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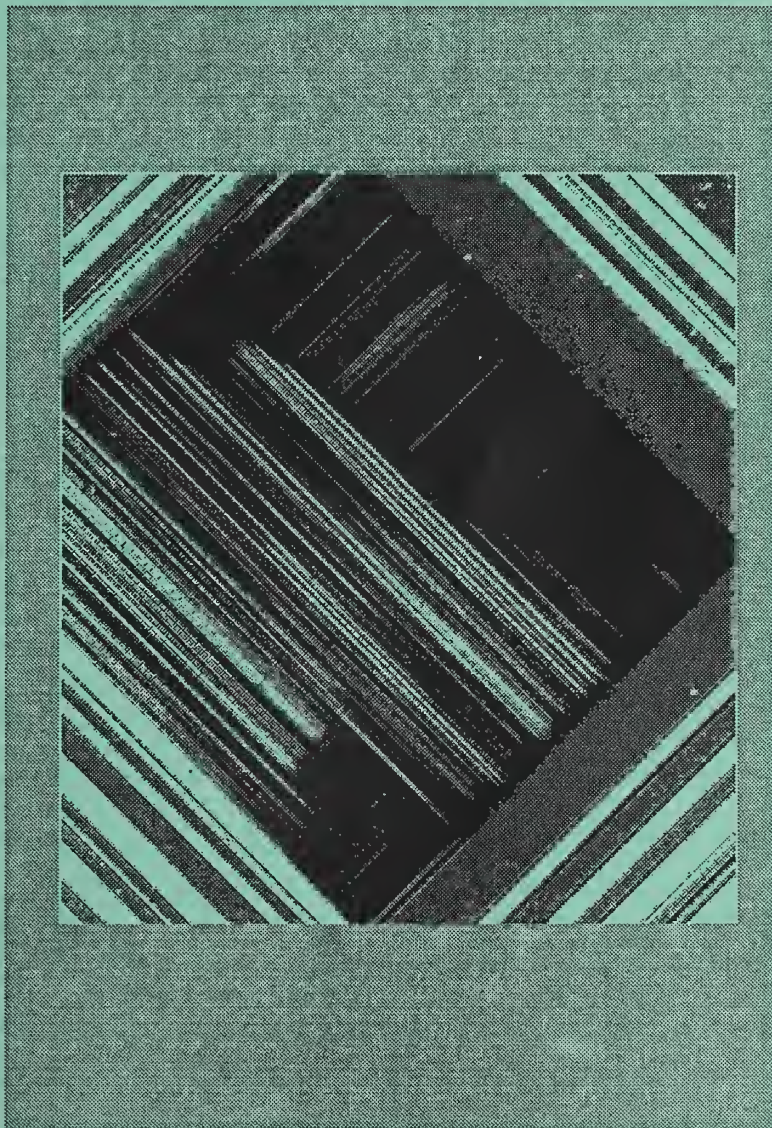
Materials Science and Engineering Laboratory

METALLURGY



NAS-NRC
Assessment Panel
January 31-February 1, 1991

NISTIR 4397
U.S. Department of Commerce
National Institute of Standards
and Technology

Technical Activities 1990

Photomicrograph of a single crystal of $Y\text{-}Ba_2\text{-}Cu_3\text{-}O_{6+x}$ high temperature superconductor material showing twinned structure (Photographed with polarized light). The twinned structure develops during the tetragonal to orthorhombic transition. Wide twin spacing at the corners is due to decreased elastic constraint during the transformation compared with the center of the crystal.

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E.N. Pugh, Chief
J.H. Smith, Deputy

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U.S. DEPARTMENT OF COMMERCE
Robert A. Mosbacher, Secretary

NATIONAL INSTITUTE OF STANDARDS
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Technical Activities 1990

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ABSTRACT

This report summarizes the FY 1990 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 cooperative programs with a professional society (the Corrosion Data Program with the National Association of Corrosion Engineers); with a trade association (the Temperature Sensor Program with the Aluminum Association) and several with industry including the Powder Atomization Consortium with two companies.

The scientific publications, committee participation, and other professional interactions of the 76 full-time and part-time permanent members of the Metallurgy Division and its 47 guest researchers are identified.

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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 radical 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 has evolved 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 eight groups which constitute the Division.

The major programs are conveniently grouped into three categories:

Processing and Process Control

Powder processing continues to be a major 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 Technology 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. 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; earlier studies showed that this laser technique can successfully measure the particle size distribution, and this year the system was 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 optimum conditions correspond to the formation of a hollow shell of liquid metal below the liquid delivery tube; impingement of the gas jets on this thin-walled shell leads to ligament and droplet formation.

Powder consolidation studies have been focused on hot isostatic pressing (HIP) 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 strategies for fabricating difficult materials such as titanium aluminides into near net shape components. NIST's role was first to develop sensors for monitoring densification in real time, and an eddy current technique has been adapted for this purpose. During the past year, effort has focussed on using the densification data to refine process models developed by M. Ashby, Cambridge University, who is collaborating in the program. Collaboration is also continuing with scientists from the BDM Corporation who are using finite element techniques to extend the information generated from experiments on simple cylindrical geometry to the more complex shapes of industrial components.

Other process-control 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 manufacturing processes in a joint project with the Aluminum Association. The method has been used successfully for a variety of extruded products (cylindrical, square and I-beam sections) and for plate and sheet, and investigations are now in progress to determine the influence of variations in alloy composition on the accuracy on the technique. The eddy current method is also being used in a new study of carbon-carbon composites. 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. Work to date has indicated that transmission of sound from the solid is not feasible because of scattering from grain boundaries, and it is clear that sound waves must be reflected from the interface from the liquid. Sensors which can be immersed in the molten metal are therefore being developed.

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 largely with continuous fibers and has focussed on the fiber-matrix interface, which controls the properties of these high performance composites. In general, the matrix and fiber are not in thermodynamic equilibrium during processing or in service, and the Metallurgical Processing Group is studying a range of possible reactions which might reduce the integrity of the interface. These include interdiffusion, phase transformations and grain growth. As part of this study, transmission electron microscopy and experimental determination of the phase diagram are being conducted on the Al-SiC system. 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. In collaborative research with American Cyanamid Company, cobalt-tungsten coatings were found to be effective barriers between graphite fibers and nickel matrices and, during this year, studies have been made of the effects of high temperature annealing on the interfacial strength and on the phases produced in the interfacial region. Work has also continued on the deposition of titanium-aluminum and other intermetallic alloys from fused salts, and a cell has been developed for continuous-fiber deposition from such electrolytes.

In addition to work in the HIP and MMC programs, titanium-aluminum intermetallic alloys are also being characterized in ongoing studies in the Metallurgical Processing Group. Interest in these ordered alloys stems from their great potential as high temperature structural materials for a range of aerospace applications, both commercial and military. Although many of the early promising property reports for intermetallics were obtained with single phase materials, further improvements in properties are anticipated to involve multiphase structures, and this requires the selection of new compositions and heat treatment schedules. For this purpose, a DARPA-supported program is focussing on the determination and/or evaluation of important alloy phase diagrams like Ti-Al-Nb and the determination of the phase transitions available to manipulate the intermetallic microstructure. In addition to studies of several Al-Al-Nb alloys, work this year has also involved Al_2TiTa, an alloy with promising oxidation resistance at high temperatures, and the NiAl-NiTi psuedobinary phase diagram.

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 the corrosion and stress corrosion cracking behavior of iron aluminide.

A new activity in 1990 also centered on intermetallic alloys. This program, supported by the U.S. Army Harry Diamond Laboratory, deals with the influence of intermetallic phases on the performance of solder joints used in the microelectronics industry. Bulk samples of the intermetallics have not been prepared elsewhere, but the Metallurgical Processing Group succeeded in producing both single phase Cu_6Sn_5 and Cu_3Sn. Together with the Mechanical Properties and Performance Group, they have used these unique samples to begin the development of a data base for the physical and mechanical properties of these materials, information which is essential for modeling the behavior of solder joints.

Activity in high T_c superconductors continued in several groups. The development of a thermomechanical process for detwinning YBCO monocrystals by the Metallurgical Processing Group in cooperation with the Ceramics Division has led to a number of studies of the effects of twin interfaces. Thus the Magnetic Materials Group confirmed that twins have a relatively small effect on flux pinning, contrary to earlier conjecture, and also established that the interaction energy between flux vortices and twins change sign near the superconducting transition temperature. The YBCO monocrystals were also used for a comparison of the magnetically-modulated-microwave-absorption (MAMMA) technique and SQUID magnetometry; MAMMA was found to resolve structures not seen by SQUID. In addition to studying mechanical properties of the YBCO monocrystals, the Metallurgical Processing Group is now turning its attention to bicrystals and polycrystals of this material in an attempt to clarify the role of grain boundaries in electrical transport. In a separate program, the High Temperature Materials Chemistry Group has continued its investigation of the formation of thin films of high T_c materials by laser ablation and vapor deposition. The thrust here is to gain insight into the mechanism of the deposition process by probing the plume. This year, time resolved mass spectra from laser-generated plumes from YBCO targets indicated the presence of many neutral and ionic species, including the bimetallic species CuBa and YCu.

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. This year, the program was extended to Co/Cu and Co/Cr multilayers. 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. An exciting development this year was the recognition that these materials may possess giant magneto-caloric effects and therefore have enormous potential for magnetic refrigeration.

Data and SRM Programs

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 1990. 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 development which examine the storage and handling of hazardous chemicals, corrosion in potable water distribution systems, and equipment used in electric power generation under sponsorship of the Materials Testing Institute of the Chemical Process Industries, International Copper Association 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.

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 carried out by the Mechanical Properties and Performance Group in collaboration with ASTM and hardness block manufacturers indicated that U.S. test blocks are not compatible with the presently used international hardness scale. This identified the need for a traceable national hardness standard in the U.S., and plans are being developed to establish a standardizing machine in the Division.

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Coriell, Sam R.	o Modeling of solidification processes o Interface stability o Convection and alloy segregation during solidification
Gayle, Frank W.	o Aluminum metallurgy o Transmission electron microscopy o Structure/property relationships o High T_c superconducting
Handwerker, Carol A.	o Interface studies o Metal matrix composites o Diffusion-induced grain boundary migration
Hardy, Stephen C.	o Alloy coarsening o Surface tension measurements o Interface segregation
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The Metallurgical Processing Group studies the processing of metals and advanced materials to support U.S. industry and government through (1) research into the basic processing mechanisms and process modelling; (2) development of measurement techniques, systems, and data important for process monitoring and control; and (3) characterization of the phases and microstructures of the product materials as a function of processing conditions. Understanding of alloy kinetics and thermodynamics is pursued to provide guidelines which can be used by industry and alloy designers to tailor alloys and processing techniques for their particular applications.

During the past year, work in the group included major efforts on intermetallics, solder joint materials, and powder processing. Titanium aluminide intermetallics were investigated for high temperature applications. Intermetallics that occur in solder joints, such as Cu_6Sn_5 and Cu_3Sn, were investigated for microelectronic applications. In the powder processing area, the third year of a successful collaboration with industry on atomization processes included achievement of automated control in the NIST supersonic inert gas metal atomizer (SiGMA). This work is directed toward development of intelligent processing systems for atomization. In similar work, real-time studies of densification during hot isostatic pressing of alloy powder have allowed design of more efficient pressing cycles.

Work on the application of processing principles to specific materials was done in response to requests by industry groups and other agencies. For example, funding was supplied by DARPA for work on intermetallics; by the Harry Diamond Laboratory for solder work; by an industrial consortium, NASA, and the David Taylor Research Center for atomization work; by NASA for work on directional solidification and alloy coarsening; by industry for studies of aluminum alloys; and by the Office of Naval Research for work on composites and interface reactions. Cooperation also continued with national efforts on high T_c superconductors.

FY 90 Significant Accomplishments

- Microstructural studies have been conducted by analytical electron microscopy techniques on ultra-high strength aluminum-copper-lithium weldable alloys. The ultra-high strength was found to arise from a fine dense precipitation of at least three different types of strengthening phases in these alloys.

- The Metallurgy Division's SiGMA system (Supersonic inert Gas Metal Atomization) has been used to produce fine spherical metal powder (50% by weight ≤ 20 μm) of materials that are difficult or impossible to produce by ingot techniques. New types of materials produced included nitrogenated steels and Cu-Sn intermetallic compounds that occur in solder joints.

- Wettabilities of tin-lead solder on fine-grained bulk samples of Cu_6Sn_5 and Cu_3Sn intermetallics and also on copper were measured in sessile drop experiments. Since such bulk intermetallic samples have not been produced elsewhere, these measurements were a first-of-their-kind. The Cu_6Sn_5 was always found to have the poorest wetting characteristics of the three materials.

- The U.S. Industry/NIST consortium on in-situ particle size measurement and control during gas atomization has successfully completed the planned three year study. The resulting particle size distribution sensor and expert system driven controller provide the necessary sensor feed-back signals to effect real-time control of the gas atomization system.

- Based on experimental results, criteria for the occurrence of diffusion-induced recrystallization at composite interfaces were developed. These criteria are concerned with stress relaxation in the diffusion zone and dislocation densities near the interface.

- Isothermal sections of the Al-Cu-Fe phase diagram at 700 and 800°C have been experimentally determined in the region of the face-centered icosahedral phase. The equilibrium composition range of the icosahedral phase has been delineated, which should facilitate studies of defect structures and structure of this unique, phason-free quasicrystalline phase.

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

Automation -- Real-Time Powder Particle Size Measurement and Control - The NIST/Industrial consortium on powder processing has completed a scheduled three year multi-disciplinary study on automation utilizing the Metallurgical Processing Group's supersonic inert gas metal atomizer (SiGMA). NIST research groups contributing to this project include the Intelligent Control Systems Group of the Factory Automation Systems Division, the Fluid Flow Group and the High Temperature Reacting Flows Group of the Chemical Process Metrology Division and the Metallurgical Processing Group of the Metallurgy Division. The NIST scientists have collaborated in this work with industrial representatives from Crucible Materials Corp., General Electric Co., and Hoeganaes Corp.

This activity included the incorporation of an in-situ particle size measurement sensor in an advanced process controller utilizing Artificial Intelligence (AI) techniques. The Fraunhofer diffraction particle size sensor uses adaptive pattern recognition software to resolve scattered laser intensity data into a distribution of 14 different sizes between 5 and 150 μm. This is done at rates exceeding 2 hz. The controller runs in an "expert system" shell which processes sensor data and sends appropriate actuator signals. The system is designed to maintain or change the particle size distribution as directed by the heuristic data base and response functions "learned" during previous programing sessions. These studies, previously conducted on 304 stainless steel, were extended to include Ni based superalloys and tool steels.

Fluid flow studies included detailed velocity and pressure profiles of the gas jets used to produce the ultra-fine (< 45 μm) powder in the SiGMA facility, high speed movies of Ni based superalloys and 304 SS atomization, and mathematical modeling of supersonic gas flow. Careful frame-by-frame examination of the high speed 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 primary droplets form as the sheet thins and holes-through. The current understanding indicates that the sheet formation phase is an essential component for stable liquid-gas interaction and production of fine powder product.

<u>HIP Consolidation of Metal Powders</u> - Hot isostatic pressing (HIP) is one of the primary methods used for consolidation of metal powders into solid parts. It is especially important for materials such as intermetallic compounds, metal-matrix composites, and high-temperature alloys which are difficult to fabricate by conventional casting, forging, and machining processes. In a program sponsored by DARPA, the Metallurgy Division has worked with BDM International, the University of Cambridge, and an Industrial Consortium to develop techniques for applying the intelligent processing of materials (IPM) to HIP consolidation of metal powders of titanium and titanium aluminide. In an additional project, sponsored by the Harry Diamond Laboratory, intermetallic phases which normally form as minute precipitates in solder joints are formed into bulk samples by HIP consolidation of atomized powders.

In the intelligent processing project, eddy-current sensors are used to measure the dimensional changes of metal powder samples within the HIP chamber, and the measured behavior is compared to that predicted by a process model. These measurements have revealed several ways in which the present process model can be enhanced to more accurately describe the behavior of the HIP samples. This capability will allow design of more efficient pressing cycles. A new mechanism of densification, related to a phase change in the titanium aluminde powder, was discovered.

A critical part of the sample behavior is the change in shape which occurs as the sample densifies. To accurately predict this shape change, it is necessary to understand the response of a powder compact to shear stresses as well as to hydrostatic pressure. For this purpose, a theoretical model of the deformation behavior of partially densified powders has been developed, and an extensive series of mechanical tests has been carried out on partially densified material. The shape change of cylindrical samples with different combinations of can materials and powders has been measured under a variety of pressure and temperature conditions, and the results have been compared to predictions obtained by finite-element calculations using different constitutive relations. This work has not only revealed the deformation behavior of the powder materials; it has also demonstrated that the mechanical properties of the canning material play a critical role in the determination of shape change.

In the copper-tin project, special HIP techniques were developed to make possible consolidation of the intermetallic phase powders without remnant porosity or cracks. These techniques included the use of a liner in the HIP can which eliminated residual tensile stresses as the sample cooled from the

HIP temperature and a pre-compaction of the powder into pellets which had enough green strength to preserve the shape defined by the HIP can. The resulting samples, with a fine grain structure and a minimum of defects, have been used for a variety of property measurements at NIST and have been provided to many outside investigators. The samples of Cu_6Sn_5 intermetallic prepared by these techniques have played a central role in a national research effort on this material, since they are the only bulk samples of this material currently available.

Nitrogen as an Alloying Addition in Gas Atomization - One of the limiting factors in the application of nitrogenated stainless steels is the difficulty of incorporating a controllable, homogenous concentration of nitrogen into the metal. Current methods include melting and casting under high pressure gaseous nitrogen or adding nitrogenous materials to the slag during electroslag remelting. A processing route which had not been previously explored is to entrain nitrogen during gas atomization and consolidate the resulting powder. By using this approach, the near-net-shape capabilities of powder metallurgy can be combined with the improved properties of nitrogenated austenitic stainless steels. Nitrogenated stainless steels are candidate materials for applications where moderately high strength, good corrosion and oxidation resistance, and high toughness are required of non-magnetic alloys.

Fully dense nitrogenated austenitic stainless steels were produced by gas atomization in the SiGMA atomizer and HIP consolidation. The base alloy, 304L, contained about 0.15 wt.% nitrogen when atomized under optimum conditions, and a modified version of 304L with 23 wt.% Cr contained 0.206 wt.% nitrogen. A series of experiments using various combinations of N_2 and Ar as the melt chamber backfill gas and atomizing gas demonstrated that the nitrogen content of the powder was controlled by the backfill gas and that the fraction of hollow particles was determined by the atomization gas. The elimination of voids in the powder spheres by nitrogen atomization in itself has significant practical implications.

Hardness and compression behavior (yield strength and flow stress) are substantially improved with the addition of nitrogen. Mechanical property studies of the consolidated alloys done at NIST indicate that the properties of nitrogenated stainless steels fabricated in this manner are comparable to other high nitrogen austenitic alloys.

Physical Metallurgy and Processing of Intermetallics and Advanced Aluminum Alloys

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Research in this task is concerned with improvements in the metallurgical data base for intermetallic and advanced aluminum alloys. This information permits a rational approach to the control of microstructure and properties through processing. Phase equilibria and phase transformation studies on a broad range of titanium aluminides containing Nb have been studied experimentally and thermodynamic ternary phase diagram modeling has been performed. Alloys in this system containing hexagonal Ti_3Al, orthorhombic phase and BCC have demonstrated a good balance of room temperature ductility and high temperature rupture strength. Other aluminide systems, NiAl-NiTi, Ta-Al and Ti-Al-Ta are also being studied. The precipitates responsible for the exceptional properties of advanced Al-Li-Cu alloys have been characterized.

Aluminum-Lithium Alloys - A new class of ultra-high strength, weldable aluminum-copper-lithium alloys was recently developed by Martin-Marietta Laboratories. These alloys, originally targeted for the Advanced Launch System, have twice the yield strength of the baseline alloy (AA-2219) used in the Space Shuttle External Tank. Research at NIST has been directed at determining the microstructural basis for high strength and toughness and other physical, chemical, and mechanical properties of these alloys. It has been found that the system contains a variety of strengthening precipitate phases, 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 possible new phase. Metastable phase diagrams have been constructed for the Al-Cu-Li system which are relevant to precipitation sequences encountered during processing of commercial alloys. These diagrams suggest that the new, low Cu, high toughness variants of the alloy are close to the optimal composition with regard to strength and toughness capability. The θ'/S' balance has been found to vary with changes in Cu and Li content. Grain boundary precipitation appears to be associated with enhanced toughness at cryogenic temperatures.

Phase Equilibrium in Alloys of Composition Al_2TiTa - Recently research at Lockheed and Pratt and Whitney has demonstrated that the composition Al_2TiTa has good oxidation resistance at temperatures approaching 1500°C. The ternary phase diagram of this system is not known. In order to provide a sound data base for heat treatment and further alloy development, the phases present in this alloy between 1200 and 1550°C have been determined by scanning electron microscopy/x-ray energy dispersive spectroscopy methods. The solidus for this alloy is ~ 1575°C. Below the solidus the alloy lies in a broad BCC solution phase region that extends from the BCC Ti,Ta solution phase. Between 1500 and 1425°C, the alloy consists of BCC and HCP. The HCP phase region extends from the Ti HCP phase. At 1400°C the alloy consists of BCC, HCP and σ phases. The σ phase extends from the binary σ phase based on the composition Ta_2Al. Between 1375° and 1350°C the alloy is two-phase HCP and σ. At 1330°C the alloy is composed of three phases: HCP, σ, and γ. The γ phase extends from the binary TiAl phase. Below 1325°C the alloy consists of σ and γ.

Ordered Omega Phases in Ti-Al-Nb Alloys - A composition region containing stable and very brittle omega phase has been located in the Ti-Al-Nb system. The existence of this phase appears to limit the amount of Nb that can be added to γ-TiAl to improve toughness. During cooling of the composition Ti_4Al_3Nb from a B2 phase field above 1100°C, a metastable trigonal (P3m1) ω-related phase, designated ω'', forms along with small amounts of DO_{19} and Ll_0 phases. The ω'' phase exhibits partial collapse of (111) planes and reordering relative to its B2 parent. An equilibrium low temperature phase with the $B8_2$

structure was found after 26 days at 700°C. Both ω'' and $B8_2$ structures were verified by means of TEM and by single crystal X-ray diffraction. The observed transformation path, B2(Pm3m) → ω''(P3ml) → $B8_2(P6_3/mmc)$, occurs in two steps. The first involves a subgroup transition during cooling that is primarily displacive with reordering consistent with the trigonal symmetry imposed by the ω-collapse. The second involves a supergroup transition during annealing that is primarily replacive and constitutes a chemical disordering.

The Crystal Structure of the Ti_2AlNb Orthorhombic Phase - The results of a neutron diffraction study confirm and refine the structure of the orthorhombic phase in the Ti-Al-Nb system. The structure is Cmcm (HgNa or Cd3Er) with a= 6.0893, b= 9.5694, and c= 4.6666. Ti(Nb) fills the 8g site, Al fills one 4c site, and Nb(Ti) fills another 4c site. The structure involves ternary ordering of the hexagonal DO_{19} phase. The ordering causes a break of the hexagonal symmetry and corresponding distortion of the unit cell; viz., contraction of the b and expansion of the a parameters. The c parameters of the two phases are essentially identical. Compared to a random mixture of Ti and Nb on the Ti sites of α_2 DO_{19} Ti_3Al, the ternary ordering, coordinate change, and distortion to the orthorhombic phase permit an increase of Al-Nb distances from 2.85-2.89 Å to 3.02-3.06 Å while slightly reducing the Al-Ti distances to 2.80-2.85. These distances are consistent with estimates of L-J potentials for Al-Ti and Al-Nb which yield minima at interatomic distances of 2.79 and 2.92 Å.

BCC to Orthorhombic Phase Transformation in the Ti_2AlNb Alloy - For the Ti_2AlNb composition, the B2 ordered bcc phase exists at high temperature, and the orthorhombic phase is the low temperature phase. The transformation between the two phases is martensitic. The observed platelet microstructure of the orthorhombic phase is the result of structural accommodation to minimize elastic energy. The transformation is accompanied by chemical ordering which introduces additional defects. The defect structure of the plates can be removed by subsequent recrystallization of the faulted orthorhombic phase into fault-free grains.

Coherent Precipitates in the BCC/Orthorhombic Two-Phase Field of the Ti-Al-Nb System - Alloys of composition Ti_2AlNb and Ti_4AlNb_3, cooled from 1400°C and equilibrated at 700°C for 26 days, were both found to consist of two phases: the Ti- and Nb-rich bcc phase and the orthorhombic phase based on Ti_2AlNb. Each phase was observed in a plate morphology in the matrix of the other phase depending on the alloy. In both cases the phases share a common direction, $[011]_{bcc} \parallel [001]_{ort}$, and interface (habit) plane, $(211)_{bcc} \parallel (110)_{ort}$. Geometrical patterns of plates of the different orientational variants were also observed. Analysis of the orientation relation, habit plane and plate patterns are consistent with the concept of a strain-type transition even though long range diffusion is required.

Crystallography and Thermodynamics of Polydomain Structure in the $\alpha_2 + Ti_2AlNb$ Two Phase Field of Ti-25at%Al-12.5at%Nb Alloys - For this alloy the three phases, high temperature BCC and low temperature ordered hexagonal α_2 (DO_{19}) and orthorhombic Ti_2AlNb, are involved in equilibrium microstructures. The

Ti-25Al-12.5Nb (at%) alloy upon cooling from the ß phase field exhibits a complex transformation path which depends on cooling rate. The last stages of the transformation - from the α_2 to α_2 + Orthorhombic phases - were investigated. Characteristic of the transition is a small transformation strain between the two phases which can be accommodated by platelet form and symmetric arrangement of rotational domains of the lower symmetry orthorhombic phase.

Ti-Al-Nb Phase Diagram Calculation - In order to interpolate and extrapolate the above data and those of other researchers to other compositions and temperatures in a thermodynamically consistent manner, calculations for the Ti-Al-Nb system were done. An isothermal section at 1200°C is shown in Figure 1. The description of the Nb-Al binary recently developed at NIST was used together with the descriptions of the Ti-Al system by Chang and co-workers and Nb-Ti by Kaufman and Nesor.

From experiments it is known that the intermetallic compounds which exhibit wide ranges of homogeneity in the Nb-Al and Ti-Al binaries also reveal wide ranges of homogeneity in the ternary Nb-Ti-Al system. Also, the isotypic compounds $NbAl_3$ and $TiAl_3$ form a continuous solid solution. In order to model the ternary ranges of the intermetallic compounds Nb_3Al, Nb_2Al, Ti_3Al and TiAl they must be described as compounds in the other systems. Assigning these compounds in the other binaries Gibbs energies sufficiently positive to ensure that they are metastable results in solubilities in the ternary system that are too small. Therefore, ternary terms were introduced in the analytical description for the final adjustment.

The calculated and experimental diagrams agree well in the Nb-rich corner of the 1200°C isothermal section. The disagreement in the Ti-rich corner is most likely caused by the fact that the B2 phase is not yet considered in the calculation. Attempts at fitting the relatively wide ternary range of homogeneities of $(Nb,Ti)Al_3$ were not successful, probably because of the extremely narrow width of this phase in the Nb-Al and Ti-Al binaries. Therefore, the analytical descriptions of the binary systems Nb-Al and Ti-Al should be further refined before a final adjustment of the ternary parameters is attempted.

Theoretical Studies of the NiAl-NiTi Pseudobinary Phase Diagram - Ab initio calculations have been performed of the NiAl-NiTi pseudobinary phase diagram. The calculations compared Linear Muffin Tin Orbital (LMTO), and Linearly Augmented Plane Wave (LAPW) total energy calculations, both of which were combined with Cluster Variation Method (CVM) phase diagram calculations. The results of these calculations indicate that the characteristic conditional-spinodal/symmetrical-tricritical-point phase relations of this system derive from both long-range strain energy effects and surprisingly strong repulsive third nearest-neighbor (3'rd nn) Al-Ti pairwise interactions. The 3'rd nn interactions are actually predicted to be more important than the long-range interactions in this case. This result is supported by both the LMTO and LAPW band theory calculations which yield almost identical results. The presence of a strong 3'rd nn Al-Ti repulsive interaction in Ni-Al-Ti BCC based alloys indicates that descriptions of BCC based M-Al-Ti alloys which ignore 3'rd nn interactions are fundamentally inadequate.

Ta-Al Phase Diagram Calculation - The equilibrium phase diagram of the Ta-Al system reveals seven equilibrium phases, liquid, (Ta), Ta_2Al, TaAl, $TaAl_2$, $TaAl_3$ and (Al). All solid compounds, except (Ta), melt incongruently. For the calculation of the Ta-Al system the liquid, the bcc (Ta) and fcc (Al) phases were described using regular solution type models. Three polynomial terms for the enthalpy and two for the entropy were used for the liquid, one polynomial term for the enthalpy and one for the entropy each were used for the bcc (Ta) phase and of fcc (Al). Ta_2Al is the only intermetallic compound revealing a wide range of homogeneity and was therefore modeled with the Wagner-Schottky model using three sublattices. The compounds TaAl, $TaAl_2$ and $TaAl_3$ were assumed to be stoichiometric. For the calculation of the Ta-Al system the lack of experimental data is a major obstacle. The result of the calculation of the Nb-Al system was used as a guideline for evaluating the available experimental data. These evaluated, as well as estimated, values have been used to derive the analytical description of the Ta-Al system.

Aluminum-Copper-Iron Phase Equilibria - The Al-Cu-Fe ternary system is of technological importance (e.g., aluminum bronzes, Fe-Al high temperature intermetallics, etc.) as well as of fundamental scientific interest. As an example of the latter, the most perfectly ordered quasicrystal to date appears to be an equilibrium phase of approximate composition $Al_{64}Cu_{25}Fe_{11}$. The phase is based on a 6-D face-centered cubic lattice, in contrast to other icosahedral phases which are simple cubic (6-D). Very little is known regarding a large region of the ternary phase diagram surrounding the icosahedral phase. This region (50-75at.% Al, 0-25% Fe) is presently being investigated experimentally. Isothermal sections have been determined at 700°C and 800°C, and all phases in equilibrium with the icosahedral phase have been identified. The range of solubility of the icosahedral phase has been determined at these temperatures. This will facilitate the study of non-stoichiometric defect structures and location of the atoms in quasicrystalline phases.

Processing and Properties of Special Materials

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A guideline in the NIST work on metallurgical processing is 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 are being pursued. Predictive modeling and experimental testing of the effect of processing conditions on microstructure is done and then the effect of microstructural distributions on properties is determined. Investigation on steels, intermetallics, and aluminum alloys were described in the previous two sections of this report. In addition, work on solder joint materials, electronic materials and high T_c superconductors is being done. Accomplishments in these work areas are described here.

Physical Properties of Solder Joints Intermetallics - The intermetallic phases formed during various joining operations in the microelectronics industry are widely considered to affect joint properties. In order to provide a physical and mechanical properties data base for intermetallics found in solder joints and in pretinned leads, single phase samples of the intermetallics, Cu_6Sn_5 and Cu_3Sn, were synthesized. Metal flake or powder made by rapid solidification, either melt spinning or atomization, were consolidated into bulk form by hot isostatic pressing (HIP). The resulting samples, in the form of fine-grained polycrystalline rods up to 2.5 cm in diameter, have been cut into sections for physical property measurements. Samples were supplied to the Mechanical Properties and Performance Group for mechanical testing.

To assess wettability, area-of-spread tests of molten solder were performed on these intermetallic samples and also on pure Cu using a non-activated and an activated rosin flux. The Cu_6Sn_5 was always found to have the poorest wetting characteristics of the three materials. Figure 2 shows photographs of the six tests.

Solidification of Alloys - During solidification of a binary alloy, temperature and solute gradients are inherently present, and cause a density gradient in the melt. The action of a gravitational field on this density gradient can give rise to fluid flow in the melt with consequent redistribution of solute. This leads to solute inhomogeneities in the solidified material, and thus affects its properties. In collaboration with the NIST Center for Computing and Applied Mathematics, the conditions for the onset of convective instability have been calculated for the directional solidification of a binary alloy vertically upwards at constant velocity. For a range of processing conditions, the thermal Rayleigh number is sufficiently small that the stabilizing role of the thermal field may be neglected, and only solutal convection need be considered. The effect of a time-periodic vertical gravitational acceleration (or equivalently vibration) on the onset of solutal convection has been calculated based on linear stability using Floqet theory. A stable base state can be destabilized due to modulation, while an unstable state can be stabilized. The flow and solute fields show both synchronous and subharmonic temporal response to the driving sinusoidal modulation.

In order to understand the interaction between fluid flow and the crystal-melt interface, a linear stability analysis has been carried out for the axisymmetric Taylor-Couette instability of the flow between infinite coaxial rotating cylinders for the case that one of the bounding cylinders is a crystal-melt interface. Results have been obtained for various rotation ratios of the outer and inner cylinders in the narrow gap approximation. For solid body rotation of the melt, the linear stability of the system is unaffected by the crystal-melt interface. For the inner cylinder rotating more rapidly than the outer cylinder, the Taylor-vortex mode is destabilized for large Prandtl numbers, and the critical Taylor number varies inversely with the Prandtl number. In the limiting case of small Prandtl number the results tend to classical values for a rigid-walled system.

Al-In-Sb Phase Diagram Calculations - Group III-V compound semiconductors offer a wide range of electrical and optical properties that are often superior to those of elemental silicon. The ability of these compounds to form miscible solutions among themselves provides a solid state device designer with a virtually continuous spectrum of properties from which to choose. Since many of the processing steps, such as liquid-phase epitaxy and bulk crystal growth, involve contact of liquid and solid phases of these materials, phase diagram data are important for specifying equilibrium boundary conditions. An assessment of the thermochemistry and phase diagram data for the Al-In, Al-Sb and Al-In-Sb systems has been conducted. The regular solution type model was used to describe liquid of the Al-In and Al-Sb system. The solid phases were assumed to be stoichiometric. The analytical description of the In-Sb system was taken from the literature. The ternary description using only the binary parameters gave a very reasonable fit to the available Al-In-Sb data. Two ternary parameters were determined from optimization of the ternary data and the resulting Al-In-Sb description agreed excellently with the ternary data. The Al-Sb system was also modelled successfully using the associate model for the liquid and the Wagner-Schottky model to describe the extremely small range of homogeneity of the AlSb compound.

High Tc Superconductors - Detwinned Single Crystals - The development of a thermomechanical process for detwinning $YBa_2Cu_3O_{6+x}$ single crystals has presented opportunities for numerous characterization studies of single crystals without the complication of twinning defects. Comparative investigations on twinned and detwinned crystals were conducted to elucidate the effect of twin boundaries on properties and to determine anisotropy of properties. Magnetic measurements have yielded the first direct evidence in any high temperature superconductor of an effect of twin boundaries on flux pinning. Furthermore, twin boundaries were shown to be weak pinning centers at low temperature (10 K).

Indentation measurements on twinned crystals have revealed that indentation cracks propagating perpendicular to the c-axis are 1.5-2.0 times as long as those running parallel to the c-axis, indicating a significant anisotropy in fracture toughness. Fracture toughness in twinned crystals was approximately 20% higher than in detwinned crystals, suggesting that twin boundaries act as toughening defects. Structure determination studies on a detwinned crystal with a superconducting transition temperature (T_c) of 89 K have revealed that the oxygen atoms in the O(4) chain sites are offset from the crystallographic b-axis, leading to zig-zag rather than linear Cu-O chains. The results of these characterization studies may be useful in applications development and in understanding the mechanism of high temperature superconductivity.

High Tc Superconductors - Bicrystals - The role of grain boundaries in electrical transport in polycrystalline samples of the high temperature superconductor $YBa_2Cu_3O_{6+x}$ (YBCO) is not well defined although an understanding of this role is a key element to the application of these materials. In this program, one class of grain boundaries in bi- and multi-crystals has been characterized with respect to structure and interaction with twin boundaries. Facetted grain boundary segments in multi-crystals having nearly coincident c-axes ([001] tilt boundaries) were characterized by optical microscopy techniques. Grain boundary orientations were widely distributed, with a slight favoring of {110}, {310} and {510} boundary planes. Most importantly, all grain boundaries, except those far from the symmetric condition and those with {110} facets, exhibited well-developed matching of the twin domain structures across the boundary, as seen in Figure 3. It is suggested that this newly-reported phenomenon of twin pattern matching occurs due to a local coordination of the tetragonal to orthorhombic transformation strain across grain boundaries and may be beneficial with regard to electrical transport in highly-textured polycrystalline material.

<u>Composites and Interface Reactions</u>

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In composite systems, the matrix and the fiber or particulate reinforcements will not in general be in thermodynamic equilibrium during processing, or in service. As a result of interdiffusion and phase transformations at composite interfaces, interface integrity can be undermined through large scale changes in the interface roughness, stress discontinuities along the interface, and changes in the load transfer from the matrix to the reinforcing agent. In the Metallurgical Processing Group, five general topics are being investigated, both theoretically and experimentally, in connection with interface reactions that can affect composite properties: (1) stability of two phase composites to grain growth; (2) plastic deformation and recrystallization resulting from interdiffusion; (3) principles governing solid-state reactions in the Al-SiC system, a model system for practical metal-matrix composite systems; (4) the effect of interfacial anisotropy on the motion and shape of reacting interfaces; and (5) coarsening of solid-liquid alloy mixtures.

<u>Stability of Two-Phase Composites</u> - A model for the stability of two-phase microstructures was developed for the case of motion by mean curvature, that is, for grain growth. In particular, this theory has been used as a basis for examining the stability of various structures to increases in the grain sizes of the two phases and to de-mixing of the two phases. The principal variables in this model are the grain boundary and interphase interfacial energies and volume fractions of the two phases. Specific microstructural features and growth kinetics derived from this model have been reproduced in Monte Carlo simulations of grain growth and are now being matched with two-phase experimental systems.

<u>Interdiffusion Effects</u> - Stresses created by diffusion can destabilize originally planar solid-liquid interfaces and solid-solid grain boundaries when solute diffusion leads to stress in the diffusion zone. Self-stresses accompanying interdiffusion and concomitant interface destabilization are observed in many multi-component metal systems, such Mo-Ni-Fe and Fe-Zn alloys, and multi-component ceramic systems, such as Al_2O_3-Cr_2O_3. Depending on the magnitude of the stress, the temperature, and the diffusion conditions in the liquid, a range of microstructures can be formed, from gross interface roughening with a "sinusoidal" interface to grain size refinement to new

grains formed by recrystallization. The critical conditions for the transition from the "sinusoidal" interface morphologies to recrystallization in the diffusion zone were determined using model experiments in the Mo-Ni and Mo-Cu-Fe system. Based on these experiments, criteria for the occurrence of diffusion-induced recrystallization were established as follows: (1) the stress produced by diffusion must be relaxed by plastic deformation in the diffusion zone, and (2) the local dislocation density in the solid adjacent to the interface must not be reduced below a critical level in order that new grains nucleate.

Reactions in the Al-SiC System - In the Al-SiC system, the differences in reactivity and microstructure between solid Al-SiC and liquid Al-SiC are being identified through transmission electron microscopy (TEM) studies of composites annealed between 500°C and 900°C. The slow reaction rate between Al and SiC makes it a model system for evaluating wettability of ceramics by molten metal, interface roughness and facetting generated by diffusion, phase connectivity, and orientation relationships between nucleating phases and the SiC substrate. Recent experiments indicate that only one carbide phase, Al_4C_3, forms during reaction of Al with SiC at temperatures below 900°C. In addition to this study, the high temperature phase diagram is also being determined experimentally in order to identify composition and temperature ranges over which Al and SiC remain the only stable phases after diffusion.

Motion of Interfaces - Kinetic models of interface motion have been reexamined in light of modern mathematical techniques for finding solutions to the partial differential equations. The applicability of a specific technique depends upon the starting partial differential equation, the initial interface shape, the velocity equation, and orientation dependence of surface energy functions. In the past year the method of characteristics was found to produce solutions to many different classes of problems, including interface-limited crystal growth, diffusion-induced grain boundary migration (DIGM), migration of thin liquid film (LFM) and discontinuous coarsening, but could not handle curvature-driven interface motion.

Coarsening of Solid-Liquid Alloy Mixtures - Previous studies of the coarsening of solid Sn particles in Sn-Pb eutectic liquid performed in this laboratory, although in general agreement with many of the theoretical features of modern coarsening theory, measured coarsening rate constants significantly higher than predicted using the accepted values of the liquid diffusion coefficient and the liquid-solid surface tension. However, the uncertainty in the measured values of these thermophysical constants is significant and could possibly account for the discrepancy between the measured and predicted rate constants. Thus, an independent and accurate measurement of these properties is essential to make a quantitative comparison of experiment and theory.

The development of grooves at grain boundary intersections with solid surfaces in contact with liquid provides a method of making these measurements. This technique actually measures a grooving rate constant which is the product of the diffusion coefficient, the surface tension, and other materials properties and is identical to the coarsening rate constant in the limit of low volume fraction solid to within a well defined numerical constant.

This technique has been used to study grooves in Sn surfaces in contact with Sn-Pb eutectic liquid. The measurement required the production of samples with grain boundaries normal to the surface and widely separated. After annealing at the eutectic temperature, the samples were quenched, sectioned, and metallographically polished. The shapes of the grooves were measured using optical microscopy and compared to theoretical groove shapes. The measured grooving rate constant was in excellent agreement with the value calculated using the accepted values of the thermophysical constants. This suggests that the discrepancy between the measured and theoretically predicted coarsening rate constants should not be attributed to an error in the values of the thermophysical properties used to make the comparison.

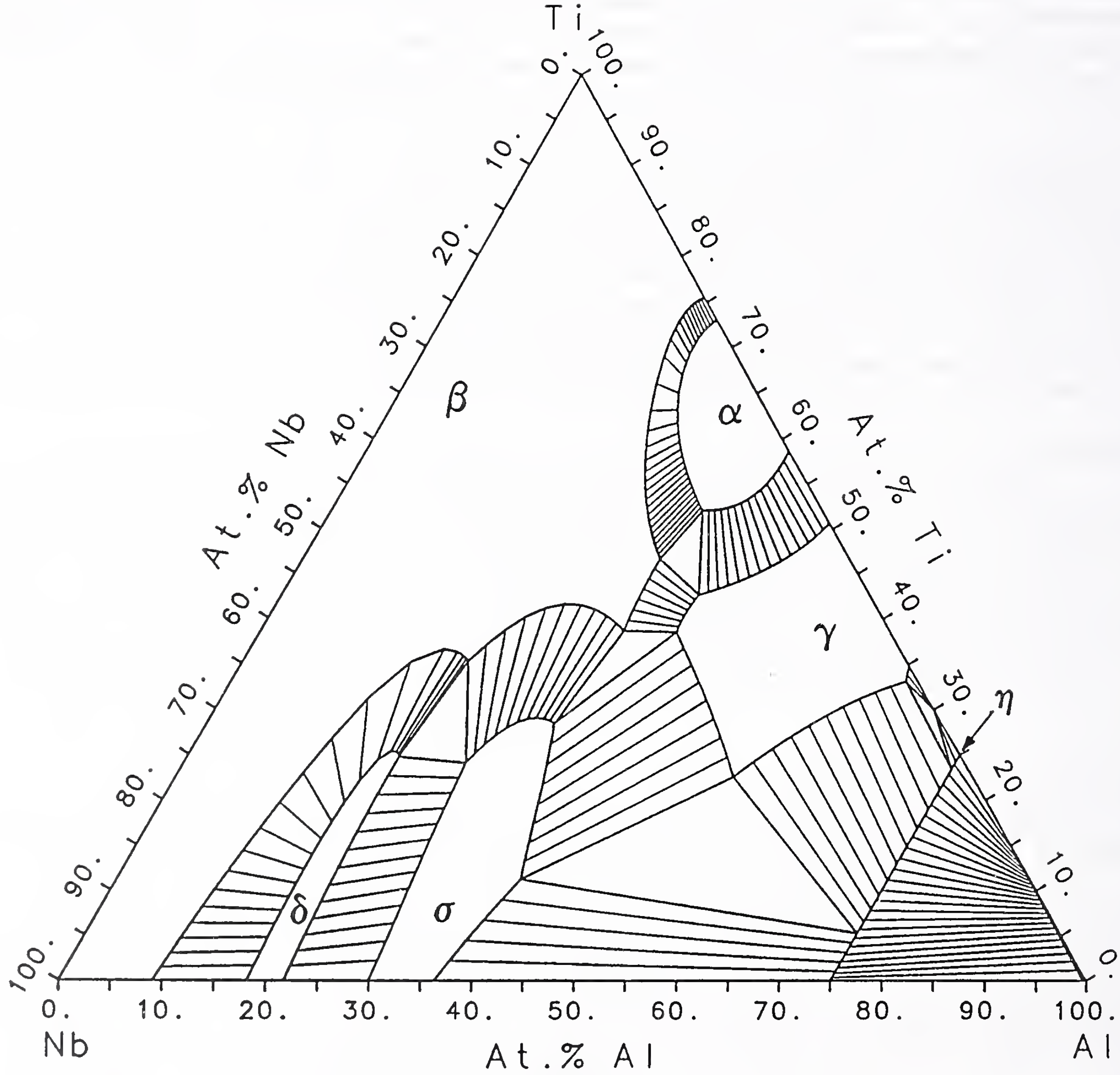

Figure 1. Calculated isothermal section at 1200°C of the Nb-Al-Ti system.

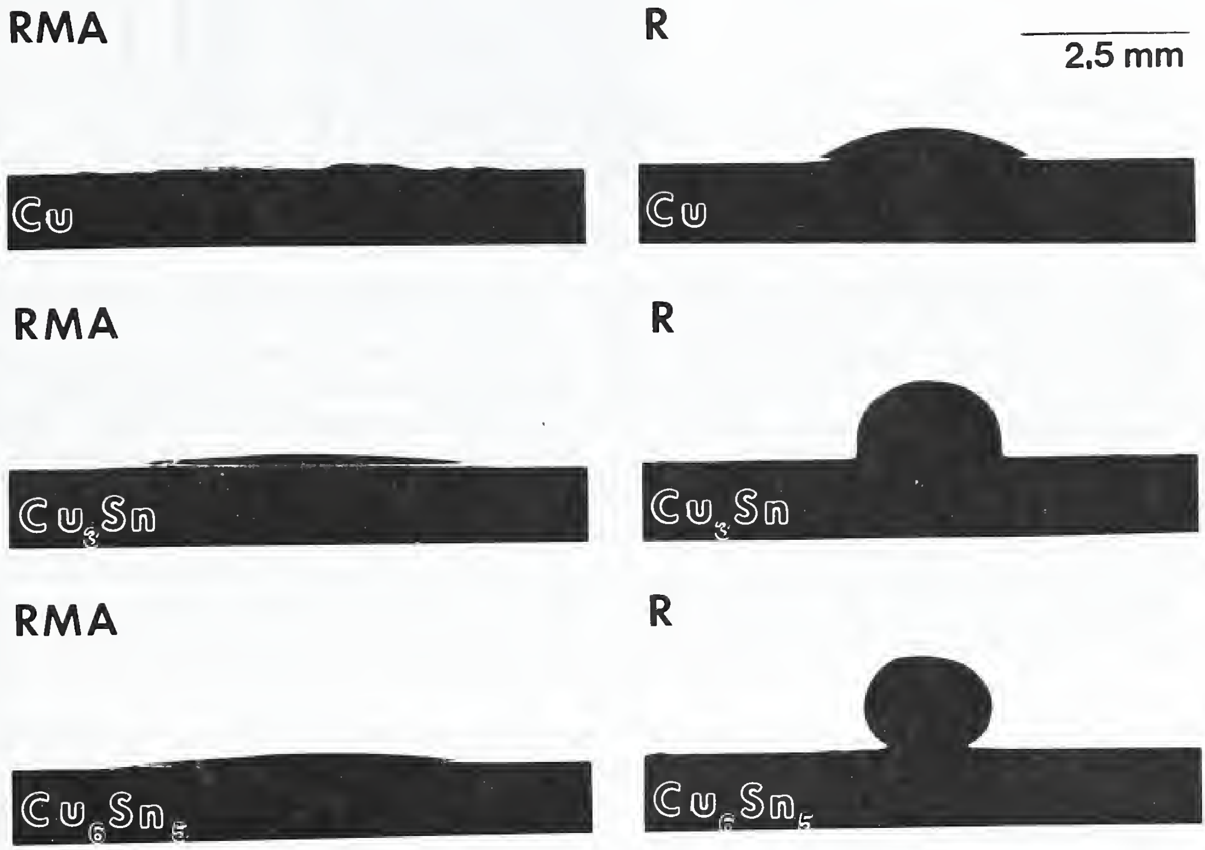

Figure 2. Side profile views of molten solder (60% Sn - 40% Pb) sessile drops on three different substrates, Cu, Cu_3Sn, and Cu_6Sn_5. Wettabilities can be determined from the contact angles. When the strong RMA flux is used to help remove oxides from the substrate surfaces, wetting is much better than when the weaker R flux is used.

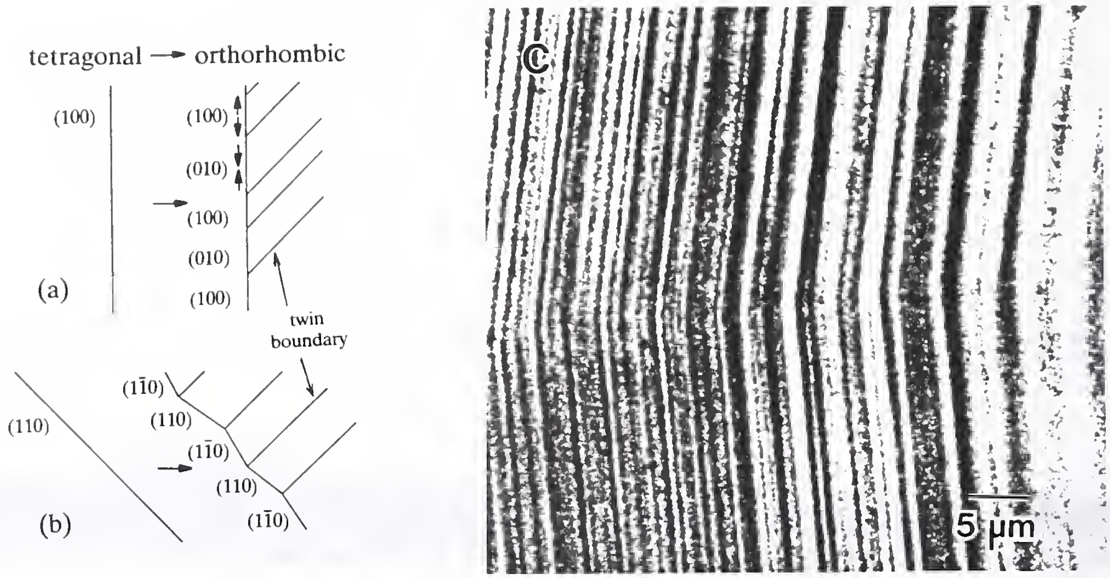

Figure 3. Schematic diagram of transformation strains and twinning in the high-T_c superconductor $YBa_2Cu_3O_{7-x}$ during the transformation from the tetragonal to orthorhombic phase. Unconstrained (100) and (110) planes or grain boundaries in the high-temperature tetragonal phase undergo linear expansion/contraction (a) or pure rotation (b), during the phase change. Transformation twins develop to minimize overall shape change. A coordination of shape change across grain boundaries with orientations similar to case (a) (but not case (b)) results in continuity of twin structures across grain boundaries, as in shown in (c). The crystallographic "micro-texture" which develops may be important for current transport in highly textured, bulk superconductors.

ADVANCED SENSING H. Thomas Yolken

The mission of the Advanced Sensing Group is to enhance the science of nondestructive evaluation sensing methods and materials process modeling and apply this capability to the needs of U. S. industry. These needs fall into several classes: non-invasive techniques of sensing for process control, methods for in-situ observation of materials phenomena for process modeling, and process modeling for process improvement and control.

A major thrust of the Group is in support of the NIST major program on intelligent processing of materials (IPM). This concept couples process models with on-line NDE sensors for monitoring the evolution of microstructure or other internal properties of the materials, 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.

Projects include a cooperative project with the General Dynamics Corporation to develop eddy current sensors for monitoring the processing of carbon/carbon composites at high temperatures. A new collaborative effort between the Group and the NIST Materials Reliability Division to develop an ultrasonic imaging system capable of imaging a variety of defects in thick section fiber-epoxy composites has been initiated. In this jointly funded DARPA/NIST project the Materials Reliability Division will develop hardware and the Advanced Sensing Group will develop new theory and algorithms. Another project, funded by the U.S. Army Harry Diamond Laboratory, involves research on electronic solder joints to develop mechanical modeling information on both conventional electronic solder joints and solder joints made with solder reinforced with particulate Cu_6Sn_5 (in effect making the solder a metal matrix composite). In addition, ultrasonic studies including acoustic microscopy are underway.

In cooperation with the Ceramics Division, the Group has developed sensors and data for high Tc superconductors for both process modeling and process control. Intelligent processing techniques have been applied to the hot isostatic pressing of intermetallic alloys. This project,which is jointly funded by DARPA and NIST, involves collaboration with the Metallurgical Processing Group, BDM Corporation, and the University of Cambridge (England) to develop process sensing methods, validated process models, and new control concepts incorporating artificial intelligence methods. Another project, funded by DARPA and NIST, involves collaboration with Georgia Institute of Technology, the University of California at Los Angeles, the University of North Dakota, and Ryukoku University (Japan) to model microstructure evolution using nonlinear methods.

In a collaborative program with the Aluminum Association, electromagnetic (eddy current) methods were developed to determine the temperature of aluminum during extrusion, and the rolling of sheet and strip. The effects of varying alloying constituents on the accuracy of the eddy current temperature measurement were determined. The effects of rolling speed and variation in sheet thickness were also investigated both theoretically and experimentally.

FY 90 Significant Accomplishments

- The lithium niobate sensor has been utilized in-situ during heat treatment of Y-Ba-Cu-O to monitor phase changes, which can have a pronounced effect on ultrasonic velocity. The kinetics of phase changes can be obtained, and first and second order phase transformations can be differentiated. In addition, the sensor can monitor, in-situ, acoustic emission produced by twinning and cracking and determine the sources and kinetics of these events.

- Acoustic measurements of the relationship between stiffness and temperature during processing of Y-Ba-Cu-O have revealed a previously undetected phase transformation between the two superconducting, ordered phases in this ceramic.

- The hot isotatic pressing (HIP) model for deformation hardening, and shape change has been verified experimentally and incorporated into finite element programs at DARPA and the University of Pennsylvania to predict the behavior of bulk HIP samples.

- New ultrasonic scattering theory has been formulated for imaging of thick section epoxy composites and algorithms have been developed to implement this capability.

- An electromagnetic-acoustic transduction technique has been developed for studying ultrasonic resonant vibrations in metallic spheres. This technique shows promise for obtaining accurate reference data on the elastic constants of polycrystalline metals at elevated temperatures and for studying the elastic properties of liquid metal droplets undergoing solidification.

- The effect of variation in alloy composition on the accuracy of temperature measurements of aluminum by eddy currents has been investigated using statistically designed experiments.

- The feasibility of on-line measurements of the conductivity of carbon-carbon composites during pyrolytic processing has been demonstrated. Prototype sensors have been fabricated for use in a cooperative project with the General Dynamics Corporation.

- The effect of product velocity on eddy current measurements of aluminum during processing has been analyzed theoretically and verified in laboratory experiments using a rotating disc.

An Acoustic Sensor for Process Modelling and Process Control of High Temperature Superconductors

Ewa Drescher-Krasicka

An acoustic sensor has been developed for obtaining data for process modeling and process control of high temperature ceramic superconductors. A lithium niobate sensor has been utilized, in-situ, during heat treatment of Y-Ba-Cu-O to monitor phase changes, which have a pronounced effect on ultrasonic velocity. The kinetics of phase changes, can be obtained and first and second order phase transformations can be differentiated. In addition, the sensor can monitor, in-situ, the acoustic emission produced by twinning and cracking and determine the sources and kinetics of these events.

Acoustic measurements of the relationship between stiffness and temperature during processing of Y-Ba-Cu-O have revealed an unknown phase transformation between the two superconducting, ordered phases in these ceramics. Oxygen ordering in the basal plane of Y-Ba-Cu-O changes its superconducting temperature and, at the same time, the stiffness of the lattice. Ordering into chains of alternating O and Cu results in two distinct superconducting phases. A 90K Tc and 60K Tc. The tetragonal, disordered lattice does not show any superconducting properties. The production of a particular phase depends on the schedule of oxygen partial pressure and temperature followed during heat treatment.

It was demonstrated that the ultrasonic sensor detects an increase of the stiffness of the sample when ordering takes place and when the nonsuperconducting ceramic (tetragonal, disordered structure) changes into a 90K ortho I phase during long term annealing. It was also found that on heating above the phase transformation from the tetragonal to the ortho I structure, a second phase transition takes place. This transformation, between ortho I and ortho II is apparently connected with the oxygen losses.

The ortho I ortho II ordering transition has the ultrasonic and kinetic features of a first order transformation. The ordering (every other row of oxygen between the copper atoms) does not reverse on cooling or during long term annealing. This structure is locked into the lattice and further processing at lower temperatures does not result in any improvement or shift to the 90K superconducting phase.

The presence of the ordered ortho II structure in samples heated above 620°C in air were confirmed by TEM results, which revealed that the superlattice consisted of every other empty row of oxygen in the basal copper-oxide plane. These results contradict the theoretical predictions of the calculations by first principle techniques [Kikuchi, Ceder et al.] for Y-Ba-Cu-O.

Modeling HIP Densification and Distortion

Roger B.Clough

The objective of this project is to provide and test constitutive models of the mechanical behavior of particulate compacts in hot isostatic pressing (HIP) processing. There are no other models available which can account for shape changes in the plastic flow regime. A model has been developed which also allows for hardening of the particulates due to deformation. It has been implemented in cooperative finite element programs at DARPA and the University of Pennsylvania. For copper and TiAl, the model is quite successful in predicting densification and shape changes of HIPed samples. A corresponding time-dependent (creep) theory has been outlined for future use which shows flow surfaces similar to the above. Data on the elevated temperature compressive flow stress of copper compacts and titanium canisters was also obtained experimentally.

Such compression tests require careful test head and specimen alignment to avoid specimen rotation. This is quite difficult to achieve at elevated temperatures, where refractory bricks are generally part of the loading column. For this reason, bend tests (which are easily aligned) are often used. The alignment problem was overcome by performing compression tests within a specially designed piston-ram geometry with center pins on the compression faces to prevent slippage.

Solder Interface Modeling
Roger B. Clough and A. Shapiro

Harry Diamond Laboratories, under Army funding, is addressing a costly problem in military electronics hardware, the unpredictability of electrical joints to properly solder during manufacturing as well as of mechanical failure of such joints during service. Our previous experience with composite materials such as these lead us to suspect that these problems originate at the interface of the solder and base material, and this hypothesis appears to be turning out to be correct.

Up to now, the microns-thick intermetallic zone of Cu_3Sn and Cu_6Sn_5 which forms at the copper/solder interface has been little characterized, and there is virtually no understanding of its mechanical properties, and the role that its chemistry and morphology play in affecting solderability. We have identified two interfacial fracture mechanisms through SEM examination of the fracture surfaces of solder joints. One is purely ductile tearing of the eutectic within a few microns of the intermetallic layers. This occurs for interfaces with thicknesses in the range of 1 micron. For thicknesses in the range of 10 microns or greater, cleavage of the Cu_3Sn phase, as well as separation between Cu_5Sn_6 phase and the eutectic bulk solder can occur (Fig-1-), leaving behind stripes of Sn on the Cu_6Sn_5 particle faces.

Ultrasonic Imaging of Thick-Section Composites
Stephen J. Norton

A collaborative effort is underway with the NIST Materials Reliability Division to develop an ultrasonic imaging system capable of imaging a variety of defects in thick-section fiber/epoxy composites. The project consists of (1) the development of the imaging hardware, comprised essentially of multi-element transducer arrays, digitizing apparatus and signal processing hardware, and (2) the development of the theory and associated software implementation of new imaging algorithms.

Conventional ultrasonic array imaging is based on the ability to compensate for the differential delays of a spherical wavefront detected over the transducer array, where the source of the spherical wavefront is a scattering point in the object. Thus, when the received ultrasonic signals are coherently added, they add constructively. Such coherent delay/sum imaging performs satisfactorily when the scatterers are embedded in a medium of constant sound velocity.

In composite materials, which are inhomogeneous and often anisotropic, the resulting distortion of the wavefronts can lead to severe image degradation or blurring. In an attempt to overcome these problems, an adaptive imaging algorithm has been developed to compensate for the distortion of the wavefronts due to inhomogeneities in the sound velocity. The adaptive, or "self-cohering," algorithm that has been developed for composite imaging is similar to an adaptive approach for phased-array imaging through an inhomogeneous atmosphere. The model developed here is a time-domain algorithm applicable to broadband ultrasonic signals. The adaptive algorithm has been successfully tested on simulated ultrasonic scattering data, assuming a hypothetical inhomogeneous material. When the imaging hardware is completed, the algorithm will then be applied to actual ultrasonic data acquired from thick composite samples.

Ultrasonic Sensing of Liquid/Solid Interfaces in Metals
Ward L. Johnson and Stephen J. Norton

The objective of this project is the development of ultrasonic techniques for monitoring liquid/solid interface position and structure in metals and alloys. This will be used in the primary metals processing industries for improved process sensing and modeling of directional solidification. To use ultrasonics to obtain real-time information on the interface liquid/solid microstructure, sound waves must be reflected off the interface from the liquid side, since waves originating in the solid would undergo scattering from grain boundaries, thus obscuring the detailed features of liquid/solid interface scattering. Immersible ultrasonic sensors capable of coupling directly to liquid metal are therefore being developed.

A series of modeling studies have been initiated to determine the effect of dendritic structure on the frequency dependence of ultrasonic backscatter. In one of these studies, ultrasonic waves were reflected at normal incidence from a parallel array of polystyrene wafers which was immersed in a water bath. The amplitude of the reflected pulse was measured while the frequency was scanned under computer control. This experiment was designed to provide insight into basic secondary arm scattering effects. Figure 2 (solid line) shows a scan from 0.3 Mhz to 1.9 Mhz with 10 wafers having a periodicity of 0.65 mm. and a thickness of 0.33 mm. The dashed line in the figure is a theoretical solution derived under the assumptions that the sound is a normally-incident plane wave and the wafers are infinitely wide. This exact one-dimensional solution, which fully accounts for multiple scattering, is derived by matching boundary conditions at each interface. The problem is formulated in matrix propagator form, in which the back-scattered or forward-scattered ultrasonic wave is numerically propagated from one layer to the next by a series of two-by-two matrix multiplications, one multiplication per layer. The discrepancy between the theoretical and experimental curves is attributed to edge effects.

A second model scattering study has explored the effects of primary dendrite arm spacing with the wavefront nonparallel to the interface. A flat periodic array of parallel wires was constructed and immersed in water. A single transducer was positioned such that the wavevector was perpendicular to the individual wires, but at an angle Θ to the normal of the plane of the array. With this geometry, constructive interference of reflections from the wires is expected at frequencies f such that

$$f = nv/2d\sin\Theta ,$$

where v is the velocity in water, d is the wire spacing, and n is a positive integer. Experimentally, well-defined peaks do appear in the frequency scans at the expected frequencies. Figure 3 shows the calculated and measured peak positions with d=1.27 mm and Θ ranging from 35° to 85°. Preliminary measurements of waves scattered off randomized wire arrays show peaks at the positions corresponding to those expected for the mean spacings, but with additional side lobes.

To determine if the attenuation is too large to use ultrasonic techniques for probing liquid/solid interfaces, an electromagnetic-acoustic transduction technique was designed for generating and detecting resonant vibrations in small solid or liquid metallic spheres. This technique allows measurements to be performed continuously during melting, because surface tension keeps the sample almost spherical. Initial development has employed 1/16" diameter hardened 52100 solid steel balls as samples. The resonant frequency spectrum shows sharp peaks which agree with the normal modes found numerically from the wave equations, using published values for the elastic constants. Future studies in this area will focus on performing measurements on aluminum alloys undergoing solidification.

<u>Eddy Current Sensing- Temperature Measurement in Aluminum Processing</u>

A.H. Kahn and M.L. Mester*

*Research Associate - The Aluminum Association

The objective of this project is to measure the temperature of aluminum during manufacturing processes. The material is to be examined during the heating of the billets in the furnace, during rolling, just after extrusion, and during aging. In all of these cases, the approach is to apply ac magnetic fields to the test material, and to measure the eddy current response, which is related to the temperature-dependent electrical resistivity. However, the electrical resistivity of a given alloy will depend on the allowed variation of alloying constituents within the alloy specification. In addition, the resistivity depends strongly upon the amount of alloying elements in solid solution, which is a function of its thermal history. Either of these can produce errors in temperature measurement by resistivity.

In order to investigate the variability of the measurement of temperature due to the above factors, Alcoa personnel supplied 15 round bars of AA-6061 alloy, of nominal 1 inch diameter, produced at the Lafayette and Messina plants. To compare the eddy current temperature measurements with thermocouple measurements as a reference, a series of samples were prepared with embedded thermocouples. The samples were heated and then placed in the eddy current sensor. During cooling from 550°C to 300°C, the temperatures as measured by the sensor and those measured by the thermocouple were concurrently recorded. It was found that the eddy current readings of the Lafayette samples correlated linearly with the thermocouple readings with a standard deviation of 2.8°C. The Messina samples behaved similarly, but with a standard deviation of 3.8°C. However, the materials from the two sources, although nominally equivalent, differed by 20°C. This demonstrates the deviations related to composition and thermal histories.

To study the effect of composition variations on temperature measured by eddy currents in sheet materials, a series of cooling experiments were performed on samples of AA-3004 supplied by Alcan, Alcoa, and Commonwealth Aluminum, AA-6061 supplied by Commonwealth, Reynolds Metals, and the R. D. Werner Co., and AA-1350 supplied by R. D. Werner. The specimens were of nominal 0.1 inch thickness. A statistically designed experiment was carried out. Cooling curves were observed, as before. A linear regression analysis was performed, relating a measured impedance angle (which determines the eddy current temperature reading) at 400°C with sample thickness and the compositions of the alloy constituents. Variabilities were observed for samples of similar (near identical) composition and are reported as standard deviations in Tables 1 and 2. Also reported are the standard deviations for repeated measurements on the same specimen. The equivalent standard deviations of eddy current temperatures are shown in parenthesis.

Table 1. Similar specimen variability and same specimen repeatability of impedance angle for commercial 3004 sheets after adjusting for differences in thickness. Variabilities are of angle alpha (in radians) at 400°C; equivalent temperature variations are shown in parentheses.

	Similar specimen variability (σ_s)	Repeatability (σ_r)
Commonwealth	0.4214 (11.7°C)	0.0412 (1.1° C)
Alcan	0.2335 (6.5)	0.0738 (2.1)
Alcoa	0.0293 (0.8)	0.1449* (4.0)

* One specimen, S4, had very poor repeatability.

Table 2. Similar specimen variability and same specimen variability of impedance angle for commercial 6061 sheets and 6061 and 1350 extruded buss bars, not adjusted for differences in thickness.

	Similar specimen variability (σ_s)	Repeatability (σ_r)
Commonwealth 6061 sheets	0.4306 (10.8°C)	0.0987 (2.5°C)
Reynolds 6061 sheets	0.	0.1995 (5.0)
Werner 6061 buss bars	0.1126 (2.8)	0.0420 (1.1)
Werner 1350 buss bars	0.1078 (2.7)	0.0494 (1.2)

Eddy Current Testing of Carbon-Carbon Composites

A. H. Kahn
Metallurgy Division

M. L. Mester
Research Associate
The Aluminum Association, Inc.

The objective of this new effort is the evaluation of eddy current methods for monitoring the electrical conductivity of carbon-carbon composite materials during processing. Carbon-carbon composites are produced by baking and pyrolyzing a prepared structure of graphite fibers in a matrix of organic resins. Processing temperatures are as high as 800°C. Large changes in electrical conductivity occur in the material between the initial stages and the final product. Use of an on-line eddy current sensor will correlate conductivity changes with various stages of the pyrolysis and will prove useful as an element in a process control system. This project is being carried out jointly by the General Dynamics Corporation, Fort Worth, Texas and NIST, with partial support by DARPA.

In the first half-year of the project, ending October 1, 1990, the objectives were to arrive at the design of an eddy current sensor to be placed in the processing reactor. A room temperature sensor model was constructed consisting of two flat spiral coils of outer diameter 4 inches, separated by a distance of 0.5 inch. Measurements were made on a sample of the composite before pyrolysis, a one foot square sheet of thickness 0.2 inch. The electronic equipment used was the same as for the monitoring of hot isostatic pressing. A plot of the real part of the transfer impedance between the primary and secondary coils with a conductive sample in place shows a maximum at a frequency determined by the resistivity of the sample. The real part of the impedance represents the ohmic loss associated with the eddy currents induced in the sample. At low frequencies, the loss increases with frequency, since the induced emf is proportional to the rate of change of magnetic flux which penetrates the sample. At high frequencies, the skin effect dominates, flux is excluded, and the loss decreases with increasing frequency. The peak loss, representing the transition between these limits, may be used to obtain a measurement of the resistivity of the test sample.

Figure 4 shows the experimental points obtained and the theoretical curve, calculated by the methods of Dodd and Deeds[2], which we have applied to a thin conductive sheet. Variation of resistivity shifts the theoretical curve along the frequency axis. The maximum of the theoretical curve was adjusted to match the experimental data, which yielded a resistivity of 4000 micro-ohm-cm for the sample before pyrolysis. A typical carbon-carbon composite, after pyrolysis, has a resistivity of 900 micro-ohm-cm. These values are in a range well within observability with the existing electronic equipment.

Modeling of Microstructure Evolution in Intermetallic Alloys

J.W. Cahn, John A. Simmons, Shui-Nee Chow*, J.K.Hale*, Ryoichi Kikuchi**, S. Osher**

* Georgia Institute of Technology
** UCLA

Advanced intermetallic alloys such as TiAl often contain metastable phases which can evolve during elevated temperature service into less creep-resistant forms. However, 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 recent NIST/DARPA 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. The principle objective of this program is the development of new techniques for cluster variation calculation of partially ordered multicomponent alloys to obtain metastable state phase diagrams and their dynamic extension using the path probability method. This requires application of new mathematical algorithms from the dynamical theory of nonlinear partial differential equations and from computational algebraic topology 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, leading to the design and implementation of experiments and nonlinear image analysis techniques to critically evaluate theoretical models and developing microstructure.

During the current year, work on the cluster variation method has laid out a group theoretic method for deriving the free energy functional for general partially ordered systems. Research is in progress to explore the local minima structure of this free energy surface to seek out possible new metastable states. This work is being done in collaboration with W. Craig Carter (Div. 420) and the Energy & Environmental Research Center at the Univ. of N. Dakota. A new formulation for computing path probabilities is under continuing development; two reports have already been prepared. The most recent of these addresses the formulation for partial ordering in ternary BCC alloys of the Ni-Ti-X type.

A one dimensional model equation describing segregation and partial ordering on planes in a two phase material is being studied at Georgia Tech and by guest workers from Georgia Tech at NIST. A numerical integration scheme has been coded and the nonlinear dynamics of the solutions are being investigated. The dynamics of the nonlinear discrete formulation of the ordering problem is being studied by Xu-Yan Chen from Georgia Tech, who is also spending part of his time at NIST.

The incorporation of stress into a one-dimensional plane stress model of the Cahn-Hilliard equation has been formulated and solved in collaboration with Francis Larche of Montpelier University (France) and Ryo Kobayashi of Ryukoku University (Japan). In this case the stress effects can be incorporated into the boundary conditions; spontaneous buckling is possible under such circumstances.

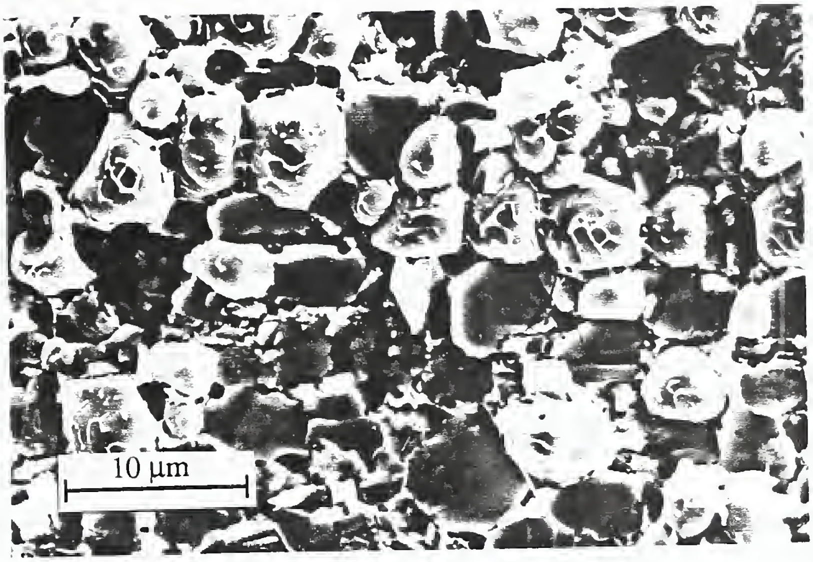

Figure 1. SEM metallograph of shear fracture surface in composite solder, looking toward the copper substrate. Fracture occurs at the interface, of 15 v/o Cu_6Sn_5 particulate/eutectic Pb/Sn composite. The interface consists of a mixture of Cu_3Sn and Cu_6Sn_5 particles. The dark gray particles are Cu_3Sn, the striped light gray particles are Cu_6Sn_5 dendrites which grew from the interface during soldering (~ 2 min at 215°C in vapor phase reflux).

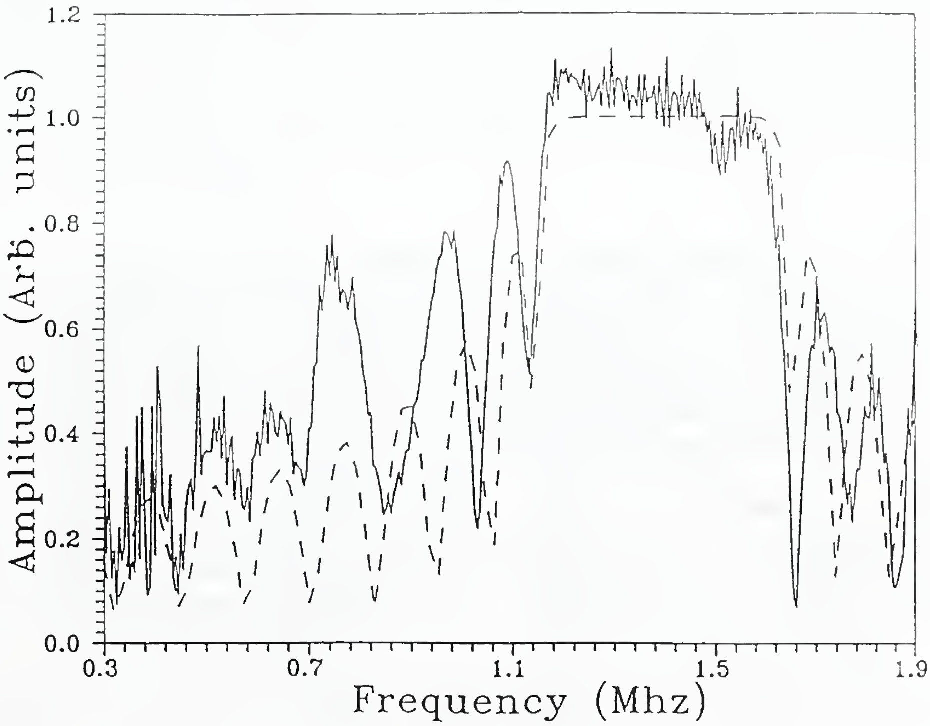

Figure 2. Amplitude versus frequency of ultrasonic waves reflected off a polystyrene/water structure. The polystyrene wafers are 0.33 mm. thick with a periodicity of 0.65 mm. (with water between them). The ultrasonic waves (generated by a 1 Mhz broadband transducer) are at normal incidence. The amplitude has been normalized with respect to the transducer sensitivity.

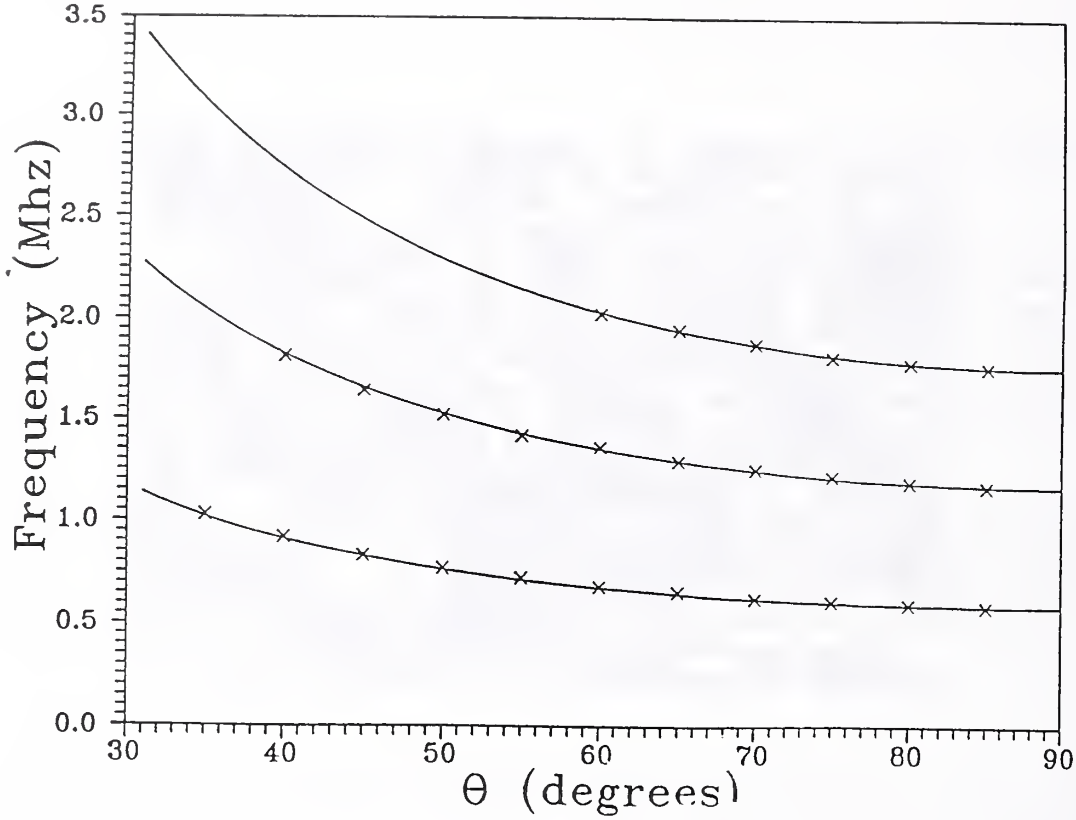

Figure 3. Measured (X) and theoretical (solid line) resonant peak positions as a function of the angle between the wavevector and the normal of an array of parallel wires with 1.27 mm spacing.

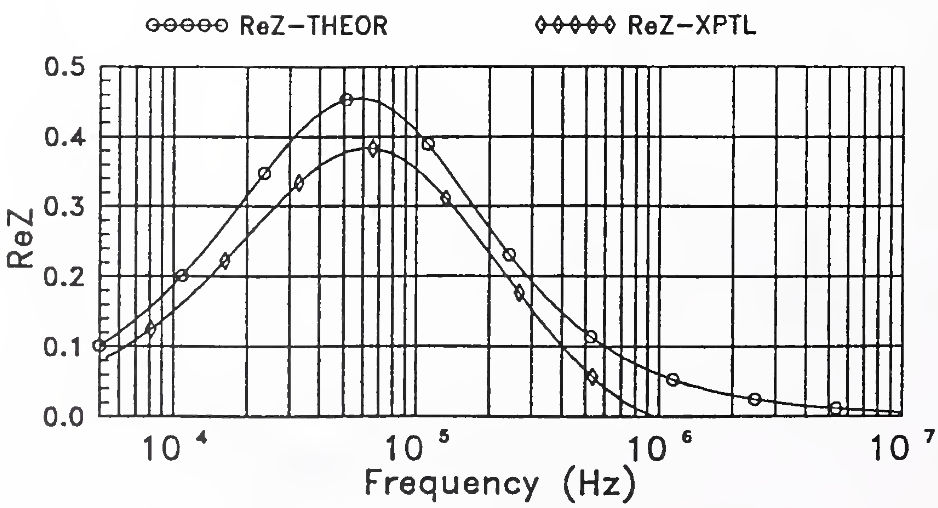

Figure 4. Real part of the two-coil transfer impedance with a carbon-carbon sample in place. The theoretical curve has been adjusted to match the measured absorption peak.

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.

To improve the understanding of corrosion mechanisms, the Corrosion Group is conducting studies into the mechanisms of stress-corrosion cracking, localized corrosion and uniform corrosion. During the past year, the Corrosion Group conducted studies into the influence of stress on the corrosion and stress-corrosion cracking of copper alloys and into the role of grain boundary precipitates in the intergranular stress-corrosion cracking of Al-Li alloys. Also, the Corrosion Group studied the influence of environmental variables such as conductivity and oxygen diffusivity on the rate and spatial distribution of corrosion damage.

To promote the development of better corrosion measurement science, the Corrosion Group has been investigating methods of measuring the electrochemical reactions that occur when the film on passive metals is ruptured. The Corrosion Group is also developing computer models based on electrochemical reaction kinetics that simulate the results of corrosion experiments and is investigating the application of these models to understanding corrosion behavior and electrochemical measurement techniques.

The Corrosion Group is also studying the application of corrosion measurement methods and the prediction of usable 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.

To assist the development and application of advanced materials, the Corrosion Group initiated new programs in FY90 on the corrosion and stress-corrosion cracking behavior of metal matrix composites fabricated by the Osprey™ process and iron aluminides. In addition to these new programs, the group is continuing its work on nickel aluminide, HSLA steel, Al-Li alloys and composites.

FY 90 Significant Accomplishments

- The NACE-NIST Corrosion Data Center expanded industry support for programs addressing industrial needs. Emphasis continued on knowledge-based expert systems using artificial intelligence concepts to aid corrosion scientists and engineers in selection of materials. Current programs focus on storage and handling of hazardous chemicals, equipment used in electric power generation and potable water systems.

- Stress corrosion cracking behavior of ductile nickel aluminide was demonstrated to be due to hydrogen absorption during exposure to the aqueous environment and the resulting embrittlement.

- The corrosion and pitting behavior of ductile nickel aluminide was demonstrated to be dominated by nickel in aqueous solutions of pH from 1 to 13 and at temperatures ranging from 25 to 80°C.

- Using an electrochemical absorption-desorption technique that the Corrosion Group developed, it was determined the diffusion coefficient of hydrogen in nickel aluminide at room temperature.

- Using a scratch repassivation experiment that the Corrosion Group developed, it was determined that lithium in solid solution in Al-Li alloys has no significant influence on the rate of repassivation in neutral pH aqueous solutions.

- The scratch repassivation technique was used to evaluate the possibility that hydrogen evolution would occur during rupture of the passive film on the surface or at a crack tip in nickel aluminide.

- The corrosion and stress corrosion cracking behavior of nickel and iron aluminide was presented to over thirty representatives of various industries at a technology transfer conference on nickel and iron aluminide sponsored by Oak Ridge National Laboratory.

- 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

D. B. Anderson, C. D. Flanigan, P. Karras*, E. J. Kuchinsky*, C. Li**, B. Mashayekhi*, C. P. Sturrock*, and J. A. Wolfson***

* NACE Research Associate
** Guest scientist sponsored by the United Nations Industrial Development Organization and Beijing University of Science and Technology.
*** Student Trainee

The Corrosion Data Center was established in 1982 as a joint program between NIST and 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 and control data for engineering materials exposed to a wide range of industrial environments.

Activities in 1990 centered on:

(1) Delivery of software program designed for users to organize and input data in a logical database structure based on proposed NACE and ASTM standardized formats.

(2) Database compilation from industrial data sources and capture of additional data focusing on materials performance in hazardous chemicals.

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

(4) Completion of additional 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).

(5) Continuation of a 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, corrosion of critical components in flue gas desulfurization systems and microbiological corrosion in service water systems.

(6) Initiation of a new program sponsored by the INCRA Technology Division of the International Copper Association to develop expert system technology for controlling corrosion in potable water distribution systems.

(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 utilizes 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 of evaluated corrosion data with industry financial support and aid in assuring consensus on the relevance and applicability of the data.

Corrosion Mechanisms and Corrosion Measurement Science
U. Bertocci, J. L. Fink, F. Friedersdorf*, J. S. Harris, T. K. G. Namboodhiri**, E. N. Pugh, R. E. Ricker and M. R. Stoudt

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Mechanism(s) of Stress Corrosion Cracking - Investigations continued on testing and evaluating the proposed film-induced cleavage mechanism for transgranular SCC. In the past FY, studies were conducted into the proposal that stress or strain significantly accelerates the rate of dealloying which would allow crack propagation over the observed distances without requiring cleavage of the unaffected matrix. An earlier study (T. K. G. Namboodhiri and R. S. Tripathi, Corro. Sci., 1986, 26, (10) p. 745), had indicated that this was a possibility; however, no evidence which would confirm this hypothesis was obtained. As part of this study, considerable information on the electrode kinetics of copper and brass in ammonia solutions was obtained. These data, which are not available in the literature, will be important in the formulation of SCC mechanisms and in computer modeling. In addition, tensile tests were carried out on Cu-Zn single crystals to determine if there is a minimum amount of Zn required for cracking susceptibility under conditions of active dissolution. Work was initiated on understanding the mechanism of intergranular SCC and evaluating the possibility that film-induced cleavage may contribute to this failure process as well as to transgranular SCC.

Computer Modeling of Electrochemical Measurements - The interpretation of electrochemical measurements tends to be difficult because of the large number of factors which influence the results. Frequently, models for electrochemical systems are based on equivalent electrical circuits with only a tenuous link to the underlying electrochemistry. As a result, the Corrosion Group has been working on the development of computer models of electrochemical systems which are based on the electrode kinetics of the system being studied and the fundamental electrochemical parameters of the system such as the reaction rate constants, the concentrations of the electroactive species and the transport properties of the solution. For this type of modeling, mathematical expressions that represent the reactions that occur in the specific system being studied are derived, and from these the response of the system to electrochemical stimulation is calculated. These formulae allow the computation of the polarization curve or the impedance spectrum at equilibrium or at any other potential for the system. Figure 1 is a polarization curve calculated with this computer model. In this figure, the diffusion layer is altered corresponding to different stirring levels and the resulting corrosion potential is seen to change. Figure 2 is an impedance spectra for this same system as calculated at different electrode potentials. This approach has made useful contributions to the interpretation of the experimental results. By varying the electrochemical quantities that constitute the input parameters and observing the corresponding changes in the curves, the experimental curves can be modeled and, in this way, test hypotheses, estimate electrochemical quantities that are not easily measured, or develop experimental strategies.

<u>Electrochemistry of Bare Surfaces</u> - During service, the surfaces of most engineering alloys are covered by a film which, in many cases, dramatically reduces the rate of corrosive attack. This "passivating" film is frequently damaged and/or removed by mechanical or chemical processes during service. The ability of the alloy to repair this film and the types of reactions that occur during the repair process may have a dramatic influence on the ability of the alloy to withstand the service environment without failing. To study the process the Corrosion Group developed a technique for the generation of bare surface by scratching that allows the evaluation of reactions on the freshly bared surface and the rate of film reformation. Figure 3 is a diagram of the experiment developed for this type of measurement. In this experiment, a weight is dropped and the surface of the metal is scratched with a sapphire scribe and the resulting potential or current transient is recorded on a digital oscilloscope. By measuring the potential transient, the thermodynamic conditions at the bare surface can be better understood and reactions that are thermodynamically impossible at the observed potential can be eliminated from consideration. For example, Figure 4 is a potential transient for nickel aluminide in an aqueous solution. Research in the Corrosion Group has shown that nickel aluminide will be embrittled by hydrogen if the potential becomes low enough to allow hydrogen absorption. There was some concern that during film rupture, the potential of nickel aluminide would be reduced momentarily to a value low enough for hydrogen absorption. As a result, scratch tests were run to test this hypothesis and it was found that the resulting potential transient, Figure 4, did not go below the potential for a hydrogen fugacity of 1 atmosphere. Another example of the application of this technique is shown in Figure 5, In this figure, the potential of the sample was held constant with a potentiostat while the sample was scratched. The current transient and the rate of current decrease after scratching is an indication of the rate of film growth. The Corrosion Group used this technique to determine if the addition of lithium to an aluminum alloy would alter the rate of repassivation and, thereby, alter its ability to resist corrosion failure mechanisms such as pitting and stress-corrosion cracking. After extensive testing of alloys with differing lithium concentrations and heat treatments, the results indicated that over the concentration range studied, there is no significant effect of solid solution lithium on the repassivation rate of aluminum in the solution examined. The Corrosion Group is continuing to work on the development and the application of this technique.

<u>Effect of Conductivity and Mass Transport on Corrosion</u> - In a large number of cases, corrosion occurs in environments, such as rock, soil or concrete, which are sufficiently porous to contain an aggressive electrolyte, but which prevent material transport by convective motion. Field experience indicates that the rate and morphology of the corrosion damage in these cases is dependent on the rate of oxygen transport in the porous medium to the metal

surface and the electrical conductivity of the medium. The Corrosion Group has been studying this system as part of its program to understand corrosion mechanisms and corrosion measurement methods. This program consisted of electrochemical experiments on model systems and the development of a computer model for these experiments. In addition, exposure tests in the model environments were conducted as part of the NRC program which will be discussed later. Sand with an aqueous solution added was used for the model system and the permeability and the conductivity of the media were varied in a systematic fashion by varying the sand particle size and the ionic content of the solution. In order to understand the electrochemical results, a computer model of the sand/solution/metal system was developed and the electrochemical measurements were simulated by 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.

<u>Advanced Materials</u>

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

* Guest Scientist, The Engineering Academy of Denmark, Lyngby, Denmark

<u>Corrosion of Ductile Nickel Aluminide</u> - Nickel aluminide (Ni_3Al) is an intermetallic compound that can be made ductile by microalloying with boron (200 ppm) and by keeping the aluminum content slightly sub-stoichiometric. 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. Electrochemical experiments were carried out on samples of this materials in solutions of pH from 1 to 13 and at temperatures ranging from 25 to 85°C. The results of the electrochemical experiments indicate that 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. Also, pitting of this material was found in strongly acidic solutions such as sulfuric and nitric acid. The results of the experiments conducted to date indicate that this behavior is due to the ohmic drop inside the pit allowing the potential within the pit to reach the active region.

<u>Stress-Corrosion Cracking of Ductile Nickel Aluminide</u> - The Corrosion Group had previously demonstrated that nickel aluminide exhibits environmentally induced cracking in aqueous environments with a low pH and hypothesized that this behavior was due to hydrogen absorption and embrittlement. To test this hypothesis, the Corrosion Group performed pre-corrosion embrittlement experiments. As shown in Figure 6, the Corrosion Group found that nickel aluminide was embrittled by pre-corrosion in a low pH solution and that this pre-corrosion effect was completely reversible by exposure to vacuum at room temperature for a period of time long enough to allow for solid state diffusion and desorption of the hydrogen absorbed during the pre-corrosion

exposure. Since hydrogen absorption and desorption is the only phenomena which can explain these observations, it was concluded that this was the mechanism of the observed environmentally induced cracking. As a result, the Corrosion Group conducted experiments to evaluate the possibility of hydrogen absorption under other environmental exposure conditions. Three possibilities were examined: (1) hydrogen absorption during the potential transient that follows film rupture (as discussed under "Electrochemistry of Bare Surfaces above), (2) hydrogen absorption as a result of galvanic coupling with a more electronegative metal, and (3) evaluation of the critical pH below which hydrogen absorption and embrittlement is observed. The results of these experiments showed that: (1) hydrogen absorption following film rupture is not significantly more likely in nickel aluminide than in pure nickel, (2) nickel aluminide is embrittled in any pH solution when the potential is reduced below the potential of hydrogen evolution ($P[H_2]=1$ atmosphere), and (3) significant hydrogen absorption and embrittlement does not occur during free corrosion in naturally aerated solutions unless the pH is less than 3.

Determination of Hydrogen Solubility and Diffusivity in Ductile Nickel Aluminide - Because nickel aluminide can be embrittled by hydrogen and may absorb hydrogen during fabrication or joining, industry needs to know the rate of hydrogen absorption and, more importantly, the rate at which it can be removed. Since the rate of hydrogen absorption and desorption is controlled by the rate of hydrogen diffusion, the Corrosion Group decided to measure the hydrogen diffusion coefficient in this material. To do this, the Corrosion Group devised an electrochemical hydrogen absorption and desorption experiment. Figure 7 is a diagram of the electrochemical cell used for this experiment and Figure 8 is a plot of the absorption and desorption transient obtained from a sample with two calculated transients for two different diffusion coefficients.

Cracking of HSLA Steels at Welds - The Corrosion Group, in collaboration with the Navy's David Taylor Research Laboratory, is conducting a study into the corrosion and cracking resistance of HSLA-100 steel. For this study, samples of a HSLA-100 and a HY-100 alloy are heat treated to represent the thermal history of the parent metal and of the weld heat affected zone. The corrosion behavior is being evaluated by electrochemical polarization experiments and the cracking resistance is being evaluated by conducting slow strain rate tensile tests in artificial seawater at constant electrode potentials and under free corrosion conditions.

Metal Matrix Composites Fabricated by the Osprey Process - The Corrosion Group initiated experiments into the corrosion behavior of aluminum alloys including aluminum-lithium alloys reinforced with particulate silicon carbide and fabricated by the Osprey™ process. During this FY, the Corrosion Group examined the electrochemical behavior of the interface between pure aluminum and silicon carbide using ac impedance and electrochemical noise. During the coming FY, the Corrosion Group will evaluate the corrosion of Osprey ™ deposited alloys by using these same techniques and the SCC behavior will be characterized by conducting slow strain rate tensile tests in different environments.

Corrosion and Stress-Corrosion Cracking of Ductile Iron Aluminide - During this FY, the Corrosion Group initiated a program into the corrosion and stress corrosion cracking behavior of iron aluminide in collaboration with Oak Ridge National Laboratory. During this FY, the Corrosion Group conducted electrochemical experiments on iron aluminide in neutral and acidic solutions. During the coming FY, the Corrosion Group will complement this work with slow strain rate tests in these solutions. Other solutions will be added to the study so that the behavior of this material over the normal range of pH's and potentials will be evaluated.

Corrosion Issues in Nuclear Waste Management

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* Nuclear Regulatory Commission
** Statistical Engineering Div., Center for Computing and Applied Mathematics

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 completely contained 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 high level waste to be disposed of will be spent fuel (UO_2). Most of the spent fuel (~90%) will still be in the form of fuel rods, most of which are clad with Zircaloy with the remainder (~3%) clad in stainless steel. Approximately ten percent of the spent fuel will be embedded in glass.

There are several potential problem areas which need to be studied and understood before the safe disposal of this waste. Several of these problem areas relate to corrosion behavior of the materials in the engineered barrier system and estimating the lifetimes of the different barriers. Of these, the Corrosion Group is addressing the following:

(1) Critical evaluations of scientific research data, measurement techniques and mechanisms used in modeling for the NIST/NRC data base are carried out. The data base has over 1088 entries. Critical evaluations were completed 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 on nuclear waste management is provided to 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 NIST staff for scientific considerations relating to predicted long term durability. Sixteen comments with associated explanations and recommendations were given. These commentaries were sent to the DOE and most were accepted and addressed in the revised version of the SCP.

(3) Laboratory experiments are conducted on basic corrosion processes. Data from the experimental programs can be used for verification of existing data and for establishing mechanisms of the corrosion processes for use in long term corrosion durability predictions. The laboratory projects for this year were (1) the determination of the effects of transport and resistivity on corrosion, (2) the determination of the effects of specific ions on the corrosion behavior of Zircaloy nuclear fuel cladding and (3) the development of a plan that could be used in the retrieval and analysis of steel specimens that have been buried under ground for twenty years.

Also, from time to time the NRC has a need for a state-of-the-art technical review on a corrosion related subject which not only reviews and summarizes the subject but goes beyond this and interprets the subject matter explaining the impact or potential impact of the important points on the high level waste program. These interpretive-review papers are provided on different subjects at the request of the NRC. During this FY90, interpretive papers were prepared on the following topics:

(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

Effects of Transport and Resistivity on Corrosion - Studies sponsored by the Department of Energy (DOE) on the corrosion behavior of candidate alloys for high level waste containers are usually conducted in media which have a high oxygen transport and high electrical conductivity. The results of these experiments indicate that the alloys under consideration will corrode uniformly, leading to a container lifetime of thousands of years. However, if the corrosion damage is not uniformly distributed, penetration of the canister could occur earlier than predicted by the DOE models. Since the transport and conductivity of the proposed repository are different from those used for the DOE experiments, a study was conducted into the effects of oxygen transport and electrical conductivity on the rate and distribution of corrosion damage.

This study investigated the rate and distribution of corrosion damage over a wide range of environmental conditions, including low conductivity as expected in the environment of the proposed repository (Yucca Mountain). The results of this study indicate that the corrosion rate of steel is directly related to the rate of oxygen transport over several orders of magnitude. 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 which reveals that as conductivity of the environment decreases and corrosion rate decreases, 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 experiments were conducted in solutions having from 1 to 10 times the ionic concentration of species found in a well near the repository site (well J13) 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 (SCE) where breakdown occurred. Corrosion rates under the conditions of the tests were negligible, but there were indications of crevice corrosion attack. These studies are continuing in an effort to obtain additional data and to improve test methods.

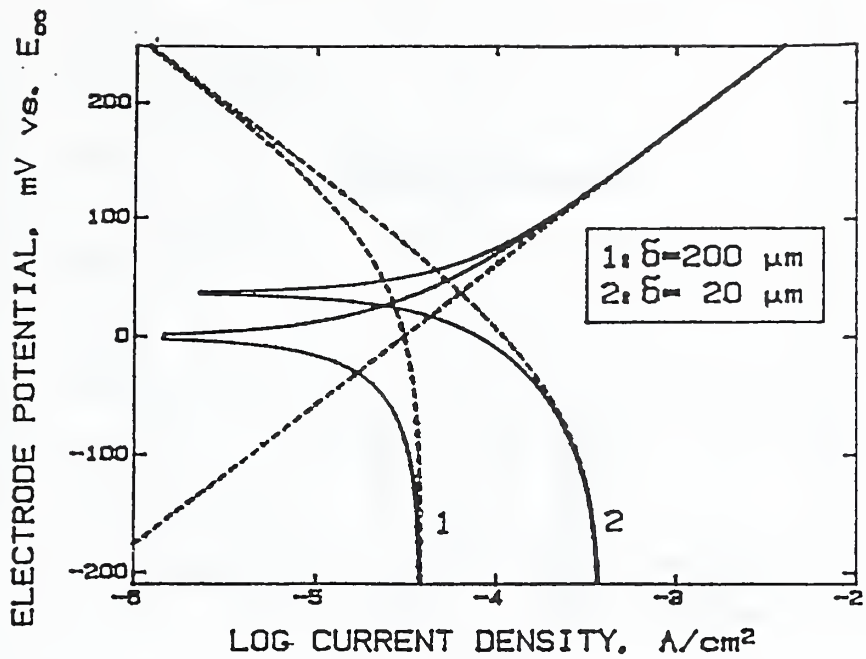

Figure 1 Polarization curves calculated for a film free metal surface corroding in an electrolyte with two different diffusion layer thicknesses and a bulk concentration of the cathodic species of $2x10^{-14}$ M. In this figure, the free corrosion potential increases with the reduction of the diffusion layer thickness which corresponds to experimental observations.

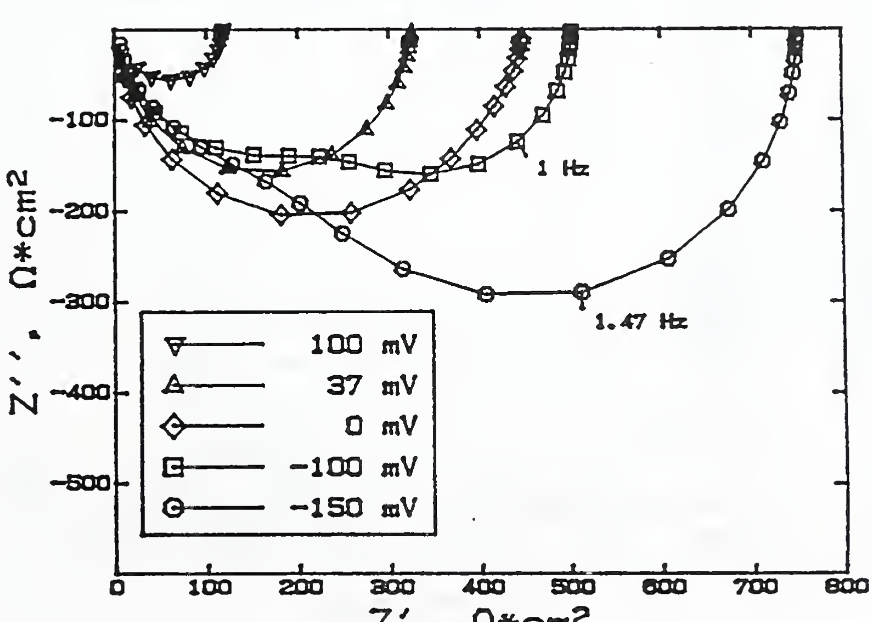

Figure 2 Impedance spectra calculated for a film free metal surface corroding in an electrolyte with a diffusion layer thickness of 0.02 mm, a double layer capacitance of 30 $\mu F \cdot cm^{-2}$, and a solution resistivity of 5 $\Omega \cdot cm^2$. The free corrosion potential is 37 mV.

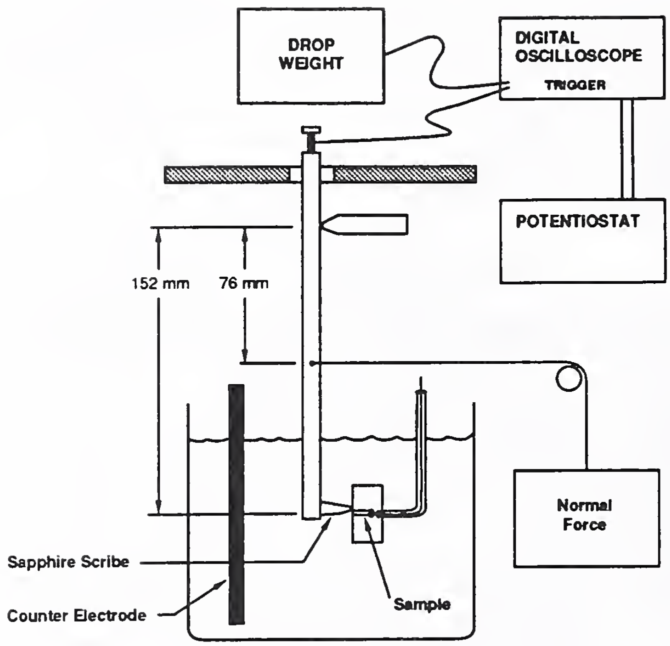

Figure 3 Schematic diagram of the system used to generate bare surface for electrochemical studies of reactions on freshly bared surfaces.

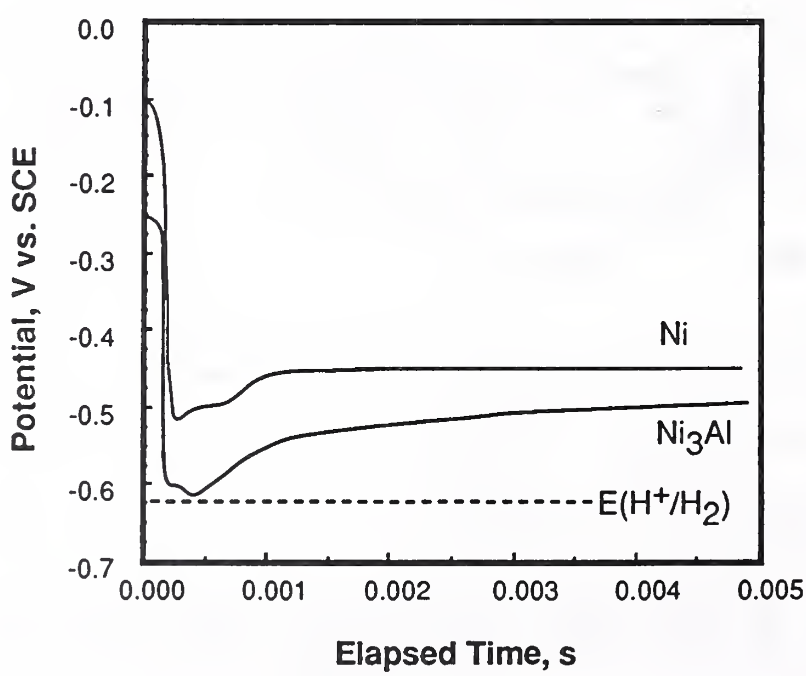

Figure 4 The potential of a pure nickel electrode and a nickel aluminide electrode with respect to a saturated calomel electrode during and immediately after the generation of bare surface by scratching in 0.5 M NaCl. The dashed line corresponds to the potential for a hydrogen fugacity of 1 atmosphere in this environment.

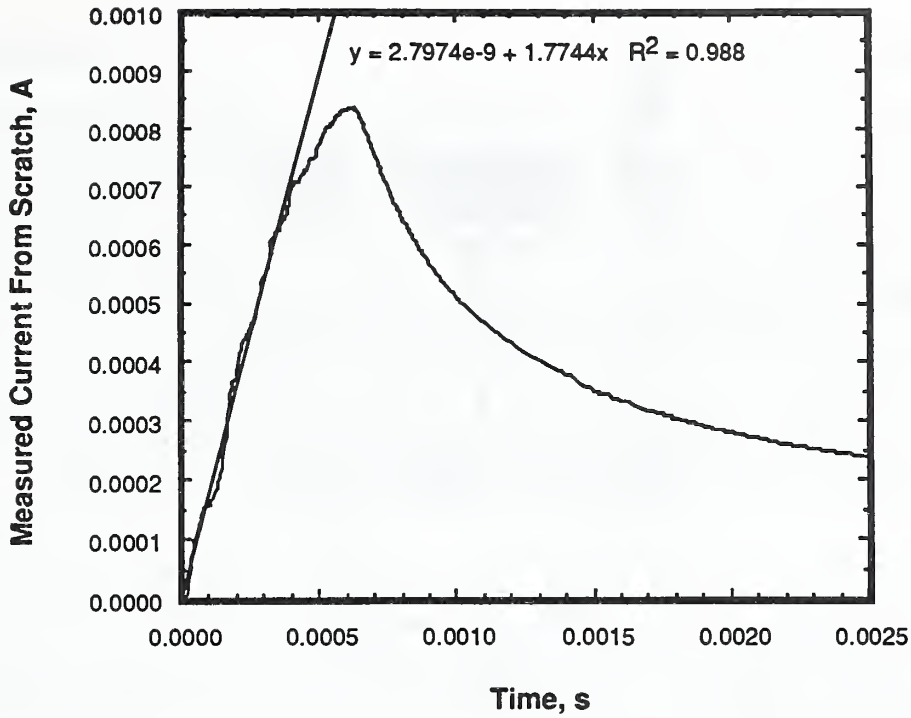

Figure 5 The current transient observed during and following the generation of bare surface by scratching a Al-1% Li electrode in a buffered solution of 0.5 M sodium chloride at a potentiostatically controlled potential of -0.8 V with respect to a saturated calomel electrode.

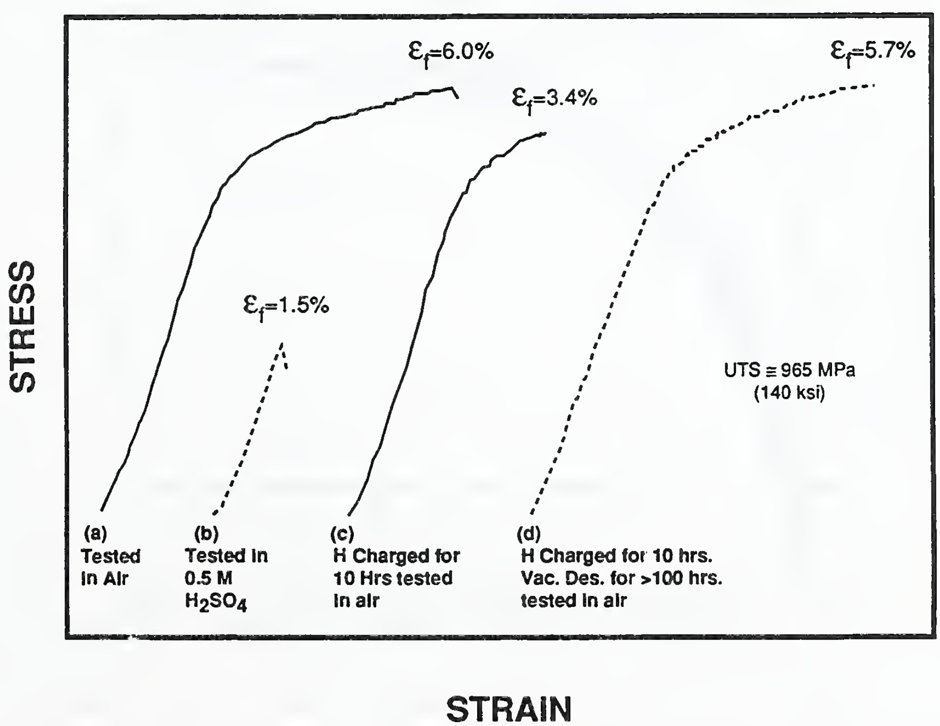

Figure 6 Diagram representing the tensile properties of ductile nickel aluminide in air (a) and in sulfuric acid (b) as compared to the properties observed in air after pre-exposure to sulfuric acid for 10 hours (c) and after pre-exposure to sulfuric acid for 10 hours followed by a vacuum "bake out" at room temperature for 100 hours (d).

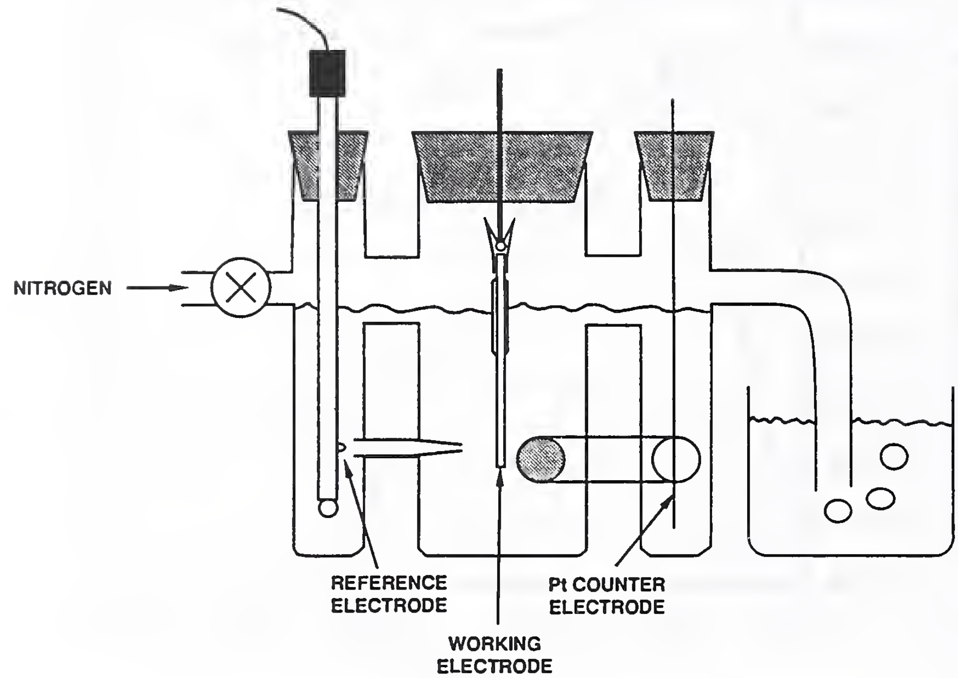

Figure 7 Diagram of the electrochemical cell used for electrochemical absorption and desorption experiments used to determine the hydrogen diffusion coefficient in ductile nickel aluminide.

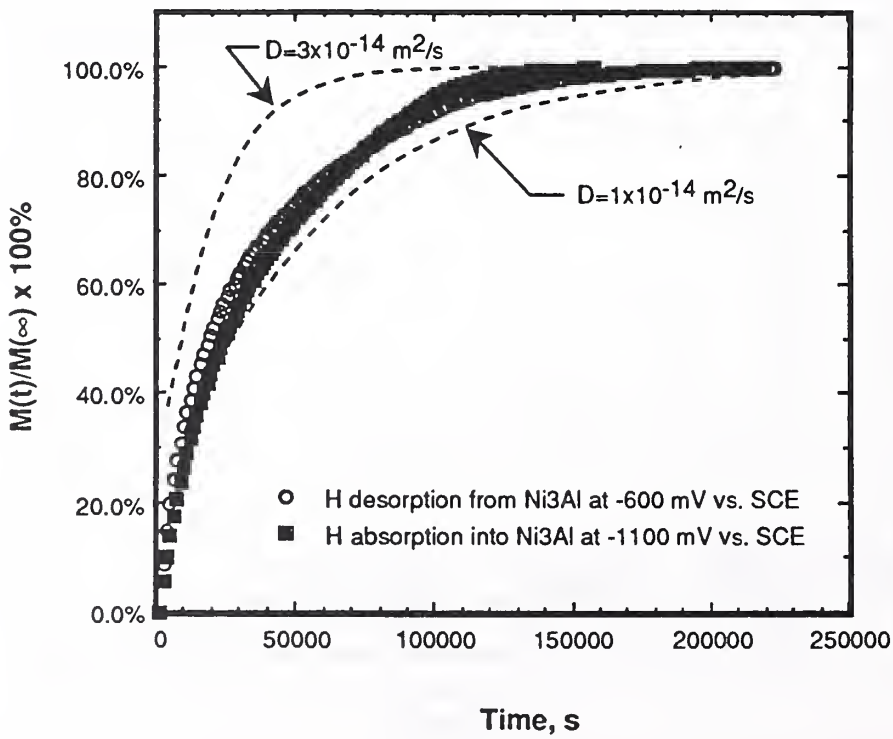

Figure 8 Diagram of the results of an electrochemical absorption and desorption experiment plotted on a normalized scale with the calculated transients that would be observed for a diffusion coefficient of $3x10^{-14}$ and $1x10^{-14}$ $m^2 \cdot s^{-1}$ clearly showing that the diffusion coefficient is between these values.

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 coatings 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 voluntary standards organizations and industry, through research associates and to the government agencies, through appropriate contracts and consulting arrangements.

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 sales about $150M per year, approximately 550K tons of metal coated wire and wire products are produced per year, tin plate account for 8800 tons with a dollar impact 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 together with $9[illegible] 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.

FY90 Significant Accomplishments

- The demonstration that small amounts of ammonium nitrate when added to hard chromium results in a coating which significantly outperforms normal hard chromium in abrasive wear conditions.

- The fabrication of a deposition cell for creating continuous fiber reinforced titanium aluminide composites for characterization of their mechanical properties.

- The demonstration of the deposition of titanium aluminide (Al_3Ti) in the Ll_2 structure. Such a structure has shown some evidence of ductility at ambient temperatures and can be stabilized by incorporating manganese.

- A sensor has been developed for the characterization of the Ti_{+2} content in the chloroaluminate process used for the deposition of titanium aluminide.

- The development of electrochemical systems capable of depositing the following multilayered alloys: (1) Co-Cr, (2) Fe-Cr, (3) Co-Cu, (4) Cr-Ni, (5) Ni-Mn, and (6) Cr-Cu.

- The demonstration that the nickel-boron carbide particulate composites can out perform hard chromium, thereby providing a non-polluting replacement for hard chromium which has serious waste pollution problems associated with it.

- The completion of a cell for the study of titanium diboride as possible fiber material. This is important because of the match of coefficient of thermal expansion of titanium diboride with the proposed titanium aluminide.

- The demonstration that hot isostatically pressed electroformed composites can be produced in idealized close packed fiber geometry.

Interfaces in Composites

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* Research Associate - American Cyanamid

The goal of the cooperative research program between N.I.S.T. and the Metal-Coated Fibers Division of American Cyanamid Company is the development and characterization of interfaces between graphite fibers and matrix materials. Interfaces that provide good bonding are required and determination of those factors which minimize deleterious interactions between fiber and matrix is necessary. Cobalt-tungsten coatings were found to behave as an effective diffusion barrier between polyacrylonitrile (PAN) graphite fibers and nickel matrices. The effect of composition and structure of the interface were studied to determine their effect fiber-matrix bond strength. The adhesion data from a fiber punch-out study of fibers with various metal coatings is shown in Figure 1. The bond between PAN graphite and the as-deposited CoW coating (CoW/PAN) was found to be 345 ± 135 MPa (50 ± 20 ksi), considerably stronger than that of Ni/pitch graphite 62 ± 21 Mpa (9 ± 3 ksi). Although the shear strength of the CoW/PAN decreased upon annealing at 800 °C for 1.5 hr (i.e., to 41 ± 7 MPa), it was still stronger than unannealed Cu/PAN (3 ± 2) or Cu/PAN annealed at 800 °C for 17 hr (21 ± 7 MPa).

Cobalt-tungsten coatings of compositions ranging from about 7 to 20 wt-% tungsten and varying in thickness from approximately 0.5 to 10 μm were electrodeposited on 7 μm PAN graphite fibers in bundles of 3000 fibers (tows). The samples were annealed at 1100 °C for 60 hours in quartz tubes having a vacuum of 10^{-5} Pa. X-ray diffraction of the unannealed samples showed the coatings to be a mixture of FCC and HCP cobalt of various relative proportions. Upon annealing, all of the coatings appeared to be entirely converted to the FCC high temperature phase, indicating that sufficient tungsten and carbon were present to stabilize this otherwise high temperature structure. This is desirable because FCC cobalt is known to have greater strength and ductility than HCP cobalt. The XRD spectra of all the samples having a high initial fcc composition indicated the presence of WC, but this phase was observed in only a few of the spectra for samples having a high initial hcp composition. Tungsten was not observed by XRD in any form other than tungsten carbide. The lack of XRD evidence for tungsten, either by itself or in the form of a cobalt tungsten or other tungsten containing phase, suggested that the tungsten was in solid solution in the cobalt lattice. Transmission Electron Microscopy/Selected Area Diffraction (TEM/SAD) of an annealed sample whose XRD spectrum had not contained WC indicated that the bulk of the coating was composed of FCC cobalt crystals approximately 5 to 10 μm in length, and there was no detectable change in the lattice parameter. In addition to the usual diffraction spots for an FCC crystal oriented along the [001] zone axis, faint spots were observed which strongly suggest the presence of an $L1_2$ ordered structure. This diffraction pattern is shown in Figure 2 and suggests that superlattice ordering may be occurring on a small scale. There is diffraction evidence, also, for a high temperature tungsten carbide phase, WC_{1-x}, on the graphite side of the interface. TEM micrographs of the cobalt side of the interface show a layer of an as yet unidentified phase. Although Laser Raman Microprobe detected Co_3O_4 in the interface region, it was not consistently present and is not expected to be in bonding of the subsequently applied matrix coating. Thus, it appears that when heated to 1100 °C for extended times that the interface probably has four parts: graphite, WC_{1-x}, an unknown layer, and fcc cobalt.

<u>Titanium Aluminide Intermetallic Electroforming</u>
D. S. Lashmore and P. Sharpless

A cell has been designed and completed for the deposition of alloys from low temperature fused salt electrolytes onto continuous fibers. The cell, shown in Figure 3, makes use of a circulating solution of silicone oil to maintain temperature to within about 1 °C. Electrical contacts to the cell are made through 60K gold plated graphite fibers, eliminating the need for mercury contacts. The cell also contains a completely separate tungsten microelectrode whose purpose is the characterization of the state of the electrolyte. This made possible measurements of the concentration of the various metallic ionic species such as titanium, aluminum and others which may be added to the electrolyte. Since the electrolyte required for the deposition of these intermetallic alloys is sensitive to the presence of water and oxygen, the cell incorporates high pressure argon airlocks separated from the atmosphere by small capillary tubes. The cell's modular design allows the replacement of the monofilament feed throughs currently in use with feed-throughs designed for tows. The static immersion 'boat' shown in Figure 3, can easily be replaced with a new 'boat' designed for forced convective transport to enhance the deposition rate. Because of the space required for this cell and the titanium diboride cell described below, an entirely new laboratory has been designed and set up to accommodate these new activities. This laboratory incorporates stainless steel hoods, polycarbonate hood doors, special ventilation and other safety devices.

Titanium Diboride Deposition
D. S. Lashmore, F. Biancaniello, P. Boyer, and R. Parks

A program on the electoforming of titanium diboride fibers has been undertaken to determine the feasibility of electrodepositing sufficient titanium diboride onto very thin graphite tows, which then effectively become titanium diboride fibers. The process involves the deposition from flouride electrolytes, operating at about 700 °C. This corrosive system mandates the use of an Inconel deposition vessel incorporating the provision for excluding oxygen and water vapor. The cell and associated support equipment has been developed in collaboration with the Metals Processing Group.

Electrochemical Deposition of Aluminum Alloys and Intermetallics
G. R. Stafford, J. P. Moran and G. M. Janowski

This study demonstrates that electrodeposition is useful in producing TiAl- and Al_3Ti-based materials. The near-net-shape capabilities and unique approach to microstructural control inherent to electrodeposition offer many advantages over more conventional processing methods. The immediate goal is to understand the mechanism of phase formation in aluminum alloy deposition from chloroaluminates and to determine the conditions required for the direct electrodeposition of aluminum intermetallic compounds, particularly the titanium aluminides. Aluminum alloys can be electrodeposited from mixtures of $AlCl_3$, NaCl and the chloride salt of the solute metal at temperatures as low as 120 °C. In the Al-Ti system, the melt chemistry is complicated by the possible presence of titanium in the 2, 3, and 4 oxidation states. In addition, titanium has poor solubility as the chloride salt and must form a tetrachloroaluminate complex; the equilibrium constants and reaction kinetics for titanium complex ion formation are not known. The preferred titanium electroactive species is Ti^{+2}.

Recent efforts have focused on studying the electrochemical oxidation of Ti^{+2} in an effort to determine its concentration in the chloroaluminate electrolyte, thereby allowing us to better understand its formation and maximize its solubility. We have determined that the electrochemical oxidation of Ti^{+2} is a reversible, 1-electron oxidation to Ti^{+}_{3}. Consequently, this reaction can be used as an in-situ measure of the Ti^{+2} concentration in the electrolyte. Ti^{+2} has been added to the electrolyte by the electrochemical dissolution of titanium metal and by the direct synthesis of $Ti[AlCl_4]_2$ by the reaction of Ti with $AlCl_3$ at 275 °C. Electrochemical dissolution appears to be the more promising of the two and we have determined that the dissolution process follows Faraday's Law for a 2-electron oxidation up to a Ti^{+2} concentration of 140 mM. At this concentration, the electrolyte is translucent and bright green; there is no indication that the solubility limit for Ti^{+2} has been reached.

A wide range of alloy compositions can be obtained by electrodepositing from melts containing varying amounts of Ti^{+2}. The highest Ti content observed thus far is 30 at.%. For electrolytes relatively dilute in Ti^{+2}, alloy composition is strongly dependent upon current density due to diffusion limitations of Ti^{+2}. In these electrolytes, alloys containing significant amounts of titanium can only be deposited at very low current densities. In contrast, as the Ti^{+2} concentration increases, the dependence of alloy composition on current density decreases. Energy dispersive spectroscopy indicates that the electrodeposits contain only aluminum and titanium; no entrained salts are present. In addition, the current efficiency for alloy deposition is greater than 95%, assuming a 3-electron and 2-electron reduction for Al and Ti, respectively.

The surface morphology of electrodeposits containing small amounts of Ti is quite similar to that of pure Al, which is nodular and somewhat dendritic. As the Ti content is increased, the deposits remain nodular but otherwise quite dense. Pulsed deposition reduces the nodule size significantly. The structure of Al-Ti electrodeposits containing up to 25 at.% Ti have been examined by TEM. As is often the case with electrodeposited alloys, the solubility limit is extended appreciably over that which appears in the equilibrium phase diagram. As-deposited alloys containing 3.6 at.% Ti are single phase FCC. This is an order of magnitude greater than the equilibrium solubility limit for Ti in FCC Al. Although the appearance of these deposits is quite similar to that of pure aluminum, the microstructure is significantly different in that the alloy is extensively twinned. This is not typically observed in pure aluminum due to its high stacking fault energy; however, the stacking fault energy is known to be a function of solute content. The presence of these twins is therefore attributed to the unusually high Ti content produced by electrodeposition.

Electrodeposits containing 25 at.% Ti are single phase and reveal electron diffraction patterns which are consistent with the ordered FCC $L1_2$ structure. The superlattice reflections are clearly apparent in electron diffraction patterns from as-deposited alloys. The equilibrium structure of Al_3Ti is the tetragonal DO_{22}; however, reports in the literature indicate that the $L1_2$ has been stabilized in ternary alloys containing at least 8 at.% of a transition metal substituted for Al.

Consolidation of Electrodeposited MMC Precursors.

C. E. Johnson, D. S. Lashmore, G. R. Stafford, R. Jiggetts, and S. W. Claggett

Four types of electrodeposited metal matrices, applied to alumina fiber tows by batch processing, have been consolidated by hot isostatic pressing (HIP) at 600 °C and 173 MPa. (25ksi). High packing densities with minimal fiber-fiber contact has been achieved.

The four precursors include: (1) Sumitomo 85 $Al_2O_3$15SiO_2 fibers coated with 0.5 μm of autocatalytic nickel-phosphorous (Ni-P) and 20 μm of electrolytic copper; (2) 3M α-Al_2O_3 fiber coated with 0.2 μm of autocatalytic Ni-P and 10μm of aluminum-manganese alloy; (3) 3M α-Al_2O_3 fibers coated with 0.2 μm of autocatalytic Ni-P and 5 μm of electrolytic Ni-P; and (4) 3M α-Al_2O_3 fibers coated with 1.5μm of autocatalytic Ni-P. Consolidation of the Ni-P matrix Al_2O_3 fibers precursors resulted in the precipitation of nickel phosphides homogeneously dispersed in the Ni-P matrix. The result is a continuous fiber reinforced composite with a dispersion strengthened or particle reinforced matrix. A scanning electron micrograph of an electroformed composite hot isostatic pressing is shown in Figure 5.

Wear Resistant Coatings for the Printing Industry

C. E. Johnson, D. R. Kelley, J. L. Mullen, and D. S. Lashmore

A 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) is in progress. The purpose of the study is (1) to evaluate, under controlled conditions, the most promising coatings, chosen from the results of NIST accelerated laboratory wear tests, 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.

Five types of coated blades have been delivered to BEP for in-situ wear testing. Blades coated with a standard hard chromium will be used as a control standard. The other four types of coatings included: (1) structure (texture) modulated chromium on W-2 and W-3 blades; (2) flame sprayed chromium oxide on W-1 and W-3 blades; (3) chromium carbide diffusion coatings on W-1 , W-2 and W-3 blades; and (4) nickel matrix particulate composite coatings on W-3 blades. For the particulate composites, laboratory investigations have shown that current density, particle loading in the electrolyte, and cationic additions to the electrolyte influence particle incorporation in the composite coatings. Electrodeposited nickel matrix composites containing 40 vol.% of 1-3 μm size boron carbide particles (B_4C) have been achieved. The B_4C particulate composite coatings have demonstrated a significant improvement in the wear performance compared with normal hard chromium deposited from the highly toxic hexavalent chromium electrolyte. It has also been shown that small additions of ammonium nitrate change the micromorphology of the normal hard chromium in such a way that the normal abrasive wear performance is improved by a factor of about two. An example of the morphology of the chromium with the addition of ammonium nitrate is shown in Figure 6.

<u>SRM Coating Thickness Standards</u>
H. G. Brown, C. R. Beauchamp, D. R. Kelley, P. N. Sharpless, and D. S. Lashmore

The Office of Standard Reference Materials now has in stock at least a one year supply of most of the thickness standards currently in production. Completion of the lead-tin composition and thickness standards has been achieved. The final statistical analysis of results is pending, with initial estimates of error being 8 to 10% for both mass/area and composition. Development of the automation of the copper on steel SRM's is continuing with efforts this past year on automating the wavelength despersive x-ray florescence instrument. A new laboratory has been designed and is currently being installed. This new installation will incorporate significantly improved ventilation, production electroplating machines designed at NIST and automated sample turning to improve current distribution.

A prototype SEM calibration panel has been electroformed, mounted, polished and viewed under a SEM. Preliminary results indicates true parallel nickel and gold lines with a highly polished, virtually scratch free surface. Progress has been significantly delayed by lack of test results.

<u>Artificial Superlattices</u>
D. S. Lashmore, C. R. Beauchamp, R. R. Oberle, L. H. Bennett, E. C. Soltani, and L. J. Swartzendruber

Electrochemical systems have been developed to grow the following layer pairs: (1) iron with cobalt, chromium or copper, (2) cobalt with copper, chromium or nickel; and (3) chromium with nickel or cobalt. A new coulometer has been designed and tested which is capable of resolving 0.1 millicoulombs. This latest capability removes all limitations on the thickness of the less noble element. This previous limitation was about 2.0 μm.

Mechanical properties measurements have been carrried using ultrasonic techniques. It has been determined that there is no enhancement in the Young's elastic modulus greater than about 10% at any wavelength. These ultrasonic measurements have only approximately been corrected for texture. This data measures the elastic behavior parallel to the film plane. EXAFS studies recently completed on (111) electrodeposited Cu-Ni suggest that there exists a slight but measurable stiffening normal to the foil and no change parallel to the foil. Electrochemical characterization of a Co-Cu process and multilayer samples are being prepared. The results so far are consistent with the previous work of Lashmore and Dariel in that cobalt seems to be deposited in the FCC structure.

Magnetic viscosity measurements have now been carried out for a series of Cu-Ni films. The line shape varies with sample and temperature, but its peak always occurs at the coercive force of the material. Comparison with copper nickel samples produced by vapor deposition revealed that this magnetic aftereffect first discovered in electrodeposited materials is a characteristic of the material itself, not a characteristic of the processing technology. Lorentz electron microscopy, conducted in collaboration with the University of Windsor has revealed that the nickel layers are single domains and that the copper codeposited with the nickel (about 6 wt.%) resides in Guinier Preston type zones within the nickel.

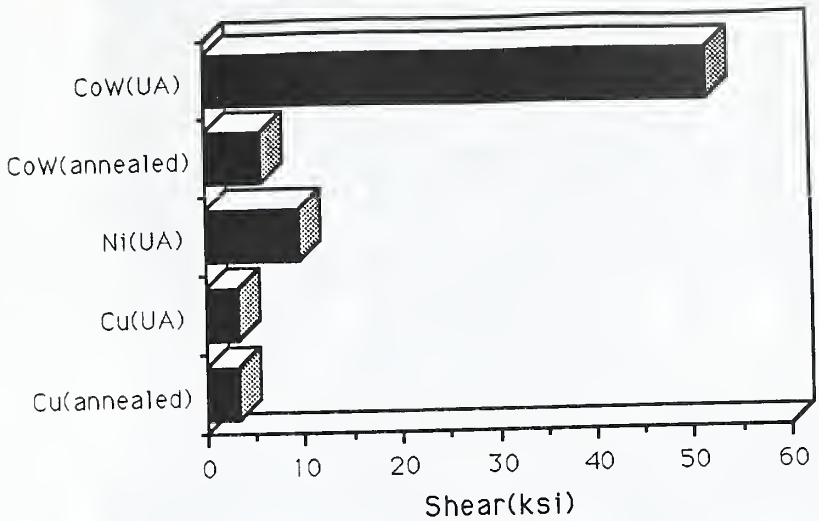

Figure 1. Adhesion data from a fiber punch-out study of fibers with various metal coatings.

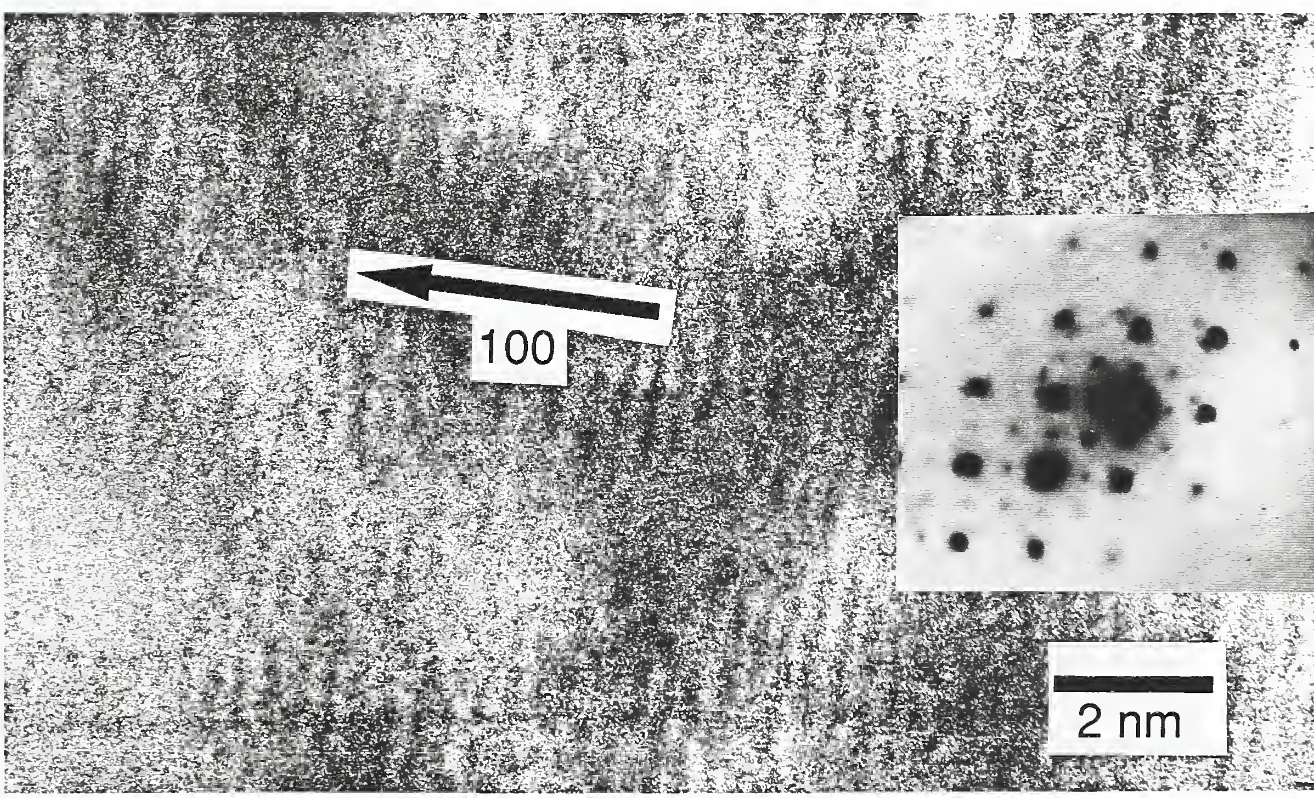

Figure 2. Selected area diffraction pattern and the corresponding bright field micrograph of cobalt-tungsten superlattice produced following exposure of 1100 °C of a CoW coated graphite tow.

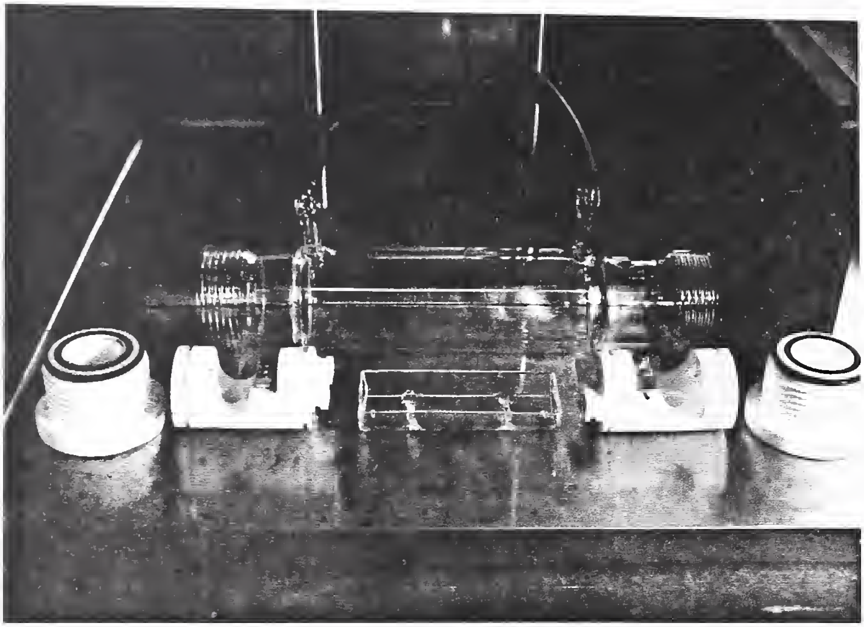

Figure 3. A photograph of the cell used for the deposition of titanium aluminides.

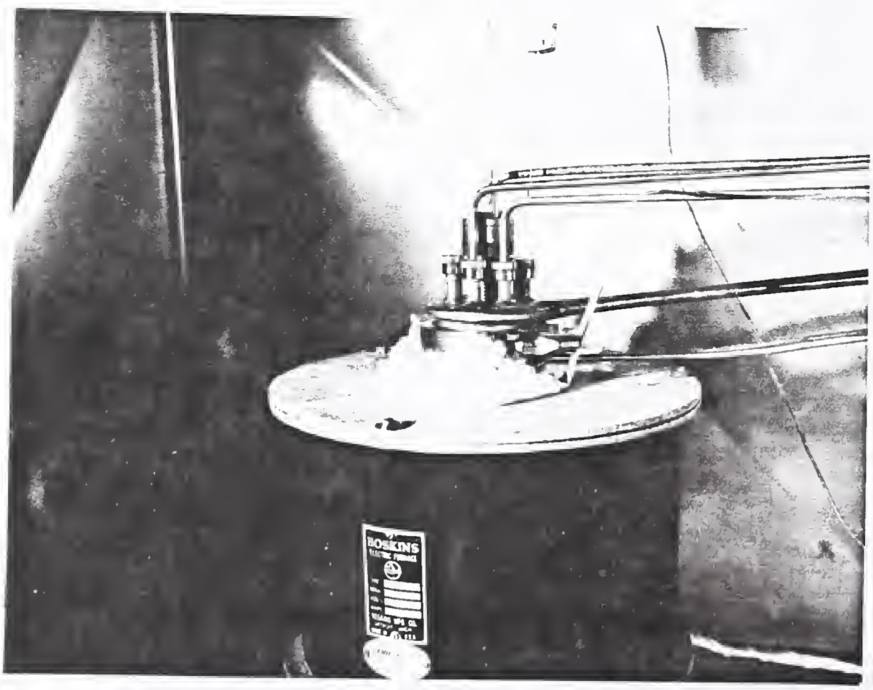

Figure 4. Photograph of the cells internal parts showing the modular design.

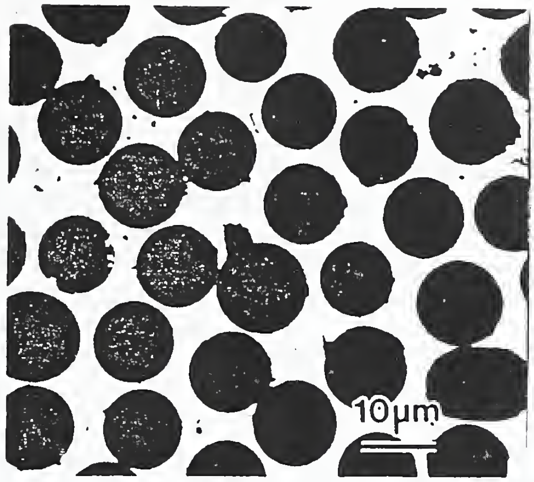

Figure 5. A scanning electron micrograph of a HIP NiP coated alumina tow illustrating the close packed geometry achievable using this technology.

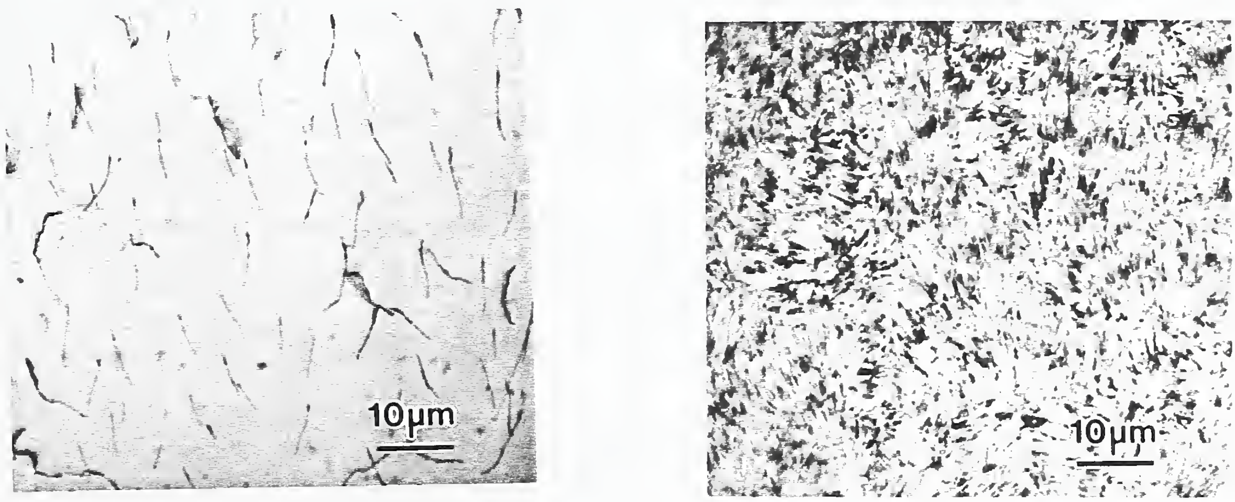

Figure 6. An example of how a chromium morphology changes with the addition of a small amount of ammonium nitrate.

MAGNETIC MATERIALS — Lawrence H. Bennett

The Magnetic Materials Group: studies advanced magnetic and superconducting materials to characterize their properties and performance, and to promote their economical processing and efficient use; develops and maintains competence in (1) the metallurgical, magnetic, and electronic structure of materials and their correlation with processing conditions, (2) magnetic measurement methods, (3) magnetic reference standards, and (4) magnetic techniques of nondestructive evaluation of materials and structures; and provides expertise to industry, universities, standards organizations, and government agencies.

Magnetic materials are important to the commerce of the nation. Sales of soft magnetic materials (information and data storage) amount to more than $25 billion per annum. Sales of hard magnetic materials (motors) are more than $1 billion per annum. Magnetic nondestructive evaluation methods are used for quality control everywhere steel is used. Conventional superconductors have many existing commercial uses, including medical magnetic resonance imaging [MRI] technology, and the new high-temperature superconductors have important potential applications, both near-term and future.

FY 90 Significant Accomplishments

- Magnetically-modulated-microwave-absorption (MAMMA) measurements of single crystals of $YBa_2Cu_3O_7$ were compared with SQUID magnetometry measurements of the same crystals. The MAMMA technique resolves structure not seen in the SQUID results.

- It was discovered that the interaction energy between flux vortices and twin planes in $YBa_2Cu_3O_7$ changes sign near the superconducting transition temperature.

- In contrast to other rare earths, substitutions of Pr for some of the Y in $YBa_2Cu_3O_7$ causes a rapid decrease in the superconducting transition temperature. Using Mössbauer and magnetic measurements it was demonstrated that this is due to an enhanced rare-earth transition-metal interaction arising from an f-electron admixture.

- Fe atoms were found to possess large magnetic moments in Fe + silica gel nanocomposites. Post preparation heat treatment with NH_3 resulted in a three-fold enhancement in the magnetic susceptibility.

- A patent application has been filed for a new type of magnetic refrigeration material. A calorimeter for the measurement of the magnetocaloric effect has been constructed and tested.

- The angular dependence of the magnetic properties of samples of commercial magnetic tapes and disks was measured to test a vector Preisach model of magnetic recording.

- It was shown that the low frequency fall off in the power density spectrum of Barkhausen noise in ferromagnetic materials cannot be explained by assuming the noise jumps are correlated. This has important consequences concerning the correct method for obtaining and analyzing Barkhausen noise for use in NDE.

<u>High-Temperature Superconductors</u>
L.J. Swartzendruber and L.H. Bennett

A major barrier to commercial development of high T_c superconductors is the lack of understanding of the effect of <u>magnetic flux pinning</u> on critical currents, including the dynamic characteristics of the currents. Using a single crystal of $YBa_2Cu_3O_7$ [YBCO] with predominantly one variant of twin boundary, small differences in magnetic behavior were observed when the field was applied perpendicular to the c-axis and either parallel or perpendicular to the twin boundaries. These differences show that twins have a direct measurable (but small) effect on flux pinning. Below 60K, the flux-vortex pinning was found to be greater when the applied magnetic field is perpendicular to the twin boundaries. However this effect was demonstrated to be temperature dependent, reversing itself at ≈80K, as is illustrated in Figure 1. In this Figure, greater flux pinning is correlated with a higher maximum of $-4\pi M$.

By comparison with traditional superconductors, high temperature superconductors have a <u>large vortex mobility</u>. Since most practical applications of high temperature superconductors will involve operation in a mixed state containing vortices, an understanding of the dynamics of vortex motion is critical. The usual method for measurement of vortex mobility is by resistivity. This has the disadvantage that it is either unusable because of experimental problems, or not sensitive enough when the vortex driving force is small. A contactless magnetic measurement was used to determine the parameters which describe vortex motion and to determine mobilities smaller by a factor of as much as 10^6 than those obtainable by direct resistance measurement. It was found that there are basically three stages of vortex motion: (i) an initial stage of flux flow, (ii) an intermediate or flux creep stage, and (iii) a final stage in which equilibrium is approached exponentially. From the flux creep stage an activation length, ℓ, which represents the average distance between effective pinning centers is determined. When combined with the time constant for approach to equilibrium, an activation energy for flux motion is determined. From the equilibrium value of magnetic induction the vortex spacing, a, is determined. In Figure 2, the activation length and vortex spacing for a highly pinned sample of YBCO (made by a melt-growth process) are displayed. It can be seen that above 70K, ℓ rises rapidly, i.e. the distance between vortices increases rapidly. The physical significance of the intersection of the curves for ℓ and a is that below the intersection temperature the vortices consist of single flux quanta, whereas above this temperature the vortices move in groups or "bundles". In order to better understand the mechanisms of vortex pinning, measurements in strongly and weakly pinned single crystals of YBCO will be made.

Two melt-grown single crystals of $YBa_2Cu_3O_{7-x}$ 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 in different ways, giving complementary information. The MAMMA technique was found to reveal structural details that are not evident in the magnetometry. For example, the presence of phases with different oxygen content can be detected by MAMMA, whereas only an average oxygen content could be determined using SQUID 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, Mössbauer effect and magnetic susceptibility measurements were performed on a series of $Y_xPr_{1-x}Ba_2Cu_{0.98}Fe_{0.02}O_{7-\delta}$ compounds. Our results indicate that a strong magnetic interaction between the Pr ions and the Cu(1) sites is responsible for suppressing the superconducting critical temperature.

An International Conference on "Advances in Materials Science and Applications of High Temperature Superconductors" April 2-6, 1990 at NASA Goddard Space Flight Center was cosponsored by NIST. A follow-on Colloquium will be held at Johns Hopkins Applied Physics Laboratory in 1991.

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

Studies are being conducted of the magnetic properties of compositionally-modulated alloys (CMA) with individual layers in the nanometer range which have been produced by the Electrodeposition Group using electrochemical deposition. Magnetic measurements on Ni/Cu multilayer thin films have demonstrated that magnetic measurements constitute an indispensable and meaningful characterization tool for them. A new computer program has been developed to efficiently carry out magnetic viscosity measurements for these films as a function of temperature and field. 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.

Two new multilayer systems, Co/Cu and Co/Cr, produced by electrochemical deposition are being investigated. Co/Cu has turned out to be of especial interest because it has been found to be possible to make even thick layers with fcc Co. The Co/Cr system has importance as the material of choice in high-density vertical recording.

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 and permit the atomic engineering of specific material properties. Thin film Ag + Fe_3O_4 nanocomposites prepared by cosputtering and field-ion microscopy were used to investigate the microstructure and microchemistry of the materials. Stable images of the films were observed and regions of high Ag and Fe concentration were seen with sizes ≈25nm, which is consistent with earlier results from Mössbauer studies.

Bulk nanocomposite materials have been prepared from homogeneous gelled composites of iron and silica containing 5-40 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 centered at $2\theta \approx 24$ degrees and the absence of any strong Bragg scattering from the iron-containing regions, indicated that these bulk materials are comprised of nanometer-sized regions of iron compounds embedded in a silica gel matrix. As cured, these materials are paramagnetic.

Analysis of the temperature dependence of the magnetic susceptibility of these materials (see Figure 3) showed that the Fe magnetic moment was quite large ($3.9\mu_B$/Fe atom in the 11%Fe+silica gel nanocomposite), indicating that a large portion of the iron was in ionic form (both Fe^{2+} and Fe^{3+}). With increasing Fe content, C_{Fe}, the magnetic moment decreased. A significant degree of antiferromagnetic interactions between the Fe atoms were also indicated in these materials by the negative intercept on the temperature axis, T_I, of the extrapolated high temperature $1/\chi$ versus T data (see Figure 4). With increasing Fe content, T_I increased in magnitude as the strength of these interactions increased.

Heat treatment of these nanocomposites in an atmosphere of hydrogen gas at T<400°C changed the room temperature magnetic state to either superparamagnetic ($C_{Fe}<15$ wt. %) or ferromagnetic ($C_{Fe}>15$ wt. %). At low temperatures, the H_2-treated materials became magnetic spin glasses. Displaced hysteresis loops at 10 K following field cooling as well as thermomagnetic history effects were observed for these latter materials. Following a treatment in ammonia gas these nanocomposites also became either superparamagnetic ($C_{Fe}<26$ wt. %) or ferromagnetic ($C_{Fe}>26$ wt. %) at room temperature. However, there was a threefold enhancement of the magnetic susceptibilities of these materials due to the NH_3 treatment. At low temperatures these latter materials were also magnetic spin glasses, as evidenced by for example the displaced hysteresis loop obtained after cooling in a field, as shown in Figure 5 for the 18%Fe+silica gel nanocomposite, but with freezing temperatures above that of the H_2-treated nanocomposites. Efforts are now underway to prepare nanocomposites using an alumina gel and also those comprised of transition element-rare earth element pairs.

Magnetic Refrigeration

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

A calorimeter has been constructed for measuring the magnetocaloric effect to evaluate materials for use as a refrigerant in a magnetic refrigeration cycle. The adiabatic chamber (evacuated to a vacuum of $\sim 10^{-5}$ Torr) which is thermally controlled by the temperature of the surrounding area (inside a 55 kOe superconducting magnet) is connected to the sample through an adjustable heat link. The magnetocaloric effect is determined from measurements of the temperature of the sample as a function of time as the sample is first withdrawn from the magnetic field and then reinserted. Measurements on gadolinium gallium garnet (Figure 6) indicated an approximate 3K effect for a change in field of 50 kOe at 30K. Measurements on the H_2-treated 11%Fe + silica gel nanocomposite have also been performed and further work on these materials is in progress. A patent application on a new class of materials (i.e., nanocomposites exhibiting superparamagnetism) for magnetic refrigeration has been filed.

Modeling of Magnetic Recording

L. J. Swartzendruber, M. Pardavi-Horvath*, F.Z. Vajda*, and L. H. Bennett
*Guest worker-The George Washington University

Modeling of the magnetic-recording process has traditionally been based on either micromagnetic models or phenomenonological models. Micromagnetic models characterize the media on a microscopic scale by numerically implementing the system of differential equations describing the behavior of the media. Phenomenological models efficiently describe the bulk behavior of the media while obscuring the physical principles. A new paradigm, the first to bridge this gap, uses a vector moving Preisach model to represent the bulk behavior of the media with "pseudo particles" which are described micromagnetically. This model, relating bulk behavior to the underlying material properties, has been experimentally tested in the Magnetic Materials Group using commercial samples of a γ-Fe_2O_3 floppy disk, a low bias γ-Fe_2O_3 magnetic tape, and a high bias Co-doped γ-Fe_2O_3 tape. Comparisons of experimental and calculated coercive force are shown in Figure 7.

Magnetic NDE

L. J. Swartzendruber

Magnetic methods of nondestructive testing are used primarily on ferritic steels to detect defects and to characterize the metallurgical and stress state of materials. Barkhausen noise analysis, which can be used to enhance the usefulness of magnetic NDE, can be characterized by various parameters: the total noise power, the total number of Barkhausen jumps, the power density spectrum, or the first or second moment of the jump amplitude spectrum. A system for obtaining the noise in digitized form over a broad bandwidth and over a complete hysteresis loop cycle has been developed and used to investigate an important question in the theory of Barkhausen noise are the elementary Barkhausen jumps statistically independent, or correlated? Using a suitable algorithm, the digitized data were analyzed to determine the field at which each Barkhausen jump occurred and the magnitude of each jump. Correlations between jumps were investigated using a so-called "return map" such as the one plotted in Figure 8. If large jumps tended to cluster together it would be immediately evident in such a plot. No evidence for a correlation or clustering of jumps was found in either iron or nickel foils.

In addition to the traditional properties of Barkhausen noise, a method was developed for characterizing such noise, termed the "jumpsum" method. In this method an algorithm is used which essentially performs a pulse height analysis of the jumps as a function of applied magnetic field. The height of these jumps is then summed as a function of field. This procedure turns the noise jumps is then summed as a function of field. This procedure turns the noise into a relatively smooth curve after only a single hysteresis cycle. Because the frequency at which Barkhausen noise is observed is usually quite low, on the order of 0.1 Hz or less, the use of this method to collect data can result in a significant time savings. This method has been used to investigate case-hardened 1050 steel and ASTM-A710 precipitation-hardening steel given different heat treatments.

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 ASTM-E709 was revised and updated based, in part, on work performed by the Magnetic Materials Group. A preliminary new ring standard using 52100 steel for use in magnetic particle inspection has being designed. The magnetic leakage fields from artificial hole patterns in these rings were measured. Based on measurements made at NIST and LTV Corp., a new hole pattern was designed. A new magnetic particle inspection method was developed based on the use of shim standards to determine the adequacy of the applied field. This purpose of the new standard is to simplify the use of the magnetic particle method by removing the considerable confusion arising from attempting to apply formulas designed for simple geometries to the many complex geometries currently being tested by the magnetic particle method.

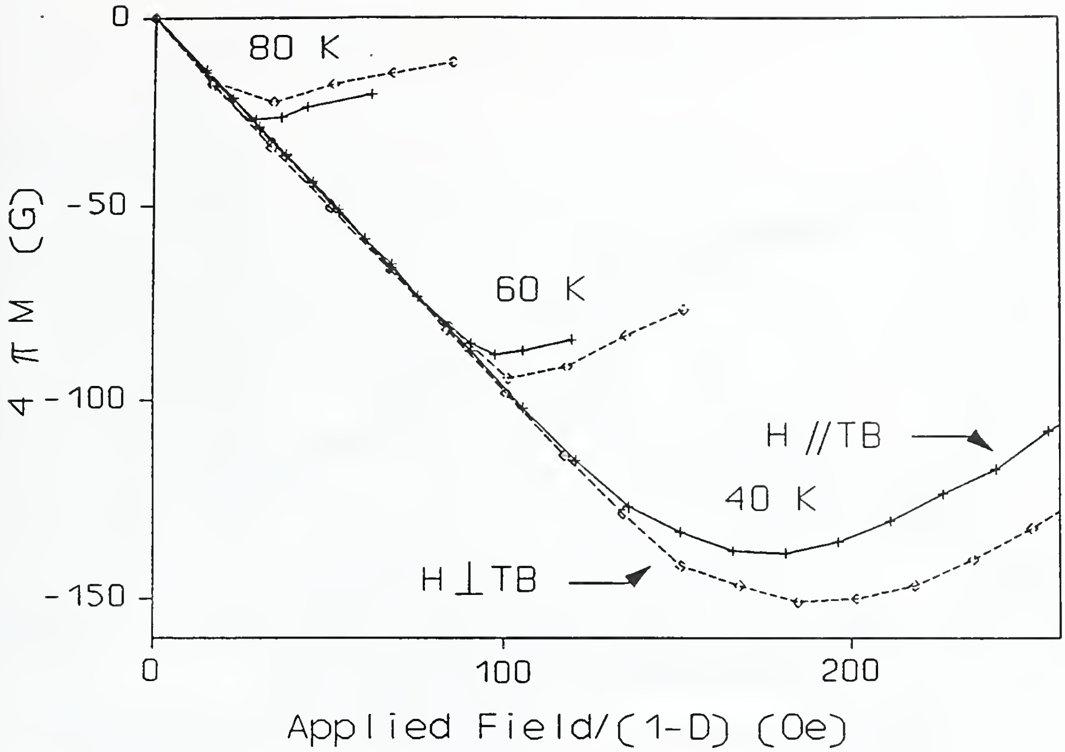

Figure 1. Magnetization vs applied field for two orientations of a single crystal of YBCO high-temperature superconductor.

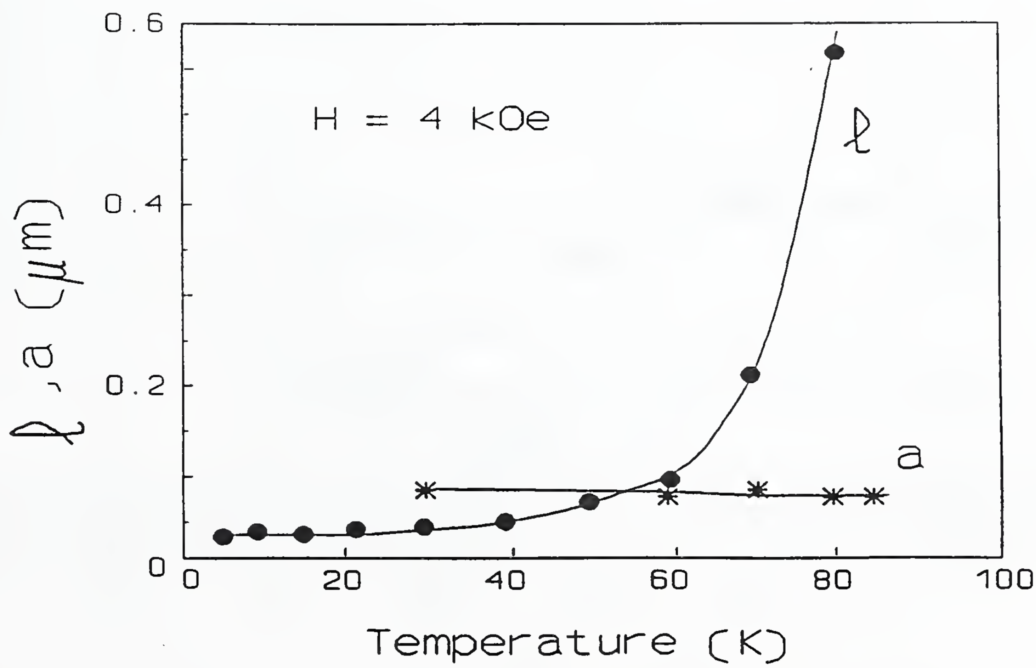

Figure 2. **Temperature dependence of the average activation length, ℓ, and intervortex spacing, a, for a highly pinned sample of YBCO.**

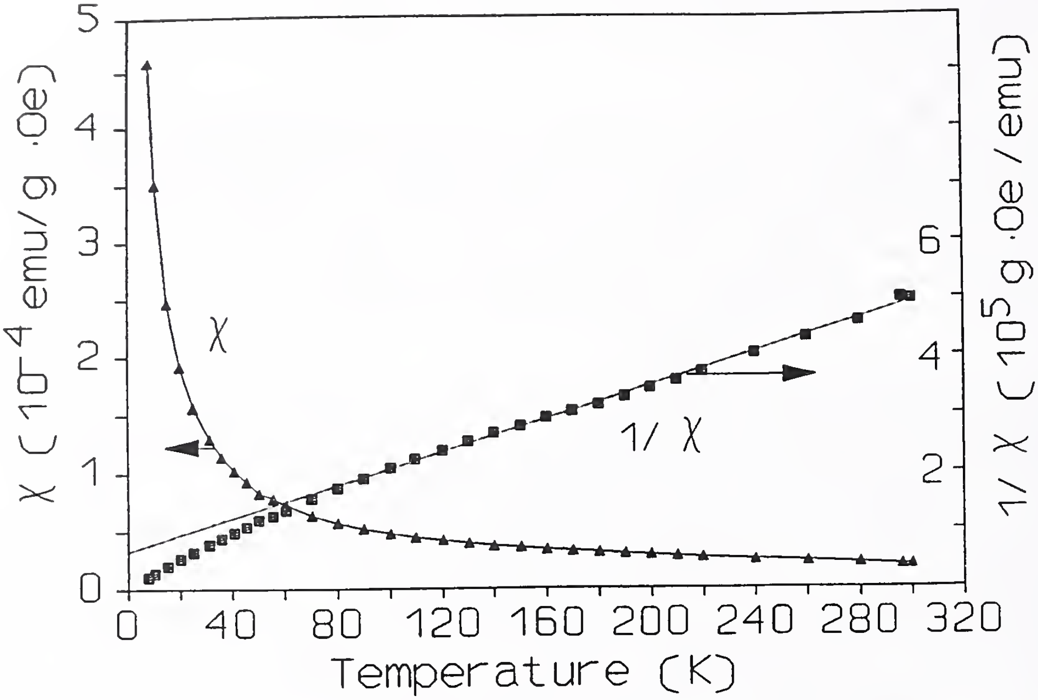

Figure 3. Magnetic susceptibility, χ, and reciprocal susceptibility, $1/\chi$, versus temperature for a 40%Fe + silica gel nanocomposite.

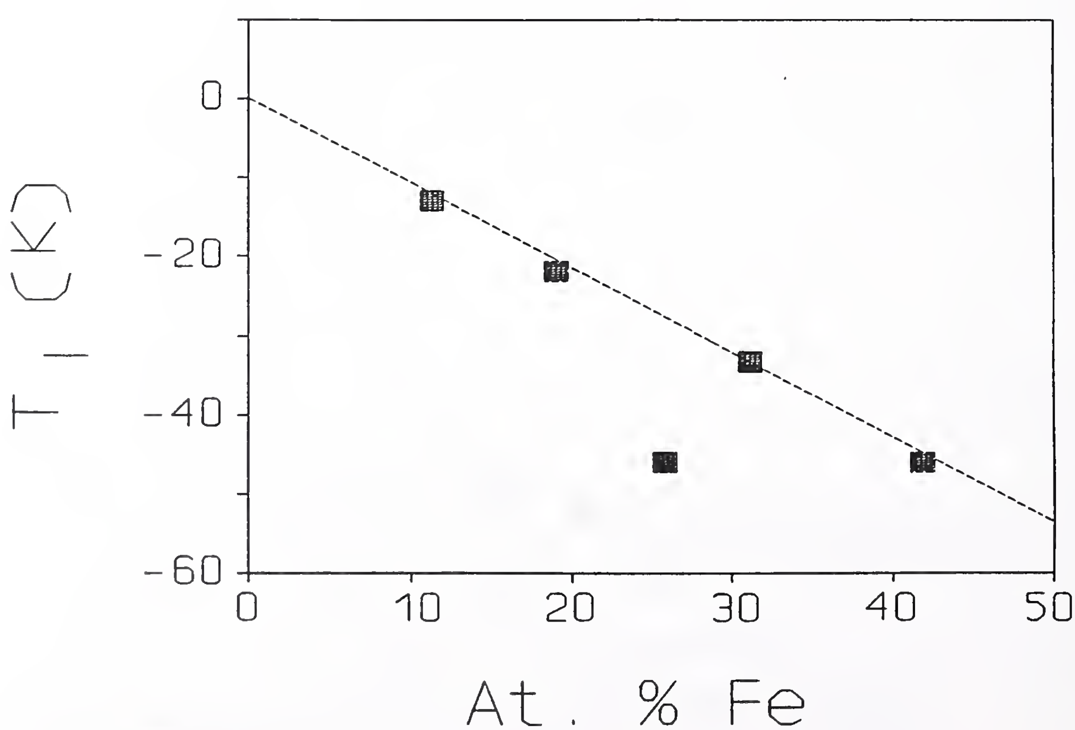

Figure 4. Interaction temperature, T_I, versus percent Fe for the same nanocomposite gel whose susceptibility is shown in Figure 3.

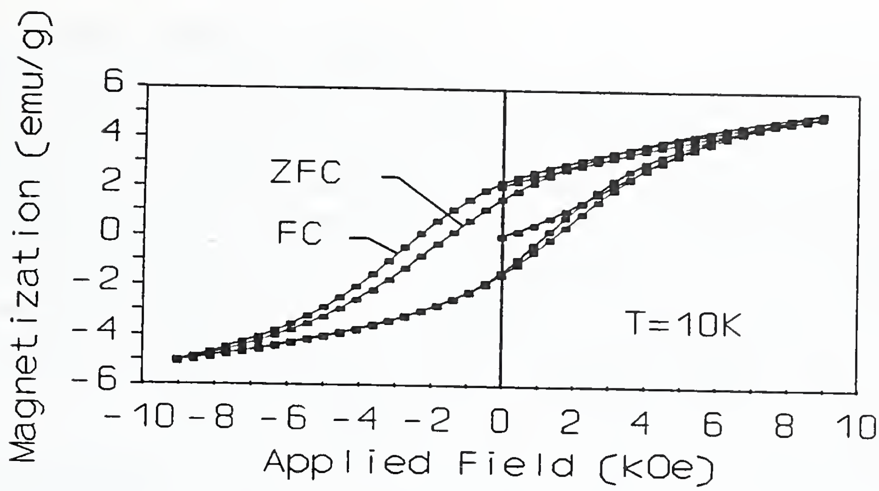

Figure 5. Hysteresis loops of a H_2 18% Fe + silica gel nanocomposite when cooled in (1) zero magnetic field (ZFC) and (2) in a 9 kOe magnetic field.

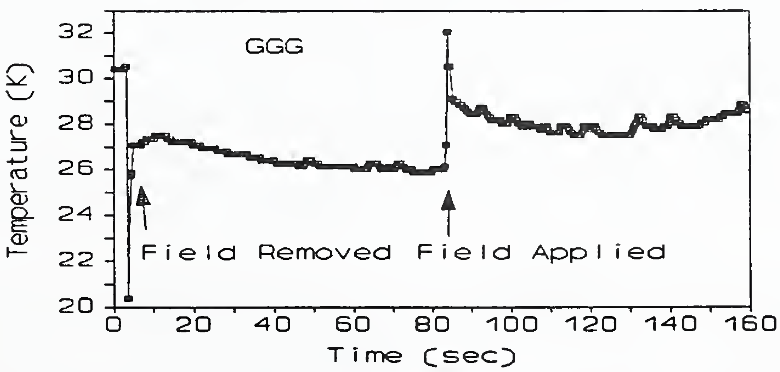

Figure 6. Calibration of the magnetocaloric probe, using gadolinium gallium garnet (GGG). The magnetocaloric effect is the temperature change (after an initial transient) upon field change.

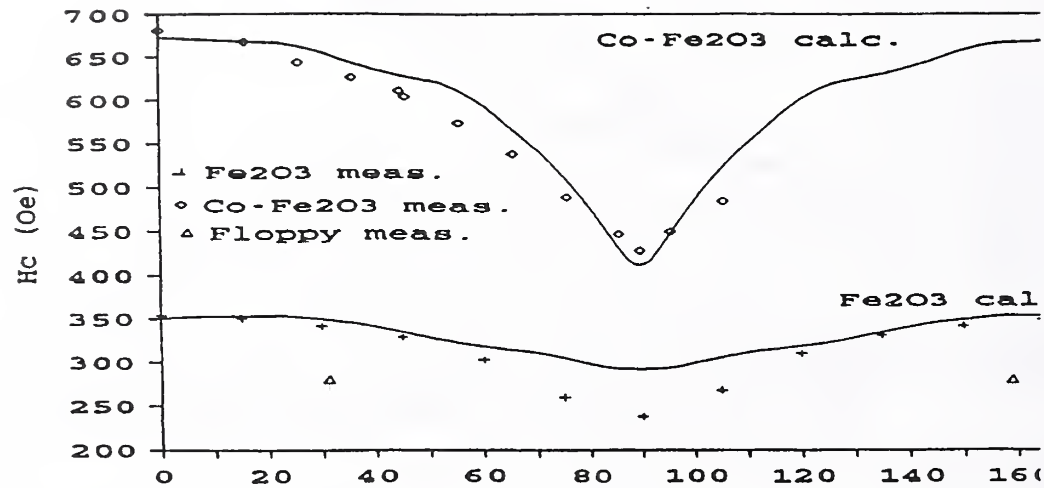

Figure 7. Angular dependence of the measured (symbols) and calculated (solid lines) coercivity. The calculation was made using a vector Preisach moving model.

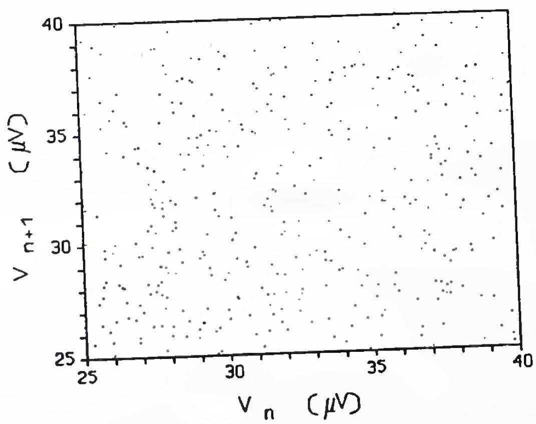

Figure 8. A portion of a return map exploring correlations for the magnitude of the Barkhausen jumps after "n+1" jumps versus the magnitude after "n" jumps observed in a 25μm thick iron foil.

HIGH TEMPERATURE MATERIALS CHEMISTRY

John W. Hastie

The high temperature materials chemistry research program 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 development of a new direct reduction iron/steel making process, through production of a thermodynamic database and solution model for prediction of slag, refractory, and inorganic inclusion thermochemistry; (2) to develop and apply (e.g., to refractory ceramics and composites) a new molecular-specific methodology for obtaining thermal and chemical stability data at ultra-high temperatures (2000 - 5000 °C). These data are needed for design of high performance materials, e.g., for hypersonic transport vehicles, defense applications, and nuclear power generation. Surveys by the National Materials Advisory Board and industry show a critical lack of thermochemical and other materials property data at these temperatures; (3) to determine basic data and process models for the preparation of thin films, including superconducting films, ferroelectric films and others, using laser or other vapor deposition techniques; (4) to carry out mechanistic studies of oxidation for enhancement of the utility of superalloys and carbon/carbon composites in high temperature oxidation environments; (5) to provide 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 through development of computer graphics for computer storage and manipulation of phase diagrams.

FY 90 Significant Accomplishments

- A previously developed thermodynamic model and its accompanying database was expanded. The model is applicable to iron-making slags and many refractories for the prediction of detailed composition and phase properties of multicomponent-multiphase iron and advanced refractory-containing oxide systems. The model database currently contains almost 200 components including many complex oxide liquids, and includes compounds of 19 different elements. Several industries and universities are beginning to substitute this model for the overly simplistic chemical models currently used in transport-based processing computer codes. Complementary experimental data for the activity coefficients of FeO in the $FeO\text{-}MgO\text{-}SiO_2$ ternary system were also obtained.

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

- In a cooperative project with the Ceramics Division and the US Army Harry Diamond Laboratories (HDL), PZT films were deposited on silicon using the

Metallurgy Division's Laser Thin Film Deposition facility. These films were successfully patterned into prototype non-volatile ferroelectric memory devices, exhibiting ferroelectric hysteresis loops that are the basis of their use as memory devices. The deposition facility was upgraded by the installation of an excimer laser system.

- A PC based program and the graphics database for Vols. 6-8 of *Phase Diagrams for Ceramists* (including nearly 3000 diagrams) was completed and the material is scheduled to be released in conjunction with a bibliographic database by NIST's Office of Standard Reference Data.

- A mass spectrometric and complementary thermochemical investigation was carried out for the ternary Nb-Al-O system. This work was performed as part of a cooperative DARPA program with CEBELCOR (M. Pourbaix, Belgium) and the University of Florida, for development of oxidation models of aerospace superalloys. Activity data on Nb-Al intermetallics and their oxidation products were obtained as part of this study. New data for NbO_2, considered to be more reliable than existing literature, were also obtained.

- A three volume conference Proceedings was prepared and is in the publication process. The manuscripts were presented as part of an IUPAC-sponsored, NIST-organized and hosted, international conference on High Temperature Materials Chemistry. Areas covered include (1) Advances in Measurement Techniques, (2) Thermochemistry and Models, (3) Processing and Synthesis, and (4) Performance under Extreme Environments. Ninety-eight comprehensive papers make up the Proceedings, which will be the first reference work covering the multidisciplinary area of materials chemistry at high temperatures in such depth.

Steel Slag -Refractory Thermochemistry

J. W. Hastie, E. R. Plante, D. W. Bonnell, M. Kowalska[1]

[1]Guest Researcher - Tech. Univ. of Wroclaw, Poland

Modeling and complementary experimental work have continued on the development of a generic predictive model of iron and steel-making slag and refractory thermochemistry. In particular, the high FeO-content systems under current study were chosen to meet the needs of the American Iron and Steel Institute/Department of Energy (AISI-DOE) national program to develop a new direct-reduction steel making process.

Experimental measurements of the FeO activity in a group of model ternary $FeO-SiO_2-MgO$ slags with constant mole ratio ($\chi(MgO)/\chi(SiO_2) = 1$) have been completed. These data were obtained and analyzed using the mass spectrometric ion ratio method developed by Belton and Fruehan. The data show an even stronger positive deviation from ideality than available literature, with values for the activity coefficient of FeO exceeding three at 20 mol% FeO and 1973 K.

The computer-based model is general, predicting detailed phase compositions of both simple and complex multicomponent, non-ideal, high temperature oxide slag systems. The model is based on the consideration of complex or associated solution components (for example, $Fe_2SiO_4(\ell)$ and $CaFe_2O_4(\ell)$ in steel slags) that account for the 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 computer model is already in use by several steel and other industrial laboratories and consortia. In addition to its present utility for existing steel-making technology, the model is designed to be applicable to new direct-reduction steel making technologies being planned or under development.

Users of the model as the chemical "engine" in process codes have expressed a need to extend the lower limits of temperature validity of current database fits below the current ~1000 - 1400 K limits. Because individual component coefficient fits in their current $\Delta_f G$ form are sensitive to changes of state in their constituent standard state elements, use of segmented fits of the database and modification of the SOLGASMIX multicomponent solution equilibrium code is being evaluated as a possible solution. The database currently contains almost 200 components covering 19 elements. Regular evaluation of the literature continues, with new species of interest to steel-making being added as quality data are found and validated.

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

D. W. Bonnell, P. K. Schenck, J. W. Hastie, M. Joseph[1]

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Coupling 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 previously inaccessible by 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.

In earlier work we have 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 LVMS 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 and optical spectrometric time-of-arrival information suggests that ions produced by the laser heating process are not all at thermal equilibrium with the neutral atomic and molecular vapor species. However, 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 sensitive to ions and neutrals.

Current work includes studies of the refractory carbides (Ta_2C, SiC), MgO, and the most refractory known oxide, HfO_2. Figure 1 shows a typical mass spectrum (ion intensity *vs.* mass-to charge ratio) obtained from laser vaporization of hafnium oxide. Of particular note is the observation of species HfO^+, Hf^+, O^+ and O_2^+, in addition to HfO_2^+. With the possible exception of O_2, time-of-arrival (TOA) analysis showed these species to be parent ions of the neutral precursors. The O_2 signal was present at barely the limit of detection, so no direct judgement could be made regarding its origin. Mass spectral ion intensity data, corrected for instrument transmission, geometric losses, and precursor cross sections are proportional to species partial pressures. Typical results derived from Fig. 1 are shown in Fig. 2. The temperature was derived from relative TOA times and the agreement with extrapolation from available literature data measured at almost half the absolute temperature is surprisingly good (see Fig. 2). Even the $O_2/O\cdot$ ratio is in good agreement with the assigned temperature.

Mechanistic Determination of Laser Deposition of Thin Films

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The objective of this project, which involves collaboration with the Magnetic Materials group and the Electronic Ceramics group of the Ceramics Division, is to determine the mechanisms of laser vaporization and deposition of thin films. Earlier work has shown that thin films, deposited by laser vaporization from bulk high temperature superconducting material, can exhibit high temperature superconductivity. Time resolved mass spectra using the LVMS technique were obtained 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 the US Army Harry Diamond Laboratories (HDL), laser deposition studies of lead zirconate titanate (PZT) thin films on silicon started in FY89 have continued. PZT is a prime candidate for use in ferroelectric thin-film non-volatile memory chips. A high speed optical multichannel analyzer was used to apply optical spectrometric techniques to the study of the laser generated plumes from PZT targets. Figures 3 and 4 show the time-resolved optical emission spectra obtained from the PZT plume at distances of 2 and 7 mm above the target surface. Special computer software and reference spectra taken of single element targets aids in emission line identification. Analysis of these spectra indicates that the laser pulse forms a plasma, containing ionized metal atoms which persist after the laser pulse. This result suggests that the interaction of the plasma with the target surface may, in part, influence the vaporization. An HDL group patterned memory devices on NIST-produced PZT films on silicon. Electrical measurements on the patterned films exhibited ferroelectric hysteresis loops, shown in Fig. 5, which are the basis of the use of this material in memory chips. Further work is underway to correlate plume species with the composition, morphology and electrical characteristics of the resulting thin films.

The Laser Thin Film Deposition facility has been using a Q-switched Nd/YAG laser, operating in the IR and visible, as the vaporization source. A new Excimer laser system has been acquired for the facility to allow vaporization of targets using UV laser light. Installation of the Excimer laser system allows the Nd/YAG laser to be used as the pump for a dye laser system to probe the laser generated plumes, or to allow for synchronous IR laser treatment of the plume or depositing film.

Phase Diagram Graphics
P. K. Schenck

Computer software has been developed to handle complex binary and ternary phase diagrams using stand alone desk top computers in support of the Phase Diagrams for Ceramists Data Center. This graphics system generated the publication-ready diagrams for the recently published (American Ceramics Society) Vols. 6 - 8 of *Phase Diagrams for Ceramists*. 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 database for Vols. 6-8 (including nearly 3000 diagrams) are scheduled to be released in conjunction with a bibliographic database by NIST's Office of Standard Reference Data.

Processing and Protection of High Temperature Structural Materials
J. W. Hastie, D. W. Bonnell, E. R. Plante, M. Kowalska[1]

[1]Guest Scientist - Tech. Univ. of Wroclaw, Poland

This activity represents a collaborative effort among NIST, the University of Florida/Gainesville, and CEBELCOR (Belgium) under a DARPA sponsored program. 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 that provide for improved design and evaluation of metallic and composite refractory systems. In particular, niobium alloys and the Nb-Al-O system are of interest, 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. Recently, an extensive literature survey and critical evaluation of thermochemical data was made for the Nb-Al-O system. Experimental measurements include studies of the vapor pressure of $NbO_2(s)$ and the interaction of Nb-Al phases with Al_2O_3. The literature data and experimental results have been provided to M. Pourbaix (CEBELCOR) for further representation as E (potential) vs. T stability diagrams.

Measurements of the vapor pressure over $NbO_2(s)$ were obtained using modulated beam Knudsen mass spectrometry. These measurements have been analyzed using both weight loss calibration and silver calibration of the spectrometer. Using weight loss calibrations, the $NbO_2(g)$ pressures are only slightly above the data previously reported by Shchukarev while analysis using the silver calibration data gives pressures close to the much lower Kamegashira data. This interpretation difference suggests that the cross section of $NbO_2(g)$ is much smaller than that estimated using cross section additivity rules. The problem of molecular cross sections for high temperature chemistry is a current project of the IUPAC High temperature committee in which this group has a lead role.

Additional measurements have been made involving the evaporation of $NbAl_3$ from an alumina cup contained in a tungsten crucible. These results yield Al_2O pressures comparable to observed Al pressures, possibly due to difficulties in maintaining saturation pressures in this type of experiment. If equilibrium is obtained, these measurements can be used to obtain the activity of Al in $NbAl_3$.

<u>Stored Chemical Energy Systems</u>

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[1]Ceramics Division

The objective of this task is to study, via mass spectrometry and collateral kinetic measurements, the reaction products of potential oxidants such as $ClO_3F(g)$, $F_2(g)$, or organic or inorganic fluoride oxidizers with reactive metal alloys such as Li-Al-Mg. Determination of reaction pathways and energetics provides necessary data for intelligent design of stored chemical energy systems. Previous data have shown that, under laboratory steady state conditions, the Cl_2 and O_2 gas pressures are orders of magnitude higher than the F_2 gas pressure, which indicates preferential fluorination of the alloys is occurring. Under higher pressure conditions (0.3 atm) in the transpiration mass spectrometer, reaction with oxygen takes place erratically as evidenced by large fluctuations in the observed O_2 signal.

Current measurements involve the study of tungsten with SF_6 because the oxidant is introduced to the alloy fuel in some devices via tungsten tubing. Knudsen mass spectrometric data suggests that $WF_6(g)$ is formed, leading to significant corrosion. Work is in progress to confirm that observation under higher pressure conditions.

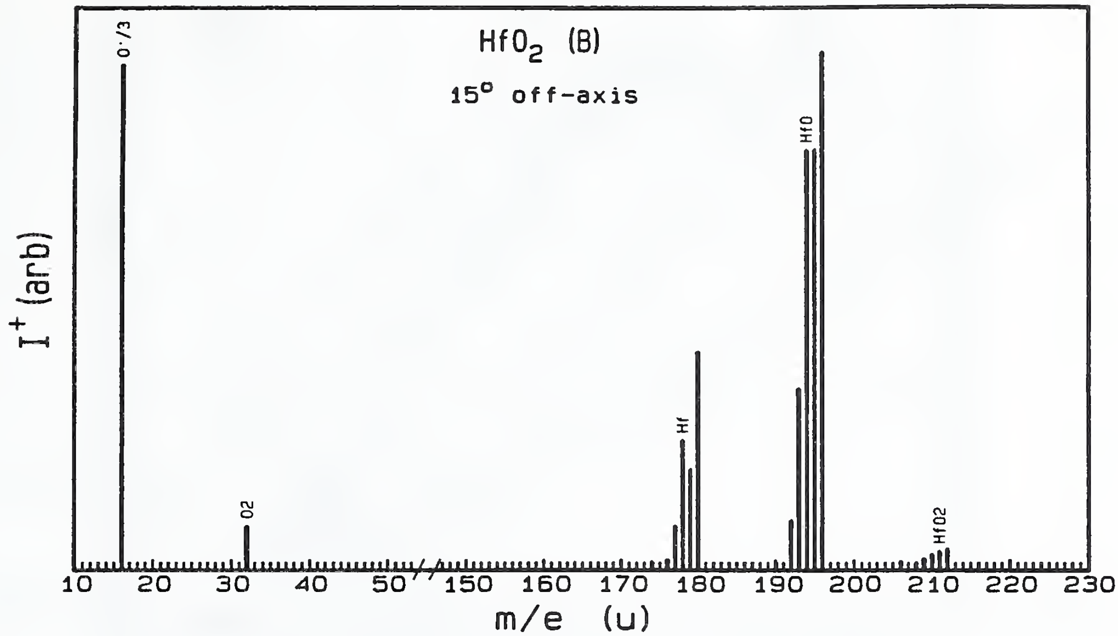

Figure 1. HfO_2 vapor species mass spectrum, obtained at 20 eV ionizing electron energy and T ~ 4900 K, showing ion intensities as a function of mass/charge ratio (nominally molecular weight).

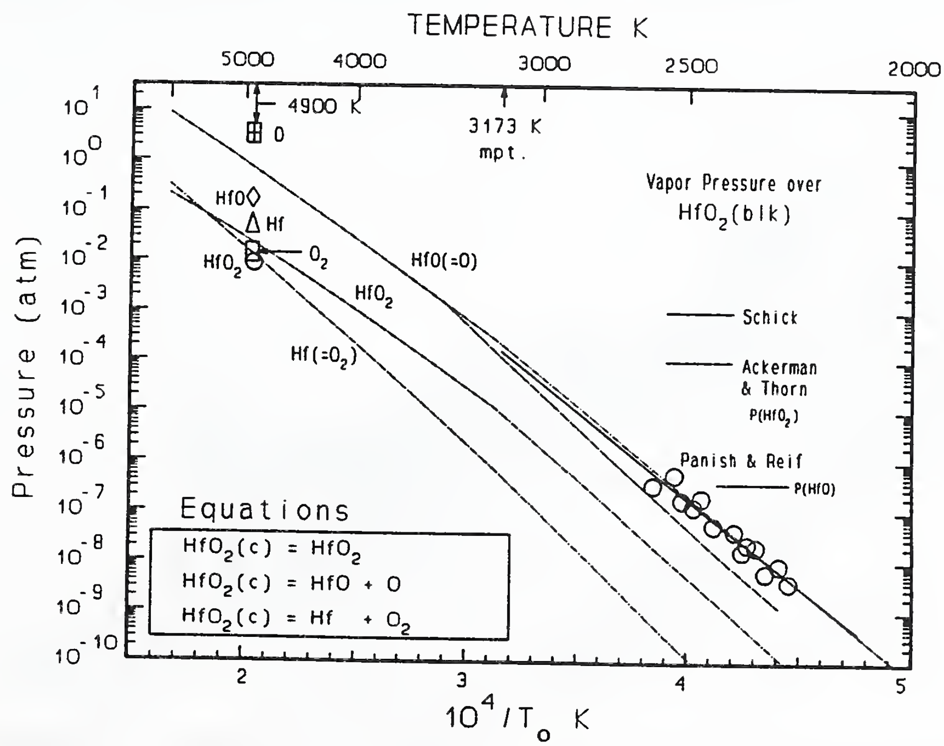

Figure 2. Vapor pressures of Hf-O species (symbols) plotted with literature extrapolations (curves).

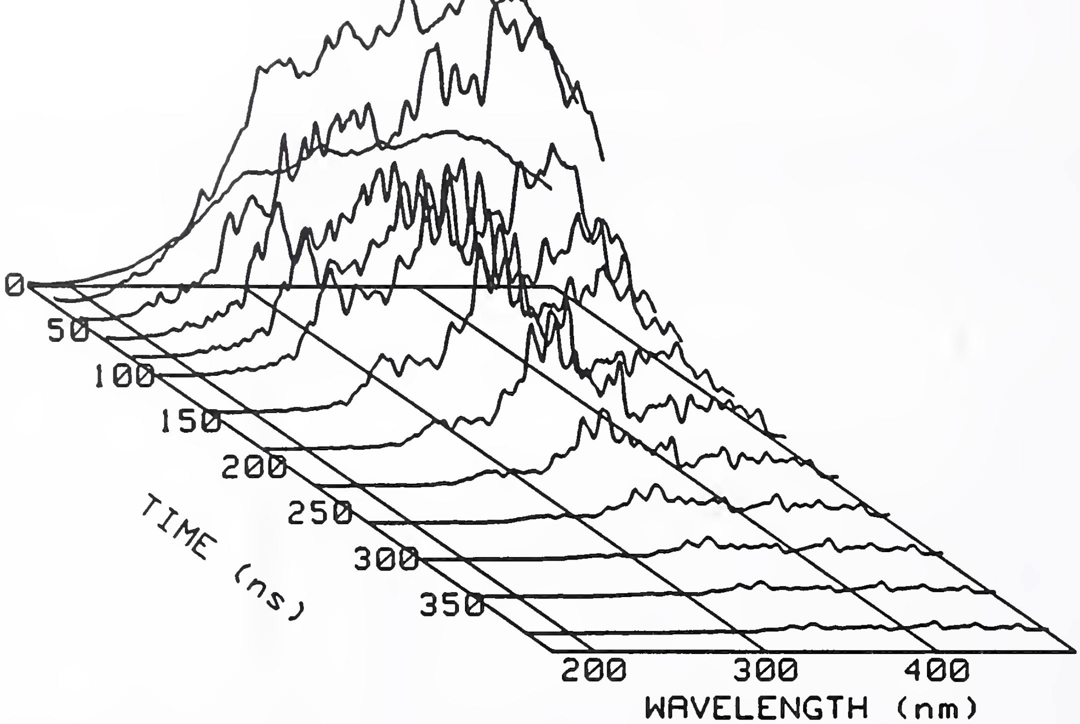

Figure 3. Time-resolved emission spectra obtained from the laser-induced plume at 2.0 - 2.5 mm above the PZT target (scale: 256048).

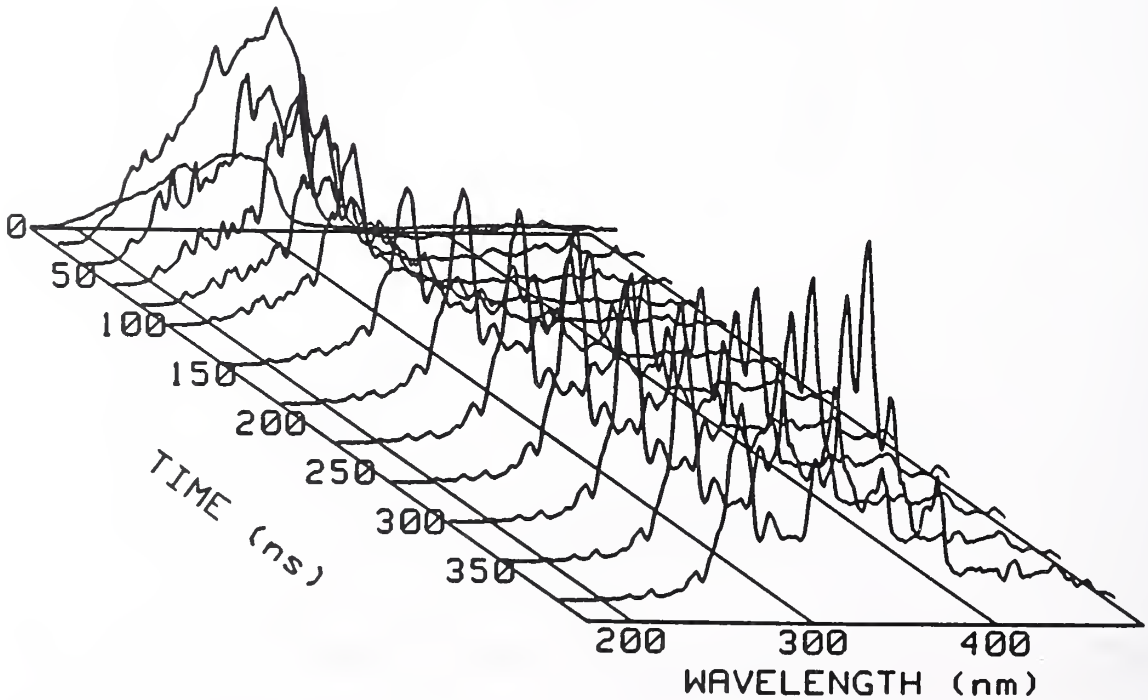

Figure 4. Time-resolved emission spectra obtained from the laser-induced plume at 7.0-7.5 mm above the PZT target (Scale:18186).

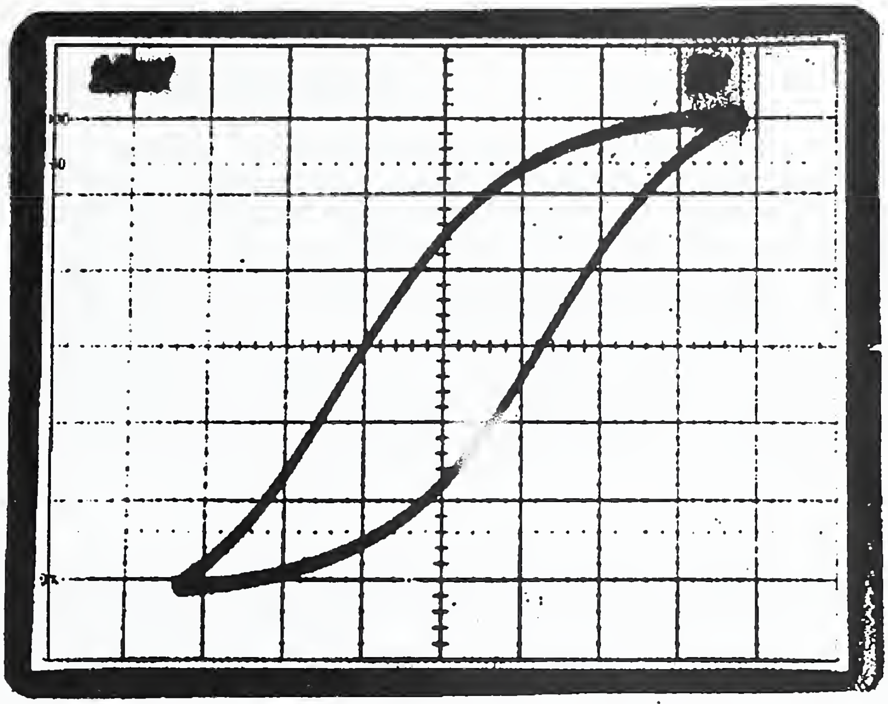

Figure 5. Ferroelectric hysteresis loop from PZT on silicon thin film. The oscilloscope trace corresponds to a plot of the polarization (-10 to +10 $\mu C/cm^2$) *vs.* applied electric field (-200 to +200 kV/cm).

MECHANICAL PROPERTIES AND PERFORMANCE — Leonard Mordfin

The Mechanical Properties and Performance Group was established during Fiscal Year 1990 in response to industrial needs for reliable data on the mechanical properties of advanced engineering materials, such as composites and intermetallics, as well as of conventional materials under the diverse environmental and operating conditions experienced in processing and service; and for new and improved mechanical test methods and standards suitable for use from the micro scale (e.g., for electronic solder connections) to the multi-million pound-force scale (e.g., for fracture tests of pressure vessel steels). The Group is also charged with providing expert consultation and testing services relating to the mechanical performance of materials and structural components, including failure analysis, for other government agencies.

Substantial effort during the year was directed toward upgrading the Group's research and testing facilities, particularly in the areas of specialized testing, data acquisition and heat treating. Test systems are newly available with enhanced capabilities for high-cycle rotating-beam fatigue; tensile and compressive strain rates down to 0.0005 cm/min; Vickers hardness testing over a wide temperature range (-60 C to 500 C); high-temperature tension, compression (2500 C), and creep (1100 C) testing in vacuum; and high-precision axial and diametral strain measurements allowing determination of Poisson's ratio. Two new automated data acquisition systems were added; a universal test machine was upgraded with a computer-assisted set-up, control, and data acquisition system, and a general purpose data acquisition system was developed for use with most of the laboratory's measuring instrumentation. The Group's heat treating facilities were also greatly expanded with the addition of three new furnaces: a high-temperature (2500 C), high-vacuum (10^{-7} Torr) system; a programmable tempering furnace; and a 1700 C heat treating furnace.

FY 90 Significant Accomplishments

- The physical and mechanical properties of the intermetallic compounds that form in lead-tin solder connections to copper were measured, most of them for the first time. These measurements revealed, among other things, that these compounds are harder than high-strength steel at room temperature but only twice as tough as glass.

- Mechanical property measurements were carried out on nitrogenated stainless steels processed by powder metallurgy techniques. One particular austenitic steel, which had been melted and atomized with nitrogen, exhibited a yield strength approximately double that for the same steel processed with argon.

- A method was developed for predicting creep under anisothermal conditions from constant temperature creep data. Comparisons with experimental results for a structural steel under simulated fire conditions showed excellent agreement.

- o Intercomparison studies of hardness test blocks produced by different manufacturers were completed for the four most commonly used Rockwell scales. The results demonstrated a serious need for uniform hardness standards. NIST is formulating a program in response to this need.

- o A method was developed for estimating grain sizes in different 3-dimensional directions from readily determined 2-dimensional measurements. In addition, and in order to test this scheme, a computer code was devised for generating 3-dimensional arrays of space-filling polyhedra which allows extraction of 2-dimensional sections.

- o A molten glass etchant was developed which highlights prior austenite grain boundaries in commercial steels.

Mechanical Properties of Electronic Solder Joint Materials
R. J. Fields, S. R. Low, III, and D. E. Harne

In a multi-year contract from the U.S. Army Harry Diamond Laboratories, the Office of Nondestructive Evaluation established a NIST-wide project last year to develop intelligent processing technology for electronic solder connections, in order to increase their reliability. One of the important features of the project is the generation of a database on the mechanical properties of the materials involved in electronic solder connections. When solder connections are made, thin layers or dispersions of brittle intermetallic compounds are nucleated near the interfaces between the solder and the base metal. In the case of conventional tin-lead solder used on copper leads, the principal intermetallics formed are Cu_6Sn_5 and Cu_3Sn. If the leads are plated with a nickel diffusion barrier, Ni_3Sn_4 is also formed. These intermetallic regions grow in size even at room temperature and strongly affect the performance of the solder connections.

The elastic, plastic and fracture properties of the various materials found in solder joints were determined using bulk samples and traditional measurement techniques. The mechanical properties of high purity 63 Sn - 37 Pb solder were determined as a function of crosshead speed at room temperature. The mechanical properties of the intermetallics were determined as a function of temperature. In addition, the thermal expansion coefficients of Cu_6Sn_5 and Ni_3Sn_4 were measured between -55 C and +200 C.

Using standard tensile bars of the solder, stress-strain curves were recorded at crosshead speeds between 0.02 and 20 inches per minute. From these curves, Young's moduli, 0.2% offset yield and ultimate tensile strengths, and elongations to fracture were determined. The results showed Young's modulus to be relatively constant while the strength values increased significantly with crosshead speed. Elongation showed a strong inverse relationship to crosshead speed.

Torsion tests of the solder were also carried out. Comparison of the yield strengths in tension with those in pure shear, for equivalent strain rates, indicates that the yield strength of solder under multiaxial stress conditions is determined more closely by the octahedral shear stress (the von Mises criterion) than the maximum shear stress (the Tresca criterion).

To measure the hardness of the intermetallic compounds at various temperatures, the device shown in Figure 1 was designed and constructed. It consists of a diamond Vickers hardness indenter mounted on a screw-driven crosshead capable of a wide range of displacement rates. The lower anvil, on which the disk-shaped specimen rests, is rotatable about an axis which is parallel to, but offset from, the loading axis, so that a series of indentations can be made over the surface of the specimen without moving it relative to the anvil. The indenter and the anvil are enclosed in a chamber that can be heated or cooled to test temperatures of interest.

The device was used to study the three intermetallic compounds over a temperature range extending from -55 to +125 C and above. Under some conditions of load and temperature the indentations produced cracks which were analyzed to determine the fracture toughness. The test data disclosed the surprising result that these compounds are harder than high-strength steel at room temperature, but only twice as tough as glass. Raising the temperature produces some softening but very little toughening.

Finally, the thermal expansion coefficients of Cu_6Sn_5 and Ni_3Sn_4 were determined using a standard dilatometric method. The results show that the expansion behavior of Cu_6Sn_5 is almost identical to that of pure copper, so that stresses are unlikely to develop between these two materials due to temperature changes. The thermal expansion of Ni_3Sn_4, on the other hand, is slightly higher than that of pure nickel and slightly lower than that of pure copper, so that low values of thermal stress could be developed in solder connections to nickel-plated copper.

Nitrogenated Stainless Steels by Gas Atomization

G. M. Janowski* and R. J. Fields

*Postdoctoral research associate; presently University of Alabama at Birmingham.

Nitrogen-containing austenitic stainless steels offer advantages as engineering materials as a result of their enhanced strength and resistance to corrosion, in comparison with non-nitrogenated alloys, while retaining the low-temperature toughness and high weldability typical of austenitic stainless steels. Research in the Mechanical Properties and Performance and Metallurgical Processing Groups addressed the development and characterization of powder-metallurgy-processed nitrogenated austenitic stainless steels. The powder metallurgy approach had not been attempted elsewhere.

The nitrogen-containing austenitic stainless steel powders were produced by melting in a nitrogen-based atmosphere and subsequently atomizing with nitrogen gas. This processing approach allowed the incorporation of up to 0.21 wt% nitrogen into a modified version of alloy 304L containing 22 wt% Cr. Atomizing with nitrogen (as opposed to argon or helium) reduces the occurrence of gas-filled bubbles in the powders. The gas contained in these bubbles is not removed during subsequent processing and may ultimately reduce the toughness, ductility, and fatigue resistance of the material.

Mechanical property measurements showed that the nitrogen acts as a potent strengthener of consolidated 304L powders. For example, the 22 wt% Cr, 0.21 wt% N alloy has approximately double the yield strength of stainless steel that was melted and atomized with argon (the usual atomizing gas) and otherwise identically processed. The hardness, yield strength, and work-hardening rate are all significantly increased by the addition of nitrogen in the range of nitrogen contents examined in these studies. These initial data indicate that the properties of P/M-processed nitrogenated stainless steels are equivalent to those of materials fabricated using other methods, while offering the benefits of near net shape. Future work on nitrogenated stainless steels will focus on determining the fracture toughness, tensile properties, fatigue properties, and processing/property relationships.

Characterization of Two Tank Car Steels Used in the Transportation of Hazardous Materials

G. E. Hicho and J. H. Smith

Research was conducted for the Federal Railroad Administration on normalized AAR TC128 grade B steel and an experimental control-rolled steel, A 8XX, considered as a replacement for the normalized AAR TC128 grade B steel. Both steels were made to fine grain practice and using a new refining process, inclusion shape control practice (ISCP), to control the shape of sulfide inclusions. Steels made using the conventional refining process contain manganese sulfide inclusions that are lenticular in shape. Using ISCP caused the inclusions to become spherical in shape. This practice reduces the anisotropy in the wrought steel and increases the upper shelf impact energy compared with steels made using normal refining processes.

A complete metallurgical characterization was performed on these steels. Charpy V-notch impact (CVN), tensile, and fracture toughness tests were carried out from -62 C to room temperature. The tensile tests showed that the yield strength of the A 8XX grade B steel was higher than that of the normalized AAR TC128 grade B steel. However, at temperatures below -12 C, the CVN impact and fracture toughness properties for the normalized and inclusion-shape-controlled AAR TC128 grade B steel were found to be better than those for the control-rolled and inclusion-shape-controlled A 8XX grade B steel. Fracture toughness tests showed that the normalized and inclusion-shape-controlled AAR TC 128 grade B steel was more resistant to both crack initiation and propagation than control-rolled and inclusion shape controlled A 8XX grade B steel. The nil-ductility transition temperature (NDT), or the highest temperature at which cleavage or brittle fracture occurs, was found to be -40 C for the AAR TC 128 grade B steel and -23 C for A 8XX steel.

Examinations of the microstructure of both steels revealed that the control-rolling conditions used on the experimental steel A 8XX did not produce the degree of grain refinement obtained in the normalized and inclusion-shape-controlled AAR TC 128 grade B steel. The use of ISCP for normalized AAR TC128 grade B steel improved the CVN, tensile, and fracture toughness properties compared to both the conventionally processed AAR TC128 grade B steel, and the control-rolled and inclusion-shape-controlled A 8XX grade B steel.

<u>Constitutive Behavior of Metal Powder Compacts</u>
R. J. Fields and S. R. Low, III

During the consolidation of metal powders, particularly by hot isostatic pressing (HIP), changes in the shape of the compact are often different from those intended. Costly test runs must be undertaken, or oversized shapes must be made, to assure that a product has the correct dimensions or can be machined to them. The present research program has as its objective the understanding and prediction of shape change during the HIP process. It involves the experimental evaluation of constitutive equations describing the deformation behavior of metal powder under general, but predominantly compressive, stress states as a function of temperature. The results of experiments are compared with finite element simulations to further improve the modelling of consolidation processes and to provide industry with reliable methods to predict shape change.

Shape change during powder consolidation usually occurs at relative densities of 0.6 to 0.85 and is a consequence of plastic or creep deformation in the presence of some deviatoric stress state. Three constitutive equations have been proposed recently to describe the powder's behavior under these conditions. By consolidating powder compacts in the specified density range under stress states having well known hydrostatic and deviatoric components, the resulting shape and volume changes were accurately measured for comparison with the proposed constitutive equations. These measurements were made at room temperature on titanium powder to assure that the measured deformation was due only to elasticity and plasticity. This work will be extended to high temperatures where creep becomes important.

<u>A Physical Basis for Crack-Arrest Toughnesses Significantly Above Those Specified in the ASME Code for Nuclear Pressure Vessels</u>
Roland deWit and R. J. Fields

The Nuclear Regulatory Commission sponsored a series of wide-plate, crack-arrest tests at NIST as part of the Heavy Section Steel Technology program. Post-test mechanical analyses revealed extremely high crack-arrest toughnesses, well in excess of those required by the ASME Boiler and Pressure Vessel Code (Section XI, Article A-4200). The objective of this project is to physically confirm that the cleavage cracks in the wide-plate tests can and did arrest at a very high applied stress intensity factor and before a macroscopic plastic zone formed. A lack of such physical evidence would compromise the validity of the mechanical analyses which are required for the resolution of safety issues relating to pressure vessel integrity.

All the data from the sixteen wide-plate tests done at NIST are being critically evaluated, assembled, and recorded in a form which permits retention of full temporal precision that is easily read by computers. Most of the instrumentation records of the wide-plate tests were originally saved on floppy disks. About two thirds of these records have been reviewed, transferred to a PC, smoothed if necessary, and converted from volts to mechanical quantities. In this effort a data format was developed to store the wave forms compactly in binary data files, and a program was developed to display the data. Each test can be fitted on a set of four 360K PC disks. Each of these sets also includes several text files of explanatory material, several files of additional data in text format, and the display program. These disks are now available for distribution for further study.

The archival data can be used to confirm the validity of the various theories used to justify the high arrest toughnesses that are indicated by the mechanical analysis. The data from test WP 2.6 were examined using a strain analysis procedure. From the strain field around the crack tip, as recorded by the nearby strain gages, a smooth curve for the crack position as a function of time from crack initiation to the first arrest was developed. The dynamic stress-intensity factor, K_{ID}, as a function of time and position was determined. The value at time $t = 0$ represents the initiation toughness, which was 202.5 MPa$\sqrt{m}$. A 3-D static finite element analysis based on the initiation load was performed by Oak Ridge National Laboratory (ORNL) and gave 174 MPa$\sqrt{m}$. It was determined that the crack arrested at $t = 0.38$ ms with an arrest toughness of 230 MPa$\sqrt{m}$. A generation-mode dynamic finite element analysis performed by ORNL gave 204 MPa$\sqrt{m}$. From the crack position data the crack velocity was calculated as a function of time. The fundamental relationship between the dynamic stress-intensity factor and the crack velocity was derived.

Additional analysis based on thickness reduction measurements, TR, made along the crack propagation plane after the test, were used to interpret the wide-plate fracture events. The thickness reduction is related to the crack tip opening displacement and hence to the fracture toughness, K. That is, the indication of plastic work rate provided by the thickness reduction near the fracture plane serves as a useful preliminary assessment of the fracture toughness. The results show good agreement between K-from-TR and K computed by ORNL from a finite element generation-mode analysis.

Prediction of Elevated Temperature Deformation of Structural Steel Under Anisothermal Conditions

B. A. Fields* and R. J. Fields

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The increased use of structural steel in high-rise buildings and bridges has created a need to further understand the mechanical behavior of such steel in a fire. In previous work an equation was developed that predicts the elastic, plastic, creep, and total strains generated for any given temperature (up to 650 C), stress, and time (up to 8 hours). However, it was assumed that the temperature was constant over the duration of the fire, no allowance being made for heating or cooling times. A method and a computer program have now been developed that will predict the strain due to creep during anisothermal tests at constant load. Comparisons were made with results from anisothermal tests for AS A149, an Australian steel close to the specification for ASTM

A36. Agreement with the experimental results is excellent for several linear heating rates and one non-linear heating rate.

Hardness Test Block Standards
T. R. Shives and J. H. Smith

At the request of ASTM Subcommittee E28.06 on Indentation Hardness, NIST undertook an intercomparison study of Rockwell hardness test blocks for the four most commonly used Rockwell scales: HRC, HRB, HR30N, and HR30T. Test blocks for the intercomparison were invited from all marketers of blocks in the United States. Seven companies, representing six different block manufacturers, responded by submitting test blocks for the study. Several companies also submitted diamond indenters for use on the HRC and HR30N scale blocks.

The intercomparison studies have been completed for three levels of hardness for each of the four scales. All tests for a given scale were conducted by the same operator using the same hardness testing machine. All tests within a given series were performed using the same indenter. The results obtained through these somewhat idealized testing conditions revealed differences in hardness values for some groups of blocks that are larger than the limit established in ASTM Standard E 18-89.

The results of this study demonstrated the need for uniform Rockwell hardness standards in the United States. NIST is formulating a program in response to this need.

Examination of Aircraft Arresting-Gear Support Assemblies
G. E. Hicho and T. R. Shives

An examination of aircraft arresting-gear support assemblies is under way for the Naval Aviation Depot, Pensacola, Florida. Hat sections, through which the tailhooks are attached to the support skins on the fuselages, are fabricated from 17-7PH precipitation hardening stainless steel. The original hat sections on a particular fleet of aircraft performed satisfactorily for more than twenty years, but at least two replacement sections have failed, one just after installation but before use, and the other the first time it was used. Ultimate and yield strengths of the replacement material are within specification, but tensile tests of notched specimens indicated that the replacement material is significantly more notch sensitive than the material of the original hat sections. Metallographic examinations of the failed components show that the material had been incorrectly heat treated, resulting in higher than expected retained austenite content. Quantitative determinations of the retained austenite were obtained using x-ray diffraction and Mössbauer techniques.

Quantitative Metallography
R. J. Fields, Roland deWit and S. R. Low, III

The utility of steel depends on its mechanical behavior, formability, and other performance characteristics such as surface finish and paintability. These properties are determined by the microstructures that result from processing. In general, the 3-dimensional inclusion and grain size and shape distributions are not known, yet are vital to understanding how processing affects properties. The objective of this project is to provide a theoretical and experimental basis for quantitative stereological analyses of steel microstructures prepared by standardized metallographic techniques. The approach is to develop (1) standardized preparation and (2-dimensional) measurement techniques for inclusions and grains; and (2) stereological methods for deriving the 3-dimensional nature of the microstructure from the 2-dimensional measurements.

Standardized procedures for preparing and measuring inclusions have been established at NIST. Automated preparation equipment is now operational. A computerized, automated microscope was programmed to measure and collect the pertinent data on inclusions from prepared samples. Stereological analyses of these data are based upon a discretized, constrained minimization scheme to solve the Abel equation for determining the size distribution of spherical particles from plane sections. This analysis was extended to prolate and oblate spheroids. Application of this analysis to an artificial 2-dimensional inclusion distribution was successful, and was applied to inclusions measured on planar sections of AISI type 304 stainless steel.

Equations relating the 3-dimensional shape and size of grains to readily measured 2-dimensional quantities were developed. To test the validity of this analysis, arrays of space-filling tetrakaidecahedra were generated in a computer. Two-dimensional sections were taken, and are being measured for comparison with the known 3-dimensional shapes and sizes in the generated array. This procedure is being extended to steel processed by various methods.

Development of a Molten Glass Etch for Determining the Prior Austenitic Grain Size of Steels
G. E. Hicho, L. C. Smith, C. A. Handwerker and D. A. Kauffman*
* Ceramics Division

A hot etching procedure was used to reveal the prior austenitic grain size of several steels. Etching experiments, using molten glass, were conducted on a HSLA precipitation-hardening steel, two alloy steels, and a control-rolled steel. Experiments were also carried out on the HSLA precipitation hardening steel in order to determine the effects of prolonged heating on the austenitic grain size. For comparison purposes, the austenitic grain size of the HSLA precipitation hardening steel, as a function of temperature, was determined using a hot-stage microscope. Results showed that molten glass was more effective in revealing the prior austenitic grain size of steels than a chemical etch.

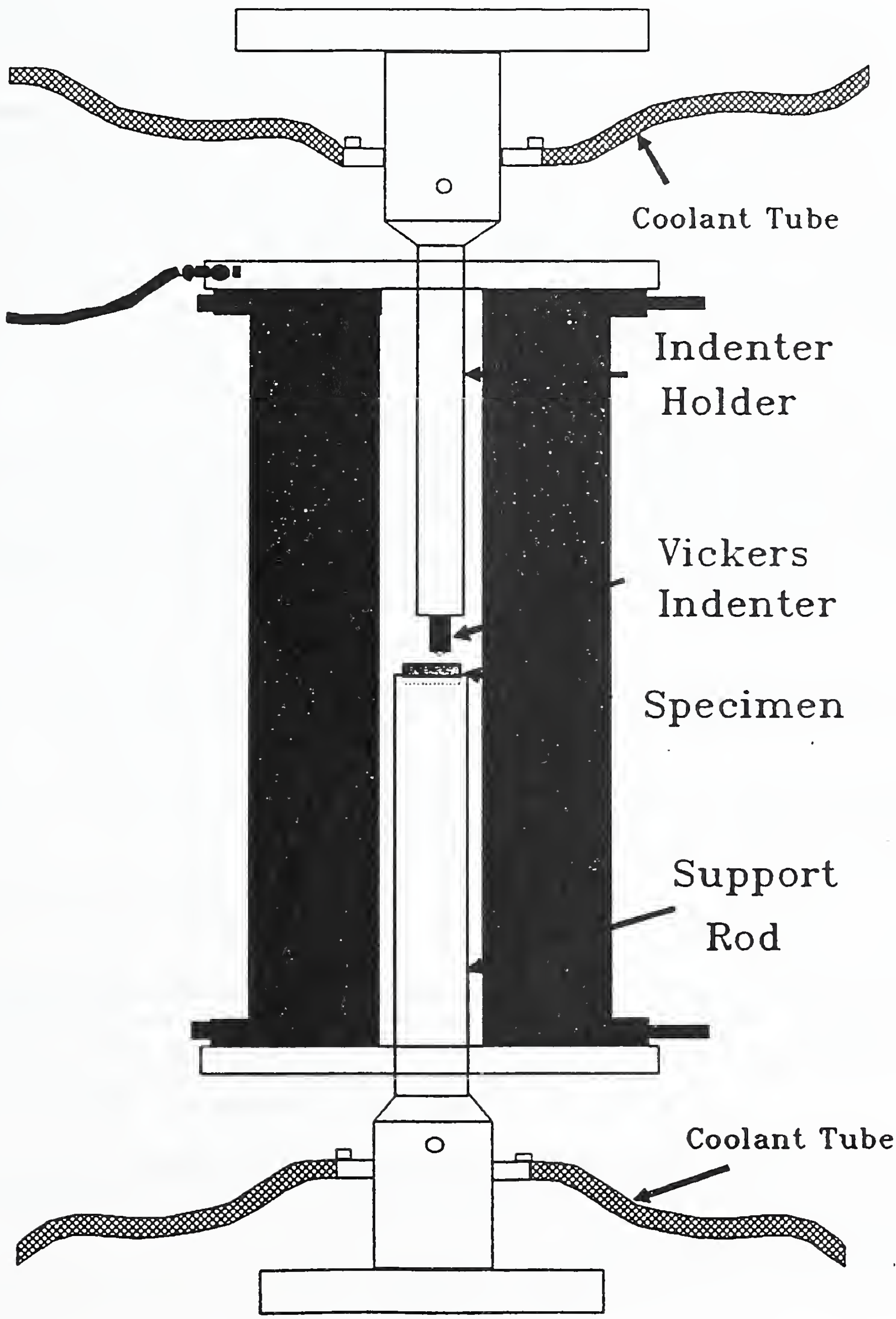

Fig. 1 Controlled temperature hardness tester.

MICROSCOPY FACILITY

John H. Smith

The mission of the Microscopy Facility is to characterize the microstructure and composition of metals and alloys.

The microstructural 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 equipment, a JEOL 840-1 electron microscope with WDS spectrometer and low light submicron backscattered electron diffraction system which was completed this year, and a ETEC Autoscan scanning electron microscope (with thermal wave imaging capability). The Microscopy Facility also has avaliable X-ray microanalyzers (Tracor Northern TN5500-5600 and TN2000) and image analysis capabilities, OMNICON image analysis system and a transmission electron microscope Philips 430 (300 kev). The facility was upgraded this year with the addition of a Tracor Northern advanced image analysis system to permit quantitative optical and electron microscopic analysis to be conducted.

Most of the activity of the Microscopy Facility 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.

FY 90 Significant Accomplishments

- The primary microstructural phases in Bi-Ca-Sr-Cu-O and Y-Ba-Cu-O high temperature superconductors have been identified and characterized by SEM analysis.
- The intermetallic phases existing at the interface in microelectronic solder joints have been identified by quantitative SEM examination.

Microstructural Characterization

A. J. Shapiro and L. C. Smith

Phase Characterization in Advanced Materials - Quantitative X-ray chemical analysis has 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 Al-Ti-Nb intermetallic compounds, and rapidly solidified Al-Mn-Fe-Si alloys and Al-Fe-Cu alloys.

Structural Characterization of Iron/Silica Gel Nanocomposites - Composite materials made from nanometer sized particles of metals and nonmetals are referred to as granular metals. Nanocomposites of iron and silica gel (11 - 40 wt% Fe) that have been polymerized at low-temperature have been studied by SEM and TEM. This material contains regions approximately 20-30 nm in diameter of at least two different phases.

Microstructural Characterization of High Temperature Superconductor Materials X-ray chemical analysis and image analysis have also been used to identify phases in the high temperature superconductors Bi-Ca-Sr-Cu-O (Pb,Sn) and Y-Ba-Cu-O systems. Thorough SEM examination of two series of ceramic samples prepared by standard solid-state sintering techniques have been performed. The stoichiometry of the series are $Bi_{1.6}Pb_xSn_{0.4-x}Sr_2Ca_2Cu_3O_z$ (where x=0.0, 0.1, 0.2, 0.3, 0.4) and $Bi_{1.9-x}Pb_xSn_{0.1}Sr_2Ca_3O_z$ (where x=0.0, 0.1, 0.2, 0.3).

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 (Cu_3Sn, Cu_6Sn_5, and Ni_3Sn_4) between the components of the solder and the materials being joined have been identified and fully characterized.

The main effort of the project has been directed toward investigation of the basic properties of the intermetallic compounds such as Cu_6Sn_5, Cu_3Sn, Ni_3Sn_4 (as well as high-purity 63 Sn-37 Pb solder) and study of their interfaces with solder under different conditions (such as aging, different wetting techniques, etc). Using SEM and X-ray analysis system (X-ray imaging, quantitative x-ray analysis) all phases have been identified. Samples of the above mentioned intermetallic compounds prepared by the HIP method revealed a single phase structure with grain size from 5 to 10 micrometers for Cu_6Sn_5 and Cu_3Sn. The sample of Ni_3Sn_4 has a substantial amount of Sn inclusions. The average grain size was near 10 micrometers. A correlation between tensile strength and appearance of the fracture surfaces of a fine dispersion of Cu_6Sn_5 powder (approximately 15% by volume)/eutectic composite solder have been made.

Microstructural Characterization of Atomized Powder of Al-5Mn-5Fe-2Si (wt%) Alloy - Aluminum-based Al-TM-Si (TM-transition metals) alloys that have been rapidly solidified and consolidated have been analyzed by SEM and X-ray microanalysis techniques. This material shows a microstructure of fine dispersoids and exhibits two major modes of crystallization. Primary crystallized cellular Al occur in particles less than 20 μm and primary crystallized nodules for larger particle sizes. The observed structure of the nodules display various growth forms of the α phase depending on degree of undercooling.

Nanocomposites prepared in a Vitreous Alumina Gel - A homogeneous gelled composite of iron and vitreous alumina containing 15% Fe prepared at room temperature by polymerization of an aqueous aluminum alkoxide solution containing ferric nitrate and nitric acid at low pH has been analyzed with SEM and TEM. It has been demonstrated that the bulk material is comprised of nanometer-sized regions of iron compounds embedded in a vitreous alumina matrix.

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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 1990, 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. 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.

2. ALCOA

   W.J. Boettinger taught a two day course for Alcoa staff members at the Alcoa Research Center (PA) on solidification path analysis.

3. Allied Signal Corporation

   Members of the Magnetic Materials Group are aiding engineers from Allied Signal involved in the design of superconducting motors using high temperature superconductors. Investigations at NIST on flux pinning and magnetic shielding are of primary interest.

4. Aluminum Association

   The fourth year of the cooperative project of NIST and the Aluminum Association has been completed successfully. Velocity effects in the measurement of electrical resistivity and temperature observed in plant trails have been modelled theoretically and the interpretation verified in laboratory experiments. Effort has been devoted to finding the effect of alloy constituent variations on temperature measurement by eddy current methods which determine resistivity.

5. American Cyanamid Corporation

   Interface studies in CoW coated graphite fibers. This program is carried out through a research associate. One patent application and one patent disclosure filed to date (N. S. Wheeler and D. S. Lashmore).

6. American Dental Association

A continuation of the cooperative program between the Electrodeposition Group and the Dental Association involves the electrochemical deposition of amorphous cobalt chromium phosphorus alloys (M. Ratzker and D. S. Lashmore).

7. American Iron and Steel Institute

Collaboration continues between K. Almand of AISI and R. J. Fields and B. A. Fields of NIST on the development of methods for predicting the mechanical behavior of structural steel exposed to fire. This year's work provided a means for dealing with the effects of the anisothermal conditions experienced in real fires.

8. 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.

9. BDM Corporation

The Metallurgical Processing Group, Advanced Sensing Group, and Mechanical Properties and Performance Group have collaborated with the BDM Corporation to develop an intelligent controller for hot isostatic pressing of intermetallic compounds. Constitutive equations for shape change during hot isostatic pressing were also developed. In this jointly funded DARPA/NIST program, NIST has developed the technology of measuring powder densification and BDM has contributed to the control software.

10. C.E.C.M./C.N.R.S.

A cooperative program is sponsored jointly by C.N.R.S. (Vitri, France) 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.

11. Collaborative Testing Services, Inc.

R. J. Fields and T. R. Shives collaborated again this year with C. Leete and J. Leete of CTS in the selection of materials, specimen fabrication processes, and procedures for interlaboratory comparison testing programs designed to measure proficiency in tensile, hardness, and fastener testing. NIST staff also conducted tests to provide input data for the comparisons.

12. Crucible Compaction Corporation

The densification of titanium powders by hot isostatic pressing is being measured and properties of partially densified titanium powders are being evaluated in a collaborative effort by NIST (R. Fields and R. Schaefer) and Crucible Compaction Corporation. The goal of this research is to develop information that can be used in finite element modelling of hot isostatic pressing processes.

13. Crucible Materials Corp., and General Electric Co.

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 third 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 in advanced sensor development and Artificial Intelligence (AI) control. Scientists from the participating companies, in collaboration with NIST scientists, have developed an in-situ particle size measurement sensor and integrated this device with an AI automatic control system.

14. DuPont

In collaboration with J.B. Parise (DuPont and SUNY-Stony Brook) and C.C. Torardi (DuPont), NIST scientists R.S. Roth (Ceramics Division) and B.P. Burton (Metallurgy Division) have synthesized and solved the crystal structures of $Ca_4Bi_6O_{13}$, $Ca_6Bi_6O_{15}$ and $Sr_2Bi_2O_5$. These are phases that may occur in oxide superconductor systems, and they are the first known crystals in which bismuth is threefold coordinated by oxide ions.

15. 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.

16. Electric Power Research Institute

EPRI continues to fund a 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 water handling equipment.

17. 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.

18. Fluoramics Corporation

The Magnetic Materials Group is cooperating with Fluoramics in their attempt to make flexible magnetic bearing material using high temperature superconductors. Flux pinning and magnetic levitation in material prepared by fluoride flux methods is under study. Results are being used by Fluoramics to help improve their production process.

19. General Dynamics

The Advanced Sensing Group is collaborating with General Dynamics, Fort Worth, TX., to develop an eddy current sensing system to monitor the electrical conductivity of carbon-carbon composites during high temperature processing.

20. General Motors

Members of the Division (L.J. Swartzendruber) have consulted with General Motors on several possible NDE projects including the measurement of case hardening depth in steel axels.

21. Harry Diamond Laboratory

In cooperation with Dr. Brody at Harry Diamond Laboratories, Dr. Peter Schenck has laser deposited lead zirconate titanate (PZT) thin films on silicon and is involved in spectroscopic characterization of the deposition vapor. PZT is a prime candidate for use in ferroelectric thin-film non-volatile memory chips.

22. IBM

Continuation of basic research on electrochemically produced artificially layered alloys. New systems have been investigated, described in the text and a new high speed coulometer integrated into the deposition system (D. S. Lashmore, C. R. Beauchamp and E. C. Soltani).

23. 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.

24. International Copper Association (ICA)

A cooperative program has been initiated in the Corrosion Data Center (D. B. Anderson) to develop expert system technology for controlling corrosion in potable water distribution systems. The program is targeted at providing guidance to system designers and operators and assure access to current technology.

25. Intersonics, Inc.

Dr. Bonnell consults with Dr. S. Krishnan regarding levitation of liquid metals, pyrometric and emissivity measurements at high temperatures, and related studies.

26. Lockheed Missiles and Space Company, Inc.

In a collaborative effort with A. Joshi of the Lockheed Missiles and Space Company R. Shull is editing a book on thermal analysis.

27. Martin-Marietta Laboratories (Baltimore), Martin-Marietta Astronautics Group (Denver)

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

28. Materials Innovation

A joint project was undertaken involving a new process for plating on difficult to plate materials. One patent disclosure resulted from this collaboration (D. S. Lashmore and D. R. Kelley).

29. Materials Technology Institute of the Chemical Process Industries (MTI)

MTI continues to support a multi-year program in the Corrosion Data Center (D. B. Anderson) to develop expert systems for selection of materials for storage and handling of hazardous chemicals. Systems are based on knowledge rules defined during discussions with consulting experts representing a broad range of industrial experience. MTI, through the National Association of Corrosion Engineers (NACE) supports two Research Associates in the Corrosion Data Center to aid in this program.

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

Dr. Bonnell collaborates and consultes on design of levitation systems for space applications.

31. National Association of Corrosion Engineers

NACE continues to support the Corrosion Data Center (D. B.Anderson) through a joint agreement with NIST. NACE continues to provide full time Research Associates at NIST plus considerable staff support for programs to collect, evaluate and disseminate corrosion data for engineering materials.

32. 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.

33. Norton Corrosion Limited, Inc.

The Corrosion Group is continuing to collaborate 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.

34. Princeton Applied Research

A joint study was undertaken on using a scanning tunnelling microscope to image electrochemically produced microlayered alloys (D. S. Lashmore and B. Rogers).

35. Society of Automotive Engineers

The Division (L.J. Swartzendruber) is cooperating with SAE in the design of a new test ring for magnetic particle inspection. Two new rings were fabricated with steel provided by Lucas Aerospace and are currently being evaluated by LTV, Dallas, Texas.

36. 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 Davis, Brigham Young University; Prof. Steve Benson, Univ. North Dakota; Prof. Tom Roberts, Milwaukee Area Technical College; Dr. C. David Rogers, Carnegie-Mellon Institute and USS of USX; Prof. Arthur E. Morris, University of Missouri-Rolla.

37. 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.

38. Technology Transfer Workshops

R. Ricker presented experimental results obtained at NIST on nickel aluminide corrosion and stress corrosion cracking at a technology transfer workshop at Oak Ridge National Laboratory. The workshop 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.

39. 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.

40. USX/Carnegie-Mellon

Dr. Rogers of Carnegie-Mellon Institute (formerly U.S. Steel of U[illegible]) 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.

41. Wilson Instruments Co. and Clark Instrument, Inc.

Cooperative NIST/industry project to intercompare Rockwell hardness test blocks marketed in the United States was completed. Rockwell hardness testing machines were loaned to NIST for this purpose by Wilson and Clark, and test blocks were donated by most of the block manufacturers that market in the United States. Rockwell indenters were loaned by most members of the domestic indenter industry.

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. Carnegie Mellon Research Institute/R. J. Lee Group

R. J. Fields and L. Mordfin collaborated with D. Rogers of CMRI and F. Schwerer of Lee to formulate a research program for developing a nondestructive method for predicting the life cycle of steel products based on ultrasonic examinations and microstructural models.

3. Department of Agriculture
Rural Electrification Administration

The Corrosion Group has continued its collaboration with REA to develop a data base on the corrosion performance of telephone cable shielding materials.

4. 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 inelastic neutron scattering.

5. Motorola/North Texas State University

Specially prepared powder of Cu_6Sn_5 intermetallic alloys prepared in the NIST metals processing laboratory were supplied to scientists at Motorola Corp. and North Texas State University for investigations of the effects of these materials in solder joints. Solder joints processed at Motorola Corp. were subsequently characterized by NIST scientists for homogeneity.

6. 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., Naval Air Development Center, NASA, Johns Hopkins Univ., UCSB, and Univ. of Wisconsin have interacted in this program.

7. Swedish Corrosion Institute

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 (NCM) and the United Nations Economic Commission For Europe (UNECE). A representative of the NIST Corrosion Group was Chairman of the three day meeting directed at evaluating the corrosion of materials in soil.

8. 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. Now, 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.

9. The International Group for Historic Aircraft Recovery (TIGHAR), Mr. R. E. Gillespie, Wilmington, DE

Twelve days before Lindberg's successful crossing of the Atlantic Ocean, two French aviators attempted to fly from France to New York. They never arrived at their destination. It was rumored that they had lost their way and crashed in Maine. TIGHAR believes that they have located the crash site, and they have collected evidence to support this possibility. After 75 years, little of the aircraft is expected to be found. However, TIGHAR found magnetic material at the site and they asked us to examine the material for possible evidence of metals.

<u>UNIVERSITIES</u>

1. 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.

2. Carnegie-Mellon University

F. Gayle is collaborating with Prof. James Howe of Carnegie-Mellon University in an investigation of a new precipitate phase in ultra-high strength aluminum alloys using High Resolution Electron Microscopy.

3. 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.

4. 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.

5. Cornell University

The Magnetic Materials Group is cooperating with Dr. F.C. Moon of the College of Engineering in developing standardized methods for measuring the levitation capabilities of high temperature superconductors. The primary goal is to develop appropriate criteria for the specification of material suitable for use in magnetic bearings.

6. Duke University

A positron annihilation study of defect structures in ultra-high strength, precipitation-hardened aluminum alloys has been initiated between F.W. Gayle (NIST) and Prof. Phillip Jones and Scott Rader (Duke University). The goal is an understanding of the extraordinary age-hardening response of this new class of Al-Cu-Li alloys.

7. 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.

8. George Washington University

A collaborative effort is underway between E. Della Torre, M. Pardavi-Horvath, and F. Vajada of George Washington University and R. Shull, L. Swartzendruber, and L. Bennett of NIST to model the magnetization behavior of assemblies of fine magnetic particle systems.

9. George Washington University

George Hicho serves as an Associate Professorial Lecturer in Engineering at GWU in the area of material property testing.

10. 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.

11. 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.

12. 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 paper has been published on this topic.

13. Johns Hopkins University

A joint program continues involving ultrasonic measurements of the elastic properties of Cu-Ni microlayered alloys (M. Rosen, R. R. Oberle R. Cammerata and D. S. Lashmore).

14. Johns Hopkins University

Studies on the mechanism of stress corrosion cracking have been pursued in cooperation with Johns Hopkins University (Dr.K. Sieradzki). The experimental studies were carried out at NBS by a graduate student (F. Friedersdorf), and the staff of the Corrosion Group was involved both in the experimental and in the analysis of the results.

15. Johns Hopkins

The magnetic properties of detwinned superconducting $YBa_2Cu_3O_{6+x}$ single crystals fabricated at NIST (D. Kaiser and F.W. Gayle) are being measured at Johns Hopkins (K. Moorjani) using the recently-developed magnetically modulated microwave absorption (MAMMA) technique. The objective of this effort is to study the details of the superconducting transition in twinned and detwinned crystals in order to further the understanding of the mechanism of high temperature superconductivity.

16. Lehigh University

R. J. Fields continued his collaboration with Professors T. J. Delph and D. G. Harlow in the quantification and modelling of creep cavitation. The research this year centered on the correct distribution function to describe the spatially nonuniform occurrence of creep cavities on grain boundaries.

17. Louisiana State University

A collaborative effort is underway between F.W. Gayle and D.L. Kaiser of NIST and Prof. Steven Watkins (LSU) to investigate details of crystal structure of detwinned single crystals of $YBa_2Cu_3O_{6+x}$ using single crystal diffractometry at cryogenic temperatures.

18. Metalurski Institut (MI), Ljubljana, Yugoslavia

Dr. Hastie is collaborating with Dr. V. Presern of MI on a NIST-Yugoslavia Joint Project research program in steel inclusion thermochemistry.

19. 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.

20. 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.

21. Oregon State University

Collaborative work on Ti-Al-Nb alloys was done by L.A. Bendersky of NIST and C.B. Shoemaker of Oregon State University, who used material prepared at NIST to determine by single crystal x-ray diffraction the collapse parameters and site occupancies for the ω" and $B8_2$ phase structures in the alloy system.

22. 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.

23. Stanford University

R. Shull of NIST is collaborating with H. Saffari of Stanford University to measure the effect of "blooming" layers on the magneto-optical effect of nickel.

24. University of Florida

Collaborative studies by the University of Florida (R. Abbaschian) and NIST (S. Coriell) on prediction and measurement of instabilities produced at solid-liquid interfaces during directional solidification were recently begun.

25. University of Houston

In this joint effort between the University of Houston (Prof. S. Moss) and NIST (D. Kaiser, F. Gayle), detwinned single crystals of $YBa_2Cu_3O_{6+x}$ are being measured by high-resolution x-ray diffraction techniques at temperatures in the range 10 - 298 K to search for a structural transition associated with the onset of superconductivity. Fully untwinned crystals are required for these measurements to eliminate ambiguities introduced by the presence of twin boundaries which are present in all as-grown crystals.

26. University of Maryland

A collaborative study between the University of Maryland (Dr. A. Roytburd) and NIST (Dr. D. Kaiser, Dr. F. Gayle, Dr. L. Swartzendruber, Dr. L. Bennett) involves theoretical aspects of twin boundary migration under an applied stress and flux pinning by twin boundaries in $YBa_2Cu_3O_{6+x}$. A model has been developed to calculate the velocity of boundary migration as a function of temperature and applied stress. Magnetization measurements demonstrating an effect of twin boundaries on flux pinning have been understood in terms of vortex/twin boundary interactions.

27. University of Maryland

R. deWit and R. J. Fields collaborate with Professor G. R. Irwin to further understanding of crack arrest and reinitiation behavior in steel. In particular, the relationship between thickness reductions and fracture toughness is being studied.

28. University of Maryland

Two graduate students from the University of Maryland are working on the Microlayered Alloys Program (C. R. Beauchamp, E. C. Soltani, M. Wuttig and D. S. Lashmore).

29. University of Notre Dame

R. Ricker served as examiner and advisor for the Ph.D. dissertation entitled "The Influence of Rare Earth Additions on the Resistance of 2 1/4 Cr - 1 Mo Steel to Hydrogen Embrittlement" by M. T. Fernandes.

30. University of Science and Technology - Beijing, China

In a program funded by the United Nations Industrial Development Organization (UNIDO), a seminar series on corrosion database development was provided by D.B. Anderson at UST-Beijing to an audience of students and corrosion researchers from throughout China. The program has also funded a fellowship for a Guest Researcher (Miss Changrong Li) for a one year visit in the Corrosion Data Center where her goal is to develop a prototype database addressing key Chinese industrial development needs.

31. University of Windsor

A joint program on conducting Lorentz microscopy of microlayered alloys was described in the text (M. Schlesinger and D. S. Lashmore).

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

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

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

Journal of Phase Equilibria, Editorial Committee
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Residual Stress Committee
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ASTM

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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:	Nuclear Fuel Cycle Waste Materials
E3: E3.07:	Chemical Analysis of Metals Acoustic Emission R. B. Clough
E4: E4.05:	Metallography Microhardness C. E. Johnson D. S. Lashmore
E4.14:	Quantitative Metallography R. J. Fields
E7:	Nondestructive Testing L. Mordfin, Executive Subcommittee L. J. Swartzendruber L. H. Bennett
E7:04:	Acoustic Emission

J. A. Simmons
R. B. Clough

E7.10:04:	Infrared NDT Methods L. Mordfin, Chairman
E7.91:	USA Committee for ISO TC 135 L. Mordfin, Chairman
E9:	Fatigue R. deWit R. J. Fields
E24:	Fracture Testing R. E. Ricker R. deWit R. J. Fields
E24:02	Fractography and Associated Microstructures G. E. Hicho
E24.04:	Environmental Cracking R. E. Ricker
E24.06:	Fracture Mechanics Applications J. H. Smith G. E. Hicho
E28:	Mechanical Testing L. Mordfin, Executive Subcommittee
E28.06.07:	Hardness Test Block Intercomparison T. R. Shives, Chairman R. J. Fields
E.28.13:	Residual Stress Measurement L. Mordfin, 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
G1:	Corrosion of Metals Mark Stoudt
G1.03.1:	Corrosion Data Formats Task Group D. B. Anderson, Chairman
G1.06:	SCC and Corrosion Fatigue R. E. Ricker
G1.10:	Corrosion of Metals in Soil E. Escalante
G1.10.01:	Measurement of pH of Soil E. Escalante, Task Group Leader
G1.10.02:	Measurement of Soil Resistivity E. Escalante
G1.11:	Electrochemical Measurements in Corrosion R. E. Ricker
G1.14:	Corrosion of Steel in Concrete E. Escalante

Electrochemical Society

Corrosion Division
R. E. Ricker

Deposition from Non-Aqueous Electrolytes Symposium
G. R. Stafford, Symposium Organizer

Division Editor, Electrodeposition
D. S. Lashmore

Electrodeposition Division
D. S. Lashmore, Chairman, Editor

Electronics Division
R. E. Ricker

Symposia Subcommittee
D. S. Lashmore

Gordon Conference, Physical Metallurgy Conference for Summer 1991
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High Temperature Science Journal
J. W. Hastie, Editorial Board Member

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

International Standards Organization

TC58.SC-3: Cylinder Design
J. H. Smith, Delegate

TC107: Metallic and Other Non-Organic Coatings
C. E. Johnson, Delegate

TC107.02: Methods of Inspection and Co-Ordination of Test Methods
C. E. Johnson, Delegate

TC107.03: Electrodeposited Coatings and Related Finishes
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TC135: Nondestructive Testing
L. Mordfin, Delegate

International Union of Pure and Applied Chemistry
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High Temperature and Solid State Chemistry
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Journal of Phase Equilibria
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Materials Research Society
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Materials Science and Engineering
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National Aeronautics and Space Administration
High Temperature Advisory Committee
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National Association of Corrosion Engineers (NACE)

T.3 Corrosion Science and Technology Committee

T.3E: Unit Committee on SCC and Corrosion Fatigue

R. Ricker

T.3K: Corrosion and Other Deterioration Phenomenon Associated with Concrete
E. Escalante

T.3L: Unit Committee on Electrochemical Techniques
R. Ricker

T.3U: Computers in Corrosion Control
D. B. Anderson

T.3U.1: Task Group on Corrosion Data Formats
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J. H. Smith, Reviewer of Invention Disclosures

Research in Nondestructive Evaluation, A Journal of the American Society for Nondestructive Testing
H. T. Yolken, Editor-In-Chief

Sigma Xi
R. deWit

Society for Biomaterials Standards
A. C. Fraker, Standards Representative on the ASTM F4 Committee

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

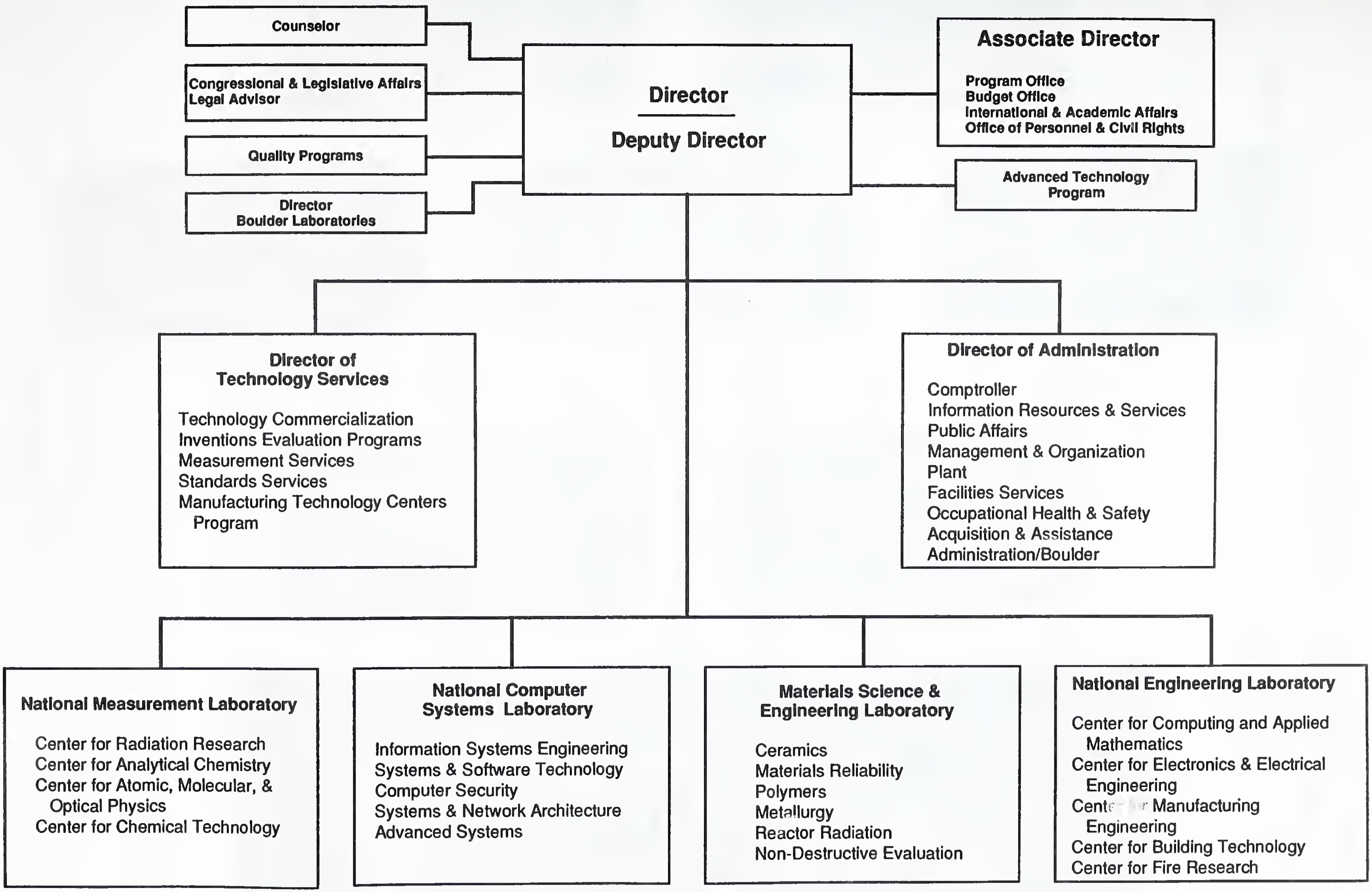
U.S. Department of Transportation
Technical Hazardous Liquid Pipeline Safety Standards Committee
R. J. Fields, Secretary

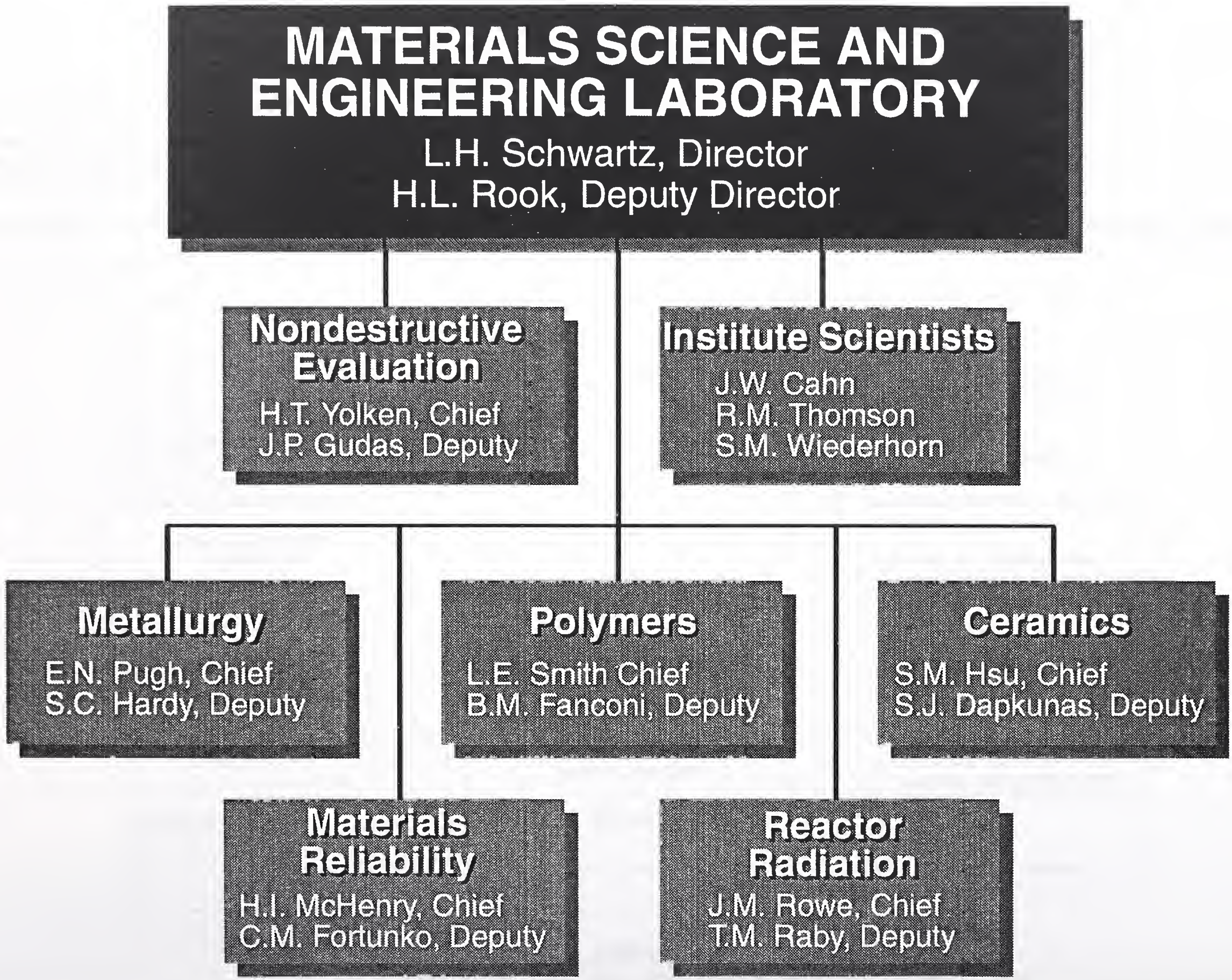
Washington Academy of Sciences
R. deWit

White House, Office of Science and Technology Policy, Council on Materials, Working Group on Materials Processing and Synthesis
H. T. Yolken, Chairman

1991 International Conference on High Temperature Materials Chemistry Organizing Committee
J. W. Hastie, Member

U.S.DEPARTMENT OF COMMERCE
National Institute of Standards and Technology

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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 1990 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 publications, committee participation, and other professional interactions of the 74 full-time and part-time permanent members of the Metallurgy Division and its 35 guest scientists 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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