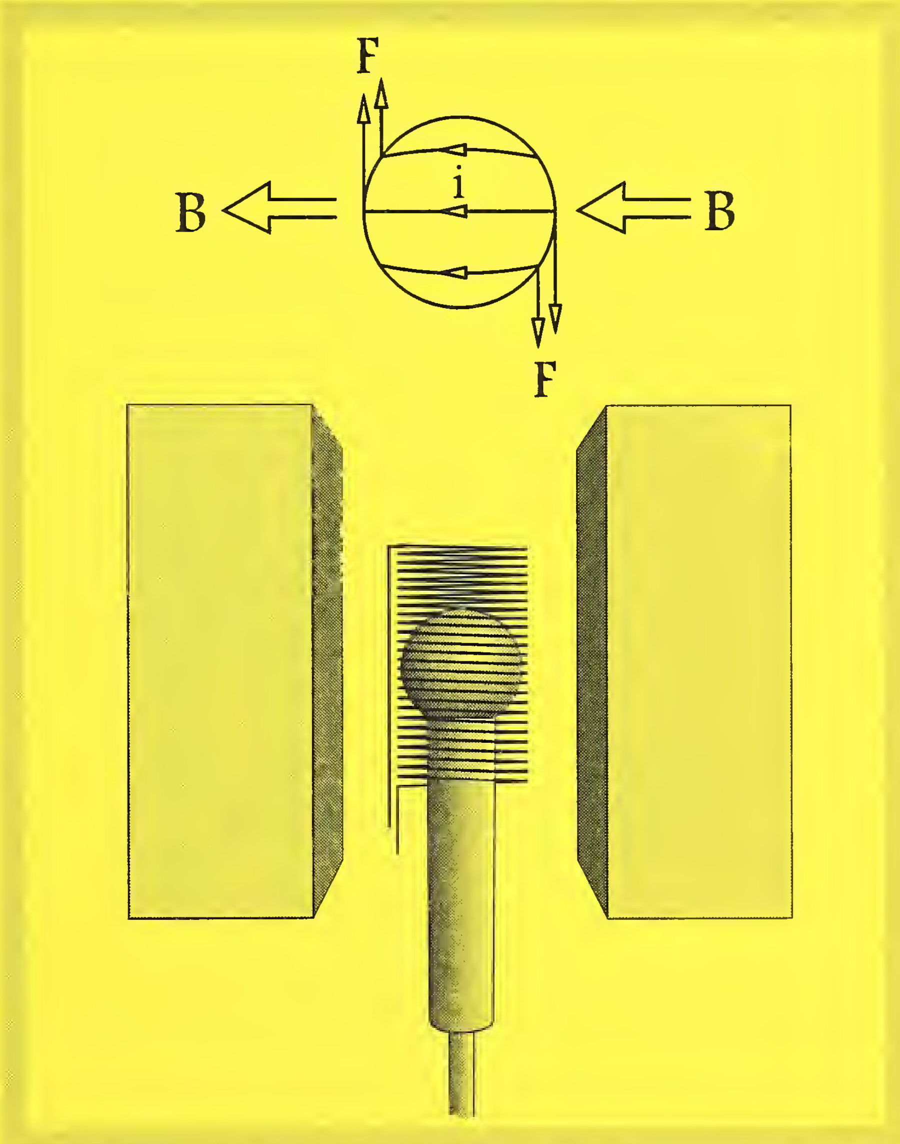
MSEL

Materials Science and Engineering Laboratory

MATERIALS RELIABILITY



NISTIR 5748
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities
1995

Materials Reliability

The cover figure depicts an electromagnetic-acoustic transduction technique for performing ultrasonic resonance measurements on spherical samples at high temperatures. The Materials Reliability Division is using this system to establish a materials science base for a proposed ultrasonic sensor that uses attenuation as a monitor of the microstructural recovery and recrystallization of cold-worked metallic alloys. The importance of monitoring these microstructural changes is particularly great for industrial annealing processes that must carefully balance increases in ductility with losses in strength.

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H. I. McHenry, Chief
T. A. Siewert, Deputy

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INTRODUCTION

MATERIALS RELIABILITY DIVISION
Harry I. McHenry, Chief
Thomas A. Siewert, Deputy

The Materials Reliability Division performs research and development on measurement technology for process control, quality assurance and materials evaluation. The measurement technologies we develop are intended to enable the producers and users of materials to improve the quality and reliability of their products.

In this report, emphasis is given to relating our research to the technical focus and goals of the Materials Science and Engineering Laboratory (MSEL). The Division research activities contribute directly to achieving the MSEL goals, as expressed in the MSEL Strategic Plan.

MSEL Goal 1: Support the measurement base and standards for materials science to serve the needs of U.S. industry.

The Division continues to concentrate its resources on three focus technologies, each of which has new measurement technology as the primary goal:

- Intelligent Processing of Materials (IPM)

  To develop on-line sensors for measuring the materials characteristics and/or processing conditions needed for real-time process control.

- Ultrasonic Characterization of Materials (UCM)

  To develop physical measurements for characterizing internal geometries of materials, such as defects, microstructures and lattice distortions.

- Micrometer-Scale Measurements for Materials Evaluation

  To develop measurement techniques for evaluating the mechanical and thermal behavior of thin films and coatings at the appropriate size scale.

MSEL Goals 2: Foster the use of advanced materials in commercial products.

Advanced materials have microstructures which are designed and controlled to provide superior properties and performance for specific functions. To consistently achieve the requisite microstructure in large-scale production, the Division is developing measurement methods for monitoring microstructural evolution during processing and for characterizing the microstructures of finished materials. We expect our measurement technologies to foster the commercialization of advanced materials by improving confidence in their properties and performance.

MSEL Goal 3: Foster the development and implementation of technologies for advanced processing of materials.

Intelligent processing is the principal approach used by the Division to achieve this MSEL goal. The central elements of IPM are (1) process understanding expressed in terms of process model, (2) real-time information on processing parameters and material condition obtained with on-line process sensors, and (3) a model-based strategy for sensing and control to achieve the desired characteristics in the finished product. Currently, we are developing sensors and process models for the control of welding, casting and thermomechanical processing. We have joint projects with industry in each area to facilitate transfer of NIST measurement technology to the control of production processes.

HIGHLIGHTS

Microstructure-Property Modeling:

We studied a short-fiber-SiC metal-matrix composite. Using sound-velocity measurements, we determined the fiber orientation-distribution function (ODF). We confirmed the ODF by two methods: acoustic-resonance spectroscopy and neutron diffraction. Using the ODF we used an extended Mori-Tanaka model to calculate the plastic-deformation properties, specifically the stress-strain curve, which we confirmed by tensile-machine measurements.

Thermal Conductivity of Ceramic Coatings:

After a complete thermal analysis and equipment upgrade to increase precision and accuracy, the high-temperature guarded hot-plate system has been used to make the first measurements of bulk thermal conductivity of a ceramic coating system and its substrate materials. Measurements on the plasma-sprayed zirconia coatings also gave a detailed evaluation of the systematic errors of the system and of the thermal resistance of the sample-apparatus interfaces.

Fatigue Testing of Thin Films:

An improved microtensile testing system based on piezo devices, upgraded data acquisition equipment, and software has been developed to measure tensile and fatigue behavior of thin films. Fatigue tests with cycles of 10 s duration have been run out to over 100 000 cycles. The apparatus has been used for tensile testing of a variety of thin films including epitaxial silicon (collaboration with Ford Microelectronics), electron beam evaporated copper, and aluminum.

Constitutive Properties of Steel:

New facilities for mechanical testing of steels in the temperature range from 900 °C to 1200 °C extended strain rate capabilities from 20 s^{-1} to 70 s^{-1}. Semi-empirical equations were developed to describe the observed stress-strain curves with strain rate, temperature and grain size as parameters.

Ultrasonic Measurement of Stress:

Electromagnetic transducers were developed for measuring the stress in steel bridge girders by ultrasonic birefringence and Rayleigh wave techniques. They were used on bridges in Virginia and West Virginia to measure service-induced loads that could not be measured by conventional strain gage techniques.

Eddy-Current Thermometer:

A noncontacting, eddy-current thermometer for monitoring the temperature of sheet aluminum as it leaves the last stand of a rolling mill at high speed and high temperature was demonstrated at the Mill Products Division of Alumax Corporation in Lancaster, PA.

Ultrasonic Inspection System:

A NIST design for couplant-free ultrasonic inspection of automotive air bag inflators was transferred to a test equipment manufacturer, and three commercial units are now performing in-line inspections at production facilities of Morton International, Inc.

Waveform-Based Ultrasonics:

An infrared Michelson interferometer for wideband ultrasonic measurements was assembled and evaluated. The interferometer is used to obtain high-fidelity ultrasonic waveforms in composite materials. Initial measurements validated the main features of the Green's-function elastic-wave-propagation theory for plate geometries.

Nonlinear Ultrasonics:

We evaluated a new, first-of-a-kind instrument for nonlinear ultrasonic studies of materials. The instrument was developed under a Cooperative Research and Development agreement (CRADA) with Ritec, Inc. The results of the NIST tests will be incorporated in a second prototype of the instrument which will be evaluated by a working group of six laboratories.

Transmission X-Ray Diffraction:

We demonstrated that a transmission x-ray diffraction technique can monitor the position of the solid-liquid interface during directional solidification. An industrial partner provided a furnace (and training on its use) for casting single-crystal turbine blades. The furnace is being used to evaluate the measurement technology under industrial conditions.

Weld Arc Sensing:

Plant trials on the Arc Sensor Module (ASM) were conducted by an automotive components manufacturer. The ASM enabled real-time detection of common welding problems which occurred in an eight-robot welding cell, thereby demonstrating the ASM as a diagnostic tool for process control.

RESEARCH STAFF

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- Process sensing and monitoring

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Schramm, Raymond E.
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- Database management of material properties
- Handbook of material properties

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- Fabrication technology

Yukawa, Sumio
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- Codes and standards
- Structural safety

TECHNICAL ACTIVITIES

INTELLIGENT PROCESSING OF MATERIALS

Intelligent processing of materials (IPM) is the conversion of materials into value-added products using model-based control of processing variables. Information for real-time process control is provided by on-line sensors which measure material characteristics and/or processing conditions. Intelligent processing will enable industry to economically produce materials with improved quality, consistant properties, and enhanced functionality. The Division has IPM projects related to welding, thermomechanical processing, and casting.

Project: WELD ARC SENSING

Project Leader: R. B. Madigan
T.A. Siewert, W.P. Dubè, T.P. Quinn, C.N. McCowan

MSEL Program	*MRD Focus Technology*
Intelligent Processing of Materials	**IPM — Welding**
Strategic Thrust	*Character of Research*
Advanced Processes	**Automotive**

Technical Description

Over the last several years we have been concentrating on the development of sensors for real-time monitoring and control of wire-fed arc welding processes. The arc sensor module (ASM) makes use of simple voltage and current sensing to provide statistical information about the welding process. The ASM executes on a personal computer and has been incorporated into automated welding systems via various high-speed communications links. The ASM continues to evolve and has recently been adapted to constant-voltage and flux-cored wire processes. A measure of contact-tip wear has also been added. We have several CRADA partners presently working to incorporate the ASM into their welding production lines.

We have also investigated the use of arc-light emissions for process monitoring and control. Using a simple photodiode and broad-band optical interference filters, we developed sensors for measuring arc length and droplet frequency. A high-speed video system with laser backlighting provides reference measurements of arc length and droplet frequency and is used to calibrate each sensor. Recently, we began using a monochromator to allow higher-wavelength resolution measurements of arc-light emissions. With this device we intend to further exploit specific portions of the emission spectra for process monitoring and control.

We have also been investigating weld spatter. Using voltage, current and arc-light measurements in conjunction with the high-speed video system, we are studying spatter generation mechanisms. Two approaches to spatter reduction are being considered: (1) the graceful elimination of, and/or (2) the prevention of the short circuit events known to be a major contributor to generation of spatter.

Technical Objectives

- Develop a better understanding of the underlying physics governing arc-welding processes.

- Develop simple, nonintrusive, and robust sensors that provide meaningful information about the status of the welding process for real-time monitoring and control.

FY95 Accomplishments

To facilitate the technology transfer of the ASM, we have been working with four CRADA partners to incorporate our arc-sensing techniques for gas metal arc welding (GMAW) into their production lines. We have made several trips to their production facilities to assist in the ASM integration. One of the CRADA companies sent a engineer to our facility to learn for a 6-month period. We have established another CRADA with a company interested in applying the ASM techniques developed for GMAW to percussive arc welding.

We have demonstrated closed-loop control of both arc length and droplet frequency using arc-light emissions. With the assistance of a visiting professor, we have identified significant differences in the ionic composition of species within the arc between gas-tungsten and gas-metal arcs. This result may have an immediate impact on the assumptions commonly used in computational arc models.

Our work in spatter reduction identified the need for a standard, quantitative method to measure spatter. As a result, we have proposed and demonstrated several techniques of spatter measurement. We have shown that short-circuit events during welding produce large spatter. Analysis of the current, voltage and arc-light signals indicated that it is possible to predict the onset of an impending short circuit. Based on the analysis of these signals, we were able to demonstrate a simple real-time control to prevent short circuiting, and therefore to reduce the amount of weld spatter.

FY95 Outputs

Awarded U.S. Patent #5349156 for Control of GMAW Using Arc Light Sensing.

"Arc Length Control in GMAW," AWS Welding Technology in Manufacturing Conference, Orlando, FL, Feb, 1995.

" Droplet Frequency Sensing in GMAW," AWS National Conference, Cleveland, OH, April, 1995.

"Arc Sensor Module Demonstration," NIST Workshop on Welding Industry Needs, NIST, Gaithersburg, MD, August, 1995.

"Review of NIST Workshop on Welding Industry Needs," Ship Panel 7 Meeting, AWS Precision Joining Center, Golden, CO, August, 1995.

"Control of Gas-Metal-Arc Welding Using Arc-Light Sensing," NISTIR 5037.

"Ionic Compositions of Gas Tungsten and Gas Metal Arcs," abstract submittal, AWS National Conference 1996, jointly with visiting scholar, Prof. Dick Richardson of Ohio State University.

"A Standard Procedure for Spatter Measurement," AWS National Conference 1996.

Project: WELD PROCESS MODELING AND CONTROL

Project Leader: T.P. Quinn
T.A. Siewert, R.B. Madigan, B.J. Filla, C.N. McCowan

MSEL Program	*MRD Focus Technology*
Intelligent Processing of Materials	**IPM — Welding**
Strategic Thrust	*Character of Research*
Advanced Processes	**Automotive**

Technical Description

Physical and empirical models and control procedures for arc welding processes are being developed in order to increase the productivity and quality of welded parts. Increases in automation in the welding industry have until now focused on automating the motion of the welding torch (the human welder's arm motion), but there has been little success in automatically controlling the process itself. (The human welder adjusts the process on the fly using audio and visual feedback.) The models of the complex welding processes can lead to understanding by welding engineers, improved welding sensors, and better feedback control procedures.

The largest obstacle to arc-welding process control is the lack of effective feedback sensors. The arc-welding processes are difficult to sense because of the ultraviolet light, smoke, molten metal spatter, and electronic noise generated by the welding arc. To gain acceptance by manufacturers, sensors must be physically small if they are attached to the torch, so as not to affect the path of the robot. Therefore, we have chosen to develop sensors that either do not require devices to be attached to the torch, or are very small. Many of our sensing procedures rely on using model-based algorithms that use the inherent process variables, the current and the voltage; this is sometimes called "through the arc sensing." The principle advantage of this sensing scheme is that the external sensors can be placed at the power supply, well away from the robot and torch.

Technical Objectives

- Develop predictive models of the gas metal arc welding (GMAW) and shielded metal arc welding (SMAW) process.

- Develop control algorithms to automate the GMAW process and to improve final quality.

- Use empirical models generated from data taken under production-line conditions to develop sensing algorithms.

FY95 Accomplishments

The final analysis of a model for SMAW electrodes was completed. The model predicts the melting rate, temperature distribution, and reactions in the covering of the SMAW electrode. Experiments were conducted on E7018, 354 mm long, 3.2 mm diameter, commercial electrodes to determine melting rates and, using thermocouples, temperature profiles. The model was able to predict the consumed length within 2 mm for currents ranging from 117.5 to 160 A. At the recommended welding current of 130 A, the rms difference in the predicted temperature between the model and experiment was 26 °C over a range of 1500 °C. At the extremes of the range in current for practical welding, 110 and 150 A, the rms differences between the model and the experiments were 108 and 150 °C, respectively. The model predicted that at 130 A the $CaCO_3$ in the covering starts to be prematurely consumed when the arc reaches the position of about 170 mm from the holder; at 140 mm from the holder all of the $CaCO_3$ is predicted to be consumed prematurely. The model can be used to select weld parameters for a particular electrode to ensure that no premature reactions occur, or can be used by designers of electrode covering to properly select materials for the covering.

To develop real-time sensing and subsequent control of GMAW for high-volume manufacturing, current and voltage data were collected in an automotive part manufacturing plant under a Cooperative Research and Development Agreement (CRADA). Empirical models of the data were constructed and used to develop sensing algorithms. The algorithms measure the process for repeatability. Data for several acceptable welds are collected, processed by the algorithms, and reduced to a model weld; subsequent welds are then compared to this model and flagged if they are outside the calculated tolerance. In tests at the automotive-parts plant, the sensing system was able to detect loss of shielding gas, oil on the parts causing subsurface porosity, burn-through, and large joint gaps. Because of the geometry (thin sheet, lap joint), the sensor was not able to detect when the torch was not welding on the seam. Work is continuing with the CRADA partner, who is implementing the system in their assembly line.

To understand the relationships between the variables that are produced by the sensing algorithms, an orthogonal design of experiments was carried out in the laboratory using the same welding conditions found in the CRADA partner's plant. Tests were again conducted with oily parts, large gaps, burn-throughs, and off-seam welds. The resulting data from the sensing algorithms were statistically tested for variable independence. The analysis showed that the algorithm's outputs were significantly coupled and no single algorithm could predict which condition was present. For example, one of the algorithms calculates a running average of the current; a low average current could be caused by a burn-through or a large gap. Work is continuing to develop a method to give a probabilistic estimate of which of the test conditions is being encountered.

To clarify the direction of the NIST welding program, a workshop was held in conjunction with the Intelligent Systems Division of NIST in Gathersburg. Representatives from shipyards, automotive manufacturers, robot suppliers, heavy-equipment manufacturers and others attended the workshop. The attendees identified the top priorities NIST should have for the coming years. Two of the top priorities identified by the attendees were developing predictive process models and a welding knowledge base for empirical models.

FY95 Outputs

"A Melting Rate and Temperature Distribution Model for Shielded Metal Arc Welding Electrodes," T.P. Quinn, A.Q. Bracarense, and S. Liu, submitted to Welding Journal

"A Melting Rate and Temperature Distribution Model for Shielded Metal Arc Welding Electrodes," paper presented at the 1995 American Welding Society Convention in Cleveland, OH.

A 12 000 line sensing and modeling code was delivered to CRADA partners.

"Sensing of Constant Voltage Gas Metal Arc Welding Process Characteristics for Welding Process Control," patent application submitted to the U.S. Patent Office.

Project: MICROSTRUCTURE SENSING FOR PROCESS MONITORING

Project Leader: G.A. Alers
S.R. Schaps

MSEL Program	*MRD Focus Technology*
Intelligent Processing of Materials	**IPM — Thermomechanical Processing**
Strategic Thrust	*Character of Research*
Advanced Processes	**Metals Producers**

Technical Description

Most material processing for commercial applications is designed to develop specific microstructures that are known to give the final product its desired properties. Therefore, the development of process-control algorithms and nondestructive evaluation procedures should be focused on developing techniques and sensors that provide information on the microstructure instead of detecting localized inhomogenities that may or may not be flaws.

Technical Objectives

The objective of this program is to investigate measurement techniques that can be applied in a production environment and that give information on the microstructure of the material being produced. Thus, the program demands establishing quantitative relationships between measurable physical properties and the microstructure, as well as relating the microstructure to the commercially desirable properties that govern the production process. The relationships must be sufficiently well defined to allow the development of models that can predict the commercial properties from the physical property values. This is actually a classic case of inversion of an ill-posed problem. Our objective is to develop models that relate a desirable commercial property (such as hardness) to the microstructure as well as a model for relating a physical property (such as sound velocity) to the microstructure, and then to invert the relationships to yield a quantitative procedure for predicting the commercial property from a physical property measurement.

FY95 Accomplishments

During the past several years, NIST has established unique capabilities in noncontact eddy current and ultrasonic transducers that can operate in the hostile environments found in industrial metal-forming operations. In particular, the sensors can measure electrical resistivity, ultrasonic wave velocities and attenuations, and some magnetic properties of hot metal sheet as it moves through a rolling mill at high speed. Thus, the emphasis during FY95 was the development of techniques for: (1) making precision ultrasonic wave velocity measurements on thin polycrystalline sheet metal; (2) establishing relationships between the velocities and the microstructure of the individual grains; and (3) investigating correlations between ultrasonic properties and the mechanical strength properties (yield strength, hardness, grain size, etc.) desired by the consumers for the sheet product. In the case of sheet steel, magnetic property measurements can also yield information on the microstructure. Therefore, development of magnetic property measurements that are compatible with the ultrasonic techniques and with rolling mill operations are being undertaken.

Task I. Simulation of On-line Testing. In order to demonstrate that the ultrasonic, magnetic and electrical properties of thin sheet can be measured under rolling mill conditions, a simple machine to move sheet metal past an array of noncontact sensors was designed and assembled. This machine consists of two 25 cm diameter drums connected together by a continuous belt of the sheet metal to be investigated. A variable speed motor drives one drum to determine the speed of the metal belt while position-adjusting screws on the other drum establish the tension in the belt. An open area of about a square meter above and below the belt is available for mounting sensors that can interact with the sheet across air gaps of appropriate size. Initial tests of the machine will be conducted in early FY96.

Task II. Precision Ultrasonic Wave Velocity Measurements. The techniques available for making precise measurements of ultrasonic wave velocity usually require bulk samples that have flat, parallel surfaces machined onto them for determining transit time or have regular polyhedron shapes that can vibrate at well defined resonant frequencies. For thin sheet metal, through-thickness-resonance methods can be used to accurately determine the ultrasonic wave velocities in the thickness dimension. Unfortunately, the desired commercial properties of sheet metal are usually attained by developing a preferred orientation in the grains that make up the microstructure. This texture makes it necessary to measure the ultrasonic wave velocities in directions other than the thickness dimension. Sound waves that propagate in the plane of the sheet are Lamb waves, and their velocities not only depend on the frequency being used but they have complicated, nonlinear relationships with the elastic constants of the constituent grains. Fortunately, the electromagnetic acoustic transducer (EMAT), whose noncontact characteristic makes it the transducer of choice in a manufacturing facility, can easily be designed to excite and detect only one Lamb wave mode at a well defined frequency. This simplifies the experimental procedures and the mathematical analysis so that phase velocities can be determined very accurately and the mathematical relationships with the elastic properties of the sheet can be written down in closed form and accurately solved by a computer.

During FY95, several types of EMATs were designed, built and tested in fixtures that allowed the phase velocities of various Lamb-wave modes to be measured to precisions approaching ±0.1%. Both shear and extensional types of waves were measured as a function of angle relative to the rolling direction in the plane of the sheet. This, in principle, provides many independent measurements that will be used to unravel the complicated expressions relating sound velocities and elastic constants of crystallite.

Task III. Correction for Texture Effects. The symmetry of the rolling process forces an orthorhombic symmetry onto the physical properties of the sheet. Thus, a complete description of the elastic or ultrasonic properties of the sheet requires specification of nine elastic modulus tensor elements. Fortunately, theoretical analysis of the properties of polycrystalline aggregates has been developed to a high level during the past several years. Now it is possible to find mathematical expressions in the literature that relate the nine elastic moduli of a textured sheet to three orientation distribution coefficients (ODCs) that describe the texture and the three single-crystal elastic constants. These describe the elastic response of an individual grain if that grain is a crystal with cubic symmetry. The more recent literature also contains mathematical expressions for the relationships between certain Lamb-wave velocities and the nine tensor elements of the elastic modulus. Some of these are the result of series expansions in small quantities, so care must be exercised in applying them to specific cases. During FY95, the expressions given in the literature were used to develop a mathematical inversion procedure for deducing the three ODCs and the three effective single-crystal elastic constants for a grain from measurements of Lamb-wave phase velocity in the plane of the sheet and of bulk wave velocities in the thickness dimension. All of the velocities can be measured by using EMATs that operate across an air gap over moving sheet metal.

Task IV. Applications. The most simple application of the experimental and theoretical techniques developed to date is to rolled brass and copper, where the texture effects are large but the grains are single-phase cubic crystals. As part of a CRADA with Olin Corporation, EMAT techniques are being developed to monitor recrystallization and grain growth during the rolling and annealing processes in an operating mill. Since Olin has supplied sheet samples of copper, bronze, and brass with various thicknesses, degrees of rolling reduction and annealing histories, the procedures to be used for following the development of texture during recovery, recrystallization, and grain growth in the rolling mill are becoming established.

Another simple case for on-line monitoring of sheet-metal properties is being investigated as part of an ATP project with Allied Signal. Here, the analysis is simplified because the material is METGLAS, which is an elastically isotropic metallic glass. Thus, there is no texture, and only two elastic constants are sufficient to describe the elastic response of the material. Measurements of the phase velocities of the So and SH Lamb wave modes were sufficient to define these two elastic constants to an accuracy of ±0.1%. Since Allied Signal supplied samples with different processing histories and different ductility properties, it was possible to seek correlations between the measured wave velocities and the mechanical strength property of ductility. A linear correlation between the ductile-to-brittle transition temperature (DBTT) and the product of the SO and SH wave velocities was observed; a model to explain this is being developed.

A third study addresses the application of Lamb-wave velocity measurements to steels. Here, we are taking advantage of an AISI/DOE program at NIST to develop constitutive relations that describe the process of hot rolling of steel in the austenitic phase. This program also seeks to develop quantitative relationships between the mechanical strength properties of the final product and the microstructures developed after the cooling of the steel through the γ-to-α phase transition and the formation of martensite. EMATs that use magnetostriction as the coupling mechanism are being developed to measure both the Lamb-wave velocities and the magnetic properties of these steels at the same time. Ultimately, this will provide both magnetic and acoustic measurements of physical properties to compare with the mechanical properties and microstructure information being developed for the steels of interest to the AISI and DOE.

FY95 Outputs

1. Proprietary letter reports to Olin Corp. and Allied Signal.

Project: THERMOMECHANICAL PROCESSING

Project Leader: Y-W. Cheng
P.T. Purtscher, B.J. Filla

MSEL Program Intelligent Processing of Materials	*MRD Focus Technology* IPM — Thermomechanical Processing
Strategic Thrust Advanced Processes	*Character of Research* Metals Producers

Technical Description

This research centers on development of quantitative relationships between the mechanical properties of steel and both its chemistry and its thermomechanical processing history. Emphasis is placed on characterization of the kinetics of microstructural evolution that occurs during the processing steps of rolling and forging. The microstructural changes included in the study are: precipitation of carbides, nitrides, and carbonitrides; dynamic and static recrystallization and grain growth; and austenite decomposition. Recent studies are also aimed at development of structure-property relationships that describe the constitutive behavior, grain refinement through recrystallization, precipitation during hot deformation of austenite, and the influence of deformation on continuous-cooling transformation characteristics of plain-carbon and microalloyed steels.

Technical Objectives

The objectives of this program are to develop, improve and validate process models that are needed to describe the thermomechanical processing of steels. If these process models are applied, steels of higher quality can be produced with less cost because of shorter design cycles and more exact process control.

FY95 Accomplishments

In 1995, we continued a major research project for the American Iron and Steel Institute (AISI) and the Department of Energy (DOE) on microstructural engineering in hot-strip mills. The objective of this particular project is to develop a predictive tool that will quantitatively link the properties of hot-rolled steel products to the process parameters of a hot-strip mill. The project is a concerted effort of four groups: NIST, the University of British Columbia (UBC-Canada), U.S. Steel, and Northwest Mettech Corporation (Canada). Major portions of the research are being done at NIST and UBC. UBC conducts research on heat transfer, quantitative characterization of the kinetics of microstructure evolution, and model verification with plant and pilot-plant trials. NIST's efforts focus on the studies of constitutive behavior under hot-rolling conditions and the development of structure-composition-property relations at the ambient temperature.

One of the goals of studying constitutive behavior of steels is to predict the flow behavior of a given steel under various processing conditions. The flow behavior is required for calculation of power requirements and roll-separating force during rolling, which in turn, is important for gage control. Furthermore, a complete and accurate description of the flow behavior in analytical form is essential to fully exploit the potential of numerical techniques, such as finite-element method, for analyzing and simulating hot rolling or forging.

During FY95, we developed models for predicting the flow of two plain carbon steels: A36 and DQSK grades. The models calculate a full stress-strain curve with input of a given combination of temperature, strain rate, and austenite grain size. Also calculated are important properties along the stress-strain curve, including the 0.2% offset yield stress, the steady-state stress prior to dynamic recrystallization, the strain at which dynamic recrystallization occurs, and the steady-state stress after dynamic recrystallization has taken place.

In addition, we upgraded the NIST mechanical testing facilities by installing a servo-hydraulic machine capable of deforming specimens at a strain rate of 70 s^{-1}, which is about three times faster than the existing facilities. The new machine is controlled by a computer and is equipped with a furnace for temperature control. The high-strain-rate, high-temperature machine is required for simulation of rolling conditions in the last few stands of a finish mill. With this new machine, we can generate experimental data for validating and improving the current models and for developing new models.

A second part of the AISI-sponsored effort is the development of quantitative relationships between the mechanical properties of steel at ambient temperature and the composition and microstructure of the steel. That is, we seek to derive relationships that describe such properties as strength, ductility, and strain-hardening characteristics in terms of microstructural and compositional parameters. The relationships can be used not only to predict the mechanical properties but also to optimize the alloy design. During FY95, we validated models that describe the yield strength and the ultimate tensile strength as a function of chemical composition and microstructural features that include ferrite grain size and pearlite volume fraction for plain, low-carbon steels. We have also developed strength models for microalloyed steels. In these cases, composition and conventional microstructural features must be augmented with processing parameters in order to describe the strength of a given steel. The important processing parameters that influence the strength of microalloyed steels include the reheat temperature, the finish-rolling temperature, the cooling rate on the runout table, and the coiling temperature. These parameters influence the ambient temperature mechanical properties because they affect the dissolution and precipitation of microalloying compounds, such as carbides, nitrides, and carbonitrides.

FY95 Outputs

1. P.T. Purtscher and Y.W. Cheng, "Prediction of the Strength Properties for Plain-Carbon and Vanadium Microalloyed Ferrite-Pearlite Steel," Proceedings of the 36th Mechanical Working and Steel Processing Conference, Oct. 16-19, 1994, Baltimore, MD.

2. N.E. Aloi, Jr., G. Krauss, D.K. Matlock, C.J. Van Tyne, and Y.W. Cheng, "Hot Deformation, Microstructure, and Properties of Medium Carbon Microalloyed Forging Steels," Proceedings of the 36th Mechanical Working and Steel Processing Conference, Oct. 16-19, 1994, Baltimore, MD.

3. R.M. Kuziak, T. Bold, and Y.W. Cheng, "Microstructure Control of Ferrite-Pearlite High Strength Low Alloy Steels Utilizing Microalloying Additions," J. Materials Processing Technology, Vol. 53, (August 1995), pp. 255-262.

4. Y.W. Cheng, "Thermomechanical Processing of Steels," presented at the Academy of Mining and Metallurgy, Krakow, Poland, May 15, 1995.

5. Y.W. Cheng, "Microstructural Engineering in Hot-Strip Mills," presented at the AISI project review meeting, May 23-24, 1995.

6. Y.W. Cheng and P.T. Purtscher, "Microstructural Engineering in Hot-Strip Mills," an annual report to AISI, May, 1995.

Project: MICROSTRUCTURAL EVOLUTION

Project Leader: W. Johnson

MSEL Program	*MRD Focus Technology*
Intelligent Processing of Materials	**IPM — Thermomechanical Processing**
Strategic Thrust	*Character of Research*
Advanced Processes	**Metals Producers**

Technical Description

This project seeks to establish a materials-science base for a proposed real-time ultrasonic sensor that will use ultrasonic attenuation to monitor the microstructural evolution of cold-worked metallic alloys at elevated temperatures. In all materials at sufficiently elevated temperatures, mechanical vibrations are highly damped as a result of the nonelastic response of dislocations to vibrational stress. Most of what is known about this phenomenon was discovered in the period from 1955 to 1975 when dislocation vibrational damping was a subject of considerable interest in basic research. The current project revisits this subject with a new perspective of applying the great sensitivity of damping measurements to the changes in dislocation density that are expected to take place during the recovery and recrystallization of cold-worked commercial materials.

Technical Objectives

The objective of this program is to investigate the possibility of using measurements of high-temperature damping as a tool in the intelligent processing of materials. Recent developments at NIST of transducers capable of performing noncontacting, real-time ultrasonic measurements at elevated temperatures indicate that such a tool would be practical at this time. It would monitor those microstructural changes that involve annihilation of dislocations such as recovery and recrystallization. The industrial importance of monitoring these microstructural changes is particularly great for annealing processes that must carefully balance increases in ductility with losses in strength, as, for example, in the production of sheet metal used in deep drawing applications. The initial objective of the project is to establish a detailed model for the high-temperature damping in cold-worked alloys.

FY95 Accomplishments

Cold-worked, polycrystalline, pure and alloyed aluminum has been the subject of the research in FY95. Aluminum and its alloys are particularly appropriate for study, because of their technological importance, the relatively large amount of relevant published data, and the readily accessible annealing temperatures. Resonance ultrasonic measurements were performed on 6.4 mm-diameter spherical samples in a low-pressure inert-gas atmosphere using noncontacting electromagnetic-acoustic transduction (EMAT) techniques. The frequencies and damping of torsional-mode resonances in the sphere were continuously recorded during heating and cooling.

Initial exploratory measurements were performed on a commercially produced 2024 aluminum sphere with an unknown state of deformation. As the sample was heated from room temperature to 330°C, the damping increased dramatically as expected for the contribution from dislocations. However, this increase was not monotonic. There was a large irreversible decrease between 240°C and 270°C that was suspected to be a result of microstuctural recovery. Corresponding to this decrease in damping, the resonant frequency reflected a relative increase in the shear constant, consistent with a reduction in dislocation density from recovery. The identification of the microstructural process is, however, complicated by the presence of alloying elements that change their degree of precipitation on heating. Dislocation damping can be significantly dependent on the presence of impurity atoms (substitutional or interstitial) that "pin" movement of dislocations.

The question of whether precipitation might be responsible for the irreversible drop was investigated in material with no precipitating elements, that is, 99.999% pure aluminum. A fully annealed ingot was deformed 10.5% and machined into a sphere. During the first annealing cycle, the damping behaved in a manner similar to that of the 2024 alloy, dropping irreversibly on heating into the temperature range from 210°C to 260°C. This demonstrated that the effect is not related to changes in precipitation. After cooling to room temperature, a second anneal was performed during heating to 425°C. During this heating cycle, the damping underwent a second irreversible drop in the temperature range from 320°C to 380°C. A subsequent thermal cycle showed no additional irreversible changes. That is, the heating and cooling data were the same with the damping increasing monotonically with temperature. The temperature range of the first drop (210°C to 260°C) is consistent with published results on the temperature dependence of recovery in unalloyed aluminum. The range of the second drop is consistent with published results on the temperature dependence of recrystallization.

These measurements indicate that vibrational damping is highly sensitive to microstructural recovery and recrystallization in cold-worked aluminum. The ability to sense recovery nondestructively in real time is particularly noteworthy, because recovery cannot be detected with optical or scanning-electron microscopy. The very high measured damping above a couple of hundred degrees Celsius indicates that dislocations are the overwhelmingly dominant source of damping in aluminum at these temperatures. This is encouraging with regard to the prospects of developing a practical sensor for monitoring changes in concentration of dislocations.

Since damping generally has a very complicated dependence on many material parameters, a process sensor will yield useful information only if a simplified model can be developed that includes a minimum number of variables found to be relevant to the application.

FY95 Outputs

The Microstructural Evolution Project was initiated in FY95. There are no publications yet.

Project: **XRD SENSING OF LIQUID-SOLID INTERFACE**

Project Leader: D.W. Fitting
T.A. Siewert, W.P. Dubè

MSEL Program Intelligent Processing of Material	*MRD Focus Technology* IPM — Casting
Strategic Thrust Advanced Processes	*Character of Research* Aeronautics

Technical Description

This project is a sensor-development activity within the NIST Consortium on Casting of Aerospace Alloys, an industry/government/university team devoted to improving quality and reducing cost through advances in materials science. The Consortium is near the middle of its projected four-year life, so our project focus is shifting from sensor development and toward practical applications of these developments (such as implementing our developments in production equipment). Other members of the team have projects in modeling, thermophysical property data collection, and validation. We are coordinating our efforts to complement those of the other members.

The consortium has identified several areas where developments in technology would substantially improve the processing procedures. In particular, no sensor to date has been capable of detecting the location of the liquid/solid boundary in the harsh environment of a turbine-blade casting furnace (high vacuum, high temperatures, and strong rf fields). Yet, knowledge of the solidification front location and shape would allow the solidification process to be optimized (through confirmation of the models), reducing scrap and increasing the production rate. Therefore, we are developing a noninvasive x-ray diffraction (XRD) technique to monitor the position of the liquid/solid boundary in single-crystal and directionally solidified jet-engine turbine-blade castings. The sensor is capable of locating the boundary even though the casting is enclosed in a thick ceramic mold and contained within a vacuum furnace. X-rays of higher energies are capable of penetrating through the furnace, mold walls, and sample, while those of lower energies yield a higher number of diffracted x-rays. An optimal x-ray energy (often between 100 and 320 keV) is chosen by using an analytical model for the XRD process.

The high contrast (many times greater than that for an x-ray imaging technique) of the XRD sensor is achieved because of the strikingly different x-ray diffraction patterns from a solid (with a high degree of structural order) and a liquid. The diffraction pattern from a solid is characterized by a spatial distribution of high-intensity spots, while that from a liquid is a diffuse ring. In addition to sensing the position of the solidification front, the XRD sensor will be used to determine the extent and shape of the mushy zone in a nickel superalloy casting, so that solidification models of the casting process may be verified.

Technical Objectives

- Develop transmission x-ray diffraction as a nondestructive means for locating, characterizing, and following the liquid/solid boundary in the casting during the directional solidification of superalloys (withdrawal through a gradient furnace)

- Demonstrate the XRD sensing technology in a turbine-blade casting facility and characterize the performance of the sensor

- Verify the models of turbine blade solidification

FY95 Accomplishments

This year, we have demonstrated the ability of the XRD technology to sense melting and solidification of metal through the walls of furnace and mold and are modifying an industrial furnace for trials on nickel superalloys. Earlier this year, we demonstrated the capability of the technology to sense the liquid/solid interface in melting experiments with copper and aluminum by penetrating the walls of our research furnace. This summer, we acquired an industrial directional solidification furnace and showed that we could produce single-crystal castings of a nickel superalloy (N5). Now, this furnace is being modified to accept the XRD solidification sensor system.

In addition, we have developed an analytical model for the transmission XRD process, which indicates an optimal range of x-ray energies for particular casting conditions. This model allows us to estimate the energy and beam intensity necessary to penetrate given thicknesses of casting, mold, and furnace walls.

FY95 Outputs

D.W. Fitting, W.P. Dubè, T.A. Siewert, and J. Paran, "A process sensor for locating the liquid solid boundary through the mold of a casting," Review of Quantitative Nondestructive Evaluation, Seattle, WA, August 1995 and Proceedings of the Review of Quantitative Nondestructive Evaluation, Plenum Press (New York), in press

T.A. Siewert, W.P. Dubè, and D.W. Fitting, US Patent Application filed on 24 March 1995, "Apparatus and Method For Monitoring Casting Process"

D.W. Fitting, W.P. Dubè, and T.A. Siewert, "Real-time monitoring of turbine blade solidification using x-ray diffraction techniques," Semi-Annual Report of the NIST Consortium on Casting of Aerospace Alloys, pp. 39-56, 7 October 1994, and revised for the Annual Report of the NIST Consortium on Casting of Aerospace Alloys, pp. 75-85, 20 April 1995.

ULTRASONIC CHARACTERIZATION OF MATERIALS

The projects on ultrasonic characterization are directed to the development of model-based methods of physical measurement which characterize the internal geometries of materials, such as defects, microstructures, and lattice distortions. Our goal is to convert these measurement methods into sensors suited for production-line and in-service measurements of materials quality and serviceability. We are working with industry to commercialize advances in noncontact ultrasonics, waveform-based acoustic emission, composites, NDE, and nonlinear ultrasonics. The Division has ultrasonic characterization projects in the areas of elastic properties, composite materials, and nondestructive evaluation.

Project: ELASTIC CONSTANTS AND RELATED PHYSICAL PROPERTIES

Project Leader: H. Ledbetter
S.A. Kim, D.R. Smith, D. Balzar

MSEL Program	*MRD Focus Technology*
Nondestructive Evaluation	**Ultrasonic Characterization**
Strategic Thrust	*Character of Research*
Advanced Materials	**Fundamental Research**

Technical Description

Our research emphasizes measurements and modeling of elastic constants and related physical properties of metals, alloys, composites, ceramics, and the new high-T_c oxide superconductors. For many studies, the temperatures range between 295 and 4 K. The elastic constants, which relate deformation to stress, sustain our interest because they relate to fundamental solid-state phenomena: interatomic potentials, equations of state, and phonon spectra. Furthermore, thermodynamics links elastic constants with specific heat, thermal expansivity, atomic volume, and the Debye temperature.

Technical Objectives

Understand, through measurements and modeling, the elastic properties of solids that possess high scientific or high technological interest. As required, develop new methods of measurement and of modeling.

FY95 Accomplishments

Invited lecture at Science University of Tokyo.

One month in Japan hosted by Japan Science and Technology Agency.

For short-fiber-reinforced composites, developed method to determine orientation-distribution function (ODF) from measured elastic constants C_{ij}. Similarly, from ODF and monocrystal C_{ij}, we can calculate textured polycrystal C_{ij} or other physical-mechanical properties such as thermal expansivity and stress-strain (plastic-deformation) behavior.

Measured successfully by ARS method, the complete elastic constants C_{ij} of a trigonal-symmetry piezoelectric monocrystal: $LiNbO_3$. Modeling of C_{ij} successful also. Modeling requires introducing dielectric tensor K_{ij} and piezoelectric tensor d_{ijk}.

Used ARS method to measure complete C_{ij} of a boron-fiber aluminum-matrix composites. Interesting, useful intercomparisons arose when we compared ARS results with those from rod-resonance, from pulse-echo, and from calculations arising from a scattered-plane-wave ensemble-average model.

FY95 Outputs

Archival publications

1. D. Balzar and H. Ledbetter, Accurate modeling of size and strain broadening in the Rietveld refinement: The double-Voigt approach, in Advances in X-Ray Analysis, Volume 38 (Plenum, New York, 1995), 397-404.

2. M. Dunn and H. Ledbetter, Poisson's ratio of porous and microcracked solids: Application to oxide superconductors, J. Mater. Res. 10 (1995) 2715-2722.

3. C. Fortunko, H. Ledbetter, and P. Heyliger, Ultrasonic-resonance spectroscopy of bulk and layered solids, in Proceedings, International Symposium on Surface Waves in Solid and Layered Structures and National Conference on Acoustoelectronics (St. Petersburg State Academy of Aerospace Instrumentation, St. Petersburg, 1995), 430-435.

4. S. Kim, Off-diagonal orthorhombic-symmetry elastic constants, Appl. Phys. Lett. 65 (1994).

5. H. Ledbetter, Elastic properties of metal-matrix composites: Measurements and modeling, Proceedings, Fourth Japan International SAMPE Symposium (SAMPE, Yokohama, 1995), 476-481.

6. H. Ledbetter, S. Datta, and M. Dunn, Elastic properties of particle-occlusion composites: Measurements and modeling, J. Eng. Mater. Technol. 117 (October 1995) 402-407.

7. H. Ledbetter, M. Dunn, and M. Couper, Calculated elastic constants of alumina-mullite ceramic particles, J. Mater. Sci. 30 (1995) 639-642.

8. H. Ledbetter, M. Dunn, S. Kim, and R. Fields, Void shape in sintered titanium in Review of Progress in Quantitative Nondestructive Evaluation, Volume 14 (Plenum, New York, 1995), 1633-1639.

9. H. Ledbetter, C. Fortunko, and P. Heyliger, Elastic constants and internal friction in polycrystalline copper, J. Mater. Res. 10 (1995) 1352-1353.

10. H. Ledbetter, C. Fortunko, and P. Heyliger, Orthohombic elastic constants of a boron-aluminum fiber-reinforced composite: An ultrasonic-resonance-spectroscopy study, J. Appl. Phys. 78 (1995) 1542-1546.

11. H. Ledbetter, C. Fortunko, and P. Heyliger, Off-diagonal elastic constants in fiber-reinforced composites, in Proceedings, Tenth International Conference on Composite Materials, Volume IV: Characterization and Ceramic-Matrix Composites (Woodhead, Cambridge, England, 1995), 19-24.

12. H. Ledbetter, P. Heyliger, K.-C. Pei, S. Kim, and C. Fortunko, Artificial crack in steel: An ultrasonic-resonance-spectroscopy and modeling study, in Review of Progress in Quantitative Nondestructive Evaluation, Volume 14 (Plenum, New York, 1995), 2019-2025.

13. Y. Shindo, H. Ledbetter, and H. Nozaki, Elastic constants and microcracks, in $YBa_2Cu_3O_7$, J. Mater. Res. 10 (1995) 7-10.

14. M. Weller and H. Ledbetter, Temperature-dependent Young's modulus of an SiC_w/Al_2O_3 composite, J. Mater. Sci. 30 (1995) 834-835.

Manuscripts submitted

1. D. Balzar and H. Ledbetter, Software for comparative analysis of diffraction-line broadening, in Advances in X-Ray Analysis Plenum, New York, 1996, forthcoming.

2. M. Dunn and H. Ledbetter, Elastic moduli of composites reinforced by multiphase particles, J. Appl. Mech, forthcoming.

3. M. Dunn and H. Ledbetter, Estimation of the orientation distribution of short-fiber composites using ultrasonic velocities, J. Acoust. Soc. Amer., forthcoming.

4. M. Dunn and H. Ledbetter, Nondestructive ultrasonic characterization of the orientation distribution of short-fiber composites, in Review of Progress in Quantitative Nondestructive Evaluation, Volume 15 (Plenum, New York, 1996), forthcoming.

5. M. Dunn and H. Ledbetter, Thermal expansion of textured polycrystalling aggregates, J. Appl. Phys., forthcoming.

6. M. Dunn and H. Ledbetter, Ultrasonic measurement of the orientation distribution of short-fiber composites, in Proceedings, MRS/SAMPE Symposium on Smart Processing of Materials (Albuquerque, October 1995), forthcoming.

7. M. Dunn, H. Ledbetter, and P. Heyliger, Free vibration of piezoelectric solids: Application to the determination of elastic and piezoelectric constants, in Proceedings, ASCE-ASME-SES Joint Mechanics Meeting (Boulder, May 1995), forthcoming.

8. M. Dunn, H. Ledbetter, P. Heyliger, and C. Choi, Elastic constants of textured short-fiber composites, J. Mech. Phys. Solids, forthcoming.

9. S. Kim and H. Ledbetter, Metal-oxide Debye temperatures and elastic constants: Estimation from interionic spacing, J. Phys. Chem. Solids, forthcoming.

10. H. Ledbetter, Acoustoelastic residual-stress measurements: Role of anisotropic dislocation arrays, J. Appl. Phys., submitted.

11. H. Ledbetter, Elastic constants and instability in face-centered-cubic metals, Phase Transitions, forthcoming.

12. H. Ledbetter, Intrinsic physical properties of $YBa_2Cu_3O_7$, Phys. Rev., submitted.

13. H. Ledbetter, Low-temperature monocrystal elastic constants of Fe-19Cr-10Ni, J. Phys. Chem. Solids, submitted.

14. H. Ledbetter, Low-temperature variability of 304-stainless-steel elastic constants, J. Appl. Phys., submitted.

15. H. Ledbetter, Sound velocity as a texture probe: Calculations for 316-stainless-steel welds, J. Appl. Phys., submitted.

16. H. Ledbetter, S. Datta, and M. Lei, Elastic constants of porous ceramics, J. Amer. Ceram. Soc., submitted.

17. H. Ledbetter, M. Lei, and Y.-Y. Li, Nickel effect on elastic constants of Fe-Cr-Ni alloys, Mater. Sci. Eng., forthcoming.

18. H. Ledbetter and S. Kim, Anisotropic ultrasonic attenuation in monocrystal copper, Mater. Sci. Eng., forthcoming.

19. H. Ledbetter and S. Kim, Bulk-moduli systematics in oxides, including superconductors, Physica C, submitted.

20. H. Ledbetter and S. Kim, Cubic-metal-oxide elastic constants: A new systematic, Physica B, submitted.

21. H. Ledbetter, S. Kim, and L. Boatner, Monocrystal elastic constants of 70Fe-15Ni-15Cr, Metall. Mater. Trans. A. forthcoming.

22. H. Ledbetter, S. Kim, D. Balzar, S. Krudele, and W. Kriven, Elastic properties of mullite, J. Amer. Ceram. Soc., forthcoming.

23. H. Ledbetter, S. Kim, C. Fortunko, and P. Heyliger, Compressibility of polycrystal and monocrystal copper: Acoustic-resonance spectroscopy, Int. J. Thermophys. 17 (1996) 263-269, forthcoming.

Talks at technical meetings

1. Nondestructive ultrasonic characterization of the orientation distribution of short-fiber composites. Twenty-first Annual Review of Progress in Quantitative Nondestructive Evaluation. Seattle (July-August).

2. Off-diagonal elastic constants of fiber-reinforced composites. Tenth International Conference on Composite Materials. Whistler, British Columbia (August).

3. Recent acoustic-resonance-spectroscopy studies at NIST-Boulder. Second Workshop on Resonant Ultrasonic Spectroscopy. Santa Fe (August).

4. Elastic properties of metal-matrix composites: measurements and modeling. Fourth Japan International SAMPE Symposium. Invited. Tokyo (September).

5. Microstructure and elastic-constant measurements of two-phase materials. Invited, 1995 IEEE International Ultrasonics Symposium. Seattle (November).

Project: **ELASTIC PROPERTIES OF SUPERCONDUCTORS**

Project Leader: H. Ledbetter
S.A. Kim

MSEL Program **Superconductivity**	*MRD Focus Technology* **Ultrasonic Characterization**
Strategic Thrust **Advanced Materials**	*Character of Research* **Fundamental Research**

Technical Description

Elastic constants represent long-wavelength phonons, which enter explicitly into the BCS relationship for the superconducting transition temperature: $T_c \sim \Theta \exp(-a/\lambda)$. The elastic constants determine the Debye characteristic temperature Θ and enter McMillan's expression for the electron-phonon parameter λ. Clearly, phonons also play key roles in the new high-T_c oxide superconductors. Furthermore, elastic constants provide a valuable probe of the second-order normal-superconducting (n-s) phase transition. And they relate closely to other important physical properties such as specific heat. The imaginary component of the elastic constants, the ultrasonic attenuation, has played a key role in studying superconductors.

Technical Objectives

We seek to understand the elastic constants, how they change with composition, crystal structure, and temperature, and how they relate to the basic n-s mechanism.

FY95 Accomplishments

Our studies of cracks in oxide superconductors show their dramatic effect on apparent macroscopic elastic constants, both dilatational-mode and shear-mode.

Our in-progress review of the Bi-O elastic constants shows that the low, even negative, values reported for the Poisson ratio are probably correct. This means that the character of interatomic bonding in the bismuth cuprates differs from bonding in all the other cuprate groups, indicating a soft compressional mode compared with bending modes.

FY95 Outputs

Archival publications

1. M. Dunn and H. Ledbetter, Poisson's ratio of porous and microcracked solids: Application to oxide superconductors, J. Mater. Res. 10 (1995) 2715-2722.

2. Y. Shindo, H. Ledbetter, and H. Nozaki, Elastic constants and microcracks in $YBa_2Cu_3O_7$, J. Mater. Res. 10 (1995) 7-10.

Manuscripts submitted

1. H. Ledbetter, Intrinsic physical properties of $YBa_2Cu_3O_7$, Phys. Rev., submitted.

2. S. Kim and H. Ledbetter, Metal-oxide Debye temperatures and elastic constants: Estimation from interionic spacing, J. Phys. Chem. Solids, submitted.

3. H. Ledbetter and S. Kim, Bulk-moduli systematics in oxides, including superconductors, Physica C, submitted.

4. H. Ledbetter and S. Kim, Cubic-metal-oxide elastic constants: A new systematic, Physica B, submitted.

In-progress manuscripts for completed studies

1. H. Ledbetter, Failure of McMillan's relationship for high-T_c superconductors.

2. H. Ledbetter, Elastic constants of Bi-O superconductors, a review.

3. H. Ledbetter and S. Kim, Dependence of T_c on Debye characteristic temperature.

4. H. Ledbetter, S. Kim, and K. Kinoshita, Low-temperature elastic constants of $La_2CaCu_2O_6$.

5. H. Ledbeter, S. Kim, and S.P. Matusuda, Low-temperature elastic constants of (Tl-Pb) $(Sr\text{-}Ba)_2$ Cu_3O_x.

6. H. Ledbetter, S. Kim, and A. Roshko, Low-temperature shear moduli of $(La\text{-}M)_2$ CuO_4 superconductors, M=Ca, Sr, Ba.

7. H. Ledbetter, S. Kim, H. Uwe, and A. Iyo, Low-temperature elastic constants of $Ba_{0.7}K_{0.3}BiO_3$.

8. H. Ledbetter, X. Xie, and M. Lei, Elastic-stiffness-compliance interconversion: Application to Nb_3Sn superconductor.

Project: **ULTRASONIC CHARACTERIZATION OF COMPOSITES**

Project Leader: C.M. Fortunko
H. Ledbetter, S.A. Kim, D.W. Fitting, N. Sizova

MSEL Program **Polymer-Matrix Composites**	*MRD Focus Technology* **Ultrasonic Characterization**
Strategic Thrust **Advanced Materials**	*Character of Research* **Automotive**

Technical Description

We need to know accurately a material's design properties not only for optimum engineering applications, but also to improve material properties. Because we can not possibly measure all properties of all materials, we need reliable quantitative models to predict and to relate properties. To define the bounds of these models, we need accurate, thorough measurements of mechanical and physical properties for well-characterized materials.

Technical Objectives

Choose a set of important properties: elastic constants, thermal expansivity, anelasticity (creep-recovery), stress-strain (plastic deformation). Use various methods to measure these properties; focus especially on uncertainties. Relate measured values to models. As required, develop new measurement and modeling methods.

FY95 Accomplishments

With M. Dunn (U. Colorado), we wrote a review of existing methods for calculating elastic constants of short-fiber composites.

With M. Dunn (U. Colorado), we developed a method for determining a short-fiber-reinforced-composite's orientation-distribution function (ODF).

We developed methods for using the ODF to calculate a composite's properties, for example, elastic constants, thermal expansivity, stress-strain curve.

Using various methods, we measured the elastic constants and internal frictions of numerous polymers and a few glass-reinforced polymers.

Using the anelasticity (creep-recovery) apparatus built with H. Kobayashi (Tsukuba), we measured numerous polymers and a few glass-reinforced polymers. To calibrate the apparatus, we measured several "soft" metals such as indium and lead.

Working with a group in Switzerland (the only group known to us now active in the method), we used Laplace-transform methods to obtain from the strain-time profiles the relaxation-time distribution function $g(\tau)$. The results agree surprisingly well with our double four-element Burgers-model calculations.

FY95 Outputs

With M. Dunn (U. Colorado), a report "Modeling the effective elastic moduli of discontinuous-fiber-reinforced composite materials."

H. Ledbetter, H. Kobayashi, N. Sizova, S. Kim, and M. Dunn, "Low-stress viscoelasticity in polyethylene, to be submitted.

H. Ledbetter, N. Sizova, S. Kim, H. Kobayashi, S. Sgobba, and L. Parrini, "Retardation-relaxation times in anelastic creep-recovery in indium", abstract submitted to ICIFUAS II, Poitiers, France, 7-11 July 1996.

Project: COMPOSITES NDE

Project Leader: C.M. Fortunko
M.A. Hamstad, E.S. Boltz, J.D. McColskey, M.J. Anderson, V.K. Tewary

MSEL Program	*MRD Focus Technology*
Polymer Matrix Composites	**Ultrasonic Characterization**
Strategic Thrust	*Character of Research*
Advanced Materials	**Automotive**

Technical Description

Experimental techniques are under development for characterizing the microstructures of advanced composite materials. Microstructure includes orientation, shape, distribution, and concentration of reinforcement phase in a matrix material. Wideband ultrasonics and acoustic emission methods are under development for both long-fiber and short-fiber composites, which are extensively used by the automotive industry. The acoustic emission work is principally aimed at studies of uniformity or lack or uniformity of microdamage during stressing. The wideband ultrasonic studies are aimed at development of quantitative methods for determining the elastic properties and relating them to the microstructure.

Technical Objectives

1. Evaluate the practicality of using noncontact, air-coupled ultrasonics for stiffness determination in composite plate geometries used in aerospace and automotive and other applications.

2. Develop wideband acoustic emission techniques to identify, locate, and characterize microdamage in short-fiber polymer composites used in automotive applications. Demonstrate that such sensors will be useful to control quality in automotive and other industrial applications.

3. Develop laboratory and in-line ultrasonic measurement methods and instrumentation for determining the stiffness moduli and fiber-orientation distributions in short-fiber reinforced composite materials. Validate the methods using metallographic and micro-mechanical modeling techniques.

FY95 Accomplishments

Completed evaluation of air-coupled ultrasonic systems for determination of the stiffness moduli of composite plates. Transferred the key elements of the technology to a CRADA partner (XXsys/Composite Retrofit Corporation).

Completed and evaluated an infrared Michelson interferometer for wideband ultrasonic measurements in composite materials. The interferometer was needed to obtain high-fidelity ultrasonic waveforms in composite materials. The interferometer is also used to characterize conventional piezoelectric contact transducers, which are used in nondestructive evaluation of composites.

Used the infrared interferometer to optimize the performance of wideband acoustic emission transducers specifically intended for studies of microdamage in composites and validate the main features of Green's theory of elastic-wave propagation for plate geometries.

Developed a quiet four-point-bend fixture and demonstrated the feasibility of using wideband acoustic emission transducers to locate and characterize microdamage states in short-fiber reinforced composites. Showed that fiber-orientation distributions obtained from ultrasonic measurements of elastic-wave velocities are consistent with those obtained by classical metallographic methods and from inversion of acoustic resonance spectroscopy (ARS) measurements. Thus, the feasibility of using ultrasonic methods to determine elastic moduli was demonstrated.

FY95 Outputs

1. M.J. Anderson, P.R. Martin, and C.M. Fortunko, Resonant Transmission of a Three Dimensional Sound Beam through a Solid Plate in Air: Theory and Measurement, J. Acoust. Soc. Am. 98(5), Pt. 1, Nov. 1995, pp. 2628-2638.

2. M.J. Anderson, P.R. Martin, and C.M. Fortunko, Gas-Coupled Ultrasonic Measurement of Elastic Stiffness Moduli of Polymer Composite Plates, in Proc. 1994 Ultrasonics Symp., B.R. McAvoy ed. (IEEE, New York, 1995) pp. 1255-1260.

3. V.K. Tewary, M. Mahapatra, and C.M. Fortunko, Greens Function for Anisotropic Half-Space Solids in Frequency Space and Calculation of Mechanical Admittance, J. Acoust. Soc. Am., accepted for publication.

4. V.K. Tewary and C.M. Fortunko, Theory of Elastic Waves in Three-Dimensional Anisotropic Plates, J. Acoust. Soc. Am., accepted for publication.

5. V.K. Tewary and C.M. Fortunko, Surface Waves in Three-Dimensional Half-Space Tetragonal Solids, J. Acoust. Soc. Am., submitted for publication.

6. V.K. Tewary and C.M. Fortunko, Lattice Correction to Mechanical Admittance of Solids, J. Acoust. Soc. Am., submitted for publication.

7. W.A. Grandia and C.M. Fortunko, NDE Applications of Air Ultrasonic Transducers, in Proc. 1995 Ultrasonics Symp., B.R. McAvoy ed. (IEEE, New York, 1995), in press.

8. H. Ledbetter, C. Fortunko, and P. Heyliger, Off-Diagonal Elastic Constants in Fiber-Reinforced Composites, in Proc. 10th Internat. Conf. on Composite Materials, Vol. IV (Woodhead, Cambridge, England, 1995) pp. 19-24.

Industrial Interactions:

Collaborated closely with industrial consortia: Automotive Composite Structures: Development of High-Volume Manufacturing and Low-Cost Manufacturing Processes and Design/Sensor Technologies for Seismic Upgrading of Bridge Columns. Both consortia received partial support from the NIST Advanced Technology Program. In collaboration with a small instrument maker and material inspection company specializing in testing of advanced materials (Quality Material Inspection), prepared and presented an invited paper entitled: NDE Applications of Air-Coupled Transducers. The paper was presented at the 1995 IEEE Ultrasonics Symposium in Seattle, WA.

Organized the 1996 Gordon Research Conference on NDE. The theme of the conference is: Microstructure Evolution, Characterization, and Property Relationships. The invited papers at the conference will reflect current trends in industry, academia, and national laboratories. There will also be foreign participation.

Project: GREEN'S FUNCTION METHOD FOR MATERIALS SCIENCE

Project Leader: V.K. Tewary

MSEL Program **Materials Theory and Modeling**	*MRD Focus Technology* **Ultrasonic Characterization**
Strategic Thrust **Advanced Materials**	*Character of Research* **Fundamental Research**

Technical Description

Green's function methods are developed for modeling of elastic/mechanical behavior of advanced materials. Modern advanced materials are often highly anisotropic and contain multiple phases and interfaces. The traditional techniques for calculation of Green's function for such materials are not computationally efficient. We have developed a delta-function representation for the three-dimensional continuum Green's function. This Green's function is computationally very efficient for calculation of both elastodynamic and elastostatic response of solids. We have also developed efficient techniques for calculation of lattice static Green's function for application to composite lattices containing interfaces and free surfaces.

Technical Objectives

The objective of this project is to develop computationally efficient models for elastostatic and elastodynamic response of advanced materials. Such models are needed to develop quantitative experimental methods for determining the mechanical properties and microstructure of advanced materials. They will also facilitate property studies leading to more efficient uses of such materials.

Main areas of interest:

A. Elastostatics

1. Lattice theory - Born-von Karman model (collaboration: Robb Thomson)
   a. Interfacial fracture, grain boundaries, chemical effects on fracture
   b. Partially ordered alloys (collaboration: John Simmons)

2. Continuum model for 2D anisotropic solids (collaboration: John Berger)
   a. Stress distribution in composite materials.
   b. Free edge effect, interfacial cracks, crack inclined at an angle to the interface.
   c. Displacement field-moire fringes.
   d. Damaged interface-phenomenological model.
   e. Boundary element method for solids with complicated geometries

B. Elastodynamics-continuum model for 3D anisotropic solids (collaboration: Chris Fortunko, Graham Mustoe)

1. Wave propagation.
   a. Pulse propagation in half-space solids and plates.
   b. Mechanical impedance.
   c. Pulse and monochromatic wave propagation in thin film coatings on anisotropic solids - linear and non-linear effects (collaboration: D. Hurley).

2. Acoustic emission.
   a. Acoustic emission waveforms in anisotropic half-space solids and plates.

FY95 Accomplishments

Developed a very efficient delta-function representation for the space-time Green's function for calculation of elastic waveforms in anisotropic solids. This representation requires only a one-dimensional numerical integration even for calculations of fully anisotropic three-dimensional space-time profiles of elastic waves. The delta-function representation is applied to calculate the waveforms in anisotropic infinite and semi-infinite solids (cubic, hexagonal, and tetragonal), and anisotropic cubic plates. Frequency-space waveforms and mechnical admittances for such solids are also calculated.

An important advance was made in the theory of mechnical admittance of solids. The mechanical admittance is the mechanical analog of electrical admittance and is an important parameter in the design of ultrasonic transducers. It is traditionally calculated by using the continuum model of solids. However, the imaginary part of the point admittance is not rigorously defined in the continuum model because of the characteristic singularity at the origin in the continuum Green's function. The singularity is unphysical and arises because the continuum model is only the asymptotic limit of the lattice model and is not valid when the distance between the source and observation points is less than the lattice spacing as in the case of mechanical admittance. We showed that the singularity in the imaginary part of the mechanical admittance can be removed by incorporating the lattice correction in the continuum Green's function. A representation was developed for the two-dimensional elastostatic Green's function for a bimaterial composite containing a damaged or defective interface. The interfacial defects are expressed in terms of parameters in the continuity conditions for the Green's function. The parameters can be determined by comparing the theoretical displacements with the observed data from moire fringes.

The application of the elastic Green's function to the boundary element analysis of anisotropic materials of industrial interest is further developed. Computer codes are written for boundary element analysis of anisotropic materials containing interfacial cracks. This work is of a strong industrial interest and several contacts with the universities and industries have been established.

Organized a Workshop (Aug. 1994), sponsored by CTCMS on Green's function and boundary element method for modeling of mechanical behavior of advanced materials. The workshop provided a platform for establishing important technical contacts between NIST, universities, and industries. During FY95, this resulted into formation of research clusters involving NIST, universities, and industries. Seven projects were identified which are now supported jointly by NIST and the National Science Foundation. Active research work has started on all these projects.

FY95 Outputs

The following archival manuscripts were completed:

1. Computationally efficient representation for elastostatic and elastodynamic Green's functions for anisotropic solids by V.K. Tewary, Phys. Rev. 51B (1995).

2. Green's function for anisotropic half-space solids in frequency space and calculation of mechanical admittance by V.K. Tewary, M. Mahapatra, and C.M. Fortunko; submitted to Journal of Acoustic Society of America.

3. Boundary-integral analysis of anisotropic bimaterials with an interface crack by John Berger and V.K. Tewary; Proceedings of the International Conference on the Boundary Element Method held at Hawaii, Aug. 1995; details to be published in a full paper.

4. Elastic Green's function for a composite solid containing a crack at an angle to the interface by V.K. Tewary and John Berger; Proceedings of the International Conference on the Boundary Element Method held at Hawaii, Aug. 1995; details to be published in a full paper.

5. Lattice correction to mechanical admittance of solids by V.K. Tewary and C.M. Fortunko; submitted to Journal of Acoustic Society of America.

6. Theory of elastic waves in three-dimensional anisotropic plates by V.K. Tewary and C.M. Fortunko; submitted to Journal of Acoustic Society of America.

7. Elastic Green's function for a damaged interface in anisotropic materials by John Berger and V.K. Tewary; accepted for publication in Journal of Materials Research.

8. Surface waves in three-dimensional half-space tetragonal solids by V.K. Tewary and C.M. Fortunko; submitted to Journal of Acoustic Society of America.

Project: **WAVEFORM-BASED ULTRASONICS**

Project Leader: C.M. Fortunko
D.A. Hurley, M.A. Hamstad, R.L. Santoyo, J.D. McColskey, M.C. Renken, E.S. Boltz

MSEL Program	*MRD Focus Technology*
Nondestructive Evaluation	Ultrasonic Characterization
Strategic Thrust	*Character of Research*
Measurement Base and Standards	Measurement Research

Technical Description

High-resolution ultrasonic techniques and instrumentation are being developed to facilitate characterization of microstructure and failure mechanisms of advanced materials. In particular, advances are being made in high-fidelity acoustic emission and nonlinear ultrasonics. The acoustic-emission work is enabled by the availability of new wideband transducers, electronics, and sound-propagation models, which should facilitate source location and damage characterization. In addition, the new techniques will improve discrimination of acoustic emission events from extraneous-noise signals. The work in the area of nonlinear ultrasonics is also motivated by a need for better microstructure-characterization methods. Therefore, emphasis is placed on quantifying the relationships between nonlinear material responses and microstructural features. Both theoretical and experimental tools are being developed for this purpose.

Technical Objectives

1. Develop wideband, high-fidelity acoustic-emission methods, including necessary measurement methods and instrumentation, for source location and damage characterization.

2. Develop an understanding of acoustic-emission phenomena in order to facilitate discrimination of acoustic-emission events from extraneous noise.

3. Develop and evaluate theoretical models and experimental apparatus for nonlinear ultrasonic studies of material microstructure.

4. Establish and validate quantitative relationships between nonlinear ultrasonic phenomena and microstructure. Apply such relationships to practical material systems, including advanced coatings.

FY95 Accomplishments

Compared the performance of wideband (NIST-developed) acoustic emission sensors with conventional, resonant sensors. Showed that the use of wideband sensors can improve discrimination between extraneous noise and acoustic emissions.

Developed wideband acoustic emission sensors for fiber-reinforced composite materials. Showed that such transducers can be used to locate acoustic emission sources in such materials as well as monitor the state of damage.

Established a CRADA with Dunegan Engineering Consultants, Inc. The purpose of the agreement is to jointly develop an improved, commercial sensor for wideband acoustic emission applications.

Established an informal working group in the area of nonlinear ultrasonics. The group now includes Johns Hopkins University, Ritec, Inc., Southwest Research Institute, NIST, NASA, and the (German) Institute for Nondestructive Testing.

Took possession of and evaluated a new, first-of-a-kind ultrasonic instrumentation system for non-linear ultrasonic studies of materials. The instrument was developed under a CRADA agreement with Ritec, Inc. The results of the NIST tests will be incorporated in a second prototype of the instrument and in production versions, which will be sold commercially.

FY95 Outputs

1. E.S. Boltz, C.M. Fortunko, M.A. Hamstad, and M.C. Renken, "Absolute Sensitivity Limits of Air, Light and Direct-Coupled Wideband Acoustic Emission Transducers," in Proc. 1994 Review of Progress in Quantitative NDE, edited by D.O. Thompson and D.E. Chimenti (Plenum Press, New York, 1995), pp. 967-974.

2. C.M. Fortunko, R.E. Schramm, C.M. Teller, G.M. Light, J.D. McColskey, W.P. Dube, M.C. Renken, "Gas-Coupled, Pulse-Echo Ultrasonic Crack Detection and Thickness Gaging," in Proc. 1994 Review of Progress in Quantitative NDE, edited by D.O. Thompson and D.E. Chimenti (Plenum Press, New York, 1995), pp. 951-958.

3. M.A. Hamstad and C.M. Fortunko, "Development of Practical Wideband High-Fidelity Acoustic Emission Sensors," in Proc. of SPIE Conf. on "Nondestructive Evaluation of Aging Infrastructure 1995," June 6-8, 1995, edited by S. Chase, Proc. SPIE, Vol. 2456, (1995), pp. 281-288.

4. E.S. Boltz and C.M. Fortunko, "Determination of the Absolute Sensitivity Limit of a Piezoelectric Displacement Transducer," in Proc. 1995 Review of Progress in Quant. NDE, D.O. Thompson and D.E. Chimenti eds. (Plenum, New York, 1995) in press.

5. M.C. Renken and C.M. Fortunko, "Impact of Quantization Noise on the Quality of Ultrasonic Signal Deconvolution," in Proc. 1995 Rev. of Prog. in Quant. NDE, D.O. Thompson and D.E. Chimenti eds. (Plenum, New York, 1995) in press.

6. C.M. Fortunko and E.S. Boltz, "Comparison of Absolute Sensitivity Limits of Various Ultrasonic and Vibration Transducers," in Proc. of the Seventh International Conf. on Nondestructive Characterization of Materials, June 19-24, Prague (Plenum, New York, 1996) in press.

7. E.S. Boltz and C.M. Fortunko, "Absolute Sensitivity of Various Ultrasonic Transducers," in Proc. 1995 Ultrasonics Symp. B.R. McAvoy ed. (IEEE, New York, 1995), in press.

Project: SENSORS FOR INDUSTRIAL NDE

Project Leader: A.V. Clark
S.R. Schaps, W. Johnson, G.A. Alers, R.E. Schramm, C.M. Fortunko

MSEL Program Nondestructive Evaluation	*MRD Focus Technology* Ultrasonic Characterization
Strategic Thrust Measurement Base and Standards	*Character of Research* Measurement Research

Technical Description

Producing industrial quantities of materials with specified properties requires sensors that can monitor the key variables during the critical processing steps. Furthermore, additional sensors are needed after the material is manufactured as well as after having been being in service in order to make sure that no defects were built in or that none have developed during service. Most of these sensors are specialized to the materials, their unique processing variables, and their service environments.

Technical Objectives

The objective of this program is to develop sensors that can survive in a mass-production environment and that can produce outputs useful for process control or for the detection of defects. To be effective, each sensor and its particular data-analysis algorithms must be designed for the specific material and its intended use. During recent years, three diverse objectives have been undertaken. First was the development of a couplant-free, ultrasonic inspection technique for a weld in a mass-produced automotive part. Second was the development of a noncontacting thermometer to be used for continuous monitoring of the temperature of sheet aluminum while it is being hot rolled. Third was a device for quantitative measurement of the residual stress built up in railroad wheels by excessive braking.

FY95 Accomplishments

On-line Inspection of Friction Welds. During the past two years, NIST has been operating under a CRADA with the Automotive Safety Products Division of Morton International, Inc. to develop a technique for detecting weld defects in a friction weld of a mass-produced part intended for an air-bag inflator. Because this inflator uses a rapid chemical reaction to produce the gas to drive the inflator bag, the manufacture of the inflator parts must be performed under very clean conditions, and the welded joints that seal the package must be free of defects that could cause a malfunction during the millisecond time period when the bag is being deployed. Conventional ultrasonic inspection could be used to detect the weld defects, but this technique requires a couplant fluid (usually water) to conduct the sound to and from the part as well as careful alignment of the transducers with the part surface to insure a correct trajectory for the sound beam inside the part. In this application, no water or other couplants could be tolerated, and the mechanical fixtures had to be capable of maintaining precise alignments for inspecting up to a million parts per month (2 s per inspection). During FY94, NIST demonstrated an electromagnetic acoustic transducer (EMAT) that could launch and detect the necessary ultrasonic waves in the aluminum inflator part across an air gap without any liquid couplant. Because the waves were generated in the part's surface, precise alignment was not critical. During FY95, a prototype inspection system based on EMATs was assembled out of laboratory instrumentation, and an algorithm for accepting or rejecting inflator units was tested on parts containing simulated weld defects. These tests were so encouraging that Morton funded a commercial subcontractor to assemble an automatic inspection system according to NIST specifications. Tests on several hundred parts manufactured under contaminated conditions and tested under operating conditions showed that the EMAT inspection system could reject parts with weak welds and accept parts with strong welds. Currently, three improved versions of the automatic inspection system have been delivered to Morton air bag production facilities and are being used for on-line inspections.

<u>Eddy Current Thermometer for Aluminum Processing:</u> Several years ago, the Aluminum Association and NIST established a CRADA to develop an on-line device to monitor continuously the temperature of sheet aluminum as it leaves the hot-finishing mill to be coiled up for shipment. Current practice consists of waiting until the material has been coiled and moved off the line to make the "official exit temperature" measurement with a hand operated, contacting thermocouple. This technique does not reflect any variations in temperature that may have occured during the rolling process and is inaccurate by the amount of cooling that has occured between the final rolling step and the actual temperature measurement. Since the exit temperature is an important control parameter for the metallurgical microstructure, the CRADA was focused on demonstrating the feasibility of a noncontacting thermometer that could operate on rapidly moving sheet metal that is covered by lubricant bubbles and bouncing around the pass line of the rolling mill. The approach taken by NIST was to develop an eddy current technique that measures the resistivity of the sheet aluminum from a pair of coils mounted nearly a meter away from the sheet. Since the resistivity of aluminum and its alloys are known functions of temperature, this measurement of resistivity is sufficient for producing a noncontact resistance thermometer in which the aluminum sheet itself forms the resistive element.

Early in FY95, a prototype of this type of eddy current thermometer was installed and tested at an Alumax Corporation rolling mill in Lancaster, PA. It demonstrated an accuracy of only $\pm 12\,^{\circ}C$ because the correction to the resistivity for the composition of the specific alloy being rolled could be estimated only from the nominal composition of the ingot at the start of the rolling operation. In the Alumax mill as well as in other mills, the incoming ingot is hot-formed into a long, thick slab before it is sent to the multi-stand, hot-finishing mill. NIST took advantage of the facts that each hot slab is stopped for trimming prior to entering the finishing mill and its temperature is measured at this time by a contacting thermocouple. A special eddy current probe was designed to accompany the thermocouple to the surface of the slab and to measure its resistivity while its temperature was being measured. This combined measurement of resistivity and temperature, in effect, calibrated the resistance-temperature characteristic for that particular alloy and allowed the accuracy of the noncontact, eddy current thermometer at the exit end of the hot finishing mill to output sheet temperatures accurate to ± 7°C. This accuracy appears to be satisfactory to the Aluminum Association because it is near the value commonly observed by modern IR and contact thermocouple devices under mill operating conditions.

Ultrasonic Measurement of Residual Stress in Railroad Wheels:

Electromagnetic Acoustic Transducers (EMATs) are particularly well suited to the generation and detection of ultrasonic shear waves in metals. Thus, they are a natural choice for the transducer with which to measure the acoustic birefringence introduced into a material by the presence of an applied or a residual stress. Unfortunately, a direct measurement of the difference in ultrasonic wave velocities (the birefringence) of the two possible shear wave polarizations along the same propagation direction is insufficient to define the stress because texture and other microstructural features in practical materials introduce effects that can mimic the stress effect. If the birefringence can be monitored during the application of an external load, or if it can be measured before and after the introduction of a residual stress, then the texture contributions can be assumed to remain unchanged and can be subtracted from the data to yield a direct measure of the stress.

The "before and after" measurement technique has been developed for application to railroad wheels that have been subjected to excessive braking during service. To stop a train, brake shoes are forced against the circumferential face of each wheel to create a large frictional drag. The energy dissipated appears as heat that increases the temperature of the wheel's rim to the point of plastic deformation. Upon cooling, the rim shrinks by thermal contraction and is left in a state of tensile residual hoop stress. After many brakings, this residual hoop stress can build up to a level where small wear cracks in the rim can grow spontaneously at catastrophic rates to shatter the entire wheel. Early detection of these tensile stresses by visual inspection of the discoloration left by the repeated heating and cooling cycles caused by braking has proven unreliable.

During FY95, an EMAT based ultrasonic birefringence measurement system developed at NIST during the past several years was packaged for use in the field by skilled technicians and shipped to the Griffin Wheel Company in Chicago, IL. where most of the American railroad wheels are manufactured. The manufacturing process has been designed to leave a compressive hoop stress in the rim of the wheel, so a nondestructive measurement of this compressive stress can be a valuable process control tool. In addition, this particular manufacturer maintains a dynamometer that simulates the drag braking process. Under a contract with the Federal Railroad Administration, quantitative measurements of the development of the tensile hoop stress under various braking conditions were undertaken. At the same time, a second ultrasonic birefringence measurement system based on conventional piezoelectric transducers was used to develop additional data and to improve confidence in the general technique. This second system was developed in Europe by the Polish Academy of Sciences and the Polish National Railways with the full cooperation of NIST personnel over several years. The results of the dynamometer tests showed essential agreement between the two ultrasonic instruments and these, in turn, agreed with destructive tests where dimensional changes were monitored while a saw cut relieved the residual stresses. Spectacular agreement was observed in a case where the ultrasonic measurements showed a large tensile hoop stress and the test wheel shattered when the saw cut became deep enough to simulate a small crack in the tread of the wheel.

FY95 Outputs

1. S. Schaps and A. Van Clark, "A Non-contact Method of Inspecting Inertia Weld Driver Inflators Using EMAT Technology," NIST Patent Application 6F90001.

2. A. Kahn, L. Phillips, S. Schaps and G. Alers, "Noncontact Measurement of the Exit Temperature of Sheet Metal in an Operating Rolling Mill," NIST Report, in preparation.

3. R.E. Schramm, A.V. Clark (NIST) and J. Szelazek (Polish Academy of Science), "Safety Assessment of Railroad Wheels by Residual Stress Measurements", in Nondestructive Evaluation of Aging Railroads, D.E.Gray and D. Stone, Editors, Proc. SPIE Vol. 2458, pp. 97-108 (1995)

4. R.E. Schramm, J. Szelazek and A.V. Clark, "Report No. 28 - Residual Stress in Induction Heated Railroad Wheels: Ultrasonic and Saw Cut Measurements," NISTIR 5038, May, 1995.

5. R.E. Schramm, J. Szelazek and A.V. Clark, "Dynamometer-induced Residual Stress in Railroad Wheels: Ultrasonic and Saw Cut Measurements," NIST Internal Report, in preparation.

Project: **ACOUSTOELASTIC MEASUREMENTS FOR GLOBAL NDE**

Project Leader: A.V. Clark
T.L. Anderson

MSEL Program	*MRD Focus Technology*
Nondestructive Evaluation	**Ultrasonic Characterization**
Strategic Thrust	*Character of Research*
Measurement Base and Standards	**Infrastructure**

Technical Description

Acoustoelastic measurements allow the stress in a structural member to be determined in a nondestructive manner with a sensor that can be taken into the field where the member forms part of a major engineering structure such as a building, a bridge, or a crane. As the structure ages or is stressed by unusual events like earthquakes or accidental overloads, acoustoelastic measurements at a few carefully chosen locations can tell whether some parts are now supporting more or less than their design loads. Thus, acoustoelastic techniques applied at a few, judiciously chosen locations may be able to assure the overall or global integrity of a large structure.

Technical Objectives

The objective of this program is to apply acoustoelastic techniques to a specific class of structures where the basic concepts of Global NDE can be demonstrated. Steel bridges have been chosen because the beams are large, and usually accessible, and have reasonably well defined design loads. Furthermore, many of the approximately 500 000 bridges in the US have structural defects but cannot be replaced for economic reasons. As a result, the bridge maintenance community is moving toward inspection techniques that yield a quantitative evaluation of the overall structural integrity of bridges. We expect that properly executed acoustoelastic measurements will be able to satisfy this Global NDE need. To be most effective in the case of steel bridges, the acoustoelastic measurements should be guided by fracture mechanics principles. Therefore, a secondary objective of the program is to develop a rational analysis for the effect of crack-like defects on bridge support beams.

FY95 Accomplishments

Fracture Mechanics of Bridge Girders. In this study, we considerd the effects of three common failure mechanisms in bridges: brittle fracture, fatigue, and plastic deformation. We also performed a comprehensive analysis of how these failure mechanisms affect bridge performance and used probabilistic methods to address questions raised by variability in the bridge material's resistance to fracture.

Brittle fracture is characerized by the stress intensity factor K. When K exceeds K c (the critical stress intensity factor), fracture occurs. We calculated K for the case of a crack in the flange as well as the case of a crack in the web of an I-beam. Thus, we can follow the evolution of K for a crack starting in the flange and propagating into the web of the beam. Such a crack has two possible ill effects: (1) loss of stiffness, which may break the deck and (2) redistribution of the load to stiffer members, which may overload them. We used our calculated values of K to determine the loss of girder stiffness from cracking and found that there was negligible stiffness change until the girder was about 80% fractured. A structural analysis using this change in stiffness revealed negligible load redistribution until the girder was about 90% fractured. For cracks deeper than 90%, load shedding occurs so K finally vanishes and there is no stress to drive the crack. Hence, in principle, total fracture should not occur.

However, such deep cracks may continue to grow due to fatigue. This kind of damage would be caused by heavy truck traffic which induces stresses above the constant amplitude fatigue limit (CAFL). In conventional fatigue-damage monitoring, the effective cyclic applied stress is determined by measuring the various stress levels resulting from bridge vibrations and converting them to an equivalent constant amplitude stress, S. The American Association of State Highway and Traffic Officials (AASHTO) has recommended the use of plots of S versus N to estimate the number of fatigue cycles needed to produce failure. These AASHTO plots are quite conservative, so that when a given bridge reaches its nominal fatigue design life it is likely to have sustained only moderate damage. During FY95, we developed an algorithm to calculate fatigue crack growth for various spectra of expected stress ranges using the case of a plate welded to the flange. This type of welded detail increases the beam stiffness but it is the least resistant to fatigue damage. We found that when the nominal fatigue life was reached, about 50% of the cracks are only slightly larger than their initial size. Even after these cracks extended through the flange, the remainder of the beam could still carry a load. To describe failure when cracks were confined to the web, we included the possibility of plastic deformation and used the failure assessment diagram (FAD) approach employed in the petrochemical industry. This approach allowed us to characterize the probability of failure for steels with varying toughness and yield strengths. We found relatively little improvement with higher yield strength steels and about a 50% increase in fatigue life with higher toughness steels. However, our calculations indicated that the number of fatigue cycles for the crack to completely penetrate the flange was several times larger than for failure due to a web crack. Hence, we expect only moderate improvement in performance by introducing more advanced steels to current bridge design practices.

Acoustoelastic Measurements. In parallel with the analytical studies, we performed field tests to measure actual stresses in highway bridges. Such stresses are conventionally measured with strain gages, which are time-consuming to install and require special precautions when lead-based paint is removed. They also measure only changes in stress and cannot detect residual stresses or the dead-load stress from the weight of the structure itself. In contrast our ultrasonic method employed noncontacting transducers which generate sound directly in the steel and require no paint removal. Furthermore, the transducers employ permanent magnets and are easily attached and removed. One type of these electromagnetic transducers (EMATs) was used to generate and receive polarized shear waves propagating through the thickness dimension of the web. The small difference in sound velocity between the two polarizations caused by the presence of stress was measured. The second type of EMAT used measured the change in stress in the flange of the I-beam when the load on the bridge changed. By using both of these sensors, liveloads (dynamic stresses) and static loads could be measured. The liveloads were measured in a bridge as a test vehicle was driven over it at a range of speeds. Good agreement with strain-gage data was obtained. Static loads (including those due to pre-stressing during fabrication) were also measured on the same bridge and compared with the stresses expected from the design. These field tests were performed in collaboration with personnel from the Virginia Transportation Research Council (VTRC) and the Constructed Facilities Center of West Virginia University.

Additional measurements were made on a second bridge at the request of the Virginia Department of Transportation (VDOT). This bridge was of novel design in that it had no expansion joints and thus eliminated problems of bearing corrosion caused by salting. Thermal expansion causes this bridge to push against backfilled soil at the abutments, with attendant compression of the girders. If this compression is too large, either plastic deformation or buckling can result. VTRC personnel installed strain-gage instrumentation on the bridge at the time of construction and monitored its status for several years. Anomalous readings were recorded at one abutment that indicated potentially dangerous stress. Our ultrasonic measurements made on several girders at opposite sides of the bridge indicated that little stress difference existed. Thus, the bridge was judged to be safe. Subsequent replacement of suspect electronics in the VTRC instrumentation confirmed this result. VTRC personnel returned to this bridge on two further occasions for further monitoring. The ultrasonic data indicated good repeatability and agreement with VTRC instrumentation.

These field tests with EMATs demonstrate that NIST has successfully completed two phases of technology transfer; training of VTRC personnel in use of the ultrasonic equipment, and delivery of transducers and associated electronics to VTRC.

FY95 Outputs

1. "Application of Electromagnetic-Acoustic Transducers for Nondestructive Evaluation of Stresses in Steel Bridge Structures," M.G. Lozev, A.V. Clark, and P.A. Fuchs, to be published as a Virginia Transportation Research Council Report.

2. "New Approaches to Life Assessment in Steel Bridges," T.L. Anderson and A.V. Clark, NISTIR, in review process.

3. "Quantitative Bridge Safety Assessment Utilizing Fracture Mechanics and Ultrasonic Stress Measurements," A.V. Clark and T.L. Anderson, to be published in proceedings of Structural Materials Technology NDE Conference to be held Feb. 20-23, 1996 in San Diego.

4. "Monitoring Bridge Fatigue Loads with Ultrasonic Transducers," P.A. Fuchs, A.V. Clark and S.R. Schaps, SENSORS Magazine, Vol. 12, No. 11, 1995, p.20

MICROMETER-SCALE MEASUREMENTS FOR MATERIALS EVALUATION

Methods for measuring the properties of materials must be minaturized to evaluate the behavior of films and coatings. Our goal is to measure material properties at a size scale such that the processing and microstructure conditions in the specimen are the same as those in the application. The influence of microstructure on performance is evaluated by simultaneously measuring and observing at high magnifications salient features, such as deformation in electronic packaging or heat flow in ceramic coatings. The Division has micrometer-scale measurement projects related to electronic packaging, ceramic coatings, and engineered surfaces.

Project: **EXPERIMENTAL MICROMECHANICS BY e-BEAM MOIRE**

Project Leader: D.T. Read
E.L. Drexler, V.K. Tewary

MSEL Program	*MRD Focus Technology*
Electronic Packaging and Interconnects	**Micrometer-Scale Measurements**
Strategic Thrust	*Character of Research*
Measurement Base and Standards	**Electronics**

Technical Description

We seek to improve the usefulness of modeling and simulation in the design and manufacture of advanced electronic packaging and interconnect structures by providing direct quantitative experimental verification of predicted deformations, and by characterizing actual failure modes. This work contributes to the areas of modeling and simulation, advanced packaging, and reliability listed in the National Technology Roadmap for Semiconductors.

Local deformations of packaging elements are measured over fields ranging from 50 by 50 μm to 500 by 500 μm.This is accomplished by preparing the specimen surface with line gratings at pitches of 100 nm to 1 μm, using electron beam lithography, and observing them in the scanning electron microscope at magnifications from 200X to 2000X. Deformations produce changes in the local moiré fringe density. These changes are analyzed to give the full-field deformation in the direction perpendicular to the electron beam raster scan. Deformations are modeled by boundary integral methods based on newly developed Green's functions to extract stresses and crack driving forces.

Technical Objectives

Develop and apply the e-beam moiré technique to measurement of strain and observation of deformation at high magnification and use the observations to characterize failure modes and to verify mathematical models and simulations of microscale mechanical behavior.

Apply the e-beam moiré measurement technique to manufacturability and reliability testing of electronic packaging and interconnect structures provided by industry/academic partners such as advanced printed circuit boards, high density interconnects, ball grid arrays, flip-chip solder joints, and conductive adhesives. Improve the experimental technique to allow writing of more durable, higher-contrast and denser gratings more consistently with less effort, and permit observation of deformation in two orthogonal directions. Use the experimental results to characterize failure modes and to verify various modeling approaches.

FY95 Accomplishments

Measured local deformations around copper vias in a multilayer copper/polymer high-density multichip module substrate manufactured by General Electric. Compared measured results to analytical predictions made by Prof. Ian Grosse and his group at the Univ. of Massachusetts. There was generally good correspondence between the experiment and the models; differences in behavior between the cross-sectioned specimen and the actual device were revealed.

Initiated a program of experiments to determine deformations in conductive adhesives and associated structures. Arranged collaborations with 3M, Ablestik, and AI Technology. Acquired specimens from each of the companies and made preliminary measurements to assess the behavior of the materials.

Improved the capability of the e-beam moiré technique by producing double and quadruple line-density grids. Demonstrated that orthogonal gratings could be written on conductive adhesives. Carried out a successful calibration and repeatability investigation of the apparatus using our copper standard.

FY95 Outputs

"Local Deformation of Plated Through Holes Under Thermomechanical Loading," by David T. Read and Elizabeth S. Drexler, in Mechanics and Materials for Electronic Packaging: Volume 2, Thermal and Mechanical Behavior and Modeling (Proceedings of the 1994 International Mechanical Engineering Congress and Exposition, Chicago, Illinois, November 6-11, 1994) edited by Michael Schen and Hiroyuki Abé, AMD-Vol. 187, American Society of Mechanical Engineers, 185-194 (1994).

"Thermomechanical Behavior of a High Density Polymer Overlay MCM Interconnect Structure: Experiments and Analysis," by D. Read, E. Drexler, I. Grosse, J. Benoit, J. DiTomasso, E. Bernard, D. Holzhauer, P. Rocci, and M. Stoklosa, in Application of Fracture Mechanics in Electronic Packaging and Materials, EEP-Vol. 11/MD-Vol. 64, proceedings from 1995 ASME International Mechanical Engineering Congress and Exposition, November 12-17, 1995, San Francisco, California, edited by T. Y. Wu, W. T. Chen, R. A. Pearson, and D. T. Read, American Society of Mechanical Engineering, New York, 1995, pp. 251-261.

Project: **MECHANICAL BEHAVIOR OF THIN FILMS**

Project Leader: D.T. Read
J.M. Phelps, R.R. Keller, F.R. Fickett

MSEL Program Electronic Packaging and Interconnects	*MRD Focus Technology* Micrometer-Scale Measurements
Strategic Thrust Measurement Base and Standards	*Character of Research* Electronics

Technical Description

Thin films are an essential component of all advanced electronic devices. Understanding of failure modes in these devices, especially interface delamination, requires a knowledge of the mechanical behavior of the films. Techniques for measuring the mechanical behavior of thin films are being developed and applied. Because the films are formed by condensation from the vapor phase, their microstructures, and hence their mechanical properties, are quite different from those of bulk materials of the same chemical composition. While the general principles of conventional mechanical testing are applicable to thin films, conventional test equipment and techniques are not. Because thin films are of the order of 1 μm thick, the failure loads are low, and the specimens cannot be handled directly.

Electromigration and stress voiding, which are important electrical failure modes, are related to the same microstructural features that control mechanical behavior. Severe triaxial stress states develop in narrow metallizations and are manifested in different forms for different grain orientations. Understanding and solving the problems of void formation is essential to the continued development of metallizations on a submicrometer scale. The 1994 SIA Roadmap specifically identifies research on stress effects and stress-induced voiding as a priority need for both interconnect and thermal/thin film technologies at the submicrometer level.

Technical Objectives

Develop experimental techniques to measure the mechanical properties of thin films, such as basic tensile properties, fatigue and fracture resistance. Relate the mechanical behavior of thin films to their microstructure.

Determine various aspects of the mechanical behavior of thin films of pure metals (copper, aluminum, gold) and technically relevant alloys. Relate mechanical behavior to microstructure, by imaging dislocation motion in the transmission electron microscope (TEM) during deformation. Correlate scanning electron microscope (SEM) and TEM observations of microstructural weak points and resulting failure mechanisms with mechanisms of stress voiding and electromigration. Assess and, when necessary, modify microstructurally based models which sufficiently describe analogous behavior in bulk metals. Extend our test techniques from their present level (1 μm thick by 200 μm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths on the order of 1 μm and thicknesses of 0.5 μm).

FY95 Accomplishments

Measured mechanical behavior of sputtered copper films for comparison with electron-beam evaporated film. The results show that sputter-deposited films are significantly stronger than evaporated ones, which is consistent with the observed finer grain structure of the sputtered films. Obtained preliminary data on hardness for these films, which indicated no consistent quantitative correlation between hardness and strength.

Measured fatigue of electron-beam-evaporated copper films and began microstructural characterization. The measurement required improvement of the data-acquisition equipment and software used in the test. Demonstrated fatigue lives of several tens of thousands of cycles, with the longest test lasting beyond 100 000 cycles.

Observed microstructural behavior of electron-beam evaporated films 0.26 μm thick during tensile strain to failure in the TEM. Cracks formed easily and propagated both through the grains and along the grain boundaries. Only a few dislocations were seen.

Measured mechanical behavior of epitaxial silicon in collaboration with Ford Microelectronics and University of Colorado. Made first set of specimens of Al-1%-Si and began mechanical property measurements.

Demonstrated the feasibility of using backscatter Kikuchi diffraction (BKD) to obtain crystallographic data from narrow metallizations that had undergone stress-induced voiding.

Collected and indexed approximately 300 BKD patterns, in digitized form, from stress-induced voiding specimens made at Cornell's National Nanofabrication Facility. Began the process of analyzing these to correlate crystallographic features to void formation. To our knowledge, such data has not been acquired before.

FY95 Outputs

Our apparatus for mechanical testing of thin films is being cloned at the University of Colorado. Their device has been fabricated and initial tests performed.

David T. Read and James W. Dally, "Mechanical Behavior of Aluminum and Copper Thin Films," Mechanics and Materials for Electronic Packaging: Volume 2, Thermal and Mechanical Behavior and Modeling (Proceedings of the 1994 International Mechanical Engineering Congress and Exposition, Chicago, Illinois, November 6-11, 1994), edited by Michael Schen and Hiroyuki Abé, AMD-Vol.187, American Society of Mechanical Engineers, 41-49 (1994).

Shawn J. Cunningham, Wan Suwito, and David T. Read, "Tensile Testing of Epitaxial Silicon Films," in Transducers '95, Vol. 2, Paper No. 255-PA8, pp. 96-99, Proceedings of The 8th International Conference on Solid-State Sensors and Actuators and Eurosensors IX, June 25-29, 1995, Stockholm, Sweden.

R.R. Keller, J.M. Phelps and D.T. Read, "Tensile and Fracture Behavior of Free-Standing Copper Films," submitted to Materials Science and Engineering A.

R.R. Keller and J.M. Phelps, "SEM observations of misfit dislocations in epitaxial $In_{0.25}Ga_{0.75}As$ on GaAs(001)," Journal of Materials Research, to appear in March 1996.

Project: THERMAL CONDUCTIVITY OF THERMAL BARRIER COATINGS

Project Leader: A.J. Slifka
J.M. Phelps, B.J. Filla, F. R. Fickett

MSEL Program Ceramic Coatings	*MRD Focus Technology* Micrometer-Scale Measurements
Strategic Thrust Advanced Materials	*Character of Research* Aeronautics

Technical Description

Accurate knowledge of the thermal conductivity of thermal barrier coatings and its relationship to processing parameters is necessary in order to produce coatings more economically and to increase reliability and performance. Increased reliability and performance will allow the future use of thermal barrier coatings in more demanding applications than allowed by the current technology. More economical coatings will replace current expensive monolithic ceramics and superalloys substrates, leading to new applications in the consumer economy. In addition to measurement of the thermal conductivity of coatings on both the macro- and micro-scale, this project uses advanced techniques of electron microscopy to characterize the microstructure and microchemistry responsible for the bulk thermal performance of coating systems.

Technical Objectives

To develop methods to measure the thermal conductivity of ceramic coatings and to relate the thermal performance to the microstructure of the coatings.

Measure the thermal conductivity of representative thermal barrier coatings, substrate materials, and monolithics to determine bulk values and interfacial resistances for the coating systems. Evaluate microscopy and spectroscopy for the various microstructural analyses. Observe the effects of varying processing parameters on microstructural features and bulk thermal conductivity. Model bulk thermal conductivity using microstructural information. Extend the model to include correlations between microstructural information and processing parameters that will result in a model that generates desired bulk thermal conductivity given appropriate processing parameters. Interact with the coatings industry to insure a supply of state-of-the art samples. Develop measurement apparatus and techniques, appropriate reference materials, and documentation to allow comparison with, and perhaps calibration of, measurement techniques used in the industry.

FY95 Accomplishments

Set up collaborations and CRADAs with industrial (Pratt & Whitney, NAL/KRC-Japan), academic (SUNY), and research laboratory (NASA Lewis, German Aerospace Research (DRL), Idaho National Engineering Laboratory) groups in an effort to obtain representative samples of state-of-the-art thermal barrier coating systems prepared with a variety of materials and processing conditions.

Completed a new thermal analysis of our unique high-temperature guarded hot-plate (HTGHP) system and modified the heat sink and sample containment setup to increase precision and accuracy. Used the system to measure bulk thermal conductivity of substrate materials and two different coating systems. Measured a model substrate material, 410 stainless steel, which is used as a base for some coating systems. Completed two-thirds of a test series on plasma-sprayed zirconia thermal barrier coatings from SUNY, including measurement of the coating thermal conductivity, evaluation of systematic errors of the HTGHP system, and determination of the effect of surface finish of the coating on the interfacial resistance between the coating and the measurement plate. This series of tests will provide a model for future coatings, allowing more rapid analysis of subsequent coating systems. Evaluated samples provided by the Japanese NAL and determined that they were not suitable for our measurement system; new samples are being prepared.

Determined the microstructure of some of the coatings using scanning and transmission electron microscopy and electron diffraction. Used energy and wavelength dispersive spectroscopies and secondary-ion mass spectrometry to determine microchemical content and distribution of chemical species for some specimens.

Continued development of an infrared microscopy system to evaluate the micron-scale thermal behavior of the constituents of the coatings. Tests and calibration to allow us to account for variations of emissivity among the materials, are nearing completion.

FY95 Outputs

A paper was presented at the Mechanics and Physics of Layered and Graded Materials Conference in Davos, Switzerland, titled "High Resolution Infrared Microscopy of a Functionally Graded Material," which dealt with the development of the infrared microscopy system.

Project: MAGNETIC SENSING FOR MICROSTRUCTURAL CHARACTERIZATION

Project Leader: F.R. Fickett

MSEL Program **Nondestructive Evaluation**	*MRD Focus Technology* **Micrometer-Scale Measurements**
Strategic Thrust **Measurement Base and Standards**	*Character of Research* **Measurement Research**

Technical Description

Modern methods for creating engineered surfaces, such as ion implantation and ion beam-assisted deposition, alter the properties of the substrate surface only to micrometer depths. Measurement of the surface condition and uniformity of the preparation is beyond the capability of most characterization methods, especially if the area to be investigated is relatively large, the sample cannot be destroyed, or the environment is less than ideal in terms of cleanliness or atmosphere. Since most engineered surfaces of industrial interest are made on ferromagnetic substrates, their characterization by magnetic techniques offers some promise. There is reason to believe that the magnetic properties of the surface layer may correlate well with the properties of interest in applications, such as surface integrity, hardness and, perhaps, friction coefficient. Similarly, thin nonmagnetic coatings on steel surfaces present new problems in determination of thickness uniformity and wear evaluation that may be susceptible to solution using magnetic techniques. Magnetic detection may also be useful in porosity determination of ceramic coatings where the pores are impregnated with ferromagnetic powder using vacuum or chemical methods.

The small size of the sensors used in this program makes them ideal not only for the applications mentioned above, but also for detection of flaws in magnetic media and other magnetic structures associated with data storage, as well as for more general metallic microstructural analysis. Initially we are concentrating on microscopic sensors based on giant magneto-resistance (GMR) for mapping of near-surface fields for regions in the range 1 μm to 100 μm. Some of these devices may be able to be configured as large-area scanning systems. Because they are not especially sensitive to environment and are relatively inexpensive and robust, they have potential for wide application in manufacturing environments. They are prepared by conventional deposition and lithography techniques; packaging methods and control electronics are well in hand for many (but not all) applications. Further along, techniques for evaluating magnetic properties of materials on a smaller scale, varying from micrometers to nanometers, such as magnetic force microscopy (MFM) and related scanned-probe techniques will be integrated into the program.

Technical Objectives

Develop innovative magnetic techniques for quantitative nondestructive microstructural characterization at the micrometer scale and below, and apply these to films, coatings, and engineered surfaces.

Determine the extent to which magnetic techniques offer an analysis capability not otherwise available, either in terms of detection ability or simplicity of application. In the early phase, concentrate on creation of a new class of sensors for mapping of near-surface fields by modification of existing and developmental recording head systems based on giant magnetoresistance (GMR). Apply these sensors to rapid and accurate characterization of materials and engineered surfaces. Initially, evaluate their applicability to determination of microstructure induced by ion implantation, focusing on determination of surface uniformity and depth distribution of the implanted region. Investigate potential for measurement of coating thickness and determination of porosity in ceramic coatings. Develop scanning systems using these sensors, and determine their suitability for manufacturing environments. Evaluate techniques for nanoscale magnetic imaging, such as tapping-mode magnetic force microscopy (MFM) and related instruments now under development that use magnetic resonance force microscopy (MRFM) to assess under-surface magnetics and element-specific structures.

FY95 Accomplishments

Created a new laboratory for magnetic measurements for microstructure evaluation. Developed and tested an apparatus for applying small magnetoresistive sensors to material measurements; more sensors are being obtained. At present these sensors are research devices and in limited supply; thus, for initial testing of ferromagnetic substrates, a more robust Hall-effect system was used. Since no actual engineered surface samples were available, samples from our ceramic coating project were used as substitutes. Hall-effect measurements were made to determine the magnetization structure in the base steel and to investigate changes caused by application of low fields.

Completed a computer search and evaluation of the literature on magnetic-surface measurement systems and techniques. Made a similar search on techniques for production of micronmeter-sized magnetic probes and associated electronics. Discussions were held with staff of NIST EEEL regarding application of their AFM and MFM systems to these problems.

Participated in the ATP Engineered Surfaces Project kickoff meeting and made preliminary arrangements for getting ion-implanted samples when they become available. Arranged a collaboration on ion-implanted stainless steel with Colorado State University and Colorado School of Mines.

FY95 Outputs

No publications in FY95. Project started in March.

OTHER PROJECTS

The Division has three projects that are not included in the three focus technologies.

1. Charpy Impact Testing is a Standard Reference Materials (SRM) project which the Division conducts jointly with the SRM Program.

2. Materials Processing ATP is a project to plan and initiate an Advanced Technoogy Program. It was completed in FY95.

3. Cryogenic Materials is a project that was initiated in 1952, remained a principal technical activity of the Division until recently, and was completed in FY95.

Project: CHARPY IMPACT TESTING

Project Leader: D.A. Vigliotti
C.N. McCowan

MSEL Program	*MRD Focus Technology*
Standard Reference Materials	
Strategic Thrust	*Character of Research*
Measurement Base and Standards	**Standards, Data, SRMs**

Technical Description

The Charpy impact test uses a swinging hammer and calibrated scale or encoder to assess the resistance of a material to brittle fracture. The low cost and simple configuration of the test have made it a common requirement in codes for critical structures such as pressure vessels and bridges. This project is handled jointly by the Standard Reference Materials Program, Office of SRMs, which oversees the administrative aspects of the program, and the Materials Reliability Division, which handles the technical and certification aspects.

NIST provides highly characterized standard reference materials (SRMs) to machine owners and independent calibration services, then evaluates the results of tests of these specimens on their impact machines. Owners of machines that meet the requirements of ASTM Standard E 23 are given a certificate of conformance, while owners of nonconforming machines are given recommendations on corrective actions.

Technical Objectives

- Provide rapid, accurate assessment of test data generated by our customers on the SRMs, and, where merited, certify the conformance to ASTM Standard E 23.
- Interact with the ASTM Committee responsible for the Charpy impact standard, to improve the service to the customers and reduce the scatter in the data, and to maintain a high-quality verification program to meet the needs of industry.
- Monitor the activity in the ISO Committees, so our specimens and procedures remain compatible with the associated international standards.

FY95 Accomplishments

We had 984 customers for this service in FY95, a number similar to that for previous years. The great majority of these machines were within tolerances required by ASTM Standard E 23, indicating a general improvement in machine maintenance over the past few years. As usual, we found that many users took advantage of our support services, as shown by our log of 1827 phone calls and 204 faxes. In our laboratory, we tested the 900 specimens necessary to confirm that nine new lots of reference specimens were suitable to go into the SRM inventory.

In spring 1995, ASTM published Special Technical Publication 1248, Pendulum Impact Machines-Procedures and Specimens for Verification. This STP is the archival record of the proceedings of a symposium which we helped to organize and to which we contributed five papers.

In cooperation with the NIST Standard Reference Materials Program and The American Society for Testing and Materials, we hosted a workshop in Norfolk, Virginia on November 16. This workshop, Materials and Heat Treatments for ASTM Charpy V-notch Verification Specimens, sought new ideas to improve the verification specimens offered the Standard Reference Materials Program. The participants identified several new projects that might improve the performance of the NIST reference materials. Research was planned to evaluate new materials in cooperation with Timken, Teledyne-Vasco, Sure Tool, and Thomas Shearer Inc. The group also suggested that a new ASTM task force be formed to monitor progress in the development of steel with reduced variability in properties.

We continue to be asked to add new impact verification services. This year, we expanded on several years of informal interaction with ASTM Committee D 20 on plastics by forming a new task group, X-10-279, to investigate the procedures and materials needed to verify the performance of plastics impact machines. We reported our impact results on a series of candidate metal alloys that should avoid the aging problems common to plastics.

FY95 Outputs

- Evaluated 984 customer machines and sent letters of confirmation

- Supported customer requests for assistance through 1827 phone calls and 204 faxes

- Edited ASTM STP 1248 on impact machine verification and contributed the following papers:

  "The Role of Strike Marks on the Reproducibility of Charpy Impact Test Results," A.K. Schmieder, P.T. Purtscher, and D.P. Vigliotti.

  "The Effect of Surface Finish of Charpy Anvils and Striking Bits on the Absorbed Energy," E.A. Ruth, T.A. Siewert, and D.P. Vigliotti.

  "The Effect of Charpy V-Notch Striker Radii on the Absorbed Energy," T.A. Siewert, and D.P. Vigliotti.

  "Proposed Changes to Charpy V-Notch Machine Certification Requirements," J.D. Splett and J. C.-M. Wang.

  "Low Cost Lower Bound Toughness Measurements," J.W. Dally, C.N. McCowan, D.P. Vigliotti, and O.S. Lee.

Project: **MATERIALS PROCESSING ATP**

Project Leader: T.A. Siewert
H.I. McHenry

MSEL Program	*MRD Focus Technology*
Other MSEL Programs	
Strategic Thrust	*Character of Research*
Advanced Processes	**Manufacturing**

Technical Description

The Advanced Technology Program (ATP) promotes the economic growth and competitiveness of U.S. industry by accelerating the development and commercialization of promising, high-risk technologies with substantial potential for enhancing U.S. economic growth. The ATP approach is to work with U.S. industry to plan, execute and cofund programs to develop enabling technologies which offer great benefits to the U.S. economy.

The Division led a NIST team that worked with industry to develop an ATP program on materials processing. The machinery manufacturers, including power generation, construction, and transportation, were the most active participants in the planning process. Industry's goal was to develop material-processing technologies which would enable the product innovations needed to capture new markets. Product innovations included improved performance through the use of advanced materials and cost reduction through improved processing technologies. After a year of planning with industry, an ATP program entitled "Materials Processing for Heavy Manufacturing" was proposed to NIST management, which selected it for implementation.

Technical Objectives

1. Work with industry to develop an ATP program in the area of materials processing which would accelerate the introduction of advanced materials into commercial products.

2. Demonstrate to NIST management that the proposed program had potentially large benefits to the U.S. economy, good technical ideas, and strong industry commitment.

3. Initiate the program through the ATP proposal process, including request for proposals, proposal evaluation, and project selection.

FY95 Accomplishments

- Tom Siewert proposed the program on Materials Processing for Heavy Manufacturing to NIST management in October 1994, and it was selected as one of six new programs for the Spring 1995 ATP competitions.

- We informed potential participants about the ATP program and the proposal evaluation criteria program at a series of briefings organized by industry groups:

Power Generation Industry	February 8, 1995	Charlotte, NC
Sponsored by: Electric Power Research Institute		
Heavy Equipment Industry	February 10, 1995	Peoria, IL
Sponsored by: American Society of Mechanical Engineers		
Steel Industry	February 22, 1995	Washington D.C.
Sponsored by: American Iron and Steel Institute		
Automotive Industry	March 1, 1995	Ypsilanti, MI
Sponsored by: MERRA, U.S. Automotive Materials Partnership, and Motor and Equipment Manufacturers Association		

- We prepared a Commerce Business Daily announcement which included a Request for Proposals, A Notice of Public Meetings, and supplemental program information. The two public meetings were Proposer's Conferences where general ATP information was presented in the morning, and the Materials Processing for Heavy Manufacturing Program was discussed in the afternoon.

- We evaluated 27 proposals and selected eight projects for funding. Harry McHenry served as Chairman of the Source Evaluation Board.

FY95 Outputs

The ATP Program on Materials Processing for Heavy Manufacturing includes eight projects, each of which was awarded, negotiated and initiated by ATP staff in 1995.

• Bonded Single- and Polycrystalline Turbine Blades	Westinghouse JV
• Intelligent Processing for Thermal Barrier Coatings	General Electric
• Aqueous Injection Molding of Silicon Nitride	AlliedSignal
• Incremental Forging of Superalloys	Wyman-Gordon
• Sand Casting of Superalloys	Precision Castparts

• Intelligent Welding of Structures	Caterpillar JV
• Ceramic Coating of Internal Surfaces	Praxair
• Near Net Shape Aluminum Castings	AlliedSignal JV

* JV means joint venture. The lead company is listed.

Project: CRYOGENIC MATERIALS

Project Leader: F. R. Fickett
N.J. Simon, D.R. Smith, A.J. Slifka

MSEL Program	*MRD Focus Technology*
Other MSEL Programs	
Strategic Thrust	*Character of Research*
Advanced Materials	**Research Services to other Agencies**

Technical Description

This year saw the end of our long-term involvement with cryogenic thermal and mechanical property measurement and data collection and analysis. Research on cryogenic materials has been underway in the Division since the creation of the Boulder Laboratories in 1952. Our research included the characterization and evaluation of materials for cryogenic service, the development of testing procedures and standards, and the collection and evaluation of materials property data. Recently, we worked on the development of materials technology for the design and construction of cryogenic systems for DoE, NASA, and the USAF. Over the past five years we have completed these projects and transferred to other laboratories the few measurement techniques that remained unique to NIST.

Technical Objectives

To complete all cryogenic materials programs and gracefully close out our involvement with the associated other agencies. To complete support of ATP collaboration with American Superconductor by characterizing the thermal behavior of superconducting coil specimens.

FY95 Accomplishments

Completed transfer and installation of a low-temperature, helium-cooled thermal conductivity apparatus to NASA Huntsville. Completed transfer and installation of two thermal conductivity apparatus to the Building Technology Division at NIST.

Completed a program of data compilation for the DoE International Thermonuclear Experimental Reactor (ITER) project. All publications and data collections have been archived on site.

Measured thermal conductivity and thermal expansion of superconducting coil segments from liquid helium (4 K) to room temperature (300 K). Thermal conductivity was measured on two different x-direction coil samples, including the latest ASC coil design, as well as y- and z-direction segments from that coil. Measured thermal expansion for all three principle directions.

FY95 Outputs

D.R. Smith and F. R. Fickett, "Low Temperature Properties of Silver," J. Res. Natl. Inst. Stand. Technol. **100**, 119-171 (1995). A document to accompany the Wall Chart on Cryogenic Properties of Silver [Natl. Inst. Stand. Technol. Technical Note 1363 (1994)]. This comprehensive review and evaluation should be the final word on this topic for many years.

N.J. Simon, "Cryogenic Properties of Inorganic Insulation Materials for ITER Magnets: A Review," NISTIR 5030 (1994).

N.J. Simon, Cryogenic properties of Inorganic Insulation Materials for ITER Magnets: A Review," DoE Project Report (March 1995).

N.J. Simon, "Radiation Limits for Nb_3Sn Superconductors for ITER Magnets: A Literature Review," DoE Project Report (February 1995).

Reports on all data gathered on the superconducting coil project with ASC in FY95 were sent to technical liason at ASC. Publication is not allowed at this time under the terms of the agreement.

RECENT PUBLICATIONS*

Anderson, M.; Hill, J.; Fortunko, C.; Dogan, N.; and Moore, R.; Broadband Electrostatic Transducers; Modeling and Experiments, J. Acoust. Soc. Am., Vol. 97, No. 1, 1995, pp. 262-272.

Anderson, M.; Martin, P.; Fortunko, C.; Gas-Coupled Ultrasonic Measurement of Elastic Stiffness Moduli of Polymer Composite Plates, Proc., IEEE 1994 Ultrasonics Symp., Cannes, France.

Balzar, D.; and Ledbetter, H.; Accurate Modeling of Size and Strain Broadening in the Rietveld Refinement: The "Double-Voight" Approach; Adv. X-Ray Analysis 38; 1995 pp. 397-404.

Berger, J.; Boundary Element Analysis of Anisotropic Solids with Planar Interfaces, Proc., 15th Boundary Element Intl. Conf., submitted.

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Boltz, E.; Fortunko, C.; Hamstad, M.; Renken, M.; Absolute Sensitivity of Air, Light and Direct-Coupled Wideband Acoustic Emission Transducers; Proc., Rev. of Progress in Quantitative NDE Conf., submitted., 1994.

Boltz, E.; Fortunko, C.; Determination of the Absolute Sensitivity Limit of a Piezoelectric Displacement Transducer, Proc., Rev. Prog. Quant. NDE submitted., 1995.

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Boltz, E.; Tewary, V.; Fortunko, C.; Fidelity of Michelson Interferometric and Conical Piezoelectric Ultrasonic Transducers, Proc., Rev. Prog. Quant. NDE, submitted., 1995.

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* Papers that were published or accepted for publication by the Editorial Review Boards of NIST during fiscal year 1995.

Clark, V.; Schramm, R.; Schaps, S.; Filla, J.; Safety Assessment of Railroad Wheels through Roll-By Detection of Tread Cracks, Proc., SPIE Conf. on Nondestructive Evaluation of Aging Infrastructure, pp. 109-119; 1995.

Clark, V.; and Schaps, S.; Measurement of Plane Stress States Using Electromagnetic-Acoustic Transducers; 1994, Rev. Prog. Quantitative NDE, submitted.

Clark, V.; and Schaps, S.; Acoustoelastic Determination of Residual Stress by Measurement of Resonance Peaks and Phase Shifts; J. Nondestructive Eval., submitted.

Fortunko, C.; Schramm, R.; Teller, C.; Light, G.; McColskey, D.; Dube, W.; Renken, M.; Gas-Coupled, Pulse-Echo Ultrasonic Crack Detection and Thickness Gaging, Rev. of Progress in QNDE, Vol. 14A, (D. O. Thompson & D. E. Chimenti, eds.) Plenum Press, New York, 1995, pp. 951-958.

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Hamstad, M.; Fortunko, C.; Development of Practical Broadband High Fidelity Acoustic Emission Sensors, Proc., SPIE submitted, 1995.

Hamstad, M.; Gary, J.; O'Gallagher, A.; A Comparison of Measured and Computed Acoustic Emission Waves in a Thick Plate, Soc. Eng. Sci., Ann. Tech. Mtg., Oct. 29, 1995, Vol. 3-4, pp. 157-170, 1994.

Hurley, D.; Fitting, D.; and Chiao, R.; Angularly Dependent Ultrasonic Velocity and Attenuation Measurements in an Anisotropic Material, 1994, Proc. Rev. Prog. Quantitative NDE, publication pending.

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Jonsson, P.; Szekely, J.; Choo, R.; Quinn, T., A Survey of Mathematical Models of Arc Welding, Processes Modelling and Simulation in Materials Science and Engineering, submitted.

Kasen, B.; Santoyo, R.; Interfacial Residual Thermal Strain, Cryogenics, publication pending.

Keller, R.; Phelps, J.; SEM Observations of Misfit Dislocations in Epitaxial Films, J. Mater. Res., pending publication.

Keller, R.; Phelps, J.; Read, D.; Tensile and Fracture Behavior of Free-Standing Copper Films, Mater. Sci. Eng. A, submitted for review.

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Kollar, L.; Springer, G.; Spingarn, J.; McColskey, D., Compression Strength of Axially Loaded Composite Cylinders, J. Reinforced Plastics and Composites, submitted.

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Ledbetter, H.; and Kim, S.; Metal-Oxide Debye Temperatures and Elastic Constants: Estimation from Interionic Spacing, J. Phys. Chem. Solids, forthcoming.

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TECHNICAL AND PROFESSIONAL COMMITTEE LEADERSHIP

American Physical Society

Instrumentation and Measurement Science Topical Group
F. R. Fickett, Executive Committee

American Society of Mechanical Engineers

Journal of Electronic Packaging
D. T. Read, Associate Technical Editor

1995 International Conference and Exhibition
Application of Fracture Mechanics to Electronic Packaging and Materials
D. T. Read, Session Organizer

American Society for Testing and Materials

A6: Magnetics
F.R. Fickett

B1.08: Superconductivity
F. R. Fickett

C16.30: Thermal Measurements of Thermal Insulation Materials
D.R. Smith

E28.07: Impact Testing
C.N. McCowan, Chairman

E28.10.02: Temperature Effects
R.L. Tobler, Task Group Leader

E42.96: Surface Analysis
J. Phelps, Chairman SC3 on Analytical Microscopy

G2: Friction and Wear
A. Slifka

American Welding Society

Welding Journal
R.B. Madigan, Reviewer
T.A. Siewert, Reviewer

Computerization of Welding Information Committee
T.A. Siewert, Vice Chairman

International Cryogenic Materials Conference

Board of Directors
F. R. Fickett

Advances in Cryogenic Engineering
F. R. Fickett, Editor

Interconnection Technology Research Institute

Project 126 on Advanced Registration
D. T. Read, Task Group Leader

International Institute of Welding

Commission V on Nondestructive Evaluation
T.A. Siewert, Chairman

Study Group 212 on Arc Physics
T.A. Siewert, U.S. Expert

Executive Committee of the American Council
T.A. Siewert

Metallurgical Society

Metallurgical Transactions, Board of Review
H. Ledbetter

Welding Research Council

Materials and Welding Procedures Subcommittee
T.A. Siewert

University Research Committee
T.A. Siewert

INDUSTRIAL AND ACADEMIC INTERACTIONS

Ablestik

E. R. Drexler of the Materials Evaluation Group is collaborating with G. Nguyen on failure-mode measurement in isotropic conductive adhesives using the electron-beam moiré technique.

AI Technology

E. R. Drexler of the Materials Evaluation Group is collaborating with T. Devereaux on reliability studies of isotropic conductive adhesives using the electron-beam moiré technique.

A.O. Smith

The Structural Materials Group has a CRADA with A.O. Smith to apply the NIST Arc Sensing Module to production welding operations for automotive components.

Allied Signal

The Process Sensing and Modeling Group is developing process sensors to monitor the rapid-solidification processing of metallic glass strips in support of an ATP project.

Aluminum Association

The Process Sensing and Modeling Group is developing an on-line version of an eddy-current temperature sensor for installation on a rolling mill at Alumax, Inc.

American Superconductor

A. Slifka of the Materials Evaluation Group measured thermal conductivity and thermal expansion of superconducting coil segments in support of an ATP program to develop high-Tc magnets for superconducting electrical machinery.

Armstrong World Industries

The Materials Evaluation Group is developing transfer standards for thermal conductivity measurements to assist Armstrong in the evaluation of new insulating materials in support of an ATP project.

Association of American Railroads (AAR)

A.V. Clark and R.E. Schramm are collaborating with AAR researchers on measurement of residual stress in railroad wheels, and on rollby inspection of wheels for defects. A.V. Clark also serves as U.S. co-investigator on wheel safety research in a four-way collaboration involving NIST, AAR, the Polish Academy of Science, and the Central Research Laboratory of the Polish National Railways.

Comalco (Melbourne)

H. Ledbetter collaborates with M. Couper on the elastic constants of Al_2O_3-mullite/Al.

Cybo Robots

The Structural Materials Group is transferring the NIST arc sensing module to the shipbuilding industry as part of a TRP program lead by Cybo Robots.

Digital Equipment Corp.

E. R. Drexler and D. T. Read of the Materials Evaluation Group are collaborating with J. Sauber and L. Lee in a project using the electron-beam moiré technique to measure strains fields in plastic ball grid arrays.

Ford Microelectronics

D. T. Read of the Materials Evaluation Group is collaborating with S. Cunningham on measurements of mechanical properties of materials for microelectromechanical system (MEMS) construction.

Fujitsu (San Jose)

D. T. Read of the Materials Evaluation Group is working with V. Holalkere on determination of mechanical properties of thin films of electroplated copper.

Gas Research Institute

The Materials Characterization Group, working with Southwest Research Institute, is transferring gas-coupled ultrasonics technology to the pipeline industry as part of a GRI-sponsored program.

General Electric

The Materials Characterization Group is developing ultrasonic methods to measure the elastic properties of injection-molded plastics in support of an ATP project being conducted by General Electric and General Motors.

E. R. Drexler and D. T. Read of the Materials Evaluation Group are collaborating with E. Bernard in a project using the electron-beam moiré technique to study strains in high-density interconnects for multi-chip modules.

General Motors

The Structural Materials Group has CRADA's with the Delphi Division of General Motors to adapt our welding-arc sensor technology to their production lines.

Griffin Wheel

A.V. Clark and R.E. Schramm are working with the Griffin Wheel Co. to compare ultrasonic and destructive measurements of residual stress in railroad wheels.

Hitachi (Japan)

H. Ledbetter collaborates with H. Tsuiki on elastic constants of Bi-O monocrystal superconductors.

H. Ledbetter collaborates with S.P. Matsuda on elastic constants of Tl-O superconductors.

Howmet

The Structural Materials Group is working with Howmet to develop an x-ray diffraction sensor to monitor the liquid-solid interface in directional-solidification casting.

Johnson Controls

The Structural Materials Group, working with Native American Technologies, is developing neural network controls to apply the NIST arc sensing methods to production welding at Johnson Controls.

3M Corp.

E. R. Drexler of the Materials Evaluation Group is using the electron-beam moiré technique to measure thermomechanical behavior of anisotropic conductive adhesives in chip-on-glass structures provided by P. Hogerton.

Miller Electric

The Structural Materials Group has a CRADA with Miller to use arc diagnostics to study the control of weld spatter.

Morrow Tech

The Structural Materials Group is transferring the arc sensing technology to Morrow Tech for possible application to percussive welding.

Morton Automotive Safety Products

A.V. Clark is collaborating with B. Barnes and R. Beaumont on issues relating to noncontact inspection of airbag inflators.

Motorola

The Materials Evaluation Group is using electron-beam moiré to measure strain fields in electronic interconnects for Motorola.

NTT (Japan)

H. Ledbetter collaborates with Y. Hidaka on oxide-superconductor elastic constants, monocrystal and polycrystal.

H. Ledbetter collaborates with K. Kinoshita on La-O-superconductor elastic constants.

Olin

The Process Sensing and Modeling Group is developing on-line ultrasonic methods to measure grain size and texture of copper and brass strip products

Pratt and Whitney

A. Slifka and F. Fickett of the Materials Evaluation Group are developing a project to measure thermal conductivity of very thin graded ceramic coatings under a newly implemented CRADA.

Precision Acoustic Devices (PAD), Inc.

C.M. Fortunko is collaborating with PAD in the development of an air-coupled ultrasonic instrument for the nondestructive evaluation of composite materials.

Quatrosonics (Albuquerque)

H. Ledbetter collaborates with M. Lei on various elastic-constants-of-solids problems.

RITEC, Inc

C.M. Fortunko is collaborating with RITEC on the development of instruments for the ultrasonic characterization of materials.

Sonoscan

The Materials Characterization Group is working with Sonoscan to develop gas-coupled methods for acoustic microscopy of electronic packaging.

United States Steel

The Process Sensing and Modeling Group is working with U.S. Steel on the AISI-sponsored project "Microstructural Engineering in Hot Strip Mills."

XXsys Technologies, Inc.

C.M. Fortunko is collaborating with XXsys on the development of noncontact methods for the ultrasonic characterization of composite materials.

Academic and Institute Interactions

Argonne National Laboratory

H. Ledbetter collaborates with Z. Li (Materials Science) on physical properties of oxide crystals.

Brigham Young University

H. Ledbetter collaborates with B. Adams (Mechanical Engineering) on elastic-constants/texture relationships.

Colorado School of Mines

T.A. Siewert is an Adjunct Professor of Metallurgy

V.K. Tewary collaborates with J.R. Berger on the development of boundary element methods for stress analysis.

Colorado School of Mines (cont.)

T.A. Siewert and T.P. Quinn are working with S. Liu in the study of metal transfer in arc welding.

Y.W. Cheng works with C. Van Tyne and G. Krauss on the thermomechanical processing of steel.

E. R. Drexler of the Materials Evaluation Group is collaborating with J. Berger in a study to apply improved electron-beam moiré techniques and advanced modeling to determination of deformation modes of conductive adhesives.

Colorado State University

H. Ledbetter collaborates with P. Heyliger (Civil Engineering) in studying the natural resonances of regular-shape solids.

H. Ledbetter collaborates with R. Leisure (Physics) on ultrasound resonance spectroscopy.

Cornell University

R. Keller of the Materials Evaluation Group is collaborating with J. Nucci of the National Nanofabrication Facility in a study of stress voiding in copper films used as conductors in microelectronic structures.

Davidson College

H. Ledbetter collaborates with L. Cain (Physics) on elastic constants of austenitic-steel.

German Aerospace Research (DRL)

A. Slifka of the Materials Evaluation Group is collaborating with U. Leushake of German Aerospace Research in determining microscale thermal conductivity of developmental functionally guarded materials (FGMs).

Idaho National Engineering Laboratory

A. Slifka of the Materials Evaluation Group is collaborating with B. Rabin in determining microscale thermal conductivity of developmental FGMs.

Institute of Metal Research (Shenyang, China)

H. Ledbetter collaborates with Y. Li in studying elastic constants of austenitic steel.

Institute of Physics and Power Engineering (Obninsk, Russia)

H. Ledbetter collaborates with S. Danilkin on neutron scattering from Fe-Cr-Ni alloys to get information on metal-nitrogen interactions.

Iowa State University: Center for Nondestructive Evaluation

A.V. Clark collaborates with R.B. Thompson on texture and formability using ultrasonics.

H. Ledbetter collaborates with R. Thompson on elastic constants of sintered iron with voids.

C.M. Fortunko serves as a NIST representative to the Center for Nondestructive Evaluation.

H. Ledbetter collaborates with D. Chimenti on elastic constants of Al-epoxy composites.

Johns Hopkins University

C.M. Fortunko collaborates with R.E. Green on the nondestructive characterization of materials.

H. Ledbetter collaborates with R. E. Green (Materials Science) on elastic constants of graphite-epoxy laminates.

Konan University (Kobe, Japan)

H. Ledbetter collaborates with N. Nakanishi (Chemistry) on elastic constants of oxides.

Massachusetts Institute of Technology

R.L. Tobler collaborates with I.S. Hwang of the Materials Sciences Department in the evaluation of nickel-base superconductor sheath alloys for applications at 4 K.

T.P. Quinn works with J. Szekeley on the modelling of gas metal arc welding.

Max-Planck-Institut für Metallforschung (Stuttgart, Germany)

H. Ledbetter collaborates with M. Weller to study the internal friction and dielectric constants of various materials.

Max-Planck-Institut für Metalphysik (Stuttgart, Germany)

H. Ledbetter collaborates with U. Essman on elastic constants of Nb monocrystals.

Michigan Technological University

E. R. Drexler of the Materials Evaluation Group is collaborating with A. Chandra in a study on reliability of conductive adhesives.

Nagoya Institute of Technology (Japan)

H. Ledbetter collaborates with K. Kawashima on elastic properties of alumina-aluminum composites.

NASA Lewis Research Center

A. Slifka of the Materials Evaluation Group is collaborating with W. Brindley on a project to evaluate thermal performance of porosity-graded thermal-barrier coatings.

National Aerospace Laboratory (Japan)

A. Slifka and J. Phelps of the Materials Evaluation Group are evaluating microstructure and microchemical composition and measuring thermal conductivity of FGMs prepared by Nippon Steel.

National Center for Atmospheric Research

J. Phelps of the Materials Evaluation Group is collaborating with C. Twohy on electron microscopy problems related to atmospheric particulate measurements

National Institute of Fusion Sciences (Japan)

R. Tobler collaborates with T. Nakamura on cryogenic materials research.

National Oceanic and Atmospheric Administration

H. Ledbetter collaborates with Y. Xie on the numerical approximation and analysis of anisotropic elastic constants.

National Research Laboratory for Metrology (Japan)

H. Ledbetter collaborates with H. Kobayashi on measuring anelastic properties of various solids.

Northwestern University

H. Ledbetter collaborates with M. Zimmerman (Materials Science) and elastic constants of Fe-Ti oxides.

Oak Ridge National Laboratory

H. Ledbetter collaborates with L. Boatner on elastic constants Fe-Cr-Ni monocrystals.

H. Ledbetter collaborates with M. Paranthaman on various aspects of oxide superconductors.

Osaka University

A.V. Clark works with H. Fukuoka and M. Hirao on the development ultrasonic measurement techniques for determining residual stress and texture.

H. Ledbetter collaborates with Y. Tsunoda (Physics) to study Cu(Fe) elastic constants.

H. Ledbetter collaborates with M. Hirao on studying internal friction in various alloys.

Polish Academy of Sciences

R.E. Schramm and A.V. Clark are collaborating with the Institute for Fundamental and Technological Research on comparison of ultrasonic measurements of residual stress.

State University of New York at Stony Brook

A. Slifka and J. Filla of the Materials Evaluation Group are measuring bulk thermal conductivity of ceramic-coated steels produced at SUNY in a collaboration with C. Berndt and G. Bancke.

Stanford University

C.M. Fortunko is collaborating with Professor B. T. Kuri-Yakub on the development of air-coupled ultrasonic transducers

Swiss Federal Institute of Technology (Zürich)

H. Ledbetter collaborates with P. Uggowitzer on studying the elastic constants of f.c.c. Fe-Cr-Ni-Mn alloys.

Tennessee State University

A. Slifka collaborates with D.K. Chaudhuri, on the tribological behavior of ion-implanted and ion-deposited materials.

Texas A&M University

H. Ledbetter collaborates with V. Kinra (Aerospace) on studying elastic constants of laminates.

A.V. Clark collaborates with T.L. Anderson (Mechanical Engineering) on application of fracture mechanics to condition assessment of bridges.

Tohoku University

H. Ledbetter collaborates with Y. Shindo (Materials Processing) on waves in composites.

R.L. Tobler interacts with Dr. H. Takahashi and T. Shoji of the Research Institute for Fracture Technology to document fracture mechanics test procedures for structural alloys at 4K.

Tsinghua University (Beijing, China)

H. Ledbetter continues studies with Y. He (Physics) on oxide superconductor elastic constants.

University of Arkansas

H. Ledbetter collaborates with Z. Sheng (Physics) on constants of elastic oxide superconductors.

University of California

H. Ledbetter collaborates with R. Fisher in studying specific heats of oxide superconductor.

University of Colorado

J. Phelps of the Materials Evaluation Group is collaborating with P. Sheridan on electron microscopy problems related to atmospheric particulate measurements

D. T. Read of the Materials Evaluation Group is providing thin film measurement and instrumentation expertise to a collaboration with M. Dunn on mechanical properties of epitaxial silicon.

H. Ledbetter is an Adjunct Professor in the Department of Mechanical Engineering and member of the graduate faculty.

H. Ledbetter collaborates with S. Datta (Mechanical Engineering) on problems of waves in composites.

H. Ledbetter collaborates with M. Dunn (Mechanical Engineering) on micromechanics of composites.

H. Ledbetter collaborates with Professor A. Hermann (Physics) on oxide-superconductor elastic constants.

D.T. Read interacts with R. May (Electrical Engineering) on use of CU's electronic microfabrication facilities.

C.M. McCowan interacts with J. Drexler (Geology) on the use of the University's microanalytical facilities.

T.A. Siewert collaborates with Y.C. Lee (Electrical Engineering) on integrity issues for electronic interconnects.

University of Geneva

H. Ledbetter continues studies with B. Seeber on the elastic constants of Chevrel-phase superconductors.

University of Idaho

C.M. Fortunko collaborates with M. Anderson (Mechanical Engineering) on the development of air-coupled ultrasonic methods for materials characterization.

University of Illinois (Urbana)

H. Ledbetter collaborates with W. Kriven (Materials Science) on elastic constants of ceramics.

University of Karlsruhe

H. Ledbetter collaborates with B. Eigenmann (Werkstoffkunde I) on elastic properties of ceramic composites.

University of Maryland

Professor J.W. Dally works with D.T. Read on high-resolution experimental mechanics and testing of thin film.

University of Michigan

D.W. Fitting collaborates with researchers at the University of Michigan on the development of silicon-based acoustical arrays.

D. T. Read of the Materials Evaluation Group is working with C. Kalmas on a project to determine mechanical properties and hardness of thin films of aluminum.

University of South Carolina

H. Ledbetter collaborates with R. Edge (Physics) on elastic and magnetic properties of Fe-Cr-Ni alloys.

H. Ledbetter collaborates with T. Datta (Physics) on magnetic properties: steels and superconductors.

University of Stuttgart (Germany)

H. Ledbetter collaborates with B. Gairola (Institute for Theoretical and Applied Physics) on elastic constants of graphite-fiber.

H. Ledbetter collaborates with E. Kröner (Institute for Theoretical and Applied Physics) on elastic constants of monocrystals and polycrystals.

H. Ledbetter collaborates with H. -E. Schaefer on elastic constants of nanocrystals.

University of Tsukuba (Japan)

H. Ledbetter collaborates with T. Suzuki (Applied Physics) on elastic constants and phase transitions.

H. Ledbetter collaborates with K. Otsuka (Materials Science) on the elastic constants of the monocrystal shape-memory alloy Cu-Al-Ni.

H. Ledbetter collaborates with M. Saito (Engineering Mechanics) on elastic constants of technical solids.

H. Ledbetter collaborates with H. Uwe (Materials Science) on elastic properties of barium-bismuthate superconductors.

University of Washington

H. Ledbetter collaborates with M. Taya (Mechanical Engineering) on elastic constants of composite-materials.

University of Zagreb

H. Ledbetter collaborates with D. Balzar (Physics) on defect properties of oxide superconductors.

H. Ledbetter collaborates with M. Stubicar (Physics) on elastic properties of composites.

U.S. Air Force Rome Laboratories

E. R. Drexler and D. T. Read of the Materials Evaluation Group are collaborating with M. Stoklosa on a study of strains in high density interconnect structures.

U. S. Geological Survey

J. Phelps of the Materials Evaluation Group is collaborating with J. Pallister and G. Meeker on electron microscopy problems related to volcanic tuft research.

VNIIMS, Gosstandart of Russia (Moscow)

H. Ledbetter collaborates with B. Kodess on elastic properties of various materials.

West Virginia University

A.V. Clark collaborates with P. Fuchs on methods of determining stress in bridges.

APPENDIX:

ORGANIZATIONAL CHARTS

MATERIALS SCIENCE AND ENGINEERING LABORATORY
Division 853

MATERIALS RELIABILITY DIVISION
H.I. McHenry, Chief
T.A. Siewert, Deputy Chief

J.E. Smith
Administrative Officer

A.M. Reidy
Division Secretary

.05 Materials Characterization
C.M. Fortunko

S.J. Barnhill

- E.S. Boltz
- M.A. Hamstad
- K.W. Hollman
- D.C. Hurley
- S.A. Kim
- H.M. Ledbetter
- J.D. McColskey
- M.C. Renken
- R.L. Santoyo
- D.R. Smith
- V.K. Tewary
- G. Mustoe(IPA)
- D. Balzar(GR)

.06 Process Sensing & Modeling
G.A. Alers

.07 Structural Materials
T.A. Siewert

V.E. Ciaranello

.06:

- Y-W. Cheng
- A.V. Clark
- B. Igarashi
- W.L. Johnson
- P.T. Purtscher
- S.R. Schaps
- R.E. Schramm

.07:

- W.P. Dube
- D.W. Fitting
- B.J. Filla
- R.B. Madigan
- C.N. McCowan
- T.P. Quinn
- D.P. Vigliotti
- S. Yukawa (INT)
- R. Castillo(Detail)

.08 Materials Evaluation
F.R. Fickett

S.J. Barnhill

- E.S. Drexler
- R.R. Keller
- J.M. Phelps
- D.T. Read
- A.J. Slifka
- R.L. Tobler

September 30, 1995

MATERIALS SCIENCE AND ENGINEERING LABORATORY

L.H. Schwartz, Director
H.L. Rook, Deputy Director

Intelligent Processing of Materials

D. Hall, Chief

NIST Fellows

J.W. Cahn
S.M. Wiederhorn
B.R. Lawn
J.J. Rush

Metallurgy

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

Polymers

L.E. Smith, Chief
B.M. Fanconi, Deputy

Ceramics

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

Materials Reliability

H.I. McHenry, Chief
T.A. Siewert, Deputy

Reactor Radiation

J.M. Rowe, Chief
T.M. Raby, Deputy

National Institute of Standards and Technology

Organizational Chart