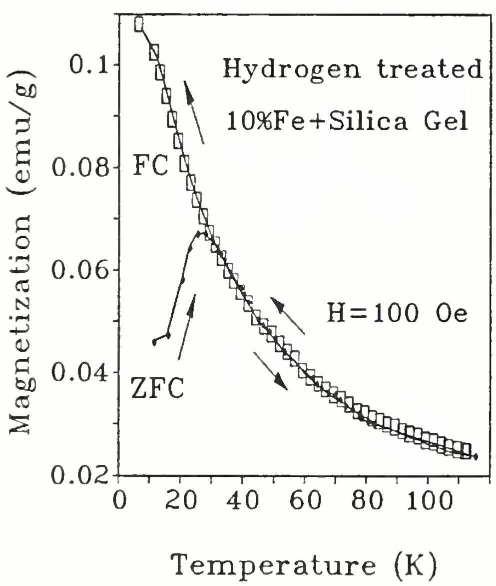


Fig. 4. Magnetization vs temperature for a nanocomposite prepared by a sol-gel process. ZFC = cooled in zero magnetic field from 295K; a field of 100 Oe was then applied. The magnetization upon heating displays a peak at ≈30K; the magnetization upon cooling in a field (FC) does not show this maximum. This behavior is an indication of a spin-glass.

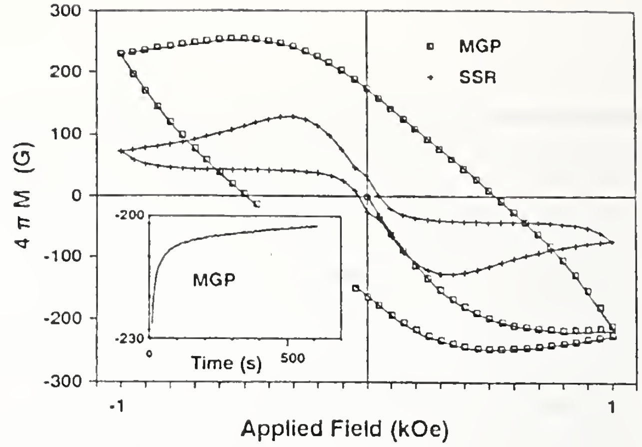


Fig. 3. Hysteresis loops taken at 77K for two BYCO high temperature superconductors prepared by different techniques. The inset show the time dependence of the magnetization (flux creep) for one of the samples (MGP) taken at 80 K after a sudden applied field change from 0 to 2 kOe.

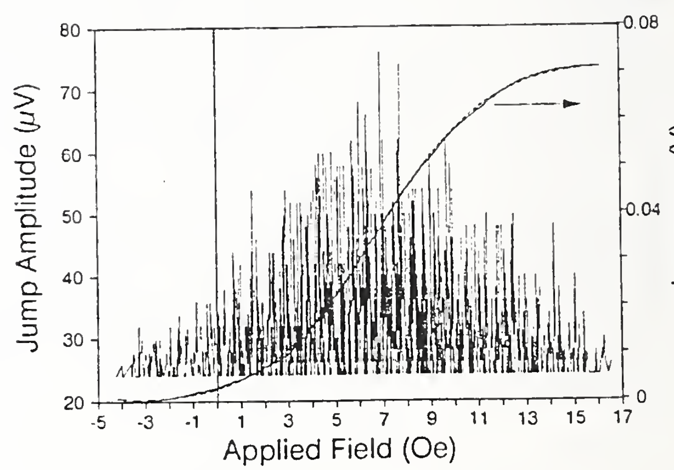


Fig. 5. Barkhausen noise data from SRM 493. The noise spikes are plotted vs. the applied field in Oe. The solid, nearly smooth, curve represents the sum of the jump amplitudes vs. applied field (right scale). The dotted line superimposed on the jumpsum is an integrated Lorentzian.

HIGH TEMPERATURE MATERIALS CHEMISTRY

John W. Hastie

The research program in high temperature materials chemistry emphasizes the thermodynamic, chemical-kinetic, interfacial microstructure, and molecular-level behavior of inorganic materials in high temperature process and service environments. Specific current objectives are (1) to support the U.S. Steel Industry through development of a thermodynamic database and solution model for prediction of slag, refractory, and inorganic steel inclusion thermochemistry; (2) to develop and apply (eg., to refractory ceramics and composites) a new molecular-specific methodology for obtaining thermal and chemical stability data at ultra-high temperatures (2000 - 5000°C) for the design of hypersonic transport vehicles and various defense applications; recent surveys by NMAB and Industry have indicated a critical need for thermochemical and other materials property data at these temperatures; (3) to develop process models and basic data for the preparation of thin films, superconducting and other, using laser or other vapor deposition techniques; (4) to enhance the utility of superalloy and carbon/carbon composites in high temperature oxidation environments through mechanistic studies of oxidation; (5) to obtain phase equilibria, kinetic, and mechanistic data for the development of stored chemical energy propulsion systems; and (6) to provide technical support to the NIST/ACerS Ceramic Phase Diagram Data Program through critical evaluation and modeling of phase diagrams and the development of computer graphics for computer storage and manipulation of phase diagrams.

FY 89 Significant Accomplishments

o A previously developed thermodynamic model and accompanying database have been expanded for the prediction of detailed composition and phase properties of multicomponent-multiphase iron containing oxide systems. The model has been shown to be applicable to typical iron-making slags and refractories used in steel processing. The model database currently contains over 125 components and has been successful in predicting high temperature thermodynamic activities and phase compositions in mixtures containing up to eight component oxides. Several industries are beginning to substitute this model for the overly simplistic chemical models currently used in transport-type processing computer codes.

o The laser-induced vaporization mass spectrometric technique (developed in-house) has been applied to vapor plumes produced from refractory carbide (SiC, TaC), and oxide (HfO_2) targets. Time-resolved mass and optical emission spectra have been obtained for many neutral and ionic species in the laser generated plumes. Ultrafast signal-averaging (100 MHz) and optical multichannel spectroscopic capabilities have been added to the laser-induced vaporization facility. These new techniques permit both mass and optical spectrometric techniques to be applied to the study of non-equilibrium processes occurring on the laser-impact time scale of a few nanoseconds.

o A new mass spectrometric and complementary thermochemical investigation of the ternary Nb-Al-O system has been initiated as part of a cooperative DARPA program with CEBELCOR (M. Pourbaix, Belgium) and the University of Florida to develop oxidation models of advanced intermetallics. Activity data on Nb-Al intermetallics and their oxidation products are being obtained under this study. Also a preliminary THERMOCALC model of the ternary Nb-Al-O system has been developed to guide the experimental work.

o An international conference, on High Temperature Materials Chemistry, was organized and hosted at NIST. This IUPAC-sponsored conference brought together the leaders in the high temperature materials chemistry field for presentations and discussions in the areas of (1) Advances in Measurement Techniques, (2) Thermochemistry and Models, (3) Processing and Synthesis, and (4) Performance under Extreme Environments. A three volume proceedings is in the publication process (Humana Press).

<u>Steel Slag -Refractory Thermochemistry</u>

J. W. Hastie, E. R. Plante, D. W. Bonnell, W. S. Horton*, M. Kowalska**

* Guest Scientist - Private Consultant
** Guest Scientist - Tech. Univ. of Wreclow, Poland

Modeling and complementary experimental work has continued on the development of a generic predictive model of iron and steel-making slag and refractory thermochemistry. The computer model is already in use by several steel and other industrial laboratories and consortia. In addition to its present utility for existng steel-making technology, the model is designed to be applicable to new direct-reduction steel making technologies being planned or under development. In particular, the high FeO-content systems under current study were chosen to meet the needs of the AISI-DOE national program to develop a new direct-reduction steel making process.

Current experimental work has involved the determination, by mass spectrometry, of activities as a function of FeO content in a model FeO-SiO_2-MgO slag utilizing the ion intensity ratio method developed by Belton.

The computer-based model is quite general, predicting detailed phase compositions of both simple and complex multicomponent, non-ideal, high temperature oxide slag systems. The model is based on assignment of complex or associated solution *components* (for example, $Fe_2SiO_4(\ell)$ and $CaFe_2O_4(\ell)$ in steel slags) that account for the known non-ideal interactions. Gibbs energies of formation functions for simple oxides and complex components are explicitly included in an extensive database for use with multicomponent solution codes. The database now contains over 150 components covering 18 elements.

Recent tests of the model against new oxide data by Rudnyi, et al, containing both Na_2O and K_2O, indicate that mixed alkali species, which are known to be significant vapor species, are not important in the slag phase. A recent evaluation of thermodynamic functions by Hildenbrand was tested and incorporated where consistant with our existing database.

Users of the model, as the chemical "engine" in process codes, have indicated a need to expand the temperature range of validity of the current database. Because of the phase sensitivity of the model to small errors in individual component fits, segmented fitting of the data base has been required. A reevaluation of the literature is also ongoing. New species of interest to steel-making are being added as quality data can be found and validated.

Recent comparisons of model and experimental data for the iron-bearing mineral illite have shown an unexpected sensitivity to the Fe-O ratio in aluminosilicate-type slags. While still under investigation, the model predicts major activity changes in the solution phase for small changes in Fe-O atom ratios, as may occur, for instance, during the redox processing step in preparing ladle heats.

Thermodynamic and Kinetic Stability of Refractory Materials at Ultra-High Temperatures

D. W. Bonnell, P. K. Schenck, J. W. Hastie, M. Joseph*

* Guest Scientist - Indira Gandhi Centre for Atomic Research, India

The coupling of laser heating with mass spectrometric and optical spectroscopic analysis has the potential for providing quantitative thermochemical data for refractory materials at temperature and pressure extremes heretofore inaccessible to conventional techniques. In addition, degradation of materials by high powered lasers is important in, for example, the design of laser fusion processes, laser welding, laser processing of ceramics (most recently, superconducting and diamond films), laser etching of semiconductor components, laser annealing of surface alloys, and in the durability of refractories in defense and space applications.

It has been shown that a Nd/YAG laser system, focused to power densities in the region of 10^8 W/cm^2, is a convenient energy source for producing controlled vapor plumes with generally negligible post-vaporization perturbation of the neutral species identities and concentrations. The NIST-LIVMS technique utilizes time-resolved mass analysis to provide time-of-flight species specific information on temperature, ionic and neutral precursors, and the time history of the laser heating process.

The mass spectrometric time-of-arrival information indicates that ions produced by the laser heating process are not at thermal equilibrium with the neutrals, even though the quantity of ions is consistent with a Saha (thermal) model. A major objective of current work is to correlate the results obtained by both mass and optical spectroscopic detection, which are are sensitive to ions and neutrals.

Current work has involved the refractory carbides (Ta_2C, SiC) and the most refractory known oxide, HfO_2. Figure 1 shows a typical mass spectrum obtained from laser vaporization from polycrystalline 98% dense SiC. Of particular note is the observation of SiC_2, SiC_3, Si_2, and Si_2C species. Current thermodynamic data indicate SiC_2 should be of the order of 4% of the vapor at 2000 K, whereas our data indicate SiC_2 to be nearly 25% of Si (the main gas species) at temperatures in excess of 3200 K.

Mechanistic Determination of Laser Deposition of Thin Films

P. K. Schenck, D. W. Bonnell, J. W. Hastie, J. Zhao*

* Guest Scientist - Academia Sinica Beijing, China

This project is being undertaken in collaboration with the Magnetic Materials group and the Ceramic Division's Electronic Ceramics group to study the mechanisms of laser vaporization and deposition of thin films. Thin films deposited by laser vaporization from bulk high temperature superconducting material have shown evidence of high temperature superconductivity. Time resolved mass spectra were obtained using the LIVMS for many neutral and ionic species in the laser generated plume from $YBa_2Cu_3O_x$ targets, including bimetallic species CuBa and YCu. A variety of ceramic materials including MgO, Al_2O_3, yttria/zirconia and mullite were also deposited as thin films using laser vaporization. In cooperation with Harry Diamond Laboratories, laser deposition studies of lead zirconate titanate (PZT) thin films on silicon have recently been initiated. A high speed optical multichannel analyzer was used to apply optical spectrometric techniques to the study of the laser generated plumes from PZT targets. Figure 2 shows the time-resolved optical emission spectra obtained from the PZT plume at distances of 5 and 10 mm above the target surface. Emission line identification is aided by special computer software and reference spectra taken of single element targets. Analysis of these spectra indicate that the laser pulse forms a plasma, containing ionized metal atoms, which persist after the laser pulse. This result suggests that the vaporization may, in part, be influenced by the interaction of the plasma with the target surface. Work is underway to further correlate plume species with the composition and characteristics of the resulting thin films.

Processing and Protection of High Temperature Structural Materials

J. W. Hastie, D. W. Bonnell, E. R. Plante, M. Kowalska*

* Guest Scientist - Tech. Univ. of Wreclow, Poland

This activity represents a new group thrust initiated late in FY 88 under a DARPA sponsored program with Univ. Florida. An industrial panel advises the program.

The purpose of this effort is to combine existing data with new experimental determinations and modeling formalisms to develop data-banked information and models which provide for improved design and evaluation of metallic and composite refractory systems. In particular, niobium alloys and the Nb-Al-O system are to be treated initially, with data on mechanisms of oxidation, species transport, thermochemical stabilities, activities, vapor phase interactions, solubilities, and diffusivities being considered important to the development of reliable models of coating/substrate/gas interactions. In the initial stage, most of the work has concentrated on the necessary literature review work and the development of a tentative ternary phase diagram to use as an experimental guide both for NIST and for the Univ. Florida and CEBELCOR groups.

In the Nb-Al-O system intermediate condensed-phase compounds exist: the binary compounds Nb_3Al, Nb_2Al, $NbAl_3$, NbO, NbO_2 and Nb_2O_5 and, along the quasibinary Al_2O_3-Nb_2O_5 section, four ternary compounds $49Nb_2O_5 \bullet Al_2O_3$, $25Nb_2O_5 \bullet Al_2O_3$, $9Nb_2O_5 \bullet Al_2O_3$ and $Nb_2O_5 \bullet Al_2O_3$, with the latter two having significant ranges of homogeneity. For the calculation of this system, the thermodynamic quantities for some phases are unknown or not enough data are available to determine them, simplifications and assumptions have been made in the current calculation.

Analysis of the literature has indicated the need for new experimental thermochemistry work on the Nb-Al system. For this work, tungsten Knudsen cells with inert liners, e.g., alumina, lucalox(r), and other inert refractories are being developed. Simple vacuum vaporization of Nb_3Al and $NbAl_3$ are planned in parallel with the data assessment efforts. Reactive studies between oxygen and these alloys will also be undertaken.

Measurements currently include the determination of vaporization characteristics of NbO_2 and Nb_2O_5-Al_2O_3 compounds. Measurements of the rate of evaporation of Al from $NbAl_2$ have also been made. Analysis of past vaporization data of NbO_2 and NbO shows that the heats of formation of these two key species are not well established.

Stored Chemical Energy Systems

E. R. Plante, D. W. Bonnell, L. P. Cook*

* Ceramics Division

The object of this work has been to make mass spectrometric observations of the reaction products of potential oxidants such as $ClO_3F(g)$ or $F_2(g)$ with reactive metals or alloys (Li, Mg, Al). The reaction of ClO_3F with Li-Al or Li-Al-Mg alloy was observed to form gaseous Li and Al mixed halides. Under laboratory steady state conditions the Cl_2 and O_2 gas pressures are orders of magnitude higher than the F_2 gas pressure indicating preferential fluorination of the alloys. However, there is no evidence of reaction of oxygen with the alloy under low pressure (10^{-5} atm) laboratory experimental conditions. Under higher pressure conditions (0.3 atm) reaction with oxygen takes place erratically as evidenced by large fluctuations in the oxygen pressure. Aluminum oxide reacts with $F_2(g)$ to form $AlF_3(g)$ and $O_2(g)$ while the addition of Al metal produces mainly AlF(g). Some evidence of kinetic inhibition of the reactions by formation of oxide or fluoride protective layers was observed.

Organic fluoride oxidizers under current study have the advantage of relative safety, high oxidant density, and high enthalpy production to the propulsion system. More recent work has been directed toward studying the reactions of liquid organic perfluoro compounds such as perfluoro-tetrahydrofuran and perfluoro-tributylamine which has been shown to react with Al to form AlF(g) and CO(g) or N_2(g) and a residue of unreacted carbon. However, the formation of permanent gases as a reaction product is considered to have a technically undersireable effect on the proposed propulsion system. Therefore, future exploratory work will involve reactions of the alloys with compounds such as perfluoryl-sulfurhexafluorides with the alloys.

Phase Diagram Graphics, Evaluation, and Modeling
P. K. Schenck, J. W. Hastie

Computer software has been developed to handle complex binary and ternary diagrams using stand alone desk top computers in support of the Phase Diagrams for Ceramists Data Center. The publication-ready diagrams for the recently completed Vol. 7 of "Phase Diagrams for Ceramists" were generated with this system. The software has been modified so that the individual work stations in the Data Center can access any diagram from a central mass storage system via the shared resource management system (local network). All digitized diagrams were transferred and are available from the central mass storage. Software was also developed to provide PC-access to the ceramics phase diagram graphics database. PC-users will have the ability to retrieve data from the screen display in a choice of user units (eg. °C, °F, or K). Mixture compositions in binary phase fields can be determined interactively by use of the lever rule and the results displayed on the PC-monitor screen. The PC based program and the graphics data base for Vol. 6 have been distributed for beta testing to the technical representatives of the more than twenty industrial sponsors of the joint NIST/ACerS phase equilibria program.

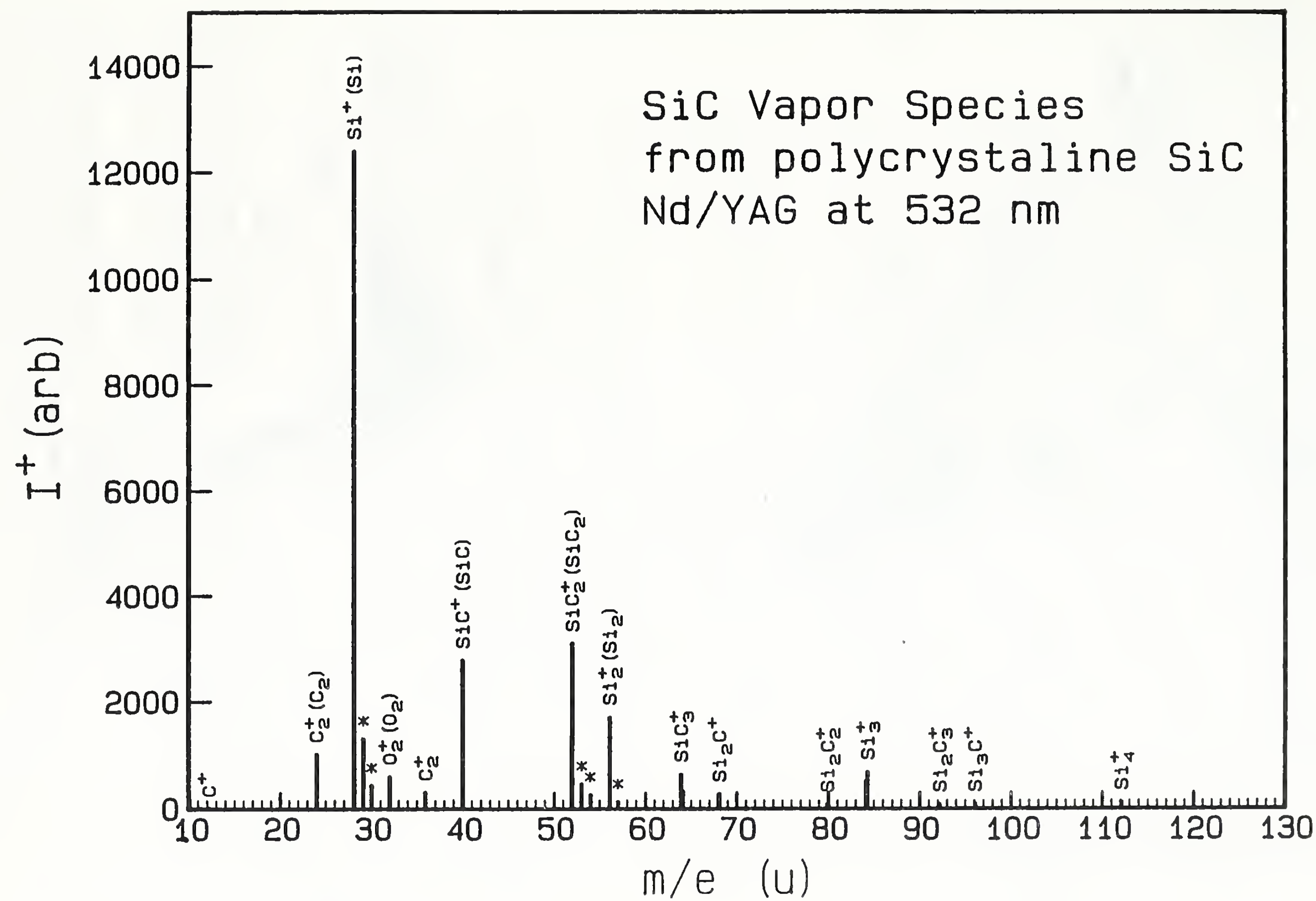


Figure 1. Mass Spectrum of vapor species above laser-vaporized polycrystalline SiC, constructed from time-resolved mass spectral data.

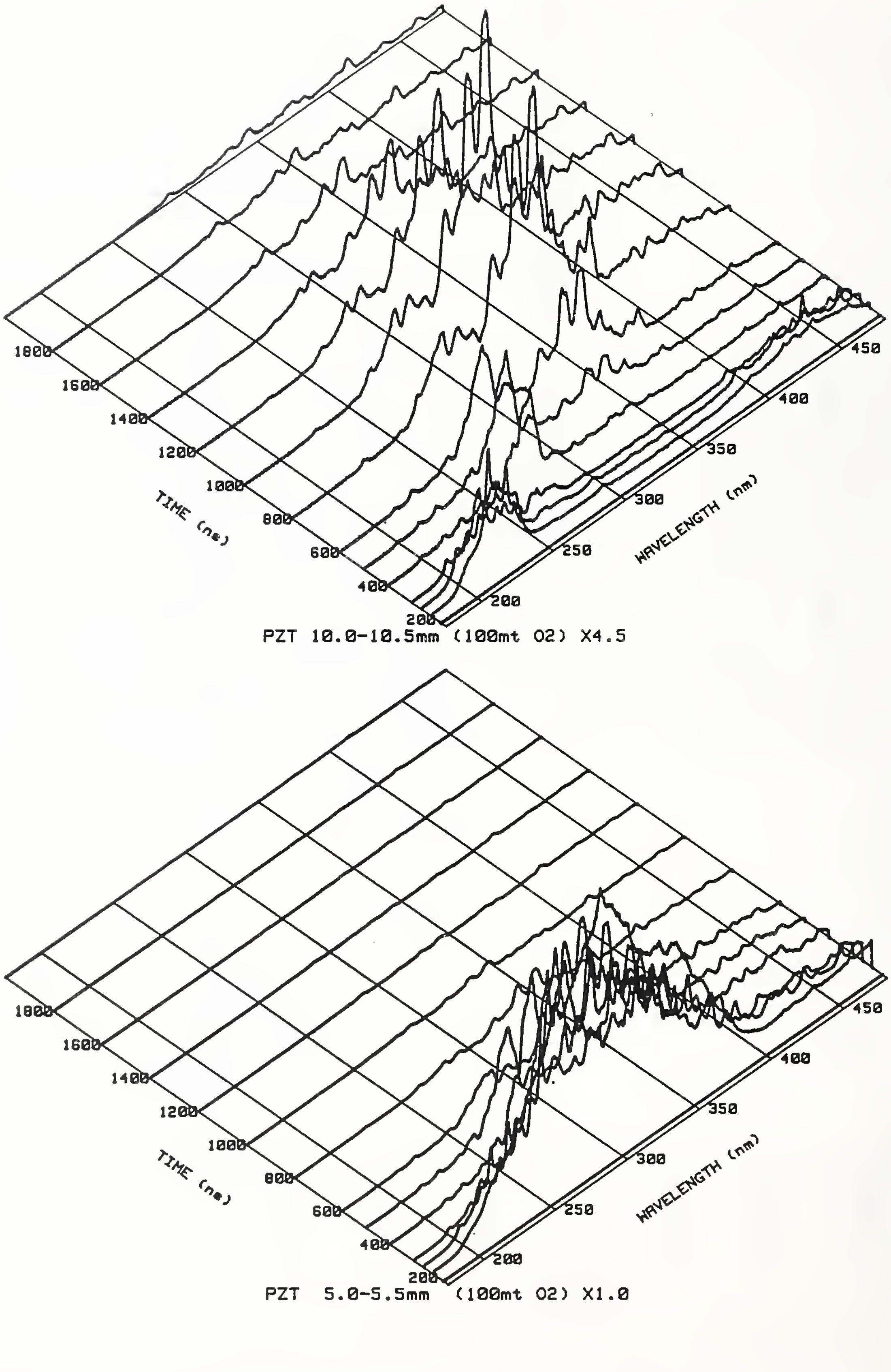


Figure 2. Time-resolved optical emission spectra from laser generated plume 5 and 10 mm above PZT target.

MECHANICAL PROPERTIES OF METALS John H. Smith

The mission of the Mechanical Properties of Metals Group is to: (1) characterize the mechanical and physical properties of metals. (2) develop improved test methods for characterizing the mechanical properties of metals (3) evaluate the structural integrity of metallic structures and components and (4) characterize the microstructure and composition of metals and alloys. The activities of this group are consist of (1) research on the mechanical properties of metals (2) development of new test and calibration methods (3) structural integrity analysis and (4) microstructural characterization of metallic materials.

FY 89 Significant Accomplishments

- A major investigation was completed for the Federal Railway Administration to determine the mechanical properties and fracture properties of new steels used in railroad tank cars. The results of this investigation will permit accurate determination of critical flaw sizes in tank cars to be determined.

- An intercomparison of Rockwell C hardness calibration test blocks has been completed and shows significant variability in the hardness values assigned by the block manufacturers.

- An assessment of failures in electric resistant welded (ERW) pipelines was completed and recommendations were made for implementing failure prevention and damage control procedures for pipelines in critical locations.

- A backscattered electron diffraction system was developed that increases the resolution of diffraction patterns to one micron.

- A multilayer deposition system was developed to deposit thin film materials by vapor deposition. Multilayer microstructures with layer thickness from a few monolayers to a few micrometers can be produced.

Mechanical Properties of Metals Research
G. E. Hicho, J. H. Smith, T. R. Shives, and B. A. Fields*

* NIST Guest Scientist

The mechanical properties of metal research emphasizes the evaluation of newly developed materials. Increasingly the emphasis is on the evaluation of advanced metallic materials. Nearly all of this research is funded by other government agencies and generally involves close collaboration with the producers and or users of the materials.

Characterization of Railroad Tank Cars Steels - Extensive research has been conducted to evaluate the mechanical properties, fracture resistance and crack arrest properties of presently used and new steels for the construction of railroad tank cars used to transport hazardous materials. Presently used, as-rolled steels are subject to fracture under some operating conditions when the tank cars are involved in accidents. However, newly developed microalloyed steels and new heat treatments of present steels, such as normalizing and stress relieving reduce the possibility of fracture. The effect of composition, temperature, and strain rate on the mechanical properties and fracture resistance of the steels have been determined. The dynamic mechanical properties of AAR M128 and ASTM A212B steels were determined using Charpy impact tests and dynamic tear tests. The transition temperature, crack initiation energy, crack propagation energy, and total fracture energy were determined. A comparison of the transition temperature of normalized TC-128 steel and a newly developed control rolled steel (A-808) is shown in Figure 1. For as rolled steels, which are used in currently existing tank cars, the ductile to brittle transition temperature was found to be aproximately at ambient temperature.

Elevated Temperature Deformation of Structural Steel - In a cooperative project with the American Iron and Steel Institute, analytical equations were developed to calculate the elastic, plastic, creep, and total strain in ASTM A-36 steel at elevated temperature. These equations have been used to construct deformation maps to determine the mode of failure at any combination of temperature and stress. These equations are useful to predict the performance of structures at elevated temperatures such as occur during fires.

Development of New Test and Calibration Methods

T. R. Shives, G. E. Hicho, J. H. Smith

The research on test methods and calibration methods is largely supported by NIST funds and is directed at improved methods for characterizing the properties of both conventional materials and of new, advanced materials.

Macrohardness Test Block Standards - Significant differences in measured values of certified Rockwell hardness test blocks from different manufacturers have been observed. The National Institute of Standards and Technology (NIST) was requested by ASTM Subcommittee E28.06 on Indentation Hardness to perform an intercomparison study of test blocks from all manufacturers who market blocks in the United States. Test blocks from Rockwell C, 30N, B, and 30T scales were obtained for this intercomparison. A Rockwell tester from the Page-Wilson Company and a superficial Rockwell tester from Clark Instruments Inc. were consigned to NIST through the ASTM subcommittee for this intercomparison studies.

The initial intercomparison has been done for Rockwell C scale hardness test blocks on the Rockwell C scale. A total of 21 HRC test blocks, 7 at each of three hardness levels were tested at NIST using the Paige-Wilson commercial hardness tester. The load applied during testing was measured with a load cell that had been calibrated by the NIST Force Calibration Service. An example of the variation in hardness of Rockwell C scale test blocks from various manufacturers can be seen in Figure 2. In these bar graphs, each point represents the difference between the average of the five measurements made at NIST and the value for the block as stated by the manufacturer. In addition, nine of the test blocks at hardness levels of HRC 25, 45, and 63 were tested on a dead weight standardizing hardness test machine at the Insituto Di Metrologia "G. Colonnetti" (IMGC) in Turin, Italy. The results of these tests are also compared with the results of tests conducted at NIST on the Paige-Wilson commercial hardness machine in Figure 2.

Previous research has identified variations in the shape of the Rockwell Brale indentor used in the C scale tests as a significant source of the variation in the measured hardness values. Therefore, a study of indenters and indenter specifications was also undertaken. The indentors were characterized by interferometry, with a stylus measuring machine and with an optical comparator. Preliminary results of the Rockwell Brale indentors evaluation have shown substantial variation from the indentor geometry required by the ASTM specification. In addition, several indentors were measured at the IMGC labortory and one indenter that satisfies the International Standards Organization (ISO) geometrical requirements, which are more complete than the ASTM standards, was selected as the standard indentor for the NIST intercomparison study.

<u>Certification of Ferrite in Weld Standard</u> - A ferrite in weld Standard Reference Materials (SRM) has been prepared and is ready for certification. This material is composed of 10% 430 stainless steel (ferritic) blended with 90% 310 stainless steel (austenitic). Powder metallurgical techniques are used to prepare these materials. The materiasl are certified by determining the percent ferrite in each specimen image and x-ray fluorescence analyses. The certified standard reference materials will be available calibrate x-ray diffraction equipment that is used in the determination of ferrite in welds.

<u>Structural Integrity Testing</u>
J. H. Smith, G. E. Hicho and T. R. Shives

This testing is done at the request of and funded by other government agencies or Congressional Committees. These investigations apply our expertise in the characterization of metals to determine the cause of failure and to evaluate the structural integrity of metallic components and structures. The results of these investigations lead to improvement in the selection and use of materials and to improvement in design and safety codes and standards.

<u>Assessment of ERW Pipelines</u> - As a result of the failure of a large oil pipeline in Missouri, an extensive review of failure incidence statistics for electric resistance welded (ERW) pipelines that are used for long distance transmission of oil and natural gas was carried out to determine if older ERW pipelines are particularly susceptible to failure. It was concluded that the relatively small number of failures in ERW pipelines does not warrant special tests and standards except in critical locations. For critical locations, it is recommended that failure prevention and damage control practices be implemented.

<u>Safety Design Criteria for High Strength Steel Cylinders</u> - Research was carried out under sponsorship of the Department of Transportation Office of

Hazardous Materials to establish a sound, technical basis for safety standards governing the safe design, manufacture, and use of seamless pressure vessels (cylinders) for the storage and transportation of compressed gases. Present research is aimed at developing a design criterion for the construction of high strength steel cylinders to prevent failure by fracture or by stress corrosion. Collaborative test programs with Taylor Wharton Inc., Norris Cylinder Inc., and T.I. Chesterfield Ltd. are being conducted to establish a fracture criterion that accurately predicts the fracture behavior of thin walled, ductile steel cylinders. This test program is being coordinated by the International Standards Orgranization (ISO) cimmittee on cyklinder design (TC-58). Results of this investigation have led to a preliminary design specification for higher strength steel cylinders.

Structural Characterization
A. J. Shapiro and L.C. Smith

The structural characterization activity provides the management of the optical microscopy, electron microscopy, and X-ray facilities for the Metallurgy Division. The facilities consist of a full range of optical metallographic equipement, two scanning electron microscopes (JEOL 840-1 and ETEC Autoscan) with X-ray microanalyzers (Tracor Northern TN5500-5600 and TN2000 and WDS spectrometer) and image analysis capabilities, OMNICON image analysis system and 300 kev transmission electron microscope Philips 430.

Most of the activity is conducted in close collaboration with the other research projects in the Division such as metals processing, magnetic materials, metal matrix composites, and other advanced materials. The emphasis has increasingly shifted to the microstructural characterization of advanced materials and to developing improved procedures and techniques to characterize the structure of these materials.

Back Scattered Electron Diffraction System - A backscattered electron diffraction system has been developed that uses image analysis capabilities of Tracor Northern TN5500. This system greatly improves the spatial resolution compared to the Electron Channeling Pattern Mode of the JEOL 840 and also improves signal to noise ratio compared to the existing Backscattered Electron Diffraction Systems. This new system increases the resolution of diffraction patterns to one micron.

Multilayered Deposition System - A multilayer deposition system based on BIO-RAD turbo coater has been developed to produce thin film materials by vapor deposition. This system has three evaporation sources and a spin table. This permits simultaneous evaporation of three different metals onto a rotated substrate. Multilayer microstructures with layer thickness from a few monolayers to a few micrometers can be produced.

Structural Characterization of Iron/Silica Gel Nanocomposites - Composite materials made from nanometer sized particles of metals and nonmetals are referred to as granular metals. When the metallic constituent has a strong magnetic moment, the granular metal composite is found to have significantly improved magnetic properties compared with the homogeneous form of the same metals. Homogeneous gelled composites of iron and silica continuing 5-30 wt% Fe, prepared by low temperature polymerization of aqua solution of ferric nitrate, tetraethoxysilate, and ethanol (with an HF catalyst), have been investigated using SEM and TEM techniques. These observations show that this matrix is characterized by presence of many interconnected pores and that the size of this pores is related to the particle size of the Fe-containing regions. An extensive electron microscope study was undertaken to evaluate the microstructure of these granular metals and establish relationships between the microstructure and the unique magnetic properties of these materials.

Microstructural Characterization of High Temperature Superconductor Materials - High temperature thin film superconductors of $YBa_2Cu_3O_7$ have been fabricated by sputter deposition. The effect of process variables such as substrate temperature, target to substrate angle and the partial pressure of oxygen in the sputtering atmosphere on the final composition of the thin film superconductor were determined by X-ray microanalysis. X-ray compositional analysis reveal the effect of oxygen partial pressure, substrate temperature, and the geometry of the source/target relationship on the compositional and microstructural phases of the superconducting films. X-ray chemical analysis and image analysis have also been used to identify phases and to measure porosity of high temperature superconducting glass-ceramics in the Bi-Sr-Ca-Cu-O and Bi-Pb-Sr-Ca-Cu-O systems.

Structural Characterization of Interfaces in Nickel-Graphite Metal Matrix Composites - Nickel/graphite-fiber metal matrix composites were prepared by electrodepositing nickel onto tows of 3000 polyacrlyonitrile graphite fibers. Scanning electron microscopic examination of these composites shows that when these composites are annealed in vacuum at a temperature of less than 800° C, the graphite diffuses into the nickel while at annealing temperatures greater than 800o C the nickel diffuses into the graphite. When a layer of electrodeposited cobalt and tungsten (CoW) is deposited on the graphite fiber the interdiffusion of graphite and nickel is suppressed.

Phase Characterization in Advanced Materials - Quantitative X-ray chemical analysis have been used to identify the microconstituent phases and to determine the chemical composition of the phases in several advanced research materials. Analysis has been done on Ti-Al-Cu quasicrystals, Nb-Al-Ti intermetallic compounds, and rapidly solidified Ag-Cu alloys.

Properties of Microelectronic Solder Joint Materials - Solder joints used in microelectronic devices are being studied to identify the causes of premature failure in solder materials. Samples of Pb-Sn-Cu solder joint materials have been prepared and the composition, grain size, microstructural constituent phases have been identified by using scanning electron microscopy and X-ray analysis. In particular, intermetallic phases between the components of the solder and the materials being joined have been identified an fully characterized.

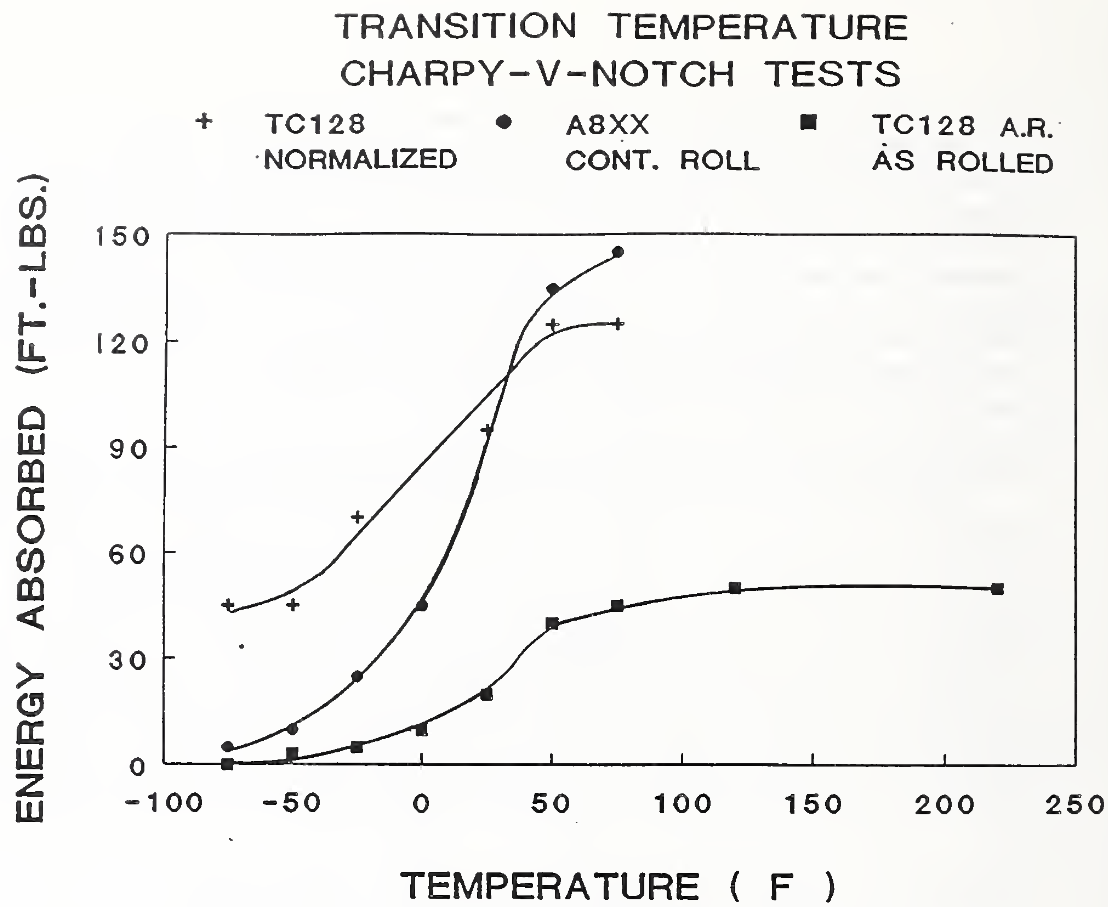


Figure 1. Transition temperature for railway tank car steels

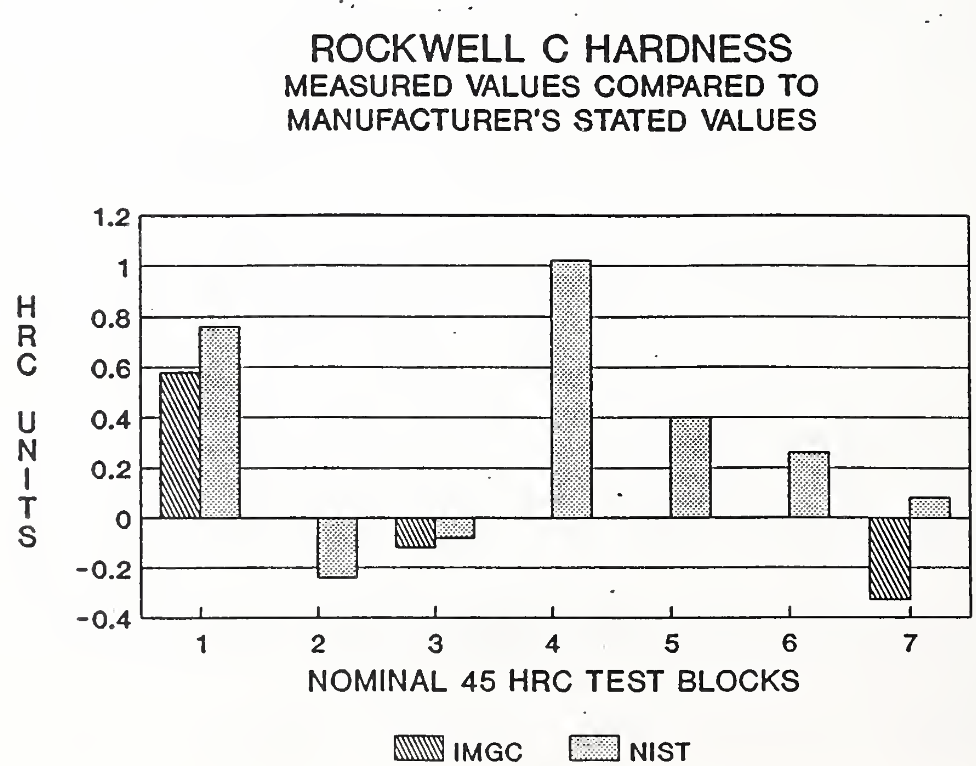


Figure 2. Variation in measured hardness values for Rockwell C tests

PUBLICATIONS

Metallurgical Processing Group

Bendersky, L. A.; McAlister, A. J.; Biancaniello, F. S.; "Phase Transformation During Annealing of Rapidly Solidified Al-based Al-Fe-Si Alloys." Met. Trans. 19, 2893, (1988).

Bendersky, L. A.; Boettinger, W. J.; Burton, B. P.; Biancaniello, F. S.; Shoemaker, C. B.; "Investigation of ω-related phases in alloys of composition Ti_4Al_3Nb." Metallurgical Transactions, in press, (1989).

Bendersky, L. A.; Cahn, J. W.; and Gratias, D; "A Crystalline Aggregates with Icosahedral Symmetry, and its Implication for the Crystallography of Twinning and Grain Boundaries." Phil. Mag. A (1989); in press.

Bendersky, L. A.; Boettinger, W. J.; "Investigation of B2 and Related Phases in the Ti-Al-Nb Ternary System." Mat Res. Soc. Symp. Proc.; 133; 45 (1989).

Bendersky, L. A.; Boettinger, W. J.; "Omega-related Phases in a Ti-Al-Nb Alloy." Proceedings of the 47th Annual Mtg of the Electron Microscope Society of America, G.W. Bailey, ed.; San Francisco Press, San Francisco, CA; 324; (1989) in press.

Biancaniello, F. S.; Conway, J. J.; Espina, P. I.; Mattingly, G. E.; and Ridder, S. D.; "Particle Size Measurement of Inert Gas Atomized Powder." Mat. Sci. Eng., in press, (1989).

Biancaniello, F. S.; Espina, P. I.; Mattingly, G. E.; and Ridder, S. D.; "A Flow Visualization Study of Supersonic Inert Gas Metal Atomization." Mat. Sci. Eng., A119, 159, (1989).

Biancaniello, F. S.; Presser, C.; and Ridder, S. D.; "Real-Time Particle Size Analysis During Inert Gas Atomization." Mat. Sci. Eng., in press, (1989).

Blendell, J. E.; Handwerker, C. A.; Shen, C. and Dang, N.-D.; "Diffusion-Induced Interface Migration in Ceramics." Ceramic Microstructures '86, ed. J. Pask and A. G. Evans, Plenum Press, (1988), 541-548.

Boettinger, W. J.; Aziz, M. J.; "A Theory for the Trapping of Solute & Disorder in Intermetallic Compounds by Rapid Solidification." Acta. Met., in press, (1989).

Boettinger, W. J.; "The Microstructure of Rapidly Solidified Alloys;" U.S.-Indo Workshop on Metastable Materials, Goa India; in press, (1989).

Burton, B. P.; "Application of the ϵ-G Approach to Binary Rhombohedral Carbonate Systems." Proceedings of CALPHAD-XVII, (1988).

Burton, B. P.; Davidson, P. M.; "Multicritical Phase Relations in Minerals." Advances in Physical Geochemistry, S. Ghose, ed., Springer-Verlag; 7; 60; (1988).

Burton, B. P. and Ross, C. R.; "A New Phase Diagram Topology with a Tricritical Point: Implications for the Omphacitic Pyroxenes and Plagioclase Feldspars." American Mineralogists, in press, (1989).

Clough, R. B., Biancaniello, F. S., Wadley, H., Kattner, U.; "Fiber and Interface Fracture in Single Crystal Aluminum-SiC Fiber Composites." Met. Trans. A., in press, (1989).

Dobbyn, R. C.; Farris, J.; Harlow, D. G.; Delph, T. J.; Fields, R. J.; "In-Situ Imaging of Creep Cavities of Synchrotron Microradiography." Scripta Metallurgica, 23, 621 (1989).

Fields, B. A.; Fields, R. J.; "Elevated Temperature Deformation of Structural Steel." NISTIR-88-3899, (1988).

Fields, R. J.; deWit, R.; "Plastic Zone Formation Around An Arresting Crack." Int'l. Journal of Fracture, in press, (1989).

Fields, R. J.; Low III, S. R.; Harne, D. E.; "Static and Dynamic Tests on Electrical Conductor Cables Specified for Airport Landing Structures." NISTIR 88-3884, (1988).

Fields, R. J.; Pugh, E. N.; Read, D. T.; Smith, J. H.; "An Assessment of the Performance and Reliability of Older ERW Pipelines." NISTIR-89-4136, (1989).

Finkelshtein, Y.; Wiederhorn, S. M.; Hockey, B. J.; Handwerker, C. A.; and Blendell, J. E.; "Migration of Sapphire Interfaces with Vitreous Bonded Aluminum Oxide." Ceramic Transactions, ed.; Handwerker, C. A.; Blendell, J. E.; and Kaysser, W. A., American Ceramic Society, in press, (1989).

Gayle, F. W.; Heubaum, F. H.; and Pickens, J. R.; "Natural Aging and Reversion Behavior of Al-Cu-Li-Ag-Mg Alloy Weldalite⊧ 049." Aluminum-Lithium Alloys (Proceedings of the Fifth International Aluminum-Lithium Conference, T. H. Sanders and E.A. Starke, eds., MCE Publications, Ltd, Birmingham, U.K., 701 (1989).

Handwerker, C. A.; Morris, P. A.; and Coble, R. L.; "Effects of Chemical Inhomogeneities on Grain Growth and Microstructure in Al_2O_3." J. Amer. Ceram. Soc. 72[1] (1989).

Handwerker, C. A.; Vaudin, M. D.; and Blendell, J. E.; "Equilibrium Crystal Shapes and Surface Phase Diagrams at Surfaces in Ceramics." Proceedings of Conference on Interface Science and Engineering, Lake Placid, NY, July 1987, J. de Physique Colloque, October 1988, C5-367- 374.

Handwerker, C. A.; "Diffusion-Induced Grain Boundary Migration in Thin Films." in: Diffusion Phenomena in Thin Films and Microelectronic Materials, ed. D. Gupta and Paul Ho, Noyes Publishing Co., (1988), 245-322.

Handwerker, C. A.; "Diffusion-Induced Grain Boundary Migration." under the major title Grain Boundaries, McGraw-Hill Yearbook of Science and Technology; (1989).

Handwerker, C.A.; Cahn, J.W; and Manning, J.R.; "Thermodynamics and Kinetics of Reactions at Interfaces in Composites." Materials Science and Engineering, in press, (1989).

Handwerker, C. A.; Cannon, R. M.; and Coble, R.L.; "Dihedral Angles in MgO and Al_2O_3: Distributions from Surface Thermal Grooves." J. Amer. Ceram. Soc., in press, (1989).

Handwerker, C. A.; Vaudin, M. D.; Kattner, U. R.; and Lee, D.-J.; "Interface Reactions and Phase Stability in the Al-SiC System." Acta Metallurgica/ Scripta Metallurgica Conference Series, ed. M. Ruhle, Pergamon Press, in press, (1989).

Handwerker, C. A.; Dynys, J. M.; Cannon, R. M.; and Coble, R. L.; "Metal Reference Line Technique for Obtaining Dihedral Angles from Surface Thermal Grooves." J. Amer. Ceram. Soc., in press, (1989).

Handwerker, C. A.; Blendell, J. E. and Kaysser W. A. (eds.); Sintering of Advanced Ceramics, in series Ceramic Transactions; American Ceramic Society, Westerville, OH, in press, (1989).

Hardy, S. C.; Voorhees, P. W.; "Ostwald Ripening in a System with a High Volume Fraction of Coarsening Phase." Met. Trans. A, 19A, 2713 (1988).

Kaiser, D. L.; Gayle, F. W.; Roth, R. S. and Swartzendruber, L.J.; "Thermochemical Detwinning of Superconducting $YBa_2Cu_3O_{7-x}$ Single Crystals." J. Materials Research, 4, 745, (1989).

Ledbetter, H. M.; Fields, R. J.; Reno, R. C.; "Texture in Recrystallized Copper Rod: An Ultrasonic-Velocity, Neutron Diffraction Study." Proceedings of the Eighth International Conference on Textures of Materials; Santa Fe, in press.

Lee, D. J.; Vaudin, M. D.; Handwerker, C. A.; Kattner, U. R.; "Phase Stability and Interface Reactions in the Al-SiC System." Mat. Res. Soc. Symp. Proc., High-Temperature, High-Performance Composites, eds. Lemkey, F. D.; Fishman, S. G.; Evans, A. G.; and Strife, J. R.; 120; 357; (1988).

Liu, T.-S.; Fields, R. J.; Fariborz, S. J.; Harlow, D. G.; Delph, T. J.; "Experimental Observations and Analysis of Creep Cavitation in AISI 304 Type 304 Stainless Steel." Acta Met., in press, (1989).

Low, III S. R.; Harne, D. E. and Fields, R. J.; "Dynamic Strength Tests on Electrical Cables Specified for Aircraft Landing Structures." Advanced Technology in Failure Prevention, Proceedings of the 43rd Meeting of the Mechanical Failures Prevention Group, October 3-6, 1988, (Shives, T. R., ed.), in press, (1989).

Manning, J.R.; "Atomic Diffusion Equations and Kinetic Forces." Diffusion Analysis and Applications, A.D. Romig, Jr. and M.A. Dayananda, eds., The Minerals, Metals and Materials Society, Warrendale, PA, in press, (1989).

Naus, D. J.; Bass, B. R.; Keeney-Walker, J.; Fields, R. J., deWit, d.; Low III, S. R.; "HSST Wide-Plate Test Results and Analysis." Nuclear Engineering and Design, in press, (1989).

Naus, D. J.; Keeney-Walker, J.; Bass, B. R.; Fields, R. J.; deWit, R., Low III, S. R.; "High-Temperature Crack-Arrest Behavior of Prototypical and Degraded (Simulated) Reactor Pressure Vessel Steels." International Journal of Pressure Vessels and Piping, in press, (1989).

Naus, D. J.; Keeney-Walker, J.; bass, B. R.; Bolt, S. E.; Fields, R. J.; deWit, R.; and Low III, S. R.; "High-Temperature Crack-Arrest Behavior in 152-mm-Thick SEN Wide Plates of Quenched and Tempered A533 Grade B Class 1 Steel." Nuclear Regulatory Commission Report NUREG/CR-5330, (1989).

Rhee, W.-H.; Handwerker, C. A.; Yoon, D.; "The Coherency Strain Induced Instability of Solid-Liquid Interfaces in the Mo-Ni System." Mat. Res. Soc. Symp. Proc.; Interfacial Structure, Properties, Design; ed. Yoo, M. H.; Clark, W. A. T.; and Briant, C. L., 122, 205, (1988).

Roth, R.S.; Rawn, C.J.; and Bendersky, L. A.; "Crystal Chemistry of the Compound $Sr_2Bi_2CuO_6$." J. Mater. Res., in press, (1989).

Roth, R. S.; Rawn, C. J.; Burton, B. P.; Beech, F.; "Phase relations and crystal chemistry in portions of the system SrO-CaO-CuO, Part-II - The system SrO-½Bi_2O_3-CuO." Journal of Research, in press, (1989).

Roth, R. S.; Rawn, C. J.; Ritter, J. J. and Burton, B. P.; "Phase relations of the system SrO-CaO-CuO." Journal of the American Ceramic Society, in press, (1989).

Shives, T.R.; Low, III, S.R.; "Tensile Tests of Type 305 Stainless Steel Mine Sweeping Wire Rope." NISTIR-89, in press, (1989).

Shoemaker, C. B.; Shoemaker, D. P.; and Bendersky, L. A.; "Crystal Structure of $\omega(Ti_3Al_{2.25}Nb_{0.75})$." Acta Cryst., in press (1989).

Vaudin, M. D.; Handwerker, C A. and Blendell, J. E.; "Electron Microscopy Studies of Diffusion Induced Grain Boundary Migration in Ceramics." Proceedings of Conference on Interface Science and Engineering, Lake Placid, NY, July 1987, J. de Physique Colloque, October 1988, C5-687-692.

Wadley, H. N. G., Biancaniello, F. S. and Cough, R. B.; "Micromechnical Studies of Model Metal Matrix Composites." Materials Res. Symp. Proc., 120, (1988), Materials Research Soc.

Advanced Sensing

Clough, R. B.; Wadley, H. N. G.; and Biancaniello, F. S.; "Micromechanical Studies of Model Metal Matrix Composites", published in Materials Res. Symp. Proc. Vol. 120, 1988, Materials Research So.

Clough, R. B; Biancaniello, F. S.; Wadley, H. and Kattner, U; "Fiber and Interface Fracture in Single Crystal Aluminum-SiC Fiber Composites." submitted to WERB, 8/89--intended for Met. Trans. A.

Johnson, W.; Kahn, A. H.; Chiang, C. K.; and Wadley, H. N. G.; "Eddy Current Sensing of Oxygen Content in High-T_c superconductors." Review of Progress in Quantitative Nondestructive Evaluation, Vol. 9, ed. D.O. Thompson and D. E. Chimenti; in press.

Norton, S. J.; Iterative seismic inversion, Geophysical Journal, 94, 457-468 (1988).

Norton, S. J.; "Tomographic reconstruction of 2-D vector fields; application to flow imaging." Geophysical Journal, 97, 161-168 (1988).

Norton, S. J.; Kahn, A. H.; and Mester, M. L.; "Reconstructing Electrical Conductivity Profiles from Variable Frequency Eddy Current Measurements." Submitted to Research and Nondestructive Evaluation.

Simmons, J. A.; Drescher-Krasicka, E.; Wadley, H. N. G.; Rosen, M.; and Hsieh, T. M. "Ultrasonic Methods for Characterizing the Interface in Composites", Review of Progress in QNDE, Vol. 7, 1988.

Wadley, H. N. G.; Simmons, J. A.; and Drescher-Krasicka, E.; "Ultrasonic Propagation at Cylindrical Metal-Ceramic Interfaces in Composites." MRS Symposium High Temperature Composites, 1988.

Corrosion Group

Ahn, T., Bertocci, U., O'Connell, W. J., Drach, R. S.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Waste Package Performance Assessment Deterministic System Model Program Scope and Specification", UCRL-53761.

Ahn, T., Bertocci, U., Wolery, T. J.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Calculation of Chemical Equilibrium Between Aqueous Solution and Minerals: The EQ3/6 Software Package", submitted to Werb September 1989, UCRL-52658.

Ahn, T., Fraker, A. C.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Prediction of Glass Durability as a Function of Glass Composition and Test Conditions: Thermodynamics and Kinetics", a paper proposed for Presentation at the Conference on Advances in the Fusion of Glass, Alfred, NY, June 14-17, 1988, DP-MS-87-157, submitted to Werb September 1989.

Ahn, T., Interrante, C. G., Mendel, J.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Waste Leaching Mechanisms Program", Chapter 6, "Phenomenological Models of Nuclear Waste Glass Leaching" PNL-5157.

Bennision, S. J., Kelly, J. F., Stoudt, M. R.; "In Situ Observation of Grain-Localized Crack Bridging in Alumina", Bulletin of the American Ceramic Society April 1989.

Bertocci, U.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Copper Corrosion in Irradiated Environments. The Influence of H_2O_2 on the Electrochemistry of Copper Dissolution in HCl Electrolyte", UCRL-95961, December 1986.

Bertocci, U.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage, "Evaluation and Compilation of DOE Waste Package Test Data", Draft Biannual Report Covering the Period February 1988 to July 1988, NIST/NRC Report.

Bertocci, U.; NBS Review of Technical Report on the High Level Waste Package for Nuclear Waste Storage: Review of: "Gamma Radiation Effects on Corrosion: I - Electrochemical Mechanisms for the Aqueous Corrosion Processes of Austenitic Stainless Steels," UCRL-92311, February 1986.

Bertocci, U.; "Electrochemical Measurements of Corrosion Processes", Proceedings of the Meeting of the Italian Automobile Technical Association, Milan, Italy, Feb 27-38, 1989, p. 1.

Bertocci, U.; "Electrochemical Noise Analysis and its Applications to Corrosion, Corrosion/89, paper No. 24., NACE, Houston, TX, 1989.

Bertocci, U., Glass, R. S., Van Konynenburg, R. A., Overturf, G. D.l; "Corrosion Processes of Austenitic Stainless Steels and Copper-Base Materials in Gamma-Irradiation Aqueous Environments", UCRL-92941.

Bertocci, U., Madsen, P. V., Fink, J. L., Ricker, R. E.; "Passivity and Passivity Breakdown on Nickel Aluminide," to be published by Pergamon Press in the Proceedings of the International Passivity Conference, Sapporo, Japan, 1989.

Bertocci, U., Pugh, E. N.; "Modeling of Electrochemical Processes During Transgranular Stress Corrosion Cracking of Copper-Base Alloys", Proceedings of the 10th International Congress on Metallic Corrosion 1987, Vol 5., p. 219, Oxford and I. B. H Pub. New Delhi, India (1989).

Bertocci, U., Pugh, E. N., Ricker, R. E.; "Environment-Induced Cracking of Copper Alloys," will be published by the National Association of Corrosion Engineers in the proceedings of the International Conference on Environment-Induced Cracking of Metals held in Kohler, Wisc, October 2-7, 1988.

Escalante, E.; "Concepts of Underground Corrosion; ASTM STP 1013, Effects of Soil Characteristics On Corrosion, 1989." American Society for Testing Materials, Philadelphia, PA.

Escalante, E.; "Elimination of IR Error in Measurements of Corrosion in Concrete." ASTM STP 1056, Ohmic Electrolyte Resistance Measurement and Compensation, in press.

Escalante, E.; "Measuring the Corrosion of Metals in Soil", will be submitted to ASTM-Special Technical Publications, submitted to WERB September 1989.

Escalante, E.; "Measuring the Rate of Corrosion of Steel in Concrete." ASTM STP 1065, Corrosion Rates of Steel in Concrete, in press.

Escalante, E.; "The Corrosion and Protection of Selected Steel Piling in a Natural Seawater Environment-13 years", NACE Conference Proceedings.

Escalante, E.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Containment Barrier Metals for High-Level Waste Packages in a Tuff Repository," by E. W. Russell, et al., UCRL-53449, October 1983, submitted to Werb September 1989.

Escalante, E., Fink, J., and Hessler, G.J.; "Corrosion Evaluation Of Underground Telephone Cable Shielding Materials After Exposure For Seven Years". Materials Protection, in press.

Fraker, A. C.; "Corrosion of Zircaloy Spent Fuel Cladding in a Repository," milestone for laboratory testing project on the Corrosion Behavior of Zircaloy Nuclear Fuel Cladding: NISTIR 89-4114, August 7, 1989.

Fraker, A. C.; "Corrosion Principles for Implantology" in preparation, Clark's Clinical Dentistry (a textbook), due September, 1989.

Fraker, A. C.; "Mechanisms of Internal Corrosion of Spent Fuel Rods", Review paper prepared for the Nuclear Regulatory Commission, September, 1989.

Fraker, A. C.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage, Review of: "Impact of Phase Stability on the Corrosion Behavior of the Austenitic Candidate Materials for NNWSI" UCRL-97562, October, 1987, NUREG/CR 4735 Vol. 5.

Fraker, A. C.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear WAste Storage, Review of: "Progress Report on the Results of Testing Advanced Conceptual Design Metal Barrier Materials Under Relevant Environmental Conditions for a Tuff Repository," UCID-210444, December, 1987, NUREG/CR 4735 Vol. 5.

Fraker, A. C.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear WAste Storage, Review of: "The Influence of Copper on Zircaloy Spent Fuel Cladding Degradation Under Potential Tuff Repository Conditions," UCRL-15993, SANL-622024, Mar, 1987, NUREG/CR 4735 Vol. 5.

Fraker, A. C., Harris, J. S.; "Corrosion Behavior of Mild Steel in High pH Aqueous Media," NISTIR 89-4173, NTIS, September 1989.

Fraker, A. C., Harris, J. S.; "Corrosion Behavior of Zircaloy Nuclear Fuel Cladding", Report to the Nuclear Regulatory Commission, August, 1989 and submitted to the Materials Research Society, September, 1989.

Fraker, A. C., Shaw, H. J.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage, Review of: "Plan for Spent Fuel Waste Form Testing for NNWSI". UCID-21272, November 1987. Submitted to Werb September 1989.

Fraker, A. C., Sung, P., Van Orden A. C., Hahn, H.; "Corrosive Response of the Interface Tissue to Dental Implants" published in Implant Prosthodontics: Surgical and Prosthetic Techniques for Dental Implants, Year Book Medical Publishers, Littleton, MA, in press.

Interrante, C. G.; "Elements of Proof for SCC", submitted for presentation in Revelation Magazine, Advanced Revelation, Scientific Notation, and Nuclear Waste", August 1989.

Interrante, C. G., Bertocci, U., Escalante, E. Fraker, A. C, Ondik, H., Ricker, R.; "Evaluation and Compilation of DOE Waste Package Test Data: Biannual Report February 1988 - July 1988," NUREG/CR-4735, Vol. 5, November 1988.

Interrante, C. G., Bertocci, U., Escalante, E., Fraker, A. C., Ondik, H, Ricker, R.; "Evaluation and Compilation of DOE Waste Package Test Data: Biannual Report August 1988 - January 1989," NUREG/CR-4735, Vol. 6, in preparation.

Interrante, C., Escalante, E., Fraker, A. C.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. "Evaluation and Compilation of DOE Waste Package Test Data," Draft Biannual Report Covering the Period February 1988 to July 1988, NUREG/CR 4735 Vol. 5.

Interrante, C. G., Messina, C., Harrison, S. A.; "A Database for Reviews and Evaluations of High-Level Waste Documents", submitted for presentation at MRS-89.

Interrante, C. G., Ondik, H., Plante, E., Mendel, J.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Final Report of the Defense High-Level Waste Leaching Mechanisms Program", Chapter 3, Environmental Interactions", PNL-5157.

Ondik, H., Interrante, C. G., Wilson, C. N. and Shaw, H. F.; NIST Review of Technical Reports on the High Level Waste Package for Nuclear Waste Storage. Review of: "Experimental Study of the Dissolution of Spent Fuel at 85•C in Natural Ground Water", UCRL-94633.

Ricker, R. E.; "The Effect of Aqueous Environments on the Fracture Behavior of Ductile Nickel Aluminide", To be submitted to Scripta Metallurgica.

Ricker, R. E., Stoudt, M. R., Fink, J. L.; "Hydrogen Absorption from Aqueous Solutions and Embrittlement of Ductile Nickel Aluminide," will be published by The Metallurgical Society of AIME in the proceedings of the 4th International Conference on The Effect of Hydrogen on Material Behavior to be held in Jackson Hole, WY, Sept. 12-16, 1989.

Sung P., Fraker, A. C.; "Activation Energy for Repassivation of 316L Stainless Steel", to be submitted to Corrosion Journal.

Electrodeposition

Beauchamp, C.; Brown, H. G.; Lashmore, D. S.; "Comparison of the NIST and European Gold Coating Standards." NIST Interagency (in press).

Bennett, L. H.; Swartzendruber, L. J.; Lashmore, D. S.; Oberle, R. R.; "Magnetic Behavior of Compositionally-Modulated Ni/Cu Thin Films." Phys. Rev. B, 40, 7 (1989).

Lashmore, D. S.; "Surface Properties and Coatings of Materials." Journal of Technology, 24, 469 (1989).

Lashmore, D. S.; Oberle, R.; Dariel M.; Bennett, L. H.; Swartzendruber, L.; "Electrodeposited Metallic Superlattices." Mat. Res. Soc. Proc.; Vol. 132, (1989).

Markrzak, C. F.; S. Satija; Neumann, D.; Rush, J.; Lashmore, D.; Johnson C.; Bradshaw, J.; Passell, L.; DiNardo R.; :Determination of Hydrogen Denisty Profiles in Thin Films and Multilayers by Neutron Reflection." 1989 MRS Fall Meeting, Boston, MA Symposium Journal: Neutron Scattering for Materials Science (1989).

Stafford, G. R.; "The Electrodeposition of an Aluminum-Manganese Metallic Glass from Molten Salts." J. Electrochem. Soc. 136, 635-639 (1989).

Grushko, B.; Stafford, G. R.; "A Structural Study of Electrodeposited Aluminum-Manganese Alloys." Met. Trans. 20A, 1351-1359 (1989).

Grushko, B.; Stafford, G. R.; "A Structural Study of a Metastable BCC Phase in LA-Mn Alloys Electrodeposited from Molten Salts." Scripta Met. 23, 557-562 (1989).

Grushko, B.; Stafford, G. R.; "Formation of the Al-Mn Icosahedral Phase by Electrodeposition." Scripta Met. 23, 1043-1048 (1989).

Wheeler, N.; "Nickel Graphite Interface." Journal of Comp. Tech. and Res.; in press.

Wheeler, N.; Lashmore, D. S.; Shapiro, A. J.; "Structural Characterization of the Nickel-Graphite Interface." American Cyanamid; in press.

Magnetic Materials

Aviram, I., Bennett, L.H., Swartzendruber, L.J. and Satija, I.I., "Complex Dynamics in the Barkhausen Effect". J. of Magn. and Magn. Materials, submitted, (1989).

Bennett, L. H., Watson, R. E., Melamud, M, "The Roles of Atomic Volume and Disclinations in the Magnetism of the Rare Earth-3D Hard Magnets". J. de Physique, C8, 537 (1988).

Bennett, L. H., Swartzendruber, L. J., Lashmore, D. S., Oberle, R., Atzmony, U., Dariel, M. P. and Watson, R. E., "Magnetic Behavior of Compositionally-Modulated Ni/Cu Thin Films". Phys. Rev., 40, 4633, (1989).

Blendell, J. E., Wong-Ng, W., Chiang, C. K., Shull, R.D., and Fuller, E. R., Jr., High Temperature Superconducting Compounds: Processing and Related Properties, Whang, S. H., and DasGupta, A., eds., The Minerals, Metals, and Materials Society Publ., Warrendale, OH, 193 (1989).

Bohandy, J., Adrian, F. J., Kim, B. F., Moorjani, K., Shull, R. D., Swartzendruber, L. J., Bennett, L. H., Wallace, J. S., "Magnetic-field-modulated Microwave-absorption Detection in a Bi-Sr-Ca-Cu-O Superconductor". J. of Superconductivity, 1, 191 (1988).

Camus, P. P., Shull, R. D., and Melmed, A. J., "APFIM Analysis of Composite Magnetic Thin Films". J. de Physique, submitted, (1989).

Chiang, C. K., Freiman, S. W., Wong-Ng, W., Cook, L. P., Shull, R. D., and Shapiro, A. J., "Preparation of the Bi-Sr-Ca-Cu-O Superconducting Composites Using Glass Technology", Materials Mechanism for Superconductivity of High T_c Materials, Conference Proceedings: Stanford, CA , submitted, (1989).

De Reggi, A. S., Chiang, C. K., Swartzendruber, L. and Davis, G. T., "Magnetic Measurements on Polymer-High T_c Superconductor Composites". High-T_c Superconductors: Magnetic Interactions, World Scientific Publishing Co., 17, 340 (1989).

Goldman, A. J., Long, G. G., Bennett, L. H., Lashmore, D. S. ,and Kuriyama, M., "Observation of Two Structurally Distinct States in Ni-P Glasses Using EXAFS". J. of Electrochemical Society, 135, 8 (1988).

Huang, C. Y., Shapira, Y., McNiff, E. J., Peters, P. N., Schwartz, B. B., Wu, M. K., Shull, R. D., "Magnetic Hysteresis of High-Temperature $YBa_2Cu_3O_x$-AgO Superconductors: Explanation of Magnetic Suspension". Modern Physics Letter B, 2, 869 (1988).

Kaiser, D. L., Gayle, F. W., Roth, R. S., and Swartzendruber, L. J., "Thermomechanical Detwinning of Superconducting $YBa_2Cu_3O_{6+x}$ Single Crystals", J. Materials Res., 4, (1989).

Kim, B. F., Phillips, T. E., Green, W. J., Swartzendruber, L. J., Shull, R. D., Bennett, L. H., Wallace, J. S., "Superconducting Thin Films of Bi-Sr-Ca-Cu-O Obtained by Laser Ablation Processing". Appl. Phys. Letter, 53, 321 (1988).

Lundy, D., Ritter, J., Swartzendruber, L., Shull, R. D., Bennett, L. H., "Studies of Magnetic Flux Penetration in a Chemically Synthesized Bi(Pb)SrCaCuO Superconductor". J. of Superconductivity, 2, 273 (1988).

Lundy, D. R., Ritter, J. J., Swartzendruber, L. J., Shull, R.D., and Bennett, L. H., "Magnetic Properties of a Chemically Synthesized Bi(Pb)SrCaCuO Superconductor". in High-T_c Superconductors: Magnetic Interactions, World Scientific Publishing Co., 263, (1989).

Lundy, D. R., Swartzendruber, and Bennett, L. H., "A Brief Review of Recent Superconductivity Research at NIST". Journal of Res. NIST, 94, 147 (1989).

Melmed, A. J., Shull, R. D., Chiang, C. K., and Fowler, H. A., "Possible Evidence for Superconducting Layers in Single Crystal $YBa_2Cu_3O_{7-x}$ by Field Ion Microscopy". Science, 239, 176-178 (1988).

Melmed, A. J., Shull, R. D. and Chiang, C. K., "Progress in Field Ion Microscopy Imaging of High-T_c Superconducting Oxides". J. de Physique, 49, C6-459 (1988).

Ostertag, C. P., Shull, R. D., Vaudin, M. D., Blendell, J. E., Stearns, L. C., and Fuller, E. R., Jr., "Alignment of Superconducting Grains by Magnetic Casting". Ceramic Superconductors II, M. F. Yan, ed., Am. Cer. Soc., Westerville, Ohio, 332 (1988).

Rubinstein, M., Swartzendruber, L. J., Bennett, L. H., Chaki, T. K., Harford, M. Z., Wolf, S. A., and Edelstein, A. E., "Normal State Properties of High T_c Compounds: Magnetism and Conductivity", in High-T_c Superconductors: Magnetic Interactions, edited by L. H. Bennett, Y. Flom, and G. C. Vezzoli (World Scientific Publishing Co.), 17, 292 (1989).

Rubinstein, M., Harford, M. Z., Swartzendruber, L. J., and Bennett, L. H., "Mossbauer Hyperfine Fields in $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ Superconductors". J. de Physique, Colloque C8, 49, 2209 (1989).

Shull, R. D., Swartzendruber, L. J., Chiang, C. K., Wu, M. K., Peters, P. N., and Huang, C. Y., "Magnetic Levitation by Attraction in Some High T_c Superconductors." in High-T_c Superconductors: Magnetic Interactions, World Scientific Publishing Co., 17, 328 (1989).

Shull, R. D., Ritter, J. J., Shapiro, A. J., Swartzendruber, L. J., and Bennett, L. H., "Magnetic Properties of Iron/Silica Gel Nanocomposites". Multicomponent Ultrafine Microstructures, MRS Symposium Proceedings (edited by L. E. McCandlish, B. H. Kear, D. E. Polk and R. W. Siegel), North Holland Publ. Co., 132, 179 (1988).

Shull, R. D., and Cline, J. P., "High Temperature X-Ray Diffractometry of Ti-Al Alloys". High Temperature Science, submitted, (1989).

Strauss, B. and Swartzendruber, L. J., "Magnetic Particle Inspection Using MIL-STD-1949". Materials Evaluation, submitted, (1989).

Swartzendruber, L. J., Bennett, L. H. and Gallo, C. F., "Measurement of Magnetic Properties in a Melt Cast Bi-Ca-Sr-Cu-O Superconductors", in High-T_c Superconductors: Magnetic Interactions, World Scientific Publishing Co., 17, 303 (1989).

Swartzendruber, L. J., "Quantitative Problems in Magnetic Particle Inspection". QNDE Conf. Proceedings, 8B, 2133 (1988).

Swartzendruber, L. J., Bennett, L. H., "Mössbauer Hyperfine Fields in $RBa_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$ Superconductors". J. de Physique, 68, 2209 (1988).

Swartzendruber, L. J., Bennett, L. H., "Mössbauer Study of the Effect of Oxygen Stoichiometry on the High T_C Superconductor $Y_1Ba_2(Cu_{0.97}Fe_{0.03})_3O_{7-x}$". J. of Superconductivity, 1, 219 (1988).

Swartzendruber, L. J., Kaiser, D.L., Gayle, F.W., Bennett, L. H., Roitburd, A., "Magnetic Properties of a Nearly Cubic Single Crystal of $YB_2Cu_3O_{6+x}$", submitted, (1989).

Wallace, J. S., Ritter, J. J., Fuller, E., Bennett, L. H., Shull, R.D. and Swartzendruber, L. J., "Synthesis & Magnetic Properties of the $BiSrCaCu_2O_x$ 80 and 110 K Superconductors", Phys. Rev. B, 39, 2333 (1988).

Wong-Ng, W., Cook, L. P, Chiang, C. K., Swartzendruber L. J., Bennett, L. H., Blendell, J., and Minor, D., "Structural Phase Transition Study of $Ba_2YCu_3O_{7-x}$ in Air", J. Mater. Res., 3, 832 (1988).

Wong-Ng, W., Cook, L. P., Chiang, C. K. Swartzendruber, L. J., and Bennett, L. H., "Structural Phase Transitions of $Ba_2RCu_3O_{7-x}$ Superconductors", Ceramic Superconductors II, 27 (1989).

Wong-Ng, W., Cook, L. P., Chiang, C. K., Swartzendruber, L. J., and Bennett, L. H., "Structural Phase Transformation Studies of High T_c Superconducting Material", Materials Research Society Proc. (1989).

Wong-Ng, W., Cook, L. P., Chiang, C. K., Vaudin, M. D., Kaiser, D. L., Beech, F., Swartzendruber, L. J., Bennett, L. H., and Fuller, E. R., Jr., in "Structural Phase Transformation Studies of the High T_c Superconducting Materials, $Ba_2RCu_3O_{6+x}$, in Air", High Temperature Superconducting Compounds: Processing & Related Properties, The Minerals, Metals & Materials Society, 553, (1989).

High Temperature Materials Chemistry

Hastie, J. W.; "Coupling Mass Spectrometry with High Pressure/High Temperature Systems," Advances in Mass Spectrometry (Heyden & Sons Ltd.) Proc. 11th Int. Mass Spectrometry Conf., Bordeaux, France (1988)

Schenck, P. K.; Bonnell, D. W.; Hastie, J. W.; "Laser Vaporization Mass Spectrometry of Refractory Materials: Graphite and $YBa_2Cu_3O_x$," J. Am. Vac. Soc. Proc. Joint Int. Laser Science and Am. Vac. Soc. Conf. (in press) (1988)

Hastie, J. W.; Bonnell, D. W.; Plante, E. R.; "Experimental Determination and Model Prediction of Glass Thermochemistry X," Proc. XV Int. Congress on Glass, Leningrad, USSR (1989)

Bonnell, D. W.; Schenck, P. K.; Hastie, J. W.; "Mass Spectrometric Probing of Laser-Induced Materials Vapor Transport: Graphite and $YBa_2Cu_3O_x$," Proc. MRS Symp B: Laser and Particle Beam Chemical Processes on Surfaces, Boston, MA (1988)

Plante, E. R.; Hastie, J. W.; "High-Temperature Mass Spectrometric Studies of Inorganic Systems," Specialist Periodical Reports: Mass Spectrometry Vol. 10, Royal Soc. Chem. London (1989)

Cook, L. P.; Plante, E. R.; Bonnell, D. W.; Hastie, J. W.; "Reaction of Liquid Aluminum with Gaseous ClO_3F," 6th Int. Conf. on High Temp. - Chemistry of Inorganic Materials, Gaithersburg, MD, (1989)

Bonnell, D. W.; Hastie, J. W.; "A Predictive Slag Phase Equilibria Model," 6th Int. Conf. on High Temp. - Chemistry of Inorganic Materials, Gaithersburg, MD, (1989) Schenck, P. K.; Bonnell, D. W.; Hastie, J. W.; "Insitu Analysis of Laser-Induced Vapor Plumes," 6th Int. Conf. on High Temp. - Chemistry of Inorganic Materials, Gaithersburg, MD, (1989)

Cook, L. P.; Schenck, P. K.; Zhao, J.; Farabaugh, E. N.; Chiang, C. K.; "Ceramic Thin Films by Laser Deposition," Proc. Symp. on Ceramic Thin and Thick Films, ACerS (1989)

Plante, E. R.; Cooke, L. P.; "Vapor Pressure and Thermodynamics of Lithium Aluminates," 2nd Int. Symp. on the Fabrication and Properties of Lithium Ceramics, Indianapolis, IN, (1989)

Mechanical Properties of Metals

Chiang, C. K.; Freiman, S. W.; Wong-Ng, W.; Cook, L. P.; Shull, R. D.; Shapiro, A. J.; "Preparation of Bi-Pb-Sr-Ca-Cu-O Superconducting Composites Using Glass Technology"; Proc. International Conf. on Materials and Mechanisms of Superconductivity; Stanford, CA; (1989); (in press).

Fields, B. A.; Fields, R. J.; "Elevated Temperature Deformation of Structural Steel" NISTIR 88-3899; (1989).

Fields, B. A.; Low, S. R.; Early, J. G.; "The Effect of Fatigue Pre-cracking Versus V-Notching on Impact Testing of Charpy Specimens"; Symposium on Charpy Impact Tests: Factors and Variables; ASTM STP 1072; J. M. Holt; Ed.; American Society for Testing and Materials; Philadelphia, PA.; (1990).

Fields, R. J.; Pugh, E. N.; Read, D. T.; Smith J. H.; "An Assessment of the Performance and Reliability of Older ERW Pipelines"; NISTIR 89-4136; (1989).

Gross, J. L; Smith, J. H.; Wright, R. N.; "Ashland Tank-Collapse Investigation"; J. of Constructed Facilities, 3, No.3, 144, (1989).

Hicho, G. E.; "Failure Characterization for the Potomac Electric Power Company Morgantown Unit 2 Waterwall Tube Samples - Characterization of Alligator Surface Present in Selected Tubes"; Letter Report - 1/13/1989.

Hicho, G. E.; "Failure characterization for the Potomac Electric Power Company - Chalk Point Unit 2, Waterwall Tube Sample Failure"; Letter Report - 2/16/1989.

Hicho, G. E.; Fields, R. J.; "Effects of Varying the Austenitizing Temperature and Cooling Rate on the Ability of HSLA-80 Steel to Achieve Impact Properties Comparable to HSLA-100"; ASM International Journal of Heat Treating; (In Press).

Hicho, G. E.; Fields, R. J.; "Effects of Varying Precipitation Hardening Temperatures and Time on the Ability of HSLA-80 Steel to a Yield Strength and Toughness Comparable to HSLA-100. ASM International Journal of Heat Treating; (In Press).

Hicho, G. E.; Smith, J. H.; "Metallurgical Evaluation of 17-4 PH Stainless Steel Castings"; NISTIR 89-4075; (1989).

Horrigan, P. M.; Finn, J. F.; Tuler, F. R.; Smith, J.H.; "Locating Fatigue Cracks by Acoustic Emission Testing"; J. of Acoustic Emission, 8, S79, (1989).

Huang, Y.; Hicho, G. E.; Fields, R. J.; "J-integral, R-curve Testing of 5052 H112 Aluminum, Utilizing DC Potential Drop and Infrared Thermographic Method"; Advances in Fracture Research; K. Salama; RaviChander; D. M. R. Taplin; P. Rama Rao; Eds.; Pergamon Press, 1, 359, (1989).

Kim, J.; Park, C.; Yoon, D.; Shapiro, A. J.; "Discontinuous Coarsening of Tetragonal Precipitates in Partially Stabilized Zirconia Induced by Diffusional Coherency Strain under Applied Stress"; Journal of American Ceramic Society; (in press).

Kreider, K. G.; Cline, J. P.; Shapiro, A. J. Pena, J. L.; Rojas, A.; Azamar, A. L.; Maldonado, J.; DelCastillo L.; "Sputtered Thin Film YBa2Cu3On" Conference Proceedings Series No. 182; American Vacuum Society Series No. 6. 1989.

Kreider, K. G.; Cline, J. P.; Shapiro, A. J.; Moreland J.; "Formation of High Tc Thin Film Superconductors on Silica Wafers" Proc. TMS Superconducting Materials Meeting; Las Vegas; 1989 (in press).

Shives, T.R.; Low,III, S.R.; "Tensile Tests and Chemical Analysis of Material from a Steel Pressure Tank"; Letter report to the National Transportation Safety Board; (June 1989).

Shives, T. R.; Low,III, S. R.; ""Tensile Tests of Type 305 Stainless Steel Mine Sweeping Wire Rope." NISTIR-4174 (1989).

Shives, T.R.; Mertaugh, L.J.; Eds.; "Detection, Diagnosis and Prognosis of Rotating Machinery to Improve Reliability, Maintainability, and Readiness through Application of New and Innovative Techniques." Proceedings of the 41st Meeting of the Mechanical Failures Prevention Group; Cambridge University Press; Cambridge, England; (1989).

Shull, R. D.; Ritter, J. J.; Shapiro, A. J.; Swartzendruber, L. J.; Bennett, L. H.; "Magnetic Properties of Iron/Silica Gel Nanocomposites" Mat. Res. Soc. Symp. Proc. 132; 179; (1989).

Smith, J. H.; "Metallurgical Examination of Pipe from Chevron Refinery"; Letter Report to the U. S. Occupational Safety and Health Administration; July 20, 1989.

INDUSTRIAL AND ACADEMIC INTERACTIONS

The research programs of the Metallurgy Division are designed and carried out in support of industrial and scientific needs. Specialized facilities within the Division, including metals processing and nondestructive evaluation, attract scientists from both academic and industrial organizations for cooperative research efforts. Interactions with industry, universities, and professional organizations are viewed as an important element of our work with collaborative programs, consulting and general involvement with outside groups being a long standing practice.

In 1989, the Division performed collaborative research with many private organizations through its Research Associate and Guest Scientist programs and other arrangements. Representative examples of such interactions include:

INDUSTRY

1. A.A.I. Corporation

   R. J. Fields and S. R. Low III provided consultation and advice to A.A.I. Corporation in the testing of components made from carbon fiber composites. Tests were carried out at NIST on various components until design requirements were satisfied. NIST also provided assistance in data acquisition and interpretation for these tests.

2. ACerS (American Ceramic Society)

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

3. Advanced Technology Commercialization Association

   Over a period of several years, the Corrosion Group developed an electrochemical, nondestructive technique incorporating portable equipment, to measure the corrosion of reinforcing steel in concrete bridge decks. The ATCA, a technology transfer organization, has picked this measurement system as a promising candidate for commercialization. Presently, the association is investigating the possibility of broadening the application of this technology to other areas of need.

4. Aluminum Association

   The third year of the cooperative project of NIST and the Aluminum Association has been successfully completed. Two designs of an eddy current sensor for measuring temperature of extruded aluminum products during processing have been demonstrated in three plant tests. The Aluminum Association and NIST are planning to continue the project, expanding the scope to include sheet and plate products rolled at high speeds. Mr. Michael Mester will continue as Research Associate for the Aluminum Association.

5. American Association of Railroads (AAR)

The Railway Tank Car Safety Research and Test Committee of the AAR Research Progress Institute has established a collaborative research with the NIST program funded by the Federal Railroad Administration (FRA) to conduct research on the properties of tank car materials.

6. American Cyanamid Corporation

The Electrodeposition Group and American Cyanamid Company have a joint program addressing the problem of interfacial failure to graphite fibers in a nickel matrix at elevated temperatures. The research is being done be a Research Associate (N. Wheeler) in collaboration with members of the Electrodeposition Group (D. S. Lashmore, C. Johnson and E. Rosset) on the electrochemical coating of the samples and with other in the Metallurgy Division (A. Shapiro, C. Handwerker, F. Gayle, and U. Kattner) on the characterization of the interface between the fiber and the matrix and (R. Clough and J. Mullen) for adhesion testing of the interface.

7. American Dental Association

A cooperative effort is underway between the American Dental Association (Dental and Medical Group at NIST) and the Electrodeposition Group (D. Kelley, D. Lashmore and C. Johnson) to design and electroform dental prostheses.

8. American Iron and Steel Institute

The Advanced Sensing Group has continued a collaboration with the American Iron and Steel Institute (AISI). This interaction began in 1983 with the signing of a memorandum of understanding and agreement to research and develop ultrasonic approaches for internal temperature distribution and pipe/porosity sensors for control of steel processing. These initial goals have been achieved, with a prototype internal temperature profiling system developed and a pipe/porosity sensor now in routine use in a least one steel mill. Further research has focused on the need for in-process sensors to locate solid-liquid interfaces. The feasibility of using laser-generated ultrasonics for this task has been demonstrated on commercially pure aluminum. Ongoing efforts are concerned with the application of this technique to alloys with extended regions of mixed liquid and solid phases.

9. American Iron and Steel Institute (AISI)

Collaborations between K. Almand of AISI and R. J. Fields and B. A. Fields of NIST continues on the high temperature deformation of structural steel. This work seeks to evaluate and quantify the fire resistance of steel used in buildings. A report on this work has been completed (NISTIR #88-3899). AISI has supplied NIST with A-36 steel for future work on this topic.

10. ASM INTERNATIONAL

As part of the ASM alloy phase diagram evaluation program, B. Burton (NIST) has evaluated the systems Fe-Li, Fe-Na, Fe-K, Fe-Rb, Fe-Pb, and Fe-Zn. The Fe-Zn evaluation was done in collaboration with Professor Pierre Perrot, University of Lille, France.

11. BDM Corporation

The Metallurgical Processing and Advanced Sensing Groups have collaborated with the BDM Corporation to develop an intelligent controller for hot isostatic pressing of intermetallic compounds. In this jointly funded DARPA/NIST program, NIST has developed the technology of measuring powder densification and BDM has developed the control software.

12. C.E.C.M./C.N.R.S., Vitri, France

A cooperative program is sponsored jointly by C.N.R.S. and NIST to study the structure and thermodynamics of a new type of material, the quasicrystal. The study involves experimental work on Al-Fe-Cu phase diagrams (F. Gayle, A. Shapiro, W. J. Boettinger, L. A. Bendersky, J. W. Cahn), neutron diffraction (B. Mozer, J. W. Cahn), and electron microscopy (L. A. Bendersky). Dr. D. Gratias and his colleagues from C.N.R.S. are the French side of the program.

13. CEM Corporation

A collaborative program with CEM Corporation in conjunction with their research associate at NIST and the Electrodeposition Group (C. Johnson and D. Kelley) is in progress. The program is focused on the development of temperature sensing devices used in a microwave environment. Commercial, off the shelf, thermocouples are being modified by electroplating a heavy gold coating on the external metal sheath which performs as a heat sink. The technique prevents the interface of thermocouple heating with temperature sensing.

14. Collaborative Testing Services, Inc.

R. J. Fields collaborated with Charles Leete, Sarah Weitzel and Ellen Trap of Collaborative Testing Services (CTS) in the selection of materials and specimen fabrication procedures for interlaboratory comparison testing. Laboratory comparisons were made in tensile testing and hardness testing. NIST also ran tests on these materials as input into the studies.

15. Crucible Materials Corp., General Electric Co., and Hoeganaes Corp.

The NIST-industrial consortium on automated processing of rapidly solidified metal powders by high pressure inert gas atomization, formed in FY 1988, has completed its second year of research activities. The

NIST supersonic inert gas metal atomizer (SiGMA) in the Metallurgy Division's metals processing laboratory has been the focal point of this pioneering work to apply the NIST concept of intelligent processing of materials. This approach consist of a system involving advanced sensors process models, and artificial intelligence (AI) control. Scientists from the three participating companies, in collaboration with NIST scientists, have developed an in-situ particle size measurement sensor and are currently integrating this device with an AI adaptive automatic control system.

16. DOMTAR Research Center

The Research Center at DOMTAR is developing a corrosion inhibiting de-icing salt designed to replace corrosive de-icing salts presently used, and the Corrosion Group (E. Escalante) has been involved in evaluating their corrosion testing program.

17. DuPont (Wilmington, DE)

Dr. John Hastie is collaborating with Dr. U. Klabunde and others of DuPont on the development of process mechanisms for titanium extractive metallurgy.

18. Electric Power Research Institute

EPRI has funded a new multi-year program within the Corrosion Data Center (D. B. Anderson) to develop computer programs to guide electric utility materials and design engineering personnel in materials selection and operating controls to minimize critical equipment failures. The program will focus on critical applications in power plant condensers, steam generators, flue gas desulfurization systems and service waters.

19. FIBA, Inc. and Union Carbide Corporation

A collaborative effort is underway between FIBA, Inc. (P. Horrigan), Union Carbide Corporation (M. Rana), and NIST (J. H. Smith) to evaluate use of acoustic emission techniques for use in the periodic inspection of large steel pressure vessels. NIST is in the process of developing specific procedures and test criteria to permit the use of acoustic emission techniques for this application.

20. General Electric Co.

The Advanced Sensing Group is collaborating with the Aircraft Engines Division of G. E. to explore potential methods for microstructure characterization during processing and for sensing the liquid-solid interface during "skull" melting of superalloys.

21. General Electric Co.

A cooperative investigation on the resistance of NIST (D. S. Lashmore, D. R. Kelley and C. E. Johnson) coated fibers to high temperature degradation is underway.

22. Harry Diamond Laboratory

In a cooperative project with the U.S. Army Harry Diamond Laboratory A. Shapiro is evaluating the composition and microstructure of microelectronic solder joints.

23. IBM Corporation

An investigation of the magnetic properties of electrochemically produced superlattices is underway under IBM sponsorship. (L. H. Bennett, D. S. Lashmore and R. R. Oberle)

24. IBM Corporation

A collaborative effort between Dr. L. A. Bendersky (NIST) and Dr. W. Krakow (IBM) is underway to study structure of interfaces of non-crystallographic twins. The research involves state-of-the-art ultra-high resolution electron microscopy.

25. IBM Corporation

An investigation of the magnetic properties of electrochemically produced superlattices is underway under IBM sponsorship. Among the accomplishments are the development of techniques to produce artificial superlattices of very high quality, the discovery of magnetic viscosity in metallic multi-layers and the correlation of alloying at the interface with saturation magnetic saturation dependence on temperature (L. H. Bennett and D. S. Lashmore).

26. Inland Steel

Drs. Plante and Bonnell have been collaborating with Dr. H. Pielet of Inland on the thermochemistry of blast furnace and steel slags and inorganic inclusions in steel. Dr. Pielet provides slag samples and data from plant experience, Dr. Plante is providing thermochemical data from high temperature mass spectrometry, while Dr. Bonnell supplies database and model techniques.

27. Luxfer USA, Inc.

A collaborative effort between NIST (J. H. Smith) and Luxfer USA, Inc. (G. Waite) is ongoing to determine the extent of cracking in seamless aluminum compressed gas cylinders and to develop a reliable test method for inspecting the cylinders in service.

28. Materials Technology Institute of the Chemical Process Industries

MTI, in conjunction with the NACE-NIST Corrosion Data Center (D.B. Anderson), continues to support a multi-year program to develop expert systems for selection of materials for storage and handling of hazardous chemicals. Systems are based on rules defined during discussions with consultant experts representing a broad range of industrial experience.

29. Martin Marietta Corporation

F. W. Gayle of NIST is collaborating with J. R. Pickens and F. H. Heubaum of Martin-Marietta Laboratories in a study of the physical metallurgy and microstructure of a new class of weldable, ultra-high strength aluminum alloys.

30. Martin Marietta Energy Systems, Inc. Oak Ridge National Laboratory

The Corrosion Group has been collaborating with Martin Marietta Energy Systems on a study of the corrosion and stress corrosion cracking of nickel aluminide alloys. The results of this work were presented at a technology transfer workshop at Oak Ridge National Laboratory. The workshops, organized by ORNL, brought together participants from several companies interested in production, fabrication, and use of nickel aluminide alloys, as well as university and government research scientists.

31. NASA/Cal Tech Jet Propulsion Lab (Pasadena, CA)

Dr. Bonnell is collaborating and consulting on design of levitation systems for space applications.

32. NASA Marshall Space Flight Center (Huntsville, AL)

A collaborative effort between P. Peters (Marshall Space Flight Center) and R. Shull (NIST) on composite high T_c superconductors resulted in the explanation of a novel method of "attractive" magnet levitation discovered at NASA/Marshall.

33. National Association of Corrosion Engineers

The NACE-NIST Corrosion Data Center (D.B. Anderson), initiated in 1982, continues to provide the scientific and technical coordination to provide evaluated corrosion data in computerized format. NACE provides three full time Research Associates at NIST plus considerable staff time in support of the program activities. Current activities involve standards activity relating to data formatting, expert system developments for industrial clients, structuring of a comprehensive corrosion database and data entry from two key industrial data collections, development of data evaluation techniques and resolution of technical deficiencies with the corrosion thermodynamics program.

34. National Geographic Society

A. Shapiro and F. W. Gayle conducted an SEM study of metallic and ceramic knife blades for a National Geographic article on advanced materials.

35. New Zealand Department of Scientific and Industrial Research (DSIR)

A cooperative program within the Corrosion Data Center (D. B. Anderson) was completed with the successful development of a standards based expert systems to guide materials selection for down hole equipment in sour gas and oil production environments. K. A. Lichi, a Research Associate from DSIR, served as program leader.

36. Norton Corrosion Limited, Inc., Bothell, WA

The Corrosion Group (E. Escalante) is continuing to collaborating with Norton Corrosion on their application of the computer controlled device, developed in our laboratories, for measuring the corrosion of reinforcing steel in concrete bridge decks.

37. Norris Cylinder, Inc.

A collaborative effort between NIST (J. H. Smith) and Norris Cylinder, Inc. (E. McSweeney) has been initiated to develop light weight high strength steel cylinders and more efficient stainless steel cylinders.

38. Packer Engineering

A cooperative program with Packer Engineering and the Electrodeposition Group (C. Johnson and D. Lashmore) was established to coat silicon carbide whiskers and particles with copper and other metals and metal alloys. The program objective is to enhance the bonding between the whiskers and particle and a matrix metal in the manufacturing of particulate composites.

39. Pepco

The Division (G. E. Hicho) has performed numerous failure analyses for the Potomac Electric Power Company (PEPCO) for two reasons: (1) to assist the public utility in its service to the community and (2) to maintain and upgrade NIST competence in materials technology for the electric power industry. As the average age of U.S. plants increases and failures become more frequent, such competence will be needed to identify and transfer state-of-the-art technology to industry.

40. Pratt and Whitney

The densification of titanium aluminide powders by hot isostatic pressing is being measured in a collaborative effort by NIST (R. Schaefer) and Pratt and Whitney.

41. Rural Electrification Administration, Dept. of Agriculture

The Corrosion Group (E. Escalante, J. Fink) has continued its collaboration with REA to develop a data base on the corrosion performance of telephone cable shielding materials.

42. Solution Model Database

Groups currently using or evaluating the IMCC model include Dr. Howard Pielet, Inland Steel; Prof. K. S. Spear, Penn State University; Dr. Chad Sheckler, Alfred University; Prof. Paul Daves, Brigham Young University; Prof. Steve Benson, Univ. North Dakota; Prof. Tom Roberts, Milwaukee Area Technical College; Dr. C. David Rogers, USS of USX and Carnegie-Mellon Institute; Prof. Arthur E. Morris, University of Missouri-Rolla.

43. Superconix, Inc.

A collaborative project is underway with C. F. Gallo, Superconix, Inc. and the Magnetic Materials Group (L. J. Swartzendruber) to measure the magnetic properties of superconducting single crystals.

44. Swedish Corrosion Institute, Stockholm, Sweden

A closed meeting, composed of scientists gathered to evaluate the acidification of water and soil by air pollution and its effect on corrosion, was called by the Nordic Council of Ministers and the United Nations Economic Commission for Europe. E. Escalante of the Corrosion Group was Chairman of the three day meeting directed at evaluating the corrosion of materials in soil. A written report describing the major conclusions was sent to the NCM and UNECE.

45. Textron, Lowell, MA

Several miles of carbon monofilament were coated with experiment alloys developed at NIST for introduction by Textron. This funded program resulted in the development of new techniques to carry high current to the fiber, new computer control software, and techniques to handle the 33 micron diameter fiber (D. S. Lashmore, E. A. Rosset).

46. Union Carbide Corporation, Taylor-Wharton, Inc., and Norris Industries

A collaborative effort is underway between the Linde Division of Union Carbide Corporation (M. Rana), Taylor-Wharton, Inc. (C. Holl), Norris Industries (E. McSweeney), and the Metallurgy Division (J. H. Smith) to develop criteria for the safe design and fabrication of high strength steel, seamless pressure vessels. Criteria have been developed, based on fracture mechanics principles, to permit the use of new, higher strength steels for the construction of pressure vessels without reducing the level of safety of these vessels.

47. USX/Carnegie-Mellon

Dr. Rogers of Carnegie-Mellon Institute (formerly U.S. Steel of USX) is developing process models for melt shop use. Dr. Bonnell is working with Dr. Rogers to develop a form of the NIST Steel Slag Model for use as the chemistry component of such process models.

48. Wilson Instruments Company

A cooperative project is being carried out by T. R. Shives of NIST to evaluate the variability in Rockwell hardness calibration blocks. Rockwell hardness test machines have been loaned to NIST by Wilson Instruments and Clark Inc. Hardness indentors for evaluation have been loaned to NIST by all major U.S. manufacturers and distributors of hardness indentors.

INDUSTRY/UNIVERSITY

1. BHABHA Atomic Research Center (Government of India)
University of Poona

A cooperative project is underway with the BHABHA Atomic Research Center (Dr. C. K. Gupta) and the University of Poona (Dr. A. P. B. Sinha). This project is part of the Indo-US Physical, Materials and Marine Sciences Collaboration Program and the objective of this project is to study the influence of nitrogen content on the stress corrosion cracking behavior of stainless steels (alloy 316L). NIST has provided material for this study and complimentary experiments will be conducted at the various institutions. A workshop on the Corrosion Science and Technology on Stainless Steels is being planned to be held in India.

2. General Electric/University of Virginia

W. Johnson and A. H. Kahn are collaborating with the Aircraft Engines Division of General Electric and Dr. Haydn Wadley of the University of Virginia to explore ultrasonic and eddy current methods for sensing the crystalline phases present in inductively coupled plasma deposited titanium aluminides.

3. NIST Metals Processing Laboratory

Facilities in this laboratory are available for preparation of special samples for various materials characterization studies. 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., General Electric Co., NASA, Johns Hopkins Univ., UCSB, and Univ. of Wisconsin have interacted in this program.

4. SRI (Stanford Research International)

Drs. Hastie and Bonnell are collaborating with Dr. D. Hildenbrand of SRI in the mass spectral analysis of complex high temperature vapors. NIST and SRI are using techniques which are unique to each laboratory but are nevertheless complementary.

UNIVERSITIES

1. Applied Physics Laboratory, The Johns Hopkins University

A collaborative effort is underway between the Applied Physics Laboratory of the Johns Hopkins University (K. Moorjani) and NIST (R. Shull) to prepare and investigate the magnetic behavior of composite materials having nanocrystalline-sized grains.

2. Applied Physics Laboratory, The Johns Hopkins University

A collaborative effort is underway between the Applied Physics Laboratory of the Johns Hopkins University (J. Bohandy) and NIST (L. Bennett) to prepare high T_c superconductors by laser ablation and investigate their magnetic properties.

3. Ben-Gurion University

A Joint U.S. Israeli Binational Science Foundation program on solid state amorphization transformations is underway between NIST (D. S. Lashmore) and Ben-Gurion University (M. P. Dariel).

4. Boris Kidric Institute (BKI), Belgrade, Yugoslavia

A collaborative activity is underway between Dr. Hastie and Dr. Zmbov of BKI for the mass spectrometric analysis of complex high temperature processes, including plasma deposition of amorphous silicon.

5. Cambridge University

Professor Michael F. Ashby and graduate students at Cambridge University (England) are developing predictive models for the hot isostatic pressing of intermetallic alloys in collaboration with Metallurgy Division scientists.

6. Chonnam University

A collaborative effort is underway between Dr. C. Handwerker of NIST and Prof. D. T. Lee of Chonnam University, Kwangju, Korea to study interface properties of Al-based metal-matrix composites.

7. E. Kardelj University, Ljubljana, Yugoslavia

A three-year collaborative project with the Department of Physical Chemistry, E. Kardelj University, Ljubljana, Yugoslavia, has been initiated with the Corrosion Group (U. Bertocci). The project will study the application of a number of electrochemical measurement methods for the in-situ monitoring of corrosion in heat-exchanger tubes.

8. Free University of Brussels

Drs. Hastie and Bonnell are collaborating with Dr. J. Drowart (Brussels) on a survey of ionization cross section usage in high temperature mass spectrometry.

9. Georgia Institute of Technology

Professors Shui-Nee Chow, Jack Hale and coworkers are developing new mathematical techniques for studying dynamical systems and applying them to the spinodal decomposition of alloys. A similar program will commence next year with Professor George Sell at the University of Minnesota.

10. George Washington University

George Hicho serves as an Associate Professorial Lecturer in Engineering at George Washington University in the area of Mechanical Properties testing.

11. Harvard University

Experimental studies of the state of long range order in intermetallic alloys subjected to rapid solidification using picosecond laser surface melting are being conducted with Prof. M. J. Aziz at Harvard University. A theoretical paper was also completed on disorder trapping in intermetallic alloys by rapid solidification.

12. Indira Gandhi Institute (IGI) Kalpakkam, India

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

13. Institute of Metals Research, Academia Sinica, Shenyang

A collaborative effort is underway with a guest scientist, Prof. Y. Huang, of the Institute of Metals Research, Academia Sinica, Shenyang, Peoples Republic of China, to investigate the fracture of metals using infrared thermography and DC potential drop techniques to measure crack length.

14. Iowa State University

A collaborative effort is underway between NIST (F. W. Gayle) and ISU (Prof. Alan Goldman) to characterize single crystals of the Al-Cu-Li quasicrystal by neutron diffraction.

15. Johns Hopkins University

Studies on the mechanism of transgranular stress corrosion cracking have been pursued in cooperation with Johns Hopkins University (Dr. J. Kruger). The experimental studies were carried out at NIST by a graduate student (T. Cassagne), and the staff of the Corrosion Group was involved both in the experimental and in the analysis of the results.

16. Johns Hopkins University

A joint investigation of the mechanical properties of electrodeposited CMS alloys is being conducted with Johns Hopkins University in cooperation with Dr. Moshe Rosen (D. S. Lashmore and R. R. Oberle).

17. Johns Hopkins University

A collaborative effort with Johns Hopkins University (D. Shechtman) which resulted in the discovery of an entirely new class of materials, the quasicrystals, was continued. Emphasis has been on studies of the structure of these unusual alloys and the processing conditions that produce them.

18. Korea Advanced Institute of Science and Technology

A collaborative effort is underway between Alexander Shapiro of the Metallurgy Division and a guest scientist, Prof. Duk H. Yoon, of the Korea Advanced Institute of Science and Technology (KAIST) to study grain boundary migration in (6 MgO + 4 CuO) + 90 ZrO_2.

19. Korea Advanced Institute of Science and Technology (KAIST)

An investigation of stress effects on diffusion and interface stability is being conducted by Dr. C. Handwerker of NIST in collaboration with Prof. D. N. Yoon and NIST guest scientist, Dr. W. H. Rhee, both of the Korea Advanced Institute of Science and Technology.

20. Korea Advanced Institute of Science and Technology (KAIST)

A collaboration effort is underway between Dr. Jin Yu of KAIST and R. J. Fields, R. de Wit and R. Thomson of NIST to study the role of microstructure in brittle fracture.

21. Louisiana State University and Iowa State University

A collaborative effort is underway with Prof. Steven Watkins (LSU) and Prof. Alan Goldman (ISU-Ames) to investigate details of crystal structure of untwinned single crystals of $YBa_2Cu_3O_{6+x}$ using single crystal diffractometry at cryogenic temperature.

22. Northwestern University

Coarsening rate measurements in solid-liquid mixtures in several metal alloy systems are being performed cooperatively with Northwestern University (P. Voorhees). The aim of this work is to perform measurements on systems which permit a quantitative comparison with theory so that a detailed understanding of coarsening processes may be developed.

23. Oregon State University

Cooperative research on structure determination of Ti-Al-Nb phases using single crystal x-ray diffractometry is being pursued between Prof. C. B. Shoemaker (Oregon State) and Drs. L. A. Bendersky and W. J. Boettinger (NIST).

24. Oregon Graduate Research Center

A collaborative effort has been initiated between NIST (J. H. Smith) and the Oregon Graduate Research Center (Prof. Atteridge) to develop test methods and standards for fracture resistant high strength steel cylinders.

25. Rice University

Dr. Bonnell continues to provide consultation support to the High Temperature Group in the Chemistry Department on levitation and thermophysical properties of liquid metals.

26. UCLA

Professors Ryoichi Kikuchi and Stanley Osher are collaborating on path probability methods underlying phase dynamics and on non-linear shock front techniques applied to image processing and crystal growth.

27. University of California and The International Group for Historic Aircraft Recovery (TIGHAR)

Fifty years ago a Lockheed Electra flown by Amelia Earhart and Fred Noonan, was lost at sea as they attempted to circle the globe. Today, new evidence suggests that they crash landed on a tropical coral atoll in the Pacific Ocean, and a group of scientists is investigating the site for evidence of the aircraft. The Corrosion Group, at NIST, has consulted with the UCA Group on the consequences of corrosion to the aircraft in several landing scenarios and in the handling and transportation of any artifacts that may be found.

28. University of Maryland

R. deWit and R. J. Fields are collaborating with G. R. Irwin and X. J. Zhang of the University of Maryland to further the understanding of cleavage crack arrest and reinitiation in steel. In particular heavy-section nuclear-vessel steels are being studied.

29. University of Maryland

D. S. Lashmore and C. R. Beauchamp are conducting a joint study with Professor Wuttig on solid state amorphization on SnNi alloys.

30. University of Notre Dame, Notre Dame, IN

Studies were conducted at Notre Dame with technical direction provided by a member of the Corrosion Group (R. E. Ricker) into the mechanism of corrosion fatigue crack initiation in a high purity Fe-Ni alloy by a graduate student (A. S. Rai) and into the influence of rare earth additions on the hydrogen embrittlement of 2.25 Cr-1 Mo steel by another graduate student (M. T. Fernandes). This collaboration resulted in both students successfully completing their degree programs in 1989.

31. University of Science and Technology - Beijing, China

In a program funded by the United Nations Industrial Development Organization (UNIDO), a lecturer from UST (Dr. C. Li) has completed a one year fellowship as a Guest Scientist in the Corrosion Data Center where she has developed a prototype corrosion database addressing Chinese industrial development needs.

32. University of Wisconsin at Madison

Cooperative programs underway with J. Perepezko and A. Chang of the University of Wisconsin have focused on studies of microstructure development and phase diagram relationships in the Ti-Al-Nb system.

TECHNICAL/PROFESSIONAL COMMITTEE LEADERSHIP ACTIVITIES

Alpha Sigma Mu, The Metallurgy and Materials Honor Society
R. E. Ricker, National Board of Trustees

Aluminum Association Standards Committee H-35 on Aluminum and Aluminum Alloys
S. D. Ridder

American Association for Crystal Growth
S. R. Coriell, Executive Committee

American Electroplaters and Surface Finishers Society
C. E. Johnson, Electrocomposites Committee
D. S. Lashmore, Alloy Deposition Subcommittee

American Institute of Mining, Metallurgical and Petroleum Engineers
The Metallurgical Society
R. D. Shull, The Chemistry and Physics of Materials Committee
R. D. Shull, The Titanium Committee
R. E. Ricker, Committee on Corrosion and Environmental Effects

American Petroleum Institute
Task Group on Tankage Brittle Fracture
J. H. Smith

American Physical Society
Materials Physics Topical Group
C. A. Handwerker, Member of Steering Committee

ASM INTERNATIONAL
Alloy Phase Diagram Appropriations Committee
E. N. Pugh

Corrosion and Environmental Effects Committee
R. E. Ricker

Editorial Committee for Advanced Materials and Processes
E. N. Pugh

Editorial Committee for Bulletin of Alloy Phase Diagrams
F. W. Gayle

Heat Treating Steering Committee
H. T. Yolken

Materials Science Seminar
1989 Materials Week
H. T. Yolken, Chairman

Subcommittee on Metallurgical Reactions and Electromigration in Electronic Devices
C. A. Handwerker, Chairman

Technology Transfer Committee
E. N. Pugh

Washington, DC Chapter - Education Committee
R. E. Ricker, Chairman

ASTM

Committee on Terminology
C. G. Interrante, Technical Committee Representative

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: B8.10:	Metallic and Inorganic Coatings General Test Methods C. E. Johnson D. S. Lashmore
B8.10.03:	Microhardness Testing C. E. Johnson, Liaison to E04 D. S. Lashmore, Liaison to E04
B9:	Metal Powders and Metal Powder Products J. R. Manning
C26: C26.07: C26.13:	Nuclear Fuel Cycle Waste Materials Repository Waste Package Materials Testing C. G. Interrante
E3: E3.07:	Chemical Analysis of Metals Acoustic Emission R. B. Clough
E4: E4.05:	Metallography Microhardness C. E. Johnson D. S. Lashmore

E7: Nondestructive Testing
L. J. Swartzendruber
L. H. Bennett

E7:04 Acoustic Emission
J. A. Simmons
R. B. Clough

E24: Fracture Testing
C. G. Interrante, Member of Executive Committee

E24:02 Fractography and Associated Microstructures
G. E. Hicho

E24.04: Sub-Critical Crack Growth
C. G. Interrante

E24.05: Terminology for Fracture Testing
C. G. Interrante, Co-Chairman
R. deWit

E24.06: Fracture Mechanics Applications
J. H. Smith
G. E. Hicho

E28: Mechanical Testing
E28.06.07: Hardness Test Block Intercomparison Task Group
T. R. Shives, Chairman

E49: Computerization of Material Property Data
D. B. Anderson, Steering Committee for Second International Symposium on Computerized Materials Databases

E49.02: Corrosion Data Formats
D. B. Anderson, Chairman

E49:91: D. B. Anderson, Awards Committee Chairman

F4: Medical and Surgical Materials and Devices
F4.02.09: Joint Section on Corrosion of Implants
A. C. Fraker, Co-Chairperson

F7: Aerospace Industry Methods
F7.04: Hydrogen Embrittlement Testing
C. G. Interrante, Chairman, Task Group on Terminology

G1: Corrosion of Metals
G1.03.1: Corrosion Data Formats Task Group
D. B. Anderson, Chairman

G1.06: SCC and Corrosion Fatigue
R. E. Ricker
C. G. Interrante

G1.10: Corrosion of Metals in Soil
E. Escalante

G1.10.01: Measurement of pH of Soil
E. Escalante, Task Group Leader

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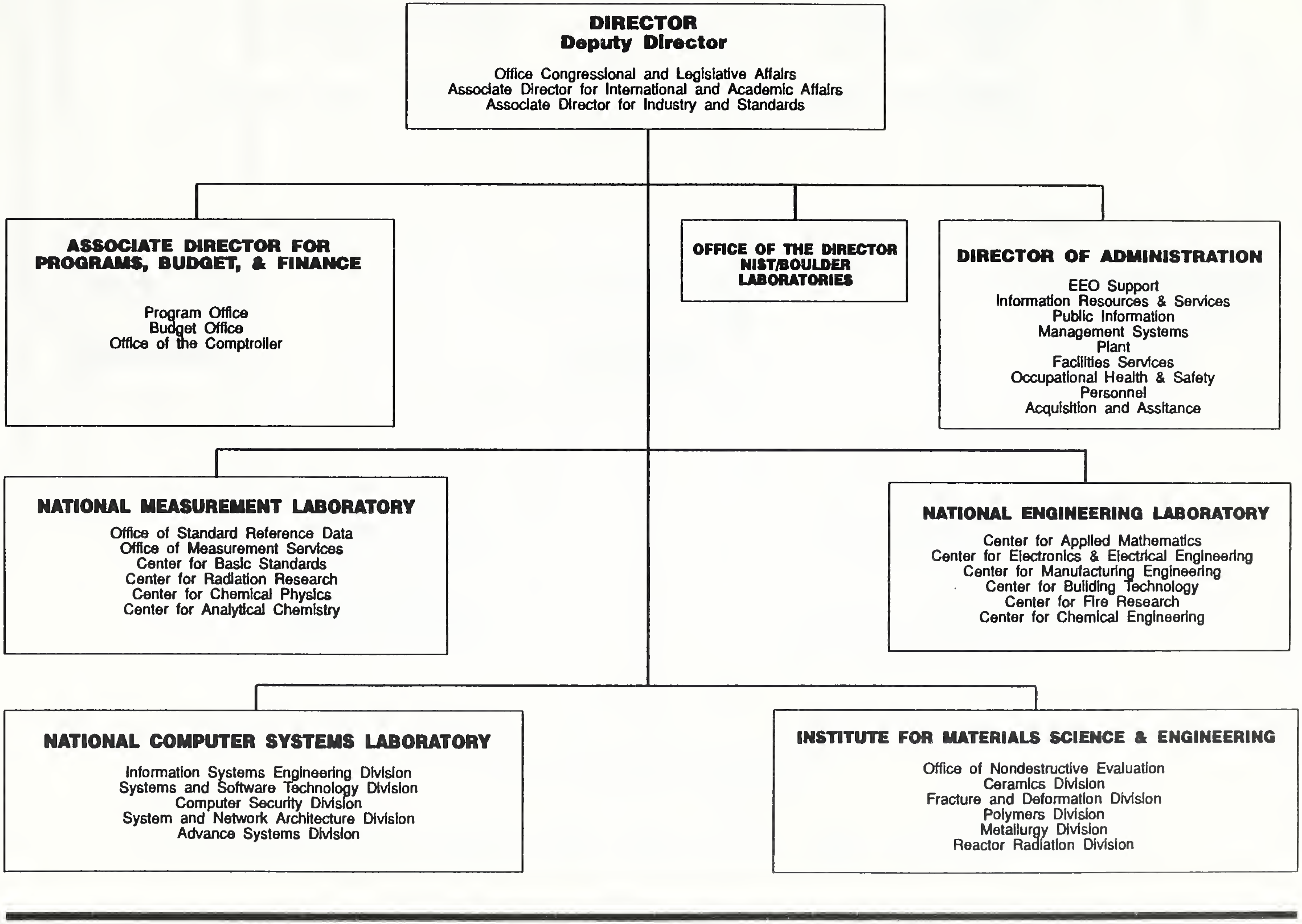

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11. ABSTRACT (A 200-WORD OR LESS FACTUAL SUMMARY OF MOST SIGNIFICANT INFORMATION. IF DOCUMENT INCLUDES A SIGNIFICANT BIBLIOGRAPHY OR LITERATURE SURVEY, MENTION IT HERE.)

This report summarizes the FY 1989 activities of the Metallurgy Division of the National Institute of Standards and Technology (NIST). These activities center upon the structure-processing-properties relations of metals and alloys and on methods of measurement; and also include the generation and evaluation of critical materials data. Efforts comprise studies of metals processing and process sensors; advanced materials, including metal matrix composites, intermetallic alloys and superconductors; corrosion and electrodeposition; mechanical properties; magnetic materials; and high temperature reactions.

The work described also includes two cooperative programs with professional societies (the Alloy Phase Diagram Program with ASM International, and the Corrosion Data Program with the National Association of Corrosion Engineers); two with trade associations (the Temperature Sensor Program with the Aluminum Association, and the Steel Sensor Program with the American Iron and Steel Institute); and several with industry including the Powder Atomization Consortium with three companies.

The scientific publication, committee participation, and other professional interactions of the 74 full-time and part-time permanent members of the Metallurgy Division and its 35 guest researchers are identified.

12. KEY WORDS (6 TO 12 ENTRIES; ALPHABETICAL ORDER; CAPITALIZE ONLY PROPER NAMES; AND SEPARATE KEY WORDS BY SEMICOLONS)

Corrosion; Electrodeposition; Magnetic Properties; Metals Processing; Metallurgy; Process Sensors

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