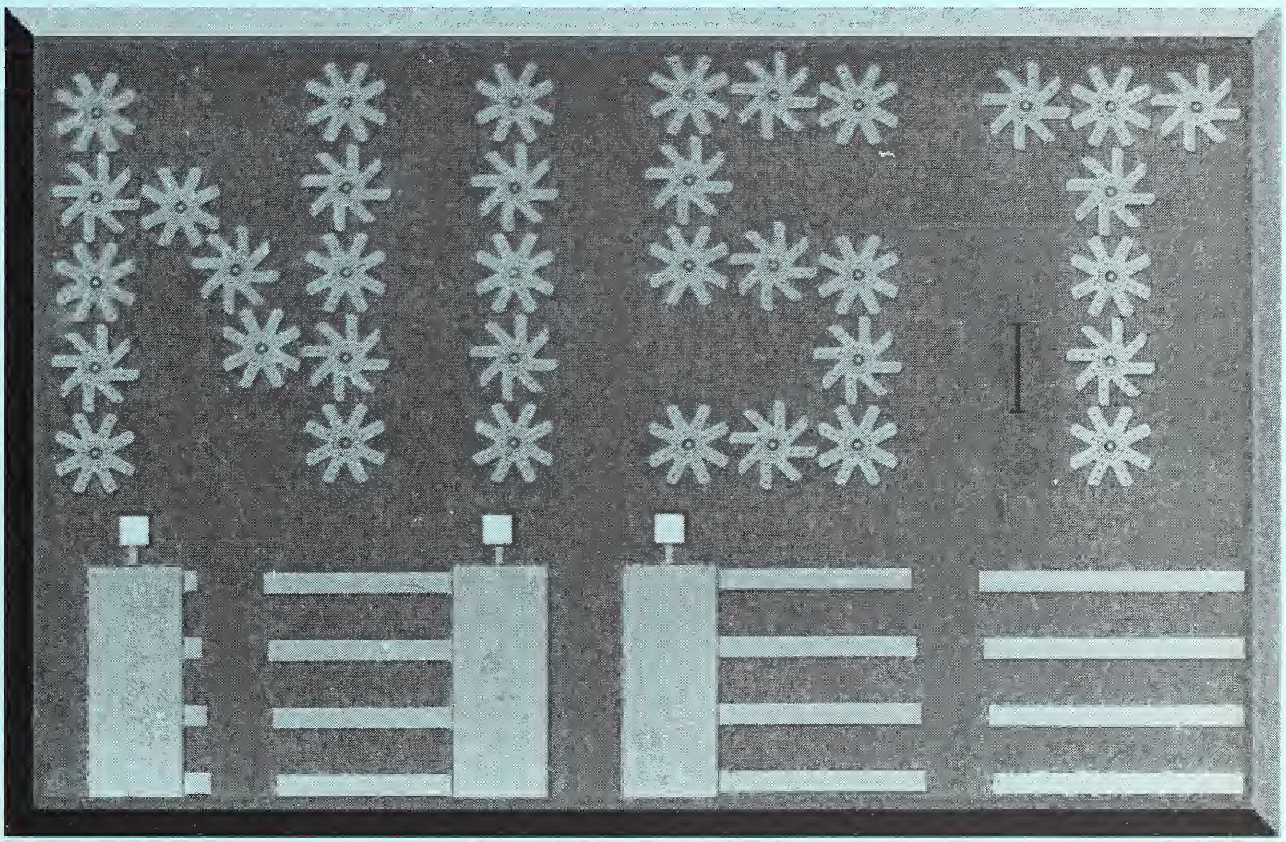
MSEL
Materials Science and Engineering Laboratory

MATERIALS RELIABILITY



NISTIR 5963
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities 1996

Materials Reliability

The gears, arranged to spell NIST, are representative of those used in MicroElectroMechanical Systems (MEMS). Chemical vapor deposition (CVD) is used to fabricate the gears from polycrystalline silicon. In an exploratory study, two mechanical properties of CVD polysilicon were measured: the fracture strength was measured in a piezo-actuated microtensile test apparatus, and the Young's modulus was measured by a resonance technique.

MSEL

Materials Science and Engineering Laboratory

MATERIALS RELIABILITY

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

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Technology Administration
National Institute of Standards and Technology

Technical Activities 1996

U.S. DEPARTMENT OF COMMERCE
Michael Kantor, Secretary
TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Arati Prabhakar, Director

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Tradenames and/or names of manufacturers are included to properly describe NIST activities. Such inclusion neither constitutes nor implies endorsement by NIST or the U.S. Government.

Executive Summary

The Materials Reliability Division develops measurement technologies which enable the producers and users of materials to improve the quality and reliability of their products. Measurement technologies are developed for process control to improve the quality and consistency of materials, for nondestructive evaluation to assure quality of finished materials and products, and for materials evaluation to assure reliable performance. Within these broad areas of measurement technology, the Division has focused its resources on three research themes.

- 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: To develop ultrasonic 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, thermal and magnetic behavior of thin films and coatings at the appropriate size scale.

This report summarizes of the technical activities of the Materials Reliability Division during fiscal year 1996. The technical activities are organized according to programs which have been defined and organized by MSEL. The Division has 21 research projects, which contribute to six MSEL Programs. Two of the MSEL Programs coincide with Division focus areas: Intelligent Processing of Materials, and Ultrasonic Characterization of Materials. Our projects on Micrometer Scale Measurements for Materials Evaluation are parts of three MSEL Programs where our measurement technologies for characterizing films and coatings can make unique and important contributions. We also have one project that is not covered by our focus areas: Charpy Impact Testing, NIST's largest Standard Reference Materials (SRM) project.

On the administrative side, FY96 was a year of continuing resolutions, government shutdowns and tight budgets. Yet there were some positive developments in staffing and facilities.

Staffing: We were not able to add new staff, but we were able to convert Bob Keller, who completed his postdoctoral appointment in 1994, from a term to a career appointment. Bob initiated a new project on stress voiding and electromigration, one of the main reliability problems for the next generation of microelectronics. We were fortunate to receive two Postdoctoral Research Associates from the 1995 NRC competition: Daryl Clerc and Christine Kalnas. Daryl will work with Hassel Ledbetter in the area of computational materials science.

Christine will work with Dave Read on mechanical properties of thin films. We now have four postdoctoral appointments, an all-time high for the Division. Eric Boltz, a temporary employee, accepted a position in industry upon receipt of his Ph.D. in Materials Science from Johns Hopkins University. Working under Chris Fortunko, Eric used laser interferometry to verify the analytical model of Vinod Tewary for acoustic wave propagation in composite materials.

Facilities: This year was the second of a three-year effort to make our facilities modern, safe and suited to the Division's research agenda. We completed transfer of five laboratories and seven offices to a new addition. Work was started on a central HVAC system for our building, a multi-million dollar project scheduled for completion in April 1997. When the construction is finished in 1997, we will have refurbished every laboratory within the past seven years, consolidated our research activities in the west end of our building, and improved the working environment.

The rest of this report consists of project summaries, each of which includes accomplishments. Selected accomplishments are listed below to highlight the technical activities of the Materials Reliability Division.

<u>Microstructural Evolution in Aluminum:</u> A series of high-temperature ultrasonic resonance measurements performed on pure aluminum revealed the dependence of recovery and recrystallization on the extent of coldwork. The high sensitivity of these measurements offers promise for the development of process-control sensors for forming and annealing operations.

<u>Constitutive Equations for Steels at High Temperatures:</u> Constitutive equations were developed for C-Mn and microalloyed steels as functions of temperature, strain rate and austenitic grain size over ranges typical for hot rolling and forging. The associated computer program has been incorporated into a process model which simulates steel rolling in hot-strip mills, and the program was delivered to the steel industry by the American Iron and Steel Institute, the research sponsor.

<u>Directional Solidification Sensor:</u> A patent was issued for the transmission X-ray diffraction method for monitoring the position of the liquid-solid interface during directional solidification. We currently use the method for detecting the liquid-solid interface during the casting of single-crystal superalloy turbine blades.

<u>Weld Arc Sensing:</u> Sensor algorithms were developed for the flux core arc welding (FCAW) process and demonstrated for welding conditions found in shipbuilding. The arc sensing module for FCAW was delivered in the form of a 19,000 line computer code to Cybo Robots and installed in their robotic welding system. The sensor/robot system was successfully demonstrated to the shipbuilding industry.

<u>Magnetostrictive Acoustic Transducers:</u> Noncontacting ultrasonic transducers that operate though magnetostrictive coupling were used to measure magnetostriction in steel and a metallic

glass. The results indicate that the magnetic field dependence of the magnetostriction coefficients correlate with yield strength in steel and with electromagnetic loss factor in the metallic glass.

Nonlinear Ultrasonics: A prototype instrument for nonlinear measurements was combined with a laser interferometer to create a nonlinear ultrasonic system with micrometer-scale resolution. The high spatial resolution is expected to enable characterization of engineered surfaces. The system was validated by measuring the nonlinearity parameter, β, for fused silica. The results were in good agreement with β-values obtained by two other detection methods.

Gas Coupled Ultrasonics: A patent was issued for a gas-coupled ultrasonic method for measuring wall thickness and detecting material flaws in natural gas pipelines. Efforts to commercialize the concept were initiated with Southwest Research Institute under a contract from the Gas Research Institute.

Stress Voiding and Electromigration: Backscatter Kikuchi diffraction was used to characterize microtextures and grain boundary structures in narrow copper interconnects subjected to stress voiding. Regions of locally weaker texture and higher-angle grain boundaries were found to be more susceptible to void formation and growth. This is the first known local characterization of microstructures associated with stress voiding in copper interconnects.

Micrometer-scale Strain Measurements: The electron-beam moire technique was extended to biaxial strain measurements. Also, significant papers were published on the measurement theory, the analysis of errors, and an inversion model for moire patterns.

Magnetic Sensing of Microstructure: The concept of "reading" microstructure was demonstrated using giant magnetoresistance (GMR) sensors from the magnetic recording industry. Computer interfacing work was initiated to convert a nanomover into a scanning system suitable for imaging.

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, consistent properties, and enhanced functionality. The IPM Program makes important contributions to three MSEL strategic thrusts: advanced processes, advanced materials, and measurement technology.

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

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

The IPM Program also contributes to MSEL's measurement technology goal. A major focus of the IPM projects is process sensors, which our industrial collaborators repeatedly identify as a crucial need. Sensor technology is a core competence of MSEL which has its roots in sensor development for nondestructive evaluation of materials. Unique MSEL capabilities are being used to measure thermophysical properties at elevated temperatures; these data are combined with model enhancements and then incorporated in industrial software for metal casting. In addition, specialized measurement capabilities, such as nuclear magnetic resonance and small-angle neutron scattering, are used to understand microstructure/morphology evolution in ceramics and polymers.

Project: WELD ARC SENSING

MSEL Program: Intelligent Processing of Materials

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

Technical Objectives

- Develop a better understanding of the underlying physics governing arc-welding processes through advanced instrumentation and data analysis techniques.
- Develop simple, nonintrusive, and robust sensors that provide meaningful information about the status of the welding process for real-time monitoring and control.
- Investigate applications of arc sensing technology to different welding processes and materials.

Technical Description

The largest obstacle to arc welding process control is the lack of feedback sensors. The arc welding processes are difficult to sense because of the ultra violet 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 torch. Therefore, we have chosen to develop sensors that either use no devices attached to the torch or are very small. Many of our sensing schemes rely on using model-based algorithms that use the inherent process variables: the current and the voltage; it is sometimes called "through the arc sensing". The principal advantage of this sensing scheme is that the external sensors can be placed at the power supply, well away from the robot and torch.

Over the last several years we have been concentrating on the development of sensors that are suitable 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 through feedback from our CRADA partners. Several of them are 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. A more sophisticated approach involves 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.

Our Welding Laboratory includes the following special equipment:

- three 486-class and two Pentium-class personal computers outfitted with high-resolution, high-speed data collection boards
- extensive analysis software and software development tools.
- analog signal conditioning and isolation manifolds
- current, voltage, wire feed speed, and light intensity transducers
- oscilloscope, function generator, and electronic signal spectrum analyzer
- optical spectrum analyzer (200 nm to 1000 nm range, 0.01 nm resolution)
- high-speed video system with laser back-lighting
- standard video cameras and VCRs
- video frame-grabber and image processing system
- 6-axis robotic manipulator, three track-type manipulators
- conventional SCR, inverter, and transistorized welding power sources
- 4-roll and capstan wire feed units

External Collaborations

Academic. Ohio State University. Dick Richardson, Professor of Welding Engineering, worked at NIST during the summer, 1996, as a guest research under the Catepillar JV ATP project. He compared arc sensing techniques for the fabrication of heavy equipment structures.

Colorado School of Mines. Steve Liu, Professor of Metallurgy, has been awarded a grant from the Colorado Advanced Materials Institute to apply NIST arc sensing technology to aluminum welding.

Industrial. Johnson Controls, Inc. CRADA. Joint program also including Native American Technologies, Inc. (NAT) and Advanced Manufacturing Engineering Technologies, Inc. (AMET), to increase weld quality for Johnson Controls' automotive seat manufacturing facility. A NAT engineer worked at the NIST Boulder site to port NIST arc sensing technology to an AMET weld process controller.

General Motor's Delphi Chassis Systems has installed NIST's Arc Sensor Module and is applying it to vehicle running gear manufacturing.

A.O. Smith, Inc. CRADA. A.O. Smith has installed NIST's Arc Sensor Module and is applying it to vehicle frame manufacturing. A.O. Smith has continued to provide feedback to us to refine the ASM program.

Internal-NIST. Manufacturing Engineering Laboratory, Intelligent Systems Division. We have a cooperative program with ISD addressing the integration of welding with advanced manipulator designs.

Accomplishments (FY96)

CYBO Robots integrated the NIST arc sensing module into their shipyard robotic welding system and demonstrated the system at the final meeting (July 1996) of the Technology Reinvestment project entitled "Portable Shipyard Robotics Technology".

General Motors, Delphi Chassis Division CRADA. 1995 - Present. Taught visiting Delphi engineer at NIST Boulder site for 4 months on the application of arc sensing techniques for the manufacture of automotive suspension components. Follow-up visits to the Delphi site are being conducted to assist in system installation and operation.

Began arc sensing in aluminum welding and planned an aluminum GMAW program with researchers at Colorado School of Mines.

Began investigation of resistance weld sensing in steel and aluminum sheet by attending technical meetings of the Auto Body Consortium's ATP entitled, "Intelligent Resistance Welding".

The American Welding Society honored this project with two poster presentation awards at their 1996 Annual Meeting: 2nd Place Award to "Ionic Composition of Gas Tungsten and Gas Metal Arcs" and 3rd Place Award to "Spatter Measurements".

Publications

1. "Characterization of Welding Arc Spectral Light Emission," R.W. Richardson, IIW Document XII-1469-96, available from the American Welding Society, Miami, 1996.

2. United States Patent 5514851, Prevention of Contact Tube Melting in Arc Welding, May, 1996.

Project: WELD PROCESS MODELING AND CONTROL

MSEL Program: Intelligent Processing of Materials

Principal Investigator: T.P. Quinn
T.A. Siewert, R.B. Madigan, B.J. Filla

Technical Objectives

- Use empirical models generated from data taken under production-line conditions to develop sensing algorithms for gas metal arc welding (GMAW) and flux-cored arc welding (FCAW).

- Develop and test arc sensing and control algorithms and transfer them to American industry.

Technical Description

Physical and empirical models and control schemes of 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 up 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 lead to better understanding by welding engineers, improved welding sensors, and can lead to feedback control schemes.

The largest obstacle to arc-welding process control is the lack of feedback sensors. Therefore, this program is tightly coupled with our weld sensor program, and helps to establish the design parameters for that program. Many of our sensing schemes rely on using model-based algorithms that use the inherent process variables: the current and the voltage; it is sometimes called "through the arc sensing." The principal advantage of this sensing scheme is that the external sensors can be placed at the power supply, well away from the robot and torch.

External Collaborations

This project participated in the CRADAs described under the Weld Arc Sensing Project.

Accomplishments (FY96)

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 part 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.

The sensor algorithms were implemented using object-oriented programming on a PC style computer. A graphical user interface (GUI) was constructed in order that the sensing system could be tested on a factory floor in a high production environment. Graphs of the outputs from the algorithms are displayed at the end of each weld. The system was also made network capable using Internet protocols so that it could be operated from a host computer. Care was taken to allow the system to process welds of any length (provided the PC has enough memory). To enable configuration of the sensor so that it could be tuned for a particular welding environment, all configuration parameters were made available through the GUI as well as the network. The data is processed through the algorithms in real time, but raw data files can be recorded after welds are made for later analysis. The program can automatically construct the model weld by averaging and smoothing records of already processed welds. The sensor is in final trials on the factory floor of a high-production automotive parts factory.

Algorithms were also developed for FCAW. Modifications to the algorithms developed for GMAW were made based on the results of laboratory tests with FCAW. The welds were made under conditions that would be found in ship-building. The modified algorithms were successfully demonstrated at a CRADA partner's site. The network enabled sensor system was delivered to a CRADA partner to embed in a ship-building robot.

A 19,000 line sensing and modeling code was delivered to CRADA partners in the automotive and ship-building industries.

Publications

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

Project: MICROSTRUCTURE SENSING FOR PROCESS MONITORING

MSEL Program: Intelligent Processing of Materials

Project Leader: G.A. Alers
S.R. Schaps, B. Igarashi

Technical Objectives

The objective of this program is to investigate measurement techniques that can be applied in a production environment and give information on the microstructure of the material being produced. The secondary objective is to develop models that relate the microstructure to properties of commercial interest so that in the long run, process control measurements can be related directly to the properties demanded by the ultimate consumer of the material.

Technical Description

Most processing methods for commercial materials are designed to develop specific microstructures that are known to give the final product its desired commercial properties. Therefore, the development of process control mechanisms and nondestructive evaluation procedures must focus on developing techniques and sensors that provide information on microstructures. To support this activity, models that relate desirable commercial properties to the microstructure and models for the relationship between microstructure and measurable physical properties must be developed. During the past few years, NIST has established unique capabilities in noncontacting magnetic, eddy-current and ultrasonic transducers that can operate on fast- moving, hot sheet metal in a commercial rolling mill. In order to exploit this knowledge, the emphasis in FY96 was placed on: (1) reducing the technique for making precision Lamb-wave velocity measurements on thin sheet metal products to a routine process; (2) analyzing Lamb-wave velocity data to characterize the state of preferred orientation in the sheet metal so that texture formation can be monitored throughout the rolling and annealing process, (3) developing magnetoelastic measurement techniques for application to magnetic sheet metal products and (4) relating magnetic and acoustic property measurements to the microstructures that are known to influence commercial properties.

External Collaborations

Industrial. Olin Research is using ultrasonic methods developed by NIST to measure texture formation in copper and brass.

Allied Signal is providing samples and process control information aimed at developing on-line, ultrasonic instruments for monitoring their METGLAS spin casting operation.

Accomplishments (FY96)

Microstructure Development in Copper and Brass Sheet. NIST has a CRADA with the Olin Corporation to develop techniques for the on-line monitoring of texture and grain size in copper, brass and bronze sheet products during rolling and annealing. Early in FY96, Olin Research supplied NIST with a large set of thin sheet metal samples that had been rolled between 10 and 60% reduction in thickness and had been annealed for 1 hour at temperatures between 100 and 600°C. They represented high conductivity copper, a 32% Zn brass and a 4% Sn bronze. Since the rolling reductions were not large, it was of interest to measure the degree of preferred orientation that was developed and how it responded to low-temperature anneals. Such an extensive study would take a very long time if conventional x-ray diffraction techniques were used to measure the degree of preferred orientation and to establish the crystallographic description at each step in the rolling and annealing process. By measuring the velocity of sound waves propagating in the plane of the sheet and applying recently developed theories for Lamb wave propagation in thin plates with texture, NIST was able to deduce the first three coefficients in the Legendre polynomial expansion equation used to describe anisotropic physical properties in polycrystalline materials. These coefficients, called Orientation Distribution Coefficients (ODCs), can be deduced from a measurement of the anisotropy of the Lamb-wave mode velocity in sheet metal. Furthermore, formability studies at NIST several years ago demonstrated how this particular Lamb wave velocity could be measured under rolling mill conditions by especially designed electromagnetic transducers (EMATs).

During FY96, special EMATs were constructed and commercial instruments purchased by Olin were assembled into a unit suitable for making routine measurements of the Lamb-wave mode velocity in the sheet samples supplied by Olin. The computer programs needed to convert the velocity values into the three ODCs were also documented and tested on data collected from the Olin samples. In May of FY96, the apparatus and software were sent to Olin Research in New Haven, Connecticut where a summer student repeated the NIST measurements and extended the study to different alloys, rolling histories and annealing schedules. The results are extensive graphical displays that expose how different textures emerge from the different rolling and annealing procedures. Thus, it is now easy to define the most efficient path to achieve a texture with particular desireable properties.

Measurement of Magnetostriction. In ferromagnetic materials and in particular steel, the application of a magnetic field will cause a change of dimensions. This phenomenon, called magnetostriction, can be used to excite ultrasonic waves by producing the magnetic field with a coil of wire carrying an RF current at the frequency of the desired wave. Detection of the wave is accomplished by allowing the coil to pick up the magnetic fields produced when the mechanical strains in the wave cause changes in magnetization through inverse magnetostriction. The amplitude of the ultrasonic wave produced under a transmitter coil or the signal detected by a receiver coil depends on the magnitude of magnetostrictive coefficients that are characteristic of the particular ferromagnetic material involved and on the direction and magnitude of a static, biasing magnetic field applied to the region around the coil.

Starting late in FY95 and continuing through FY96, NIST has made a concerted effort to verify experimentally certain aspects of the theoretical model, to investigate the ultrasonic waves produced by unusual configurations of the coils used, and to establish relationships between the magnetostrictive coefficients observed and the microstructural features of the ferromagnetic material involved. The last effort is motivated by the desire to use measurements of the magnetostrictive coefficients as nondestructive materials characterization tools.

During FY96, instrumentation was assembled to allow the efficiency of Magnetostrictive Ultrasonic Transducers (MUTs) to be measured as a function of the applied biasing magnetic field. Meander coils were used to define the wave length of the ultrasonic wave and the frequency of operation was chosen to focus attention on only one type of wave mode. By changing the direction of the applied magnetic bias field relative to the long dimension of the meander coil, either extensional or shear waves could be excited and detected preferentially. Since several other Division programs are directed at sheet metal products and the rolling process, a sample geometry in the shape of a thin rectangular plate was chosen. By cutting the samples with the long axis of the rectangle at various angles to the rolling direction of the sheet, the effect of texture on the magnetostrictive coefficients could be studied. A novel arrangement of the transmitter and receiver meander coils allowed the rectangular plate to be driven in a resonant vibrational mode. This made it possible to measure the intrinsic damping capacity, to deduce very accurate values for the elastic constants, and to measure the transduction efficiency with modest drive powers and simple preamplifiers.

To date, the important results of this study have been to verify the essential features of analytical models for magnetostrictive transducers. This indicates that noncontact measurements of ultrasonic transducer efficiency can be used as a replacement for the difficult strain gage methods currently being used to measure the magnetostriction coefficients of ferromagnetic materials. It has also been possible to demonstrate that the magnetic field dependence of the magnetostriction coefficients can be correlated with certain strengthening mechanisms in HSLA steels and with the electromagnetic loss factor in some metallic glass products. These two observations indicate that a nondestructive ultrasonic measurement of magnetostriction could be used as an on-line predictor of useful commercial properties of thin sheet metal products during their production.

Project: THERMOMECHANICAL PROCESSING

MSEL Program: Intelligent Processing of Materials

Principal Investigator: Y-W. Cheng
P.T. Purtscher, R.L. Tobler

Technical Objective

The objectives of this program are to develop, improve and validate process models that are needed in the thermomechanical processing of steels. With these process models, steel products can be manufactured at less cost through shorter design cycles and with higher quality through better process control.

Technical Description

Our research interest centers on development of quantitative relationships between the mechanical properties of steel, its chemistry, and the thermomechanical processing parameters used during its rolling and forging. Emphasis is on the characterization of the kinetics of microstructural evolution during hot working, such as precipitation of carbides, nitrides and carbonitrides, dynamic and static recrystallization, grain growth, and austenite decomposition. In addition, constitutive equations and composition-structure-property relationships are being developed. Specifically, the structure-property relations at ambient temperature and the constitutive behavior under hot rolling conditions are being determined for plain-carbon and microalloyed steels. Mathematical equations are being developed to describe the structure-property relations and the constitutive behavior based on physical-metallurgy principles coupled with experimental observations. These equations are being coded into computer programs which can be used either as stand-alone tools for calculating the mechanical properties at ambient temperature and stress-strain curves under hot-rolling conditions or for incorporation into process models that simulate the steel rolling process in hot-strip mills and calculate the mechanical properties of the hot-rolled products.

External Collaborations

Academic. University of British Columbia, Vancouver B.C. The Center for Metallurgical Process Engineering and NIST are coinvestigators for an AISI project entitled, "Microstructureal Engineering for Hot-Strip Mills. Professor J.K. Brimacombe is the principal investigator for UBC.

Colorado School of Mines. Professor C.J. VanTyne and G.T. Verlarde, a graduate student, collaborated with Yi-Wen Cheng on a study of the hot deformation behavior of microalloyed steels. Mr. Velarde's MS studies were supported by NIST's PREP program.

Industrial. U.S. Steel is the steel industry sponsor for the AISI project conducted by UBC and NIST.

International. Ferrous Metallurgy Institute, Poland. Dr. Roman Kuziak is the principal investigator on a U.S./Poland cooperative project on thermomechanical processing of forging steels.

Accomplishments (FY96)

In 1996, we continue a research project for the American Iron and Steel Institute (AISI) on microstructural engineering in hot-strip mills. The objective of the 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 cooperative effort between four groups: NIST, the University of British Columbia (UBC-Canada), U.S. Steel, and Norwest Mettech Corporation (Canada). Major portions of the research are being done at NIST and UBC. UBC conducts research on heat transfer, quantitative characterization of 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 on structure-composition-property relations at 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 separation 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 methods, for analyzing and simulating hot rolling or forging. In 1996, we have developed models for predicting the flow behavior of a niobium-treated high-strength low-alloy (HSLA) steel, in addition to refining and improving the models for two plain-carbon steels, which were developed in 1995. The models calculate a full stress-strain curve for a wide range of temperatures (800-1300 °C), strain rates (1-400/s), and austenite grain sizes (20-500 μm). The models also calculate the important properties along the stress-strain curve, including 0.2% 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.

The mechanical properties of a steel at ambient temperature are closely related to composition and microstructure. In 1995, structure-property relations for plain-carbon steels were validated. In 1996, the strengthening effects due to precipitations of VC, VN, and V(CN) were quantified and described with a modified Shercliff-Ashby model, which considers the effects of solid-solution hardening in addition to precipitation strengthening. Traditionally, only the latter is considered. The magnitude of the precipitation strengthening is treated as a function of coiling temperature in industrial practice. Currently, this model is being extended to include Nb-treated steels.

The equations developed for the constitutive behavior and for the structure-property relations have been successfully incorporated into a process model which is being developed at UBC-Canada. The process model simulates the rolling processes involved in making flat steel slabs in hot-strip mills and calculates the mechanical properties of the hot-rolled products. The equations and the process model have been disseminated to AISI member companies through project-review meetings and workshops.

Publications

1. "Design and Performance of an Automated Test Facility for Hot-Compression Testing", R. L. Tobler, B. J. Filla and Y. W. Cheng, Submitted to Journal of Testing and Evaluation, ASTM.

2. "Hot-Deformation Behavior of Several Microalloyed Forging Steels," G. T. Velarde, C. J. Van Tyne, and Y. W. Cheng, presented at the second International Symposium on Micoralloyed Bar & Forging Steel, Colorado School of Mines, Golden, CO, July 8-10, 1996.

3. "Optimization of the Ferrite-Pearlite Microstructure of Vanadium-Treated Medium-Carbon Steels by Means of Mathematical Modeling of Forging Process," R. Kuziak, M. Pietrzyk, and Y. W. Cheng, presented at the second International Symposium on Microalloyed Bar & Forging Steel, Colorado School of Mines, Golden, CO, July 8-10, 1996.

4. “Prediction of Strengthening Due to V Additions in Direct-Cooled Ferrite-Pearlite Forging Steels”, P.T. Purtscher, Y-W, Cheng, R. Kuziak and R.P. Foley, Proceedings, 37th Mechanical Working and Steel Processing Conference, Iron and Steel Society, Warrendale, PA, pp. 405-416, 1996.

Project: MICROSTRUCTURAL EVOLUTION

MSEL Program: Intelligent Processing of Materials

Project Leader: W.L. Johnson
G.A. Alers

Technical Objective

This project seeks to establish a materials-science base for a proposed real-time ultrasonic sensor that will use measurements of vibrational damping to nondestructively monitor the microstructural evolution of cold-worked metallic alloys at elevated temperatures. Recovery and recrystallization are the microstructural changes of primary interest. The importance of monitoring these changes is particularly great for industrial annealing processes that must carefully balance increases in ductility with losses in strength, as, for example, in the production of sheet metal used in forming applications.

Technical Description

In all materials at sufficiently elevated temperatures, mechanical vibrations are highly damped. This phenomenon has been studied relatively little, usually being regarded simply as a nuisance in studies that have focused on other high-temperature effects, but it is generally believed to result from the nonelastic response of dislocations to vibrational stress. The current project revisits this subject from the perspective of intelligent processing of materials. Since vibrational damping is highly sensitive to dislocation structure, it can serve as a real-time nondestructive monitor of microstructural changes that involve annihilation or reconfiguration of dislocations, such as recovery and recrystallization in cold-worked commercial alloys. Observed changes in damping with recovery and recrystallization in pure and alloyed aluminum were reported in FY95. Further studies of these effects were pursued in FY96. A unique experimental system employing noncontacting electromagnetic-acoustic transduction was used to excite resonant modes of spherical samples and measure the frequency and damping of these modes. Since the usefulness of an ultrasonic monitor of recovery or recrystallization rests partly on an understanding of the physical mechanisms involved in the high-temperature damping in alloys, research efforts have also been focused on establishing a physical model for this damping.

Accomplishments (FY96)

Following the procedure used in FY95, a series of high-temperature ultrasonic resonance measurements were performed on pure aluminum that had been deformed different amounts. The temperature ranges where large changes in damping occur were found to be consistent with the expected dependence of recovery and recrystallization annealing stages on the extent of coldwork.

Measurements of damping in annealed pure aluminum samples were performed as a function of temperature, frequency, and vibrational amplitude to provide information leading to a physical model. Based on these results, a tentative model was formulated that explains the high-temperature damping in terms of a thermally activated change in pinning of dislocations by point defects.

Publications

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Project: XRD SENSING OF LIQUID-SOLID INTERFACE

MSEL Program: Intelligent Processing of Material

Principal Investigators: D.W. Fitting
T.A. Siewert, W.P. Dubé

Technical Objectives

- Develop transmission x-ray diffraction as a nondestructive means for locating, characterizing, and following the liquid-dendrite-solid region in an investment casting during directional solidification.
- Map isocontours of fraction solid in the mushy zone of a superalloy casting
- Demonstrate the XRD sensing technology on a turbine-blade casting furnace and characterize the performance of the sensor
- Verify finite-element models of turbine blade casting solidification

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 has identified several areas where developments in technology would substantially improve the investment casting process. In particular, no sensor to date has been capable of detecting the location of the liquid-dendrite-solid region 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 solidification models), reducing scrap and increasing the production rate and yield. Therefore, we are developing a noninvasive x-ray diffraction (XRD) technique to monitor the position and shape of the liquid-dendrite-solid region in single-crystal and directionally solidified jet-engine turbine-blade castings. The sensor is capable of sensing the state of solidification even though the casting is enclosed in a thick ceramic mold and contained within a vacuum furnace. 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, and to verify solidification models of the casting process. X-rays of high energy are capable of penetrating through the furnace, mold walls, and sample, while those of low energy yield a higher number of diffracted x-rays. An optimal x-ray energy (often between 100 and 320 keV) has been chosen using an analytical model for the transmission XRD process.

External Collaborations

Industrial. Howmet Corporation has been an active collaborator in developing the XRD sensor. Howmet engineers have (1) furnished us with an industrial directional solidification furnace, (2) fabricated casting molds in a variety of shapes, (3) provided us with superalloy casting charges, (4) assisted during metal casting experiments, and (5) provided a wealth of information on investment casting.

Accomplishments (FY96)

This year, we have attached our XRD sensor to an industrial directional solidification furnace and successfully monitored the solidification of nickel superalloy (N5) single-crystal castings. The high x-ray energies (up to 320 keV) permit transmission XRD to be performed on a 6 mm thick nickel alloy specimen. XRD images from solid and molten N5 alloy were obtained even though the x-ray path through the furnace included 20 mm of Pyrex (furnace ports), 3.2 mm of molybdenum (furnace windings), 9 mm of aluminum oxide (furnace heater coil support), and 12.8 mm of mold material (refractory oxides). The x-ray source to imager distance was 1180 mm.

We have added new modules to our analytical model for the transmission XRD process. This model allows us to estimate the energy and beam intensity necessary to penetrate given thicknesses of casting, mold, and furnace components. The model has proven useful for assessing the feasibility of particular sensing applications.

Publications

1. W.P. Dubè, D.W. Fitting, and T.A. Siewert, "Real-Time Sensing of the Liquid-Solid Interface of Castings Using Transmission XRD: Experiments and an Analytical Model", Denver X-Ray Conference, 5-8 August 1996 (Denver, CO).

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

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ULTRASONIC CHARACTERIZATION OF MATERIALS

The Program on Ultrasonic Characterization of Materials is 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.

A primary focus of the Ultrasonic Characterization Program is microstructural characterization of metals and alloys, composite materials, and engineered surfaces. The idea is that models relate microstructure and physical properties. Thus, by measuring quantities related to physical properties, the salient microstructural features can be ascertained. For example, sound velocity is related to elastic properties, and thus, ultrasonic measurements can be used to characterize fiber-orientation distributions in composites or texture in metals. These model-based measurements enable industry to replace microscopy with nondestructive methods for the microstructural characterization needed to assure the quality of advanced materials.

The Ultrasonic Characterization Program is making significant contributions to measurement technology and materials modeling. We have worked with industry to commercialize advances in non-contact ultrasonics, waveform-based acoustic emission, nondestructive evaluation of composites and nonlinear ultrasonics. Modeling advances include Green's function methods for wave propagation in anisotropic materials, obtaining elastic constants from resonance spectra, and determining texture based on ultrasonic measurements.

Project: ELASTIC PROPERTIES OF SUPERCONDUCTORS

MSEL Program: Ultrasonic Characterization of Materials

Project Leader: H. Ledbetter
S. Kim, D. Clerc*, D. Balzar**

Technical Objective

This project focuses on oxide superductors and related metal oxides. We seek to understand the elastic constants, how they change with composition, crystal structure, and temperature, and how they relate to the basic normal-superconducting (n-s) mechanism. For the nonsuperconducting oxides we try to understand interrelationships among crystal structure, elastic constants (and other physical properties), and valence.

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.

Accomplishments (FY96)

Using acoustic-resonance spectroscopy, we measured the low-temperature internal friction of a detwinned Y-Ba-Cu-O monocrystal. Our results show that the superconducting-pair wave function is less symmetrical than isotropic s, the logical candidate being anisotropic $d_{x^2-y^2}$. We submitted our results to the upcoming large superconductor meeting: M^2S-HTSC-V. (Beijing, February - March 1997).

* NRC-NIST Postdoctoral Research Associate.
** Visiting Scientist from Ruđer Bošković Institute, Zagreb.

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Project: ELASTIC CONSTANTS AND RELATED PHYSICAL PROPERTIES

MSEL Program: Intelligent Processing of Materials

Project Leader: H. Ledbetter
S. Kim, D. Clerc*, D. Balzar**

Technical Objective

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

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, the Debye temperature, and the Grüneisen parameter.

Accomplishments (FY96)

Gave invited lecture at ICMFM96: International Conference on Microstructures and Functions of Materials (Tokyo, 9-11 September).

Gave invited lecture at Fraunhofer-Institut für zerstörungsfreie Prüfverfahren (Saarbrücken, Germany, 17 July).

Spent one week at Los Alamos National Laboratory neutron-diffraction facility measuring texture and internal strain in several materials. Subsidized by LANL based on our successful competitive research proposal.

Spent two weeks in Japan supported partially by Japan Agency of Industrial Science and Technology (MITI).

* NRC-NIST Postdoctoral Research Associate.
** Visiting Scientist from Ruđer Bošković Institute, Zagreb.

Spent one week at Brookhaven National Laboratory Synchrotron - radiation facility measuring texture and internal stress in several materials. Subsidized by BNL.

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

Measured successfully by ARS method, the complete elastic constants C_{ij} of diamond-cubic-symmetry monocrystals. Measured also companion internal friction and interpreted using Koehler-Granato-Lücke model.

Began ab initio calculations of elastic constants of several materials including metals, oxides, and oxide superconductors.

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Project: SENSORS FOR INDUSTRIAL NDE

MSEL Program: Ultrasonic Characterization of Materials

Project Leader: G.A. Alers
S.R. Schaps, R.L. Santoyo, R.E. Schramm, C.M. Fortunko

Technical Objective

Producing industrial quantities of materials with specified properties requires sensors that can survive in the mill environment and monitor the key variables during critical processing steps. Furthermore, similar sensors are needed after the material is manufactured and after having been in service in order to make sure that no defects were built in or that none have developed during service. The objective of this program is to develop sensors that can both survive in a mass-production environment to produce outputs useful for process control as well as to detect defects during and after manufacturing under hostile conditions.

Technical Description

Most sensors must be specialized to the materials on which they are to be used. Furthermore, the sensors often need to be designed for the unique processing variables used in manufacturing the material as well as for the environments in which the materials must ultimately survive. During recent years, four industrial sensor problems have been investigated. First was the development of noncontacting ultrasonic transducers for the inspection of mass-produced automotive parts and for the detection of cracking in buried gas pipelines. Second was the development of a noncontacting thermometer for use at the final stand of an aluminum rolling mill. Third was a device for quantitative measurement of the residual stress built up in railroad wheel rims by the over heating that accompanies in-service braking. Fourth was an application of resonant ultrasonic spectroscopy (RUS) to a new kind of load cell for the accurate weighing of heavy objects and to a new method of measuring case depths in automotive drive shafts.

External Collaborations

Industrial. IEM, Inc., for EMAT inspection of railroad rails.

Southwest Research Institute, for gas pipeline inspection (CRADA).

The Aluminum Association, for a noncontact thermometer (CRADA).

Morton International, for flaw detection in air bag inflators (CRADA).

Sponsors. The Federal Railway Administration (FRA) sponsors the development of a residual stress measuring device for railroad wheels.

The Gas Research Institute (GRI) sponsors the development of gas-coupled transducers for pipeline inspection.

Accomplishments (FY96)

Electromagnetic Transducers for Rapid Parts Inspection. During FY96, NIST completed a CRADA with the Automotive Safety Products Division of Morton International, Inc. It was established in FY93 to develop an ultrasonic inspection technique for the closure weld in their aluminum air bag inflators. The part had be kept dry and the inspection process had to be completed in less than ten seconds. During FY94 and FY95, NIST developed a miniature Electromagnetic Acoustic Transducer (EMAT) and an inspection algorithm that would meet these requirements. Sonic Sensors, Inc. of San Louis Obispo, California then worked with NIST and Morton production engineers to produce a robust, automatic version of the laboratory prototype that could be tested on the air-bag production line in Provo, Utah. Extensive testing of this on-line system demonstrated that inferior parts could be rejected in a reliable manner. In FY96, NIST assisted Sonic Sensors in the production of three improved copies of the system, and these are currently being used for routine inspection on three other production lines. A patent application was filed in FY96 so that the proprietary interests of Morton International can be protected.

Gas Coupled Transducers for Gas Pipeline Inspection. The gas industry needs better methods for inspecting their gas-transmission pipelines for flaws such as loss of wall thickness and stress-corrosion cracking. Ultrasonic inspection methods have been used successfully in other applications, but their use in gas-pipeline inspection has been impeded by the difficult problem of providing a suitable means to couple the ultrasound into the pipe. In the past, NIST has demonstrated the feasibility of using the compressed gas itself as the ultrasonic couplant. We are now working with the Gas Research Institute and Southwest Research Institute to determine the practicality of this concept. Specifically, we are working on the identification and quantification of the underlying physical phenomena in order to improve the measurement technique.
In FY96, we worked with Southwest Research Institute (SWRI) under a contract from the Gas Research Institute (GRI) to determine whether the gas-coupled ultrasonic inspection approach can achieve the sensitivity necessary for detecting stress-corrosion cracking (SCC) in pressurized natural gas transmission pipe. Previous work, carried out at NIST using a special pressure vessel and custom pulser/receiver electronics and transducers, demonstrated the feasibility of detecting crack-like flaws under the simplifying conditions of smooth, flat test plates, and smooth, flat artificial flaws. The present work extends the previous studies to the more realistic conditions of curved, somewhat rough pipe surfaces and actual cracks which are very irregular in shape. To

address these practical issues, a focused experimental and modeling effort is being conducted. Specifically, we designed and fabricated a new transducer-scanning fixture for the pressure vessel and considerably improved our pulser/receiver electronics and transducers. We also made extensive use of mathematical modeling to improve our understanding of the underlying physical phenomena and optimize the experimental parameters. By briefing the industry on the objectives and status of our project, we have obtained realistic specimens for our experimental investigations.

Eddy Current Thermometer for Aluminum Processing. At the end of FY95, a system designed by NIST for measuring the resistivity of fast moving, hot aluminum sheet metal was tested under-rolling mill conditions at the Alumax Mill Products Division rolling mill in Lancaster, Pennsylvania. This system had been under development for several years under a CRADA with the Aluminum Association to test the idea of making the aluminum sheet itself act as the resistive element in a resistance thermometer. To accomplish this, one must have an accurate equation relating the measured resistivity to the temperature. For the aluminum alloys commonly rolled into sheet products, this equation could be represented by the well known resistivity versus temperature relationship for pure aluminum plus a temperature-independent term characteristic of the particular alloy being processed. Extensive tests performed in previous years showed that the alloy term had to be measured on each billet before it entered the final rolling stages of the hot mill. In the final series of tests in late FY95, the NIST instrumentation measured the resistivity at two locations in the mill. At the upstream location, a contacting eddy-current probe and a thermocouple were placed on each aluminum alloy slab as it sat motionless at the entrance to the hot mill to have its leading edge cut square and to have its temperature transmitted to the mill operator. Data from these two instruments allowed the alloy term to be measured directly, thereby effectively calibrating the slab as a resistance thermometer. At the downstream location, a second resistivity measurement was made on the sheet as it moved out of the final rolling stand into a coiling machine. This measurement was performed without physical contact with the sheet by two eddy current coils placed above and below the pass line of the mill at a large separation distance. The same instrumentation that was used with the upstream, contacting eddy-current probe was used for the downstream resistivity measurement, although different frequencies and data reduction algorithms had to be employed.

During FY96, all the measurements taken at the Alumax mill on 59 slabs of 10 different alloy types were analyze to determine the accuracy to which the temperature at the exit end of the hot mill could be determined. This approach to the accuracy question was the only one available because there is no reference thermometer capable of independently measuring the temperature on the moving sheet of aluminum at the mill exit. Each of the two probes had to be analized separately because they used different calibration techniques and different measurement procedures. The upstream probe was calibrated for the mill conditions by directly measuring the alloy contribution to the resistivity on samples cut from each alloy slab. The final result was a 3σ uncertainty of ± 0.138 μΩ•cm in the alloy contribution to the resistivity. Most of this uncertainty could be traced to a $\pm$ 3 °C error in the temperature reading of the contact thermocouple. The downstream probe that measured the resistivity of the moving sheet metal was calibrated by determining the individual random errors in the three elements of the equation that related measured quantities to the resistivity. These tests showed a 3σ uncertainty of ±0.054 μΩ•cm in the resistivity measured by the downstream eddy current probe. Combining all these uncertainties in resistivity measurements led to an estimated total 3σ random error of ±0.148 μΩ•cm. This corresponds to a temperature error of for the sheet metal leaving the last stand of the mill ± 11 °C.

Analysis of the various sources of this total error showed where improvements in later systems should be made. If the error in the contacting thermocouple used in the upstream measurements could be reduced from ± 3 °C to ± 1 °C and all the resistivity measurements errors reduced to the level of the best measurement (i.e. ± 0.6%), then the overall 3σ temperature uncertainty could be reduced to ±6 °C, which is close enough to the original goal set by the Aluminum Association to warrant continued effort. Future NIST efforts will be directed toward transfering its technology to a commercial supplier of eddy-current instruments who can deliver an on-line temperature sensing system to the aluminum industry at a reasonable cost.

Ultrasonic Inspection of Railroad Wheels and Rails. For many years, NIST has worked with the Federal Railroad Administration to develop electromagnetic transducers (EMATs) for measuring residual stress in wheel rims and for detecting cracks in the running surface of wheels during service. Both of these applications take advantage of the coupling reliability of EMATs when used on the variety of surfaces encountered in field inspections of railroad equipment. In FY96, the results of a detailed study of the residual stresses left in the rim of wheels by excessive braking was published as a NIST Internal Report (NISTIR 5043) and in a Final Report to the FRA. These reports demonstrated essential agreement between the results of destructive saw cut tests and nondestructive measurements by both EMATs and conventional piezoelectric transducers. As a result, the FRA has awarded NIST a contract to assemble a compact and portable EMAT-based instrument for measuring the residual stress level in wheel rims while the wheels are inservice on a train. Much of this development will be carried out in FY97.

During FY96, the FRA awarded a Phase I SBIR program to International Electronic Machines Corporation (IEM) of Albany, New York to investigate techniques that could detect subsurface cracks in the head of rails. Since NIST had investigated the use of EMATs for detecting cracks in the running surface of wheels, IEM asked NIST for assistance in performing their feasibility demonstration of EMAT techniques applied to rail heads. NIST was able to loan IEM some electronic instruments that had been designed especially for EMATs and was able to advise IEM on the design of EMAT probes that would generate and detect a Shear Horizontal wave in the head of the rail. This particular wave type had been shown in previous FRA studies to have unique sensitivity to the flaw of interest to the SBIR program. Because of this assistance, IEM was able to complete their Phase I plan successfully by demonstrating improvements over previous studies concerning rail flaw detection. A continuation into a Phase II program is expected.

Resonant Body Sensors. Excitation and measurement of the vibrational modes of spheres, cubes, cylinders and bars is becoming a very useful tool for ultrasonic materials characterization. Unfortunately, application of these techniques to industrial sensing problems is difficult because the shape of the resonant body plays a fundamental role in the relationship between the measured resonant frequency and the elastic properties of interest. Furthermore, the method of supporting the body and the transducer used to excite and detect the resonant modes must couple so weakly that they do not shift the resonant frequencies or modify the quality or Q factor of the resonance. During the past few years, special EMATs that satisfy the weak-coupling requirement have been developed for operation on cylindrical bodies, and a new technique for trapping the vibrational energy away from attachment points is satisfying the support requirements. Two industrial applications of this new, trapped-mode technology were investigated in FY96. The first measures the force on a cylindrical bar supporting an axial load as might be found in a load cell or weighing application. The second measures a series of particular resonant modes in a case-hardened automotive drive shaft to deduce the depth of hardening.

The method of measuring large forces with ultrasonic resonance provides several advantages over commercial load cells based on resistive strain-gage techniques. These include greater resolution, insensitivity to transducer and bond degradation, durability in harsh environments, and insensitivity to bending moments. In the resonance technique, a solid aluminum cylinder serves as a resonant load-bearing element and a noncontacting electromagnetic-acoustic transducer measures the stress-dependent resonant frequency. Vibrational damping from the application of the force is avoided by employing trapped resonant modes which have negligible amplitude at the ends of the cylinder where the force is applied. In practical applications, an individual resonant-frequency measurement will be influenced by the temperature. However, if other resonant modes are measured nearly simultaneously, temperature effects can be compensated by forming ratios of the mode frequencies, with nearly equal temperature sensitivities.

For measuring the depth of case hardening in automobile drive shafts, ultrasonic resonance measurements of a series of overtone modes were used to nondestructively profile the hardness in the cylindrical shafts. A noncontacting electromagnetic-acoustic transducer was used to measure the frequencies of a series of very particular resonant modes whose vibrational patterns penetrate to various depths in the shafts. Because analytical expressions for the displacements associated with these modes are available, an inversion algorithm could be developed that predicts the curve for acoustic wave velocity versus depth from the measured mode frequencies. The results indicate that there are two factors that determine the wave velocity profiles: hardness and residual stress. Both of these quantities can be profiled from a single set of ultrasonic measurements.

Publications

1. S. Schaps, A.V. Clark, and B. Barnes (Morton), "Noncontact Method of Inspecting Inertia Weld Driver Inflator Using EMAT Technology," U.S. Patent Appl. 08/951,597.

2. W. Johnson, G. Alers, and B. Auld, "Acoustic Resonator for Measuring Force," U.S. Patent Application filed on 4 September 1996.

3. W. Johnson and G. A. Alers, "Force Measurement Using Vibrational Spectroscopy", Rev. Sci. Instrum. Vol. 68, no. 1, 1997.

4. A. Kahn, L. Phillips, S. Schaps and G. Alers, "Noncontact Measurement of the Exit Temperature of Sheet Metal in an Operating Rolling Mill", NISTIR 5058.

5. R.E. Schramm, J. Szelazek and A.V. Clark, “Dynamometer-induced Residual Stress in Railroad Wheels: Ultrasonic and Saw Cut Measurements," NISTIR 5043, Mar. 1996.

Project: WAVEFORM-BASED ACOUSTIC EMISSION

MSEL Program: Ultrasonic Characterization of Materials

Principal Investigator: C.M. Fortunko
M.A. Hamstad, J.D. McColskey, J.M. Gary*

Technical Objectives

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

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

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result of local transient energy releases in a material. Monitoring these waves can provide fundamental information about the location and mechanism of the transient energy release. Often the energy release is due to a local micro-damage process. The anticipated outcome of this acoustic-emission effort will be fundamental micro-information about deformation and defect response of various materials to applied stress. This information will provide unique input to basic studies by material scientists and enhance the application of nondestructive characterization of the response of structures to applied loads. The technical approach follows a multifaceted development of all the key components which are relevant to a wideband application of AE technology. These components include development of wideband high-sensitivity sensor preamplifiers, high-speed wide-dynamic-range digital recording data gathering systems, finite element modelling of the predicted far-field displacement waves from relevant AE sources, wideband experimental AE displacement waveforms from materials of interest, and the development of signal processing techniques to extract accurate source locations and source type identification. The scope in FY96 covered three phases: (1) 3-D finite element modelling and experimental verification in the far-field of thick plates; (2) Wideband characterization of extraneous AE during fatigue cycling as contrasted to the old AE technology using resonant sensors and fixed-threshold AE; and (3) Development of new source location approaches along with comparison with existing methodologies for location of AE sources in automotive-type chopped-glass composites using wideband data developed during bending of two different material systems. Special equipment and facilities include a specially

* NIST, Applied and Computational Mathematics Division

modified hydraulic materials testing system, multiple coupled waveform recorders, specially developed wideband AE sensor/preamplifiers, and a waveform-based AE recording system (NASA Langley system).

External Collaborations

Academic. CRADA - High sensitivity, wideband AE sensor/preamplifier development with Dunegan Engineering, Inc.

ATP. Characterization of microdamage in automotive-type chopped glass/polymer/filler composites by waveform-based AE with Michigan State University Advanced Materials Engineering Experimental Station.

Government Laboratory. Evaluation of multichannel waveform-based AE system and NIST-Boulder wideband sensors to characterize damage accumulation in fiber composites with NASA Langley.

Private Laboratory. Relationship of the physical mechanism of an AE crack source in steel to the mathematical moment-tensor AE characterization of the same source with SRI International.

Government Agency. The Federal Highway Administration sponsored the development of waveform-based AE for the NDE of bridges.

Accomplishments (FY96)

Demonstrated that 3-D finite element code prediction of far-field displacements in 25 mm thick steel plate matched those experimentally measured for pencil lead breaks. Investigated finite element parameter limited necessary to accurately model displacements at frequencies up to those in Rayleigh waves.

Developed a database of multi-channel waveforms for AE events during four-point bending of automotive type composites. Analyzed and developed approaches to accurate source locations. Showed that small velocity variations hinder accurate locations.

Completed gathering of extraneous AE signals which were generated during fatigue of steel. Contrasted the difference between these signals when measured with wideband versus resonant sensors. Developed the implications of the results and the advantages gained when using wideband waveform sensor data.

Publications

1. M.A. Hamstad, J. Gary, A. O'Gallagher, "Far-field Acoustic Emission Waves by Three-Dimensional Finite Element Modelling of Pencil Breaks on a Thick Plate", accepted for publication by the Journal of Acoustic Emission.

Project: NONLINEAR ULTRASONICS FOR MATERIALS CHARACTERIZATION

MSEL Program: Ultrasonic Characterization of Materials

Principal Investigator: **D. C. Hurley**
M. C. Renken, C. M. Fortunko

Technical Objectives

- Identify quantitative relationships between nonlinear-ultrasonic measurements, microstructure, and mechanical/physical properties.

- Develop and evaluate appropriate experimental techniques; develop analytical models to predict and explain experimental results. Use methods to characterize specific systems such as diamond-coated or ion-implanted surfaces.

Technical Description

We are investigating the applicability of nonlinear-ultrasonic techniques to understand the relationships between microstructure and mechanical or physical properties. Nonlinear ultrasonics can, in principle, probe certain microstructural properties more sensitively than standard linear ultrasonics. For instance, linear-ultrasonic methods are typically ill-suited to interrogate properties such as film adhesion and surface hardness. Our long-term goal is to develop nonlinear ultrasonic techniques for nondestructive materials characterization, particularly in relation to material reliability and remaining lifetime.

Our technical approach first involves the development of sensitive and accurate nonlinear ultrasonic techniques. Experimental methods will be validated by performing baseline measurements and comparing the results to those obtained with established methods. Idealized or "model" bulk systems can then be studied. In a parallel effort, instrumentation and methods will be adapted to enable measurements on surfaces. Model surface-modified samples will be identified, and experiments performed on these specimens to understand nonlinear surface-wave behavior. For both bulk and surface studies, analytical models will be developed to predict and explain experimental results. Results will also be correlated with mechanical measurements. In this way, quantitative relationships between the parameters measured and the microstructural properties can be established. Finally, the feasibility of using our methods on industrial materials such as diamond-coated tools or ion-implanted parts will be examined. Obstacles to success will be identified and practical solutions will be developed. We anticipate that out of this work, tools utilizing nonlinear ultrasonics will be developed to understand microstructure and its relation to mechanical/physical properties. In particular, we seek techniques for nondestructive assessment of properties such as adhesion, hardening, and microcracking or fatigue.

The scope of FY96's effort was to establish laboratory techniques for performing nonlinear ultrasonic measurements reliably, accurately, and with sufficient sensitivity. An important element was validation of our methods through comparison with established techniques. In FY96, we also established ties with groups working in nonlinear ultrasonics and related fields. Time was spent furthering our knowledge of the physics of nonlinear ultrasonics, and special issues for coatings and surfaces.

External Collaborations

Industrial. Through a CRADA with RITEC, Inc., we have obtained specialized electronics for nonlinear-ultrasonic measurements. The instrument, which is the first of its kind and not a turnkey system, represents an instrumentation advantage for NIST. In FY96, we evaluated the prototype system and integrated it into our experimental apparatus. Modifications based on our findings were incorporated in a second "beta" version currently under development and scheduled for release in mid-FY97.

Academic. Collaborative ties with a number of academic groups were established in FY96, including: R. B. Thompson, Iowa State University; W. K. Arnold, University of Saarbruecken/IzfP (Germany); and M. F. Hamilton, University of Texas. Relations were developed through personal visits and at technical conferences, which provided opportunities to discuss topics of mutual interest.

Government Laboratory. A working collaboration with W. T. Yost at NASA's Langley Research Center was established. Yost is a leader in the field of bulk nonlinear measurements, and his capacitive detection technique is the most commonly used nonlinear-ultrasonic method. Experiments to determine the bulk nonlinearity parameter in fused silica and single-crystal silicon specimens were performed at Langley by NIST staff. The experimental results from this collaboration provided confidence in our own apparatus.

ATP. Interactions with producers of plasma-source ion-implanted (PSII) and diamond-coated materials were initiated. NIST staff visited Los Alamos National Laboratory to learn about the PSII process and program needs, and to obtain PSII test samples. We discussed program goals and mechanical-property/ microstructual measurement needs for diamond-coated tools with Crystallume staff. Preliminary diamond-coated tungsten carbide samples from Crystallume and Norton Diamond Films were obtained.

Accomplishments (FY96)

We have developed an optical probe, based on a Michelson laser interferometer, to measure linear and nonlinear ultrasonic wave propagation. We have chosen this approach because an interferometer enables direct measurement of absolute ultrasonic displacement, which is essential for nonlinear studies. Unlike existing nonlinear-ultrasonic techniques, interferometric detection is inherently nonintrusive, possesses micrometer-scale resolution, and has the potential to scan large, curved areas quickly. The interferometer was combined with the electronics discussed above to create a system for nonlinear measurements. The system was validated by using it to determine the bulk nonlinearity parameter beta in a fused silica specimen, and comparing the results to those obtained with two other detection methods: a capacitive receiver and a piezoelectric transducer. The values obtained from the three methods are in good agreement with each other and with previously published results. We therefore have gained confidence in using the interferometer as an accurate, reliable tool to study nonlinear wave propagation.

Late in FY96, efforts were begun to adapt the laboratory apparatus for surface studies. Means to generate surface waves of sufficient amplitude and spectral purity to observe nonlinear effects are under investigation. In addition, preliminary nonlinear experiments were performed on a PSII specimen provided by Los Alamos National Laboratory using the interferometer and bulk-wave techniques described above. Some potential nonlinear effects were observed, but correct interpretation of the results will require further experiments on other samples (planned for FY97).

Publications

1. D. C. Hurley, W. T. Yost, E. S. Boltz, and C. M. Fortunko, "A Comparison of Three Techniques to Determine the Nonlinear Ultrasonic Parameter Beta," in *Review of Progress in Quantitative NDE* 16, eds. D. O. Thompson and D.E. Chimenti (Plenum Press, New York 1997), to be published.

2. D. C. Hurley, W. T. Yost, E. S. Boltz, and C. M. Fortunko, "Experimental Comparison of Ultrasonic Techniques to Determine the Nonlinearity Parameter Beta," *Proceedings of the 1996 IEEE Ultrasonics Symposium*, to be published.

3. D. C. Hurley and C. M. Fortunko, "Determination of the Nonlinear Ultrasonic Parameter Beta Using a Michelson Interferometer," submitted for publication.

Project: ULTRASONIC MEASUREMENTS OF STRESS

MSEL Program: Ultrasonic Characterization of Materials

Project Leader: A.V. Clark
G.A. Alers, S.R. Schaps, R.E. Schramm

Technical Objective

The objective of this program is to apply ultrasonic techniques for stress measurement to the assessment of the aging infrastructure. In particular we are concerned with field measurements on bridges.

Technical Description

Since stress changes the velocity of sound, in the stressed materials, a measurement of the sound velocity can be used to measure stress in a variety of materials and structures. Most applications to date have been limited to laboratory settings because the effect is small and the measurements are hindered by the fact that variations in the microstructure have a competing effect. To deal with this problem we use information about the stress state in the structure to separate the influence of microstructure and stress. Furthermore, to be practical the stress measurement system must be portable, durable, inexpensive, and capable of reproducible results in the hands of personnel with minimal training. Our approach is to use noncontacting electromagnetic-acoustic transducers (EMATs) which require no couplant to generate sound in the structure and are easily fixtured. These devices are inexpensive and reduce operator dependence. We are collaborating with researchers in the area of bridge safety to prove out concepts of ultrasonic stress measurement in the field and in realistic laboratory simulations.

External Collaborations

Academic. Constructed Facilities Research Center, West Virginia University

Government agencies/laboratories. Virginia Transportation Research Council and the Federal Highway Administration

Accomplishments (FY96)

Field Test Results: We provided support for field tests performed by Virginia Transportation Research Council (VTRC) personnel. Some of their previous tests on a particular bridge using strain gages indicated that stresses near the yield stress were present at one side of the bridge. Ultrasonic measurements made at opposite sides of the bridge showed combined bending and compression in the girders but no excessive stresses. We predicted rotation of the girders due to bending and this was confirmed by visual inspection. We predicted that the bridge was in a "safe" condition and this was confirmed when some suspect strain-gage electronics was replaced by VTRC personnel. Based on the experience gained in these field tests, we have initiated a major redesign of our stress measurement system. We replaced our computer, monitor and oscilloscope with a single portable unit. This will improve reliability and reduce bulk for future field tests.

Measurements on Painted Steel: During the tests described above, it was often necessary to remove paint to get good signals. Paint removal can present a potential lead-poisoning hazard and, hence, requires special precautions. We performed experiments to characterize the effect of painted surfaces on ultrasound. The results showed that paint can have acoustic properties close enough to those of steel to allow the sound to propagate into the paint and be attenuated. Layers as small as 10 μm thick can a significantly reduce signal strength. We simulated measurements on older bridges with thick paint layers in the laboratory to learn that compliance and thickness variations in the paint could decrease the precision of our stress measurements by a factor of two. We have designed a special 4-element transducer to suppress this effect.

Pin and Hanger Connections: Pin and hanger connections are used to suspend an interior span from the outer spans in order to compensate for thermal expansion effects. If corrosion at the pin occurs, it can cause torsion in the pin and bending of the hanger. We anticipate that ultrasonic measurements of the bending stress in the hanger will be a simple and most robust way to determine status of the connection. Measurements of the sound speed in uniaxial tension specimens have been made to simulate a "good" connection. For simulating measurements on a hanger with a "bad" connection, we have designed a cantilever section to fit in our mechanical testing machines. It should be ready for testing early in FY97.

Technology Transfer: We are establishing a CRADA with SonicForce, Inc. to apply some of our ultrasonic stress measuring techniques to the measurement of earthquake-induced damage in steel buildings and to measure the thermally-induced stresses in continuously welded rails. This company is already marketing an EMAT-based ultrasonic system for measuring the fatigue loading of bridges while in service. In addition, we are collaborating with researchers at Federal Highway Administration in analyzing the performance of another EMAT-based system for fatigue loading.

Publications

1. M.G. Lozev, A.V. Clark and P.A. Fuchs, "Application of Electromagnetic-Acoustic Transducers for Nondestructive Evaluation of Stresses in Steel Bridge Structures," VTRC 96-R30, Virginia Transportation Research Council, Charlottesville, VA, April, 1996.

2. A.V. Clark and T.L. Anderson, "Quantitative Bridge Safety Assessment Utilizing Fracture Mechanics and Ultrasonic Stress Measurements," Structural Materials Technology: An NDT Conference, San Diego, CA, Feb. 20-23, 1996, Technomic Pub. Co., Lancaster, PA.

3. A.V. Clark, T.L. Anderson, M.G. Lozev and P.A. Fuchs, "Bridge Safety Assessment from Fracture Mechanics and Ultrasonic Stress Measurement," Nondestructive Evaluation of Civil Structures and Materials, Boulder, CO, Sept. 9-11, 1996, Express Press, Boulder, CO.

4. A.V. Clark, M.G. Lozev and P.A. Fuchs, "Bridge Safety Evaluation Using Ultrasonic Stress Measurement," to be published in Nondestructive Evaluation Techiques for Aging Infrastructure and Manufacturing, SPIE, Scottsdale, AZ, Dec. 2-5, 1996.

Project: GREEN'S FUNCTION METHOD FOR MATERIALS SCIENCE

MSEL Program: Ultrasonic Characterization of Materials

Principal Investigator: V.K. Tewary

Technical Objectives

- To develop computationally efficient methods for calculating elastostatic and elastodynamic Green's functions for bounded solids.

- To develop the Green's-function-based theoretical methods for modeling and analysis of linear and nonlinear elastic properties of anisotropic materials.

- To apply the Green's functions in the development of boundary element formulation for stress analysis of anisotropic solids.

- To develop a theoretical formulation for interpretation of experimental results on elastic wave propagation in anisotropic plates.

Technical Description

A Green's function gives the response of a solid to a unit force. It gives the solution of the Christoffel equations of elastic equilibrium for prescribed boundary conditions and an arbitrary integrable force distribution. Since it gives the response of the whole solid, it provides a convenient mathematical technique to model the elastostatic and elastodynamic properties of anisotropic solids. The elastodynamic Green's function is used to calculate the elastic waveforms and other characteristics of the elastic wave propagation induced by a pulsed load. The elastostatic Green's function is used to calculate stress distribution due to a static load in solids containing cracks and/or other discontinuities such as interfaces and free surfaces. Combined with the boundary-element analysis, the Green's function method provides a powerful numerical tool for stress analysis of engineering materials of different geometrical shapes.

A major computational advantage of the Green's function is that it is a characteristic of the material and its geometry, and is independent of the loading. The Green's function can be calculated in steps of increasing geometrical complexities using the previous value as an input. It is, therefore, possible to calculate and store the Green's functions for basic geometrical shapes and different material parameters for use in further calculations. The strategy is that we calculate the Green's function analytically for simple geometrical shapes and use the boundary-element method for application to complicated geometrical shapes for engineering applications.

The Green's function method is also applicable for perturbation solution of the nonlinear elastic problems. We are working on using the Green's function method for obtaining approximate solutions of the nonlinear Christoffel equation for modeling the nonlinear elastic wave propagation in anisotropic solids. Study of nonlinear properties of elastic waves can be used for microstructural characterization of advanced materials.

We calculate the Green's functions by using a delta function representation that we had developed earlier. In the delta function representation, we use a linear combination of the space and time variables instead of using the two separately. This representation has been found to be computationally very efficient. We have already applied it to various problems on elastic wave propagation such as elastic waveforms in anisotropic half-space solids and plates due to a pulsed load. We are trying to develop it further to solve the nonlinear Christoffel equation.

In elastostatics the delta function representation in two dimensions becomes similar to the Stroh's representation. For solids containing elastic discontinuities, the requirement of satisfying the prescribed boundary conditions involves solution of a Hilbert problem. We have developed an orthogonal complex transform to solve the Hilbert problem and applied it to free edges, to interfacial cracks, and to cracks inclined at an arbitrary angle to the interface in bimaterial composites. Using the calculated Green's functions, we have developed boundary-element formulations for interfacial cracks and analysis of moire fields in anisotropic materials. In three dimensions, to which the Stroh's representation is not applicable, the delta function representation provides a computationally efficient method for calculating the Green's function and the stress distribution in anisotropic solids. These calculations will serve as the basic input to the three-dimensional boundary element formulation and storage of Green's functions for engineering applications.

External Collaborations

Collaboration with Prof. John Berger of Colorado School of Mines on elastostatic calculations on ansiotropic materials.

Collaboration with Frank Rizzo of Iowa State University on exploring the possibility of developing a library of Green's functions for industrial applications (idea originally proposed by Frank Rizzo).

Collaboration with Prof Graham Mustoe (Guest Researcher at NIST) of Colorado School of Mines on elastic wave propagation.

Collaborations with several universities (Colorado School of Mines. University of Arizona/ Michigan Technological University, Iowa State University, Vanderbilt University), and industries (Motorola, Gates Rubber Company, United Technologies) on four NIST/NSF projects dealing with the application of Green's function/boundary element method to industrial problems (projects identified at the workshop sponsored by the Center for Theoretical and Computational Materials Science).

Accomplishments (FY96)

Elastic waveforms were calculated for surface waves in tetragonal solids, and thick anisotropic plates with cubic and general orthorhombic symmetry. The theoretical results on plates were used to interpret the experimental results and to estimate the elastic constants. Waveforms in frequency space were calculated for harmonic loads in cubic and orthorhombic half-space solids. Mechanical impedance of anisotropic half-space solids was also calculated. The singularity in the imaginary part of the mechanical admittance is an artifact of the continuum model and does not arise if a correction is introduced to account for the discrete structure of the lattice.

The elastic Green's function was calculated for a damaged interface in anisotropic bimaterial composites. A boundary element formulation was developed for interfacial cracks and interpretation of moire field data in anisotropic bimaterial composites. The Green's function and stress intensity factor were calculated for a crack inclined to the interface in an anisotropic bimaterial composite. The stress near the crack tip remains oscillatory for small angles but not for large angles between the cack and the interface.

Four projects were sponsored jointly by NIST and NSF as a direct outcome of the workshop on Green's functions and boundary element analysis that we organized last year. This workshop was sponsored by the Center for Theoretical and Computational Materials Science. In all these projects, NIST has the coordinating and enabling role. The list of these projects and the institutions and industries which are involved is given below:

1. Interface and near-interface cracking in automotive V-belts (Gates Rubber Company, NIST, Colorado School of Mines).

2. Cracking in thermal barrier coatings (United Technologies, NIST, Vanderbilt University, Colorado School of Mines).

3. A Green's function library (NIST, Iowa State University, Colorado School of Mines, Michigan Technological University).

4. Reliability of conductive adhesives (Motorola, 3M, NIST, Michigan Technological University, Colorado School of Mines).

Publications

1. V.K. Tewary and C.M. Fortunko: "Surface waves in three-dimensional half-space tetragonal solids," J. Acoust. Soc. Am. **100**, 86 (1996).

2. V.K. Tewary and C.M. Fortunko: "Lattice correction to mechanical admittance of solids," J. Acoust. Soc. Am. **100**, 89 (1996).

3. J. Berger and V.K. Tewary: "Elastic Green's function for a damaged interface in anisotropic materials," J. Mat. Res. **11**, 537 (1996).

4. V.K. Tewary, M.Mahapatra, C.M. Fortunko: "Green's functions for anisotropic half-space solids in frequency space and calculation of mechanical admittance," J. Acoust. Soc. Am., **100**, 2960 (1996)

5. V.K. Tewary and C.M. Fortunko: "Theory of elastic waves in three-dimensional anisotropic plates," J. Acoust. Soc. Am. **100**, 2964 (1996).

6. V.K. Tewary: "Elastic Green's functions for anisotropic solids (review)," Proceedings of the NIST workshop on Green's functions and boundary element analysis, NIST Special Publication SP 910 (1996).

7. V.K. Tewary and J. Berger: "Elastic Green's function for a bimaterial composite solid containing a crack inclined to the interface," J. Comp. Mech. (to be published).

8. J. Berger and V.K. Tewary: "Boundary-integral analysis of anisotropic bimaterials with an interface crack," J. Comp. Mech. (to be published).

9. J. Berger and V.K. Tewary; "Boundary element analysis of moire fields in anisotropic materials," J. Comp. Mech. (to be published).

10. G. Mustoe, V.K. Tewary, and C.M. Fortunko: "A computational Green's function procedure to determine bulk and surface waves within anisotropic solids," J. Comp. Mech. (to be published).

11. J. Berger and V.K. Tewary (Editors) "Proceedings of the NIST workshop on Green's functions and boundary element analysis," NIST Special Publication SP 910 (1996).

12. E.S. Boltz, V.K. Tewary, and C.M. Fortunko, "Fedelity of Michelson interferometer and conical piezoelectric ultrasonic transducers," Review of Progress in Nondestructive Evalaution, (Plenum Press, N.Y.) 15A 971 (1996).

Project: ULTRASONIC CHARACTERIZATION OF COMPOSITES

MSEL Program: Ultrasonic Characterization of Materials

Project Leader: C.M. Fortunko
H. Ledbetter, S. Kim, M. Dunn*, D. Balzar, N. Sizova*****

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.

Technical Description

We need to know accurately a material's 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.

Accomplishments (FY96)

Organized 1996 Gordon NDE Conference. The conference focused on microstructure evolution, characterization, and property relationships.

At the 1996 Gordon Conference on Nondestructive Evaluation, we gave an invited lecture: Modeling and measurements of two phase materials: short-fiber composites.

We extended and applied 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.

* Visiting scientist from University of Colorado.
** Visiting scientist from Ruđer Bošković Institute, Zagreb.
*** Visiting scientist from University of Colorado.

Using the pseudoshear-mode 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.

We converted the above pseudoshear-mode apparatus to a pure-torsion apparatus. We expect to make creep-recovery measurements using this deformation mode.

We measured the elastic constants and internal friction of graphite-fiber epoxy-matrix composites with several layup geometries: 0°, 0° - 90°, 0° - 45°, 0° - 30° - 60°. Companion modeling studies are in progress.

Publications

1. M. Dunn, "Viscoelastic damping of particle-reinforced and fiber reinforced composites", J. Acoust. Soc. Amer. 98 (1995) 3360.

2. M. Dunn and H. Ledbetter, "Acoustic charcterization of short-fiber composites," manuscript in preparation.

3. M. Dunn and H. Ledbetter, "Elastic moduli of composites reinforced by multiphase particles", J. Appl. Mech. 62 (1995) 1023.

4. M. Dunn and H. Ledbetter, "Elastic-plastic behavior of textured short-fiber composites", Acta Materialia, forthcoming.

5. M. Dunn and H. Ledbetter, "Estimation of the orientation distribution of short-fiber composites using ultrasonic velocities", J. Acoust. Soc.Am. 99 (1996) 283.

6. M. Dunn and H. Ledbetter, "Ultrasonic characterization of the orientation distribution of short-fiber composites", Proceedings 27th SAMPE Techn. Conf. (SAMPE, 1995) 150.

7. M. Dunn, H. Ledbetter, P. Heyliger, and C.-S. Choi, "Elastic constants of textured short-fiber composites", J. Mech. Phys. Solids 44 (1996) 1509.

8. M. Dunn, H. Ledbetter and Z. Li, "Thermal expansion of textured short-fiber composites," manuscript in preparation.

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10. K.W. Hollman, C.M. Fortunko and D.W. Fitting, Through-thickness elastic constant for aramid-epoxy/aluminum composite materials, Review of Progress in QNDE, 1996, Brunswick, ME, eds: D. O. Thompson and D.E. Chimenti, (Plenum Press, New York, NY).

11. K.W. Hollman, C.M. Fortunko, and D.W. Fitting, Ultrasonic characterization of aramid-epoxy/aluminum laminates form pulse-echo data, 1996 IEEE Ultrasonics Symp. Proc., San Antonio, TX, eds: M. Levy, S.C. Schneider, and B.R. McAvoy, (Institute of Electrical and Electronic Engineers, New York, NY).

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15. H. Ledbetter and M. Dunn, "Microstructure, properties, and modeling: Elastic and related properties of two-phase materials," Proceedings ICMFM, Tokyo, Japan, September 1996, forthcoming.

16. H. Ledbetter, M. Dunn, S. Kim, "Internal friction and elastic constants of sintered titanium, Proceedings ICIFUAS-11, J. de Phys., forthcoming.

17. H. Ledbetter, M. Dunn, S. Kim, and R. Fields, "Elastic constants and internal friction of sintered titanium", manuscript complete.

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19. H. Ledbetter, S. Kim. C. Fortunko, and M. Dunn, "Elastic constants and internal friction in sintered titanium," Proceedings ICIFUAS-11, Poitiers, France, July 1996, forthcoming.

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

21. H. Ledbetter, N. Sizova, S. Kim, H. Kobayashi, S. Sgobba, and L. Parrini, "Retardation-relaxation times in anelastic creep-recovery in indium", Proceedings ICIFUAS 11, Poitiers, France, July 1996, forthcoming.

Project: ULTRASONIC STANDARDS FOR MATERIALS CHARACTERIZATION

MSEL Program: Ultrasonic Characterization of Materials

Principal Investigators: C.M. Fortunko
H. Ledbetter

Technical Objectives

(1) Develop the ultrasonic measurement methods, instrumentation, and models for quantitative characterization of materials microstructures, and (2) develop the standard reference materials and calibration methods need to evaluate ultrasonic measurements used in process control and quality assurance. For example, elastic-wave velocities can be measured with 1% accuracy. Many industrial and scientific applications require 0.1% accuracy, and higher.

Technical Description

Advanced materials have microstructures that are designed and controlled to provide superior properties and improved performance for specific functions. To consistently achieve the desired microstructure, new physical measurements will be needed for process control and quality assurance. To use ultrasonics for materials microstructure characterization, determination of certain mechanical and physical properties, and validation of micromechanical models, absolute elastic-wave velocities must often be measured with better than 1% accuracies. Our goal is 0.1%. Accurate methods are also needed for elastic-wave absorption and nonlinearity determination. In our laboratory, we have already demonstrated the feasibility of such measurements and their enormous potential value for microstructure characterization and validation of micromechanical models.

Measurements that would be enabled by better than 1% accuracy of elastic-wave velocities include: texture, grain or particle size, phase content and internal flaw characteristics, internal strain, and pressure effects and stress relaxation. Accurate measurement of absorption are essential for grain-size and internal-friction determinations.

Fundamentally, the determination of elastic-wave velocities involves the measurement of time delay and acoustic propagation path, or resonance frequency and dimensions of the specimen. Such measurements are typically affected by factors related to details of physical modulation and demodulation of the elastic-wave signals, signal-to-noise performance and resolution of the instrumentation, various geometrical factors related to the shape of the specimen and transducers, signal-processing and data-reduction methods. In our laboratory, we operate several experimental systems that are subject to different experimental errors. Very similar considerations affect absorption measurements. Because of the fundamental importance of such measurements, we are constantly improving the capabilities of our existing measurement approaches and developing new ones.

At the present time, we use Resonant Ultrasound Spectroscopy (RUS) and amplitude spectroscopy for accurate elastic-wave velocity determinations. We also routinely use other techniques: pulse-echo overlap, phase-advance spectroscopy, impulse excitation, and compound mechanical resonator. Currently we use primarily piezoelectric transducers. However, development of noncontact transducers (capacitive, EMAT and laser) and point-transducer methods is also under way.

Our long-term plan includes five elements: (1) Identify and validate stable reference materials with known elastic-wave velocities and elastic constants; (2) systematically study the sources of error of the typical measurement methods and develop models to compensate for such errors; (3) develop suitable standard reference materials and calibration procedures for evaluating the performance of elastic-wave velocity and absorption measurement systems; (4) determine the accuracy of ultrasonic characterazation of microstructural features such as grain size, phase content, texture internal stress; and (5) identify and validate the appropriate measurement models. At the present time we are concentrating our efforts on elements 1, 2, and 3. The idea of a workshop is also under consideration.

External Collaborations

Industrial. Ritec, Quatrosonics.

Academic. Prof. M. Dunn, University of Colorado
Prof. P. Heyliger, Colorado State University
Prof. B. DeFacio, University of Missouri

National Laboratory. Dr. A. Migliori, Los Alamos

Accomplishments (FY96)

The project was started in FY 1996. During this initial phase of the project we concentrated on an experimental comparison of our current experimental systems for elastic-wave and absorption measurement, review of literature and our data-analysis techniques, and identification of candidate materials for fabrication of standard reference specimens.

Currently, the instrumentation industry produces metal artifact block-length standards, which are used in calibration of ultrasonic thickness gages and flaw detectors. However, true elastic-wave velocity standards are not generally available. We have initiated discussions with makers of ultrasonic measurement instrumentation to identify specific needs and collaborations.

Preparations are under way to participate in a workshop at Iowa State University on Model-Based Standards in Nondestructive Evaluation. The workshop will take place in April 1997.

The role of standards in NDE will also be discussed at the European/North-American Validation of NDE Reliability Workshop in Berlin on June 18-20, 1997. NIST (C.M. Fortunko) and BAM (Berlin, Germany) are cosponsors of the workshop.

We measured the elastic constants of high-quality monocrystalline silicon whch is part of a multinational standards effort focusing on mass density, average atomic mass, and Avogadro's number.

Publications

1. E.S. Boltz, C.M. Fortunko, Absolute sensitivity limit of various ultrasonic transducers, Proc. IEEE Ultrasonic Symposium, Nov. 1995, forthcoming.

2. E.S. Boltz, V.K. Tewary, C.M. Fortunko, Fidelity of Michelson interferometric and conical piezoelectric ultrasonic transducers, Proc., Rev. Prog. Quant. NDE, Vol 15, forthcoming.

3. H. Ledbetter and M. Dunn, "Microstructure and Elastic Constants of Two-Phase Materials", in Proc. 1995 IEEE Ultrasonics Symp., (IEEE Press, Piscataway, NJ, 1995) pp. 625-634.

4. H. Ledbetter and M. Dunn, "Microstructure Measurements and Modeling: Elastic Properties of Two-Phase Materials", in Proc. Int. Conf. On Microstructure and Functions of Materials (Tokyo, Sept. 1996), in press.

5. H. Ledbetter, S. Kim, and M. Tanaka, Internal friction and elastic constants of monocrystal silicon, manuscript complete.

ELECTRONIC PACKAGING AND INTERCONNECTION

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

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

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

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

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

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

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

More information about this program, and other NIST activities in electronic packaging, interconnection and assembly, is contained in *Electronics Packaging, Interconnection and*

Assembly at NIST: Guide and Resources (WWW link), NISTIR 5817. This publication presents a complete inventory of NIST's activities in this area and lists information on how to contact project leaders.

Project: EXPERIMENTAL MICROMECHANICS BY e-BEAM MOIRÉ

MSEL Program: Electronic Packaging and Interconnects

Principal Investigator: D. T. Read
E. S. Drexler

Technical Objectives

Develop and apply the e-beam moiré technique to measurement of strain and observation of deformation at high magnification. 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, chip-on-glass, and conductive adhesives. Improve the experimental technique to allow writing of more durable, high-contrast gratings so that e-beam moiré may be used to characterize the thermal fatigue behavior of electronic packages. Determine the feasibility of producing higher density gratings to permit the study of finer-scale packaging features. Use the experimental results to characterize failure modes and to verify various modeling approaches.

Technical Description

Failure of electronic packaging is a major source of concern in modern electronics. In this project 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 and crossed-line grating at pitches of 200 nm to 1 μm, using electron-beam lithography, and observing them in the scanning electron microscope at magnifications from 200× to 2000× for line gratings and magnifications of 200× to 500× for cross-line gratings. 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 if single line gratings are used, or complete normal and shear deformations if cross-line gratings are used. Deformations are modeled by boundary-integral methods based on newly developed Green's functions to extract stresses and crack-driving forces.

External Collaborations

Academic. E. S. Drexler is collaborating with J. R. Berger of the Colorado School of Mines on a study to apply improved electron-beam moiré techniques and advanced modeling to determination of deformation modes of conductive adhesives.

Industrial. E. S. Drexler is using the electron-beam moiré technique to evaluate a series of chip-on-glass specimens from 3M, assembled with different curing procedures. The specimens are loaded thermally and the qualities of the anisotropic conductive adhesive cures are compared using electron-beam moiré.

Accomplishments (FY 96)

Electron-beam moiré was used in the study of two isotropic conductive adhesives, one a paste and the other a film. A specimen of each type was loaded mechanically and thermally to observe and measure local deformations. Comparisons were made between the two materials.

An anisotropic conductive adhesive was thermally loaded and moiré patterns observed at regularly spaced intervals along the length of a chip-on-glass specimen. The deformation behaviors for the different locations were analyzed.

Orthogonal (cross-line) gratings were routinely produced using a control box that switches the x and y output. This method allows for a 90° rotation with great (20 s) precision. Crossed-line gratings have been used in thermal and mechanical loading tests, allowing both normal strains and shear strain to be calculated for the first time.

Publications

1. "Error Analysis and Thermal Expansion Measurement with Electron-beam Moiré," by J. R. Berger, E. S. Drexler, and D. T. Read, submitted to *Experimental Mechanics*.

2. "Boundary element analysis of moiré fields in anisotropic materials, " V.K. Tewary and J.R. Berger, J. Comp. Mech., forthcoming.

3. "Theory of Electron Beam Moiré," by D.T. Read and J.W. Dally, J. of Res. of the NIST, Volume 101, Number 1, January - February, 1996, pp. 47-61.

4. "Micro-Strain Distribution Around a Crack Tip by Electron Beam Moiré Methods," by O.S. Lee and D. T. Read, KSME Journal, Volume 9, Number 3, 1995, pp. 298-311.

5. "Thermomechanical Behavior of a High Density Polymer Overlay MCM Interconnect Structure: Experiments and Analysis," D.T. Read, E.S. Drexler, I. Grosse, J. Benoit, J. DiTomasso, E. Barnard, D. Holzhauer, P. Rocci, and M. Stoklosa, in Application of Fracture Mechanics in Electronic Packaging and Materials, ASME EEP-Vol. 11/MD-Vol. 64, edited by T. Y. Wu, W.T. Chen, R.A. Pearson, and D.T. Read, 1995, pp. 251-262.

Project: MECHANICAL BEHAVIOR OF THIN FILMS

MSEL Program: Electronic Packaging and Interconnects

Principal Investigator: D.T. Read
J.M. Phelps

Technical Objectives

Develop experimental techniques to measure the mechanical properties of thin films, including basic tensile properties, fatigue, and fracture resistance. Relate thin-film mechanical behavior to microstructure.

Extend test techniques from their present level (1 μm thick by down to 10 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).

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 physical vapor deposition, 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 vapor-deposited films are of the order of 1 μm thick, the failure loads are of the order of gram-forces, and the specimens cannot be handled directly.

In addition to metallic thin films, ceramic thin films are now being used in advanced commercial products such as sensors and accelerometers. Many of these devices have mechanical functions, and so their mechanical properties are of interest. Specimen designs and experimental techniques are being developed to extend the metal-film measurements to films of polysilicon. From a purely mechanical point of view, the behavior of polysilicon differs from metals in two important respects: polysilicon is much more brittle, and it is stronger.

Anticipated outcome. There are three general types of anticipated outcomes (multiyear) from this work: data; experimental techniques; and standard test methods. Data have been generated during this and previous reporting years. The apparatus and experimental techniques have been developing, but this year the microtensile tester reached a state of development where it was described to and offered to outside organizations. NIST formally decided to offer the concept and design as public-domain information, without a patent. The standard test method remains in the future. The key will be the acceptance by the technical community of the silicon-framed tensile specimen. However, there are several university groups who are having good results with this general type of specimen, so I think the prospects are good.

Technical approach. The main track in the technical approach continues to be to develop techniques to perform tensile tests that are the same in principle as standard macroscopic tensile tests. The key element with thin films is that the specimens are so small and delicate that they cannot be handled directly. Hence a handling strategy must be utilized. The silicon-framed tensile specimen is the answer. This specimen consists of a silicon frame that carries a tensile coupon. The frame is conveniently handled, for attachment to the grips of the testing device and for alignment; then the silicon is cut. This year we started using a dental drill to cut the silicon, and we have had good results.

Although the tester produces data on force and grip displacement, the quantity needed for analysis is the gauge length strain. An electronic speckle pattern interferometry (ESPI) approach has been developed to provide the needed data.

A resonance method for measuring Young's modulus was explored further, but two problems were found: first, this method will work only with very flat specimens, but many thin films bow when released from the substrate; second, the vibrating cantilever must be separated by many micrometers from nearby surfaces such as the substrate. This method remains promising for situations where these problems can be avoided.

It is questionable whether this approach will work for specimens with widths below 10 μm. The specimens may be too delicate. A "skyhook" approach which tests the film *in situ* on the wafer was explored with some success. The idea here is that the specimen is produced on the substrate, and then a sacrificial layer between the specimen and the substrate is removed. At this point the specimen remains firmly attached to the substrate at one end, but only by its corners at the other end. The chip is attached to the moveable grip of the testing machine, to utilize the controlled motion and the force measurement. The "skyhook" reaches in and hooks on to the nearly-free end of the specimen. The moveable grip, carrying the substrate and the other end of the specimen, is driven away from the "skyhook", pulling the specimen to failure. Technical problems to be solved include avoiding stress concentrations and misalignments in the gauge section due to the "skyhook", and isolating the specimen strain from the total displacement of the moving grip.

Scope. In FY 96, tensile tests were conducted on the following thin films: electrodeposited copper from an industry collaborator, normal and ion-beam-assisted-deposition (IBAD) aluminum from a university collaborator, sputtered copper from a National Lab collaborator, electron-beam-evaporated copper produced in house, and polysilicon (polycrystalline silicon) produced at MCNC. The "skyhook" and resonating cantilever techniques were explored on polysilicon specimens.

Special equipment and facilities. The microtensile tester and the associated computer software, the laser and photodetector for the vibrating cantilever technique, and the laser and twelve-bit, extended-area cooled-CCD camera for the ESPI technique are being developed.

External Collaborations

Academic. University of Colorado. D. T. Read is serving on Ph. D. committee of Wan Suwito. Discussions, loan of apparatus, etc. A suggestion that Wan investigate fracture at corners is having a big influence on his work.

University of Michigan. D. T. Read hosted Christine Kalnas for two weeks of tensile testing using our apparatus.

Industrial. Fujitsu Computer Packaging Incorporated, San Jose. D. T. Read tested electro-deposited films and reported preliminary results.

Accomplishments (FY 96)

Results and discussion. Tested the thickest specimen so far, 7 μm thick electrodeposited specimens. No trouble with strength properties, but failed to reach full elongation of 10 percent, compared to 1 percent for electrodeposited films. Improved load resolution of microtensile tester by about a factor of 5. Tested narrowest specimens yet, 10 μm wide polysilicon. Started getting reliable Young's modulus data using ESPI as indicated above. Specimens of copper from four different wafers have given values between 98 and 110 GPa, in contrast with the bulk average value of 128 Gpa. Explored approaches to testing specimens with widths below 10 μm.

Workshops, conferences. "Application of Fracture Mechanics in Electronic Packaging and Materials" symposium of 31 technical papers held at the 1995 ASME International Mechanicanical Engineering Congress and Exposition, November 12-17, 1995, San Francisco, California, chaired by T.Y. Wu and W.T. Chen of IBM, R.A. Pearson of Lehigh University, and D.T. Read.

Technology transfer. Discussed microtensile tester with two potential commercializers.

Technical leadership. Associate Technical Editor of the *Journal of Electronic Packaging*.

Publications

1. "Analysis of fixed-fixed beam structures," by J. C. Marshall, D. T. Read, M. Gaitan, *Microlithography and Metrology in Micromachining II* -- SPIE V. 2880, 1996, pp. 46-55.

2. "Measurements of fracture strength and Young's modulus of surface-micromachined polysilicon", by D. T. Read and J. C. Marshall, *Microlithography and Metrology in Micromachining II* -- SPIE Volume 2880, 1996, pp. 56-63.

3. "Tension-tension fatigue of copper thin films," *Journal of Fatigue* (in press)

4. "Piezo-actuated microtensile test apparatus," *ASTM Journal of Testing and Evaluation* (in press).

Project: THERMAL CONDUCTIVITY OF THIN FILMS

MSEL Program: Electronic Packaging and Interconnects

Principal Investigator: D. R. Smith
F. R. Fickett

Technical Objectives

Develop an apparatus for measuring, by absolute steady-state methods, thermal conductivity of thin metallic films of the dimensions typically used (about one micrometer) in modern large-scale integrated circuits (ICs), for both in-plane and out-of-plane thermal transport. Acquire the requisite technology for manufacturing suitable specimens of semiconductor and metallizations for measuring their heat transport at length scales of the order of about 1 µm. Measure metallization traces and semiconductor elements and determine the separate contributions of interfacial and bulk thermal resistances for the thin metallization films and interfaces between conductive elements. Compare results of the absolute steady-state measurements with the results of transient methods to evaluate the precision and uncertainty of the transient methods.

Technical Description

The use of, and dependence on, modern computers and electronic communication technologies underlies almost every technical and commercial aspect of modern society. Great advantages in increased technical efficiency and reliability of ICs, as well as reduced unit cost, can be achieved by reducing the dimensional scales within the package and on the chip. Typical width scales for elements within ICs are now evolving to significantly less than 1 µm. The advantages of reduced length scales are necessarily accompanied by greater packing density of the individual devices, with attendant increase in density of power (heat) generation. Unless the increased heat production can be transferred from the element to the surrounding environment, the lifetime to failure of critical elements of the package may be severely compromised due to diffusive degradation of semiconductor elements or may suffer outright catastrophic failure.

The reduced size of the metallization and semiconducting elements within present and planned future devices is approaching the size where the classical physics of transport of electrons and phonons begins to break down. That is, within a typical IC the greatly reduced size of the metallization traces, for example, leads to much greater importance of surface effects that could previously be ignored for devices and elements of larger size. The surfaces where new behavior is expected are the free surfaces of the semiconductor or metallization, as well as the interfacial boundary between them. Surface physics can reduce the conduction of electrons along the metallization traces as well as the transport of phonons both along and into or out of the metallization. While much theoretical work has been done to model conductive transport of electrons and phonons in metals and semiconductors, it is not at present useful in predicting such phenomena in solids with small length scales. In the absence of a general theory of solid-state transport applicable to IC elements and devices at the small scales presently used, experimental studies are required to determine the transport properties for specific geometries.

Accurate measurement of thermal conductivity is difficult. Use of absolute steady-state techniques requires that sufficient time elapse for a stable steady-state to be set up. Careful guarding is required to ensure that all (and only) the metered heat flows through the specimen. A third difficulty is related to the other modes of heat transfer: convective and radiative transport. Transient methods determine the thermal diffusivity, from which the conductivity may be calculated. There is on-going debate as to their accuracy. Evaluation of the individual accuracies of such methods is a major objective of the present work, in order to inform the users of the transient methods as to whether the conductivities obtained by their methods are reliable for their purposes.

During FY97 initial, exploratory measurements will be made that will form the basis for design of the more sophisticated second-round devices. The measurement system will be proved and improved, and specimen design features needing improvement will be identified and carefully studied. In the second phase this work will provide data, measurement systems and standards for thermal measurements of thin films.

External Collaborations

In July 1996, D. R. Smith attended a 3-day workshop at MCNC (formerly Microelectromechanical Center of North Carolina) at Research Triangle Park, NC. Attendees were introduced to the principles and practices of using the mature methods used to manufacture integrated circuits for development of electromechanical devices at the 1-μm scale, at least for MCNC's specific technology, Multi-User Micromechanical Processes. MUMPs provides a fixed set of semiconductor, insulating, and metallic layers. Attendees were also introduced to a commercial software package for the design of micromechanical devices, including microbridges needed for the NIST microscale thermal conductivity systems. After returning from the MUMPs workshop the PI acquired the layout-editor software package and designed a suite of prototype devices for measuring thermal conductivity. The device designs were submitted to MCNC on 11 November 1996 for their MUMPs-16 device manufacturing run (one run is offered every other month).

Accomplishments (FY96)

A comprehensive review has been made of the literature to find all apparatus that claim to allow measurement of thermal conductivity of thin films. The devices have been evaluated in detail and it was determined that there are no actual steady state thermal conductivity apparatus available in this size range. An initial design of such an apparatus was developed that could be fabricated by relatively conventional large-scale integration (LSI) processing methods.

Project: STRESS VOIDING AND ELECTROMIGRATION

MSEL Program: Electronic Packaging and Interconnects

Principal Investigator: R. R. Keller

Technical Objectives

Develop a mechanistic understanding of the microstructural processes controlling stress voiding and electromigration in interconnect structures.

Perform detailed characterizations of the microstructures of interconnects that undergo thermal- and current-induced stressing, using advanced electron microscopy techniques. Correlate local variations in microstructure to differences in stress voiding and electromigration behavior. Assess and, when necessary, modify microstructurally-based models that sufficiently describe similar behavior in bulk metals.

Technical Description

Stress voiding and electromigration are failure phenomena that presently limit the reliability of narrow interconnects. They occur during thermal- and electrical current-induced stressing, respectively. The end result is the formation and growth of voids in the metal due to the development of severe triaxial tensile stresses; such stresses result from differential thermal expansion among the metal, substrate and rigid passivation overlayer. Their impact is projected to worsen as the dimensions of interconnect structures continue to scale downward, unless a more complete mechanistic understanding of the phenomena is developed. Interconnects become less homogeneous as dimensions scale downward, since the structures are then composed of individual grains through the film thickness and across the line width. Behavior also becomes less homogeneous, and even small variations in microstructure can have detrimental effects on reliability. Understanding and solving the problems of void formation and growth at the microstructural level is essential to the continued development of metallizations on a submicron scale, as specifically identified in the 1994 SIA Roadmap.

Electron microscopy and, in particular, electron diffraction techniques are used to quantitatively characterize on a local scale the microstructures of narrow metallizations for interconnects. Backscatter Kikuchi diffraction in the SEM and convergent beam electron diffraction in the TEM are the primary measurement techniques. Specifically, variations in microtextures, grain boundary structures, dislocation configurations, and lattice parameters are measured and related to the observed void behavior. The results are interpreted in terms of both the energetics and kinetics of void formation and growth. An orientation imaging microscopy system for acquiring and analyzing backscatter Kikuchi diffraction patterns is used for determinations of microtextures and grain boundary structures. Scanning transmission electron microscopy and a cold stage for acquiring convergent-beam electron diffraction patterns are used to determine local lattice parameters which are used in assessing residual strains and stresses.

External Collaborations

Academic. R. Keller is collaborating with J. Nucci and Y. Shacham-Diamand of Cornell University in a study of stress voiding and thin film microstructures in copper films used as conductors in microelectronic structures.

R. Keller is collaborating with J. Sanchez, Jr. of the University of Michigan in a study of stress voiding in copper films used as conductors in microelectronic structures.

Industrial. R. Keller is collaborating with G. Alers of Lucent Technologies in a study of stress voiding, electromigration and internal stresses in aluminum alloy films used as conductors in microelectronic structures.

R. Keller is collaborating with D. Field of TexSEM Laboratories in a study of stress voiding and thin film microstructures in copper films used as conductors in microelectronic structures.

Accomplishments (FY96)

Performed analysis of approximately 300 backscatter Kikuchi diffraction patterns obtained from narrow copper metallizations which had undergone stress voiding. Reported results in the forms of standard pole figures, orientation distribution functions, and misorientation angle distributions. Identified distinctions between data associated with regions of interconnects that remained intact after thermal stressing and those associated with regions immediately adjacent to voids. The results suggested that there can exist significant local variations in microstructure within thin film interconnects. Those regions associated with void formation and growth were composed of microtextures and grain boundary structures indicative of higher local diffusivities. The importance of the available kinetic pathways for void growth was suggested. This is, to our knowledge, the first known local crystallographic characterization of the microstructures associated with voiding in copper interconnects.

Began the process of developing a TEM test specimen preparation procedure for narrow aluminum alloy interconnect structures subjected to stress voiding and electromigration.

Workshops, conferences. R. R. Keller is co-organizing and co-editing the Materials Research Society symposium "Materials Reliability in Microelectronics VII," in preparation for the 1997 MRS Spring Meeting.

Publications

1. R.R. Keller, J.M. Phelps, and D.T. Read, “Tensile and Fracture Behavior of Free-Standing Copper Films,” Materials Science and Engineering A214, 42-52 (1996).

2. J.A. Nucci, R.R. Keller, J.E. Sanchez, Jr., and Y. Shacham-Diamand, “Local Crystallographic Texture and Voiding in Passivated Copper Interconnects,” Applied Physics Letters 69, 4017-4019 (1996).

3. J.A. Nucci, R.R. Keller, and Y. Shacham-Diamand, “Effects of Grain Boundary Character on Stress-Induced Voiding in Passivated Copper Interconnects,” Electronic Proceedings for TECHCON 96, Semiconductor Research Corporation (1996).

4. D.P. Field, R.R. Keller, and J.A. Nucci, “Interconnect Failure Dependence on Crystallographic Texture,” Proceedings of 22nd International Symposium for Testing and Failure Analysis, ASM, 351-355 (1996).

5. J.A. Nucci, R.R. Keller, D.P. Field, and Y. Shacham-Diamand, “Grain Boundary Misorientation Angles and Stress-InducedVoiding in Oxide Passivated Copper Interconnects,” Applied Physics Letters, in press (to appear March 1997).

CERAMIC COATINGS

The Coatings program is a measurement and characterization effort which addresses the processing reproducibility and performance prediction issues associated with, primarily, thermal-spray deposited ceramic coatings. The program focuses on plasma-spray-deposited ceramic thermal barrier coatings used in aircraft gas turbines and expected to be used in land-based turbines and diesel engines. Sales in the thermal-spray industry are currently valued at over one billion dollars annually, a significant portion of which is ceramic thermal-barrier coatings. Collaborations have been established with industrial organizations including Pratt and Whitney, General Electric, Caterpillar, METCO, MetTech and Zircoa as well as the Thermal Spray Laboratory at the State University of New York at Stony Brook and the Thermal Spray Laboratory at Sandia National Laboratory. The program includes collaboration with the National Aerospace Laboratory and the National Mechanical Engineering Laboratory, both in Japan, to examine functionally gradient materials. Research is also conducted on the processing and properties of chemical-vapor-deposited (CVD) diamond films in collaboration with Westinghouse, an Advanced Technology Program (ATP) awardee, and on Physical Vapor Deposited (PVD) ceramic coatings in collaboration with Praxair, another ATP awardee.

Participants in the NIST program are located in the Ceramics, Materials Reliability, and Reactor Radiation Divisions of the Materials Science and Engineering Laboratory as well as in the Chemical Science and Technology Laboratory.

The approach taken in the plasma-spray (PS) research has been to build on the analytical capabilities at NIST and the material processing capabilities of collaborators. The program has the following elements:

- development of techniques for characterization of physical and chemical properties of stabilized-zirconia feedstock to provide data for increased processing reproducibility as well as data required for production of a Standard Reference Material suitable for calibration of light-scattering size-distribution instruments used in industry for analysis of PS powder;

- development of scattering techniques to determine the quantity, size and orientation of porosity and microcracks in PS ceramic coatings suitable for use in modeling the thermomechanical behavior of these materials;

- development of methods to measure chemical, elastic modulus, and thermal properties on a scale suitable for use in microstructural models of behavior;

- development of techniques to model thermomechanical behavior of thermal-barrier coatings to enable more reliable performance prediction; and

- development of techniques for accurate measurement of the thermal conductivity of PS coatings, by use of the guarded-hot-plate technique suitable for incorporation in ASTM standards and by the pulsed laser-heating technique, to provide a method for comparison with routine industrial techniques.

- development and refinement of more sensitive methods for accurate analysis of oxide phases and residual stresses which affect performance and durability of coatings.

Research on chemical mapping of powders and microstructures is conducted in the Microanalysis Division of the Chemical Science and Technology Laboratory. Thermal property research is conducted in the Materials Reliability and Metallurgy Divisions, and the Reactor Radiation Division participates in both the powder analysis and scattering projects. A strong attribute of the PS coatings research is the use of common materials for which complementary data can provide a more complete understanding of processing-microstructure-property relationships.

Project: THERMAL CONDUCTIVITY OF THERMAL BARRIER COATINGS

MSEL Program: Ceramic Coatings

Principal Investigator: A.J. Slifka
J.M. Phelps, B.J. Filla, F.R. Fickett

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 worker to determine bulk values and interfacial resistances for the coating systems. Evaluate microscopy and spectroscopy techniques for the various microstructural analyses. Observe the effects of varying processing parameters and processing techniques on microstructural features and bulk thermal conductivity. Model bulk thermal conductivity using microstructural information and processing parameters which will result in a model that generates desired bulk thermal conductivity given appropriate processing parameters. Interact with the coatings industry to ensure 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 industry.

Technical Description

Accurate knowledge of the thermal conductivity of thermal barrier coatings and its relationship to processing parameters is necessary in order to more economically produce coatings 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 superalloy 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 electron-microscopy techniques to characterize the microstructure and microchemistry responsible for the bulk thermal performance of coating systems.

Standard reference materials will be developed that relate the thermal conductivity of various classes of ceramic coatings to the thermal diffusivity. Industry commonly measures thermal diffusivity because it is a relatively fast, user-friendly measurement. Models of the heat flow in complex coatings will be developed.

An absolute, steady-state measurement of thermal conductivity is used in order to obtain the thermal conductivity of coatings with the greatest accuracy and reliability possible. A modified guarded hot plate has been constructed for these measurements. In addition, infrared microscopy will be used to monitor heat flow on the micrometer scale and to measure the thermal conductivity of coating constituents and the thermal resistance of constituent interfaces. Modelling of the heat flow in coatings will be done using input from the infrared microscope measurements that generates bulk thermal conductivity values as obtained from the guarded hot plate.

Special facilities used include an infrared microscope system capable of 5 micrometer spatial resolution from room temperature to 500 K and the world's only guarded-hot-plate thermal conductivity apparatus operating from 400 to 1200 K.

External Collaborations

Academic. A. Slifka has made arrangements to collaborate with K. Barmak of Lehigh University in determining thermal conductivity of developmental FGMs.

Industrial. A. J. Slifka and J. M. Phelps are collaborating with Brad Beardsley of Caterpillar on measurement of thermal conductivity of FGMs using our absolute, steady-state technique and comparing with a transient, laser-flash method.

A. J. Slifka and B. J. Filla are measuring thermal conductivity of very thin monolithic coatings and layered coatings from Pratt & Whitney under a CRADA.

Government agencies/laboratories. A. J. Slifka is collaborating with B.H. Rabin of the Idaho National Engineering Laboratory in determining microscale thermal conductivity of developmental FGMs.

A J. Slifka is collaborating with U. Leushake and W.A. Kaysser of the German Aerospace Research Establishment in determining bulk and microscale thermal conductivity of developmental coatings and FGMs.

The entire project staff is collaborating with Dr. A. Kumakawa of NAL/KRC (Japan) by evaluating microstructure and microchemical composition and measuring thermal conductivity of FGMs prepared by Nippon Steel.

Accomplishments (FY96)

Completed bulk thermal conductivity measurements on atmospheric-plasma-spray coatings from SUNY. Measured the thermal conductivity of a nickel superalloy material, PWA 1484, which is the substrate material for the coatings to be supplied soon by Pratt & Whitney. Began testing on MgO, which is a good model material for high thermal conductivity. This monolithic material will be used to define the performance of the bulk thermal conductivity apparatus at the high-thermal-conductivity end. Two functionally graded specimens from Nippon Steel Corp. were tested. These NiCr / 8YSZ specimens were tested as part of our collaboration with NAL/KRC. We did multiple tests of each of these low-pressure-plasma-sprayed coatings to determine the effect of thermal shock on the thermal conductivity of these coatings. Continued to refine our understanding of the effect of surface finish on the interfacial thermal resistance between coatings and measurement plates.

Determined the microstructure of coatings using electron microscopy techniques. Used energy- and wavelength-dispersive spectroscopies to determine microchemical content and distribution of chemical species for a number of specimens.

Continued development of an infrared microscopy system to evaluate micrometer-scale thermal behavior of the constituents of the coatings. Calibration allows us to account for variations in emissivity among coating constituents and quantitative measurements of thermal conductivity are being attempted.

Publications

1. "The Effect of Thermal Shock on the Thermal Conductivity of a Functionally Graded Material," A.J. Slifka, A. Kumakawa, B.J. Filla, J.M. Phelps and N. Shimoda, submitted to *Proceedings of the FGM 96 Conference: Transactions of the Materials Research Society of Japan.*

2. "Thermal Conductivity of a Zirconia Thermal Barrier Coating," A.J. Slifka, B.J. Filla, J.M. Phelps, G. Bancke, and C.C. Berndt, submitted to the *ASM Journal of Thermal Spray Technology.*

MAGNETIC MATERIALS

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

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

Areas of present study include the following:

- processing of magnetic multilayers for optimal giant magnetoresistance effects
- measurement and modeling of the enhanced magnetocaloric effect in nanocomposites
- observation and micromagnetic modeling of magnetic domains for understanding magnetization statics and dynamics in advanced and conventional materials
- nanotribology of magnetic hard disk; measurement of stiction, friction, and wear at the nanometer scale
- development of magnetic sensors of mechanical properties for incorporation as *in situ* controls in a steel mill

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

- development of a magnetoresistance microscope for micrometer-scale magnetic measurements in industrial environments

- a study of magnetic signatures of engineered surfaces such as those created by ion implantation of ferrous materials

By experimentally addressing important issues in magnetism, by bringing together the industrial and scientific communities through the organization of workshops and conferences in the area, and by the development and preparation of appropriate standards, NIST acts to accelerate the utilization of advanced magnetic materials by the industrial sector, and to enable industry to take advantage of new discoveries and innovations. In addition, close linkage with the national storage industry consortium (NSIC) which consists of 38 companies and a score of universities allows industrial relevance and partnership. Additional collaborations with Xerox, General Motors, Hewlett Packard, IBM, Seagate, and Motorola Corporations, for example, enable NIST to leverage its activities with the much larger, but complementary, capabilities of other organizations.

Project: MAGNETIC SENSING FOR MICROSTRUCTURAL CHARACTERIZATION

MSEL Program: Magnetic Materials

Principal Investigator: F. R. Fickett
W. P. Dubé

Technical Objectives

Develop magnetic techniques for nondestructive microstructural characterization over the range from hundreds of micrometers to tens of nanometers and apply these to films, coatings, and engineered surfaces. Determine the extent to which magnetic techniques offer a microstructural analysis capability not otherwise available, either in terms of detection ability or simplicity of application. Investigate creation of a new class of magnetic mapping sensors created by modification of existing and developmental recording-head systems based on anisotropic magnetoresistance (AMR) and giant magnetoresistance (GMR). Study application of these sensors to characterization of materials and engineered surfaces. Develop a scanning system (magnetoresistive microscope-MRM) using these sensors and determine its suitability for use in manufacturing environments. Observe and evaluate scanning system potential for use in evaluation of current-flow path problems in interconnects and on-chip electrical circuits. In all these areas, evaluate applications of magnetic force microscopy (MFM), which allows measurement on yet a smaller scale, and acquire the capability to make these measurements.

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 project makes them ideal not only for the applications mentioned above, but also for flaw detection in magnetic media and other magnetic structures associated with data storage as well as for more general metallic-microstructure analysis. The possibility of magnetic mapping of operating high-frequency microcircuits also exists, leading to possible on-chip nondestructive evaluation.

This project is anticipated to result in a wide-ranging micromagnetic sensing capability for the Division. This measurement technology will be applied to research in evaluation of thin film and surface-modified microstructures in fields ranging from ion-implanted surfaces for heavy industry to electronic packaging and applications of thin-film magnetic structures in data storage systems.

We are concentrating on microscopic sensors based on giant magneto-resistance (GMR) for mapping of near-surface fields for regions in the 1-100 μm range. 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. Over the next year, 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.

In FY96 a prototype scanning MRM system was developed and applied successfully to a few magnetic-media measurements for equipment testing purposes. The complete software and instrument control package is still under development. We are evaluating ion-implantation-induced microstructure, focusing on determination of surface uniformity and depth distribution of the implanted region. We are also investigating the potential for measurement of nonmagnetic coatings on magnetic substrates.

A scanned probe microscope (SPM) was procured for the magnetics laboratory and was delivered in October (FY97). Its main application in this project will be in magnetic-force microscopy (MFM), but it is a Division resource for atomic-force microscopy (AFM), scanning tunneling microscopy (STM), and a number of more exotic SPM modes.

External Collaborations

Academic. A collaboration on magnetic measurements of ion-implanted stainless steel with Colorado State University and Colorado School of Mines has been initiated. Preliminary MFM measurements have been made on a few samples.

Industrial. Interactions with producers of implanted materials in the ATP project *Plasma-Based Processing of Lightweight Materials for Motor-Vehicle Components and Manufacturing Applications* (Environmental Research Institute of Michigan) are underway. GM has promised to deliver implanted samples of chrome-plated steel late in 1996.

Government agencies/laboratories. Also related to the ATP project above, Fred Fickett visited Carter Munson at Los Alamos late in FY96. Los Alamos has since delivered several test samples of implanted tool steel.

Accomplishments (FY 96)

A computer controlled magnetoresistive microscope (MRM) was developed using "commercial" recording heads integrated into a three-axis scanned-probe system based on commercial nanomovers. The heads are very difficult to obtain in quantities less than 100,000 and they thus remain a major problem with this device because they are quite sensitive to electrostatic discharges, and a given head does not have a very lengthy lifetime. We are pursuing a collaboration with EEEL that will allow us to make our own, more rugged, sensors for this system in the future. Initial tests of the system indicated that it has the expected magnetic detection capabilities, but the transportable system is still some distance in the future.

Tests were made on ion-implanted stainless steel samples developed in a collaboration with local universities that show the formation of strongly magnetic surface structures on the essentially nonmagnetic substrate. The stainless steel measurements required a magnetic-force microscope (MFM), since the domains were very small, on the order of tenths of a micrometer. Last-minute preliminary measurements were made on two LANL samples of ion implanted H13 steel using the MFM. They show possible magnetic signatures, but meaningful analysis requires measurements of unimplanted surfaces and samples with a wider range of implantation parameters.

STANDARD REFERENCE MATERIALS

The NIST Standard Reference Materials Program serves as the nation's primary source of reference standards used to develop accurate methods of analysis, calibrate measurement systems and assure the long-term adequacy of measurement assurance programs. The aim is to assist industry, science, and academia to achieve the level of product conformance and measurement quality required for national and international commerce and trade.

As the world commerce and trade markets have become more global, Standard Reference Materials (SRMs) have become more important. All data derived from measurements in which SRMs are part of the measurement system have the capability of being traceable to a common and recognized set of standards and, consequently, data compatibility can be realized.

The technical staff of the Materials Science and Engineering Laboratory produces a series of standards for materials suppliers and users that are key elements in assisting the industry to develop and/or improve its competitive edge in the global arena. MSEL designs, develops and produces many SRMs related to ceramics, polymers, metals and related materials. These SRMs are routinely employed in the production and processing of materials. Many projects are conducted in cooperation with applicable industries and are an integral part of the Laboratory's research efforts.

Typical SRMs address chemical composition of specific materials, particle size distribution and x-ray diffraction parameters for instrument calibration, and reference properties such as fracture toughness and hardness.

SRMs are sometimes developed to complement standard analytical methods such as those developed by consensus through the ASTM.

Project: CHARPY IMPACT TESTING

MSEL Program: Standard Reference Materials

Investigators: D.P. Vigliotti
C.N. McCowan, T.A. Siewert

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.

Technical Description

The Charpy impact test uses a swinging hammer and calibrated scale, and encoder or integrated signal from an instrumented striker 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.

Our special facilities include the three master Charpy impact machines (all roughly 300 J capacity). These three machines are used to establish reference energies for the NIST reference materials sold through the Standard Reference Materials Program Office. In addition, we have several more machines (3 to 400 J capacity) that are used for research purposes.

Accomplishments (FY96)

We had 787 customers for this service in FY96, 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 170 faxes and numerous phone calls. In our laboratory, we tested the 700 specimens necessary to confirm that seven new lots of reference specimens were suitable to go into the SRM inventory.

In cooperation with the NIST Standard Reference Materials Program and The American Society for Testing and Materials, we hosted a workshop in Norfolk, Virginia during November 1995. This workshop, Materials and Heat Treatments for ASTM Charpy V-notch Verification Specimens, sought new ideas to improve the verification specimens offered through the Standard Reference Materials Program. The participants identified several new research projects that might improve the performance of the NIST reference materials. In the following months, we coordinated the machining and heat treating tasks among the volunteer organizations: Timken, Teledyne-Vasco, Sure Tool, and Thomas Shearer Inc. In addition, Bob Gassner volunteered to perform some ultrasonic measurements of the round-robin specimens to supplement ultrasonic measurements being performed by researchers at NIST. These ultrasonic measurements are being cross correlated to hardness and impact energy measurements for a better understanding of the microstructural unifo

MATERIALS RELIABILITY DIVISION

Harry I. McHenry, Chief
(303) 497-3268

Thomas A. Siewert, Deputy
(303) 497-3523

Joyce E. Smith, Administrative Officer
(303) 497-7380

Group Leaders

Materials Characterization
Christopher M. Fortunko
(303) 497-3062

Process Sensing and Modeling
George A. Alers
(303) 497-7899

Structural Materials
Thomas A. Siewert
(303) 497-3523

Materials Evaluation
Fred R. Fickett
(303) 497-3785

RESEARCH STAFF

Alers, George A.
- Nondestructive evaluation
- Electromagnetic acoustic transducers
- Process sensing and monitoring

Boltz, Eric S. (resigned 6/96)
- Ultrasonic NDE
- Elastic wave propagation
- Optical measurements

Cheng, Yi-Wen
- High temperature mechanical behavior
- Thermomechanical processing of steels
- Ferrous metallurgy

Clark, Alfred V., Jr.
- Ultrasonic NDE
- Engineering mechanics
- Residual stress measurements

Clerc, Daryl G.
- Elastic constants by *ab initio* electronic structure theory
- Theory and measurement of elastic constants
- Hardness fundamentals and correlations

Drexler, Elizabeth S.
- Electron-beam moire
- Electronic packaging
- Micromechanical property measurement

Dubé, William P.
- X-ray diffraction
- Ultrasonics
- Electromechanical instruments

Fickett, Fred R.
- Materials evaluation
- Magnetic sensors
- Electronic processing

Filla, James B.
- Thermal conductivity
- High temperature measurements
- Robotics

Fitting, Dale W.
- Sensor arrays for NDE
- Ultrasonic and radiographic NDE
- NDE of composites

Fortunko, Christopher M.
- Ultrasonic transducers and instrumentation
- Nondestructive evaluation
- Analog measurements

Hamstad, Marvin A.
- Acoustic emission
- Composite materials
- Nondestructive evaluation

Hollman, Kyle W.
- Ultrasonic measurements
- Determination of elastic coefficients
- Nondestructive evaluation

Hurley, Donna C.
- Physical measurements
- Nonlinear ultrasonics
- Solid state physics

Igarashi, Brian
- Ultrasonic measurements
- Magnetostriction
- Solid state physics

Johnson, Ward L.
- Ultrasonic measurements
- Internal friction
- Process sensing

Keller, Robert R.

- Materials science
- Electron microscopy
- Mechanical behavior

Kim, Sudook

- Elastic properties
- Low-temperature physical properties
- Ultrasonic measurements

Ledbetter, Hassel

- Physical properties of solids
- Theory and measurement of elastic constants
- Martensite-transformation theory

Madigan, R. Bruce

- Welding engineering
- Welding process sensors
- Process control

McColskey, J. David

- Acoustic emission
- Composite materials
- Mechanical testing

McCowan, Chris N.

- Welding metallurgy
- Charpy impact testing
- Metallography and fractography

McHenry, Harry I.

- Fracture mechanics
- Materials processing
- Fracture control

Phelps, John M.

- Electron microscopy
- Chemical microanalysis
- Compositional mapping

Purtscher, Patrick T.
- Fracture properties of metals
- Metallography and fractography
- Ferrous metallurgy

Quinn, Timothy P.
- Control theory
- Welding automation
- Process modeling

Read, David T.
- Electronic packaging
- Elastic-plastic fracture mechanics
- Mechanical behavior of thin films

Schaps, Stephen R.
- Electrical design
- Nondestructive evaluation
- Sensor systems integration

Schramm, Raymond E.
- Ultrasonic NDE of welds
- Ultrasonic measurement of residual stress
- Electromagnetic acoustic transducers

Siewert, Thomas A.
- Welding metallurgy of steel
- Gas-metal interactions during welding
- Welding database management

Slifka, Andrew J.
- Thermal conductivity
- Thermal barrier coatings
- Surface characterization

Smith, David R.
- Thermal conductivity
- Thermal expansion
- Low-temperature physics

Tewary, Vinod K.

- Solid state physics
- Green's function methods
- Elastic wave propagation

Tobler, Ralph L.

- Fracture mechanics
- Material properties at low temperatures
- Low-temperature test standards

Vigliotti, Daniel P.

- Charpy impact testing
- Standard reference materials
- Fabrication technology

Yukawa, Sumio

- Fracture mechanics
- Codes and standards
- Structural safety

APPENDIX

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

D.G. Clerc
M.A. Hamstad
K.W. Hollman
D.C. Hurley
S.A. Kim
H.M. Ledbetter
J.D. McColskey
M.C. Renken
R.L. Santoyo
V.K. Tewary
D. Balzar(GR)

.06 Process Sensing & Modeling
G.A. Alers

.07 Structural Materials
T.A. Siewert

V.E. Ciaranello

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

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

.08 Materials Evaluation
F.R. Fickett

S.J. Barnhill

E.S. Drexler
R.R. Keller
J.M. Phelps
D.T. Read
A.J. Slifka
D.R. Smith

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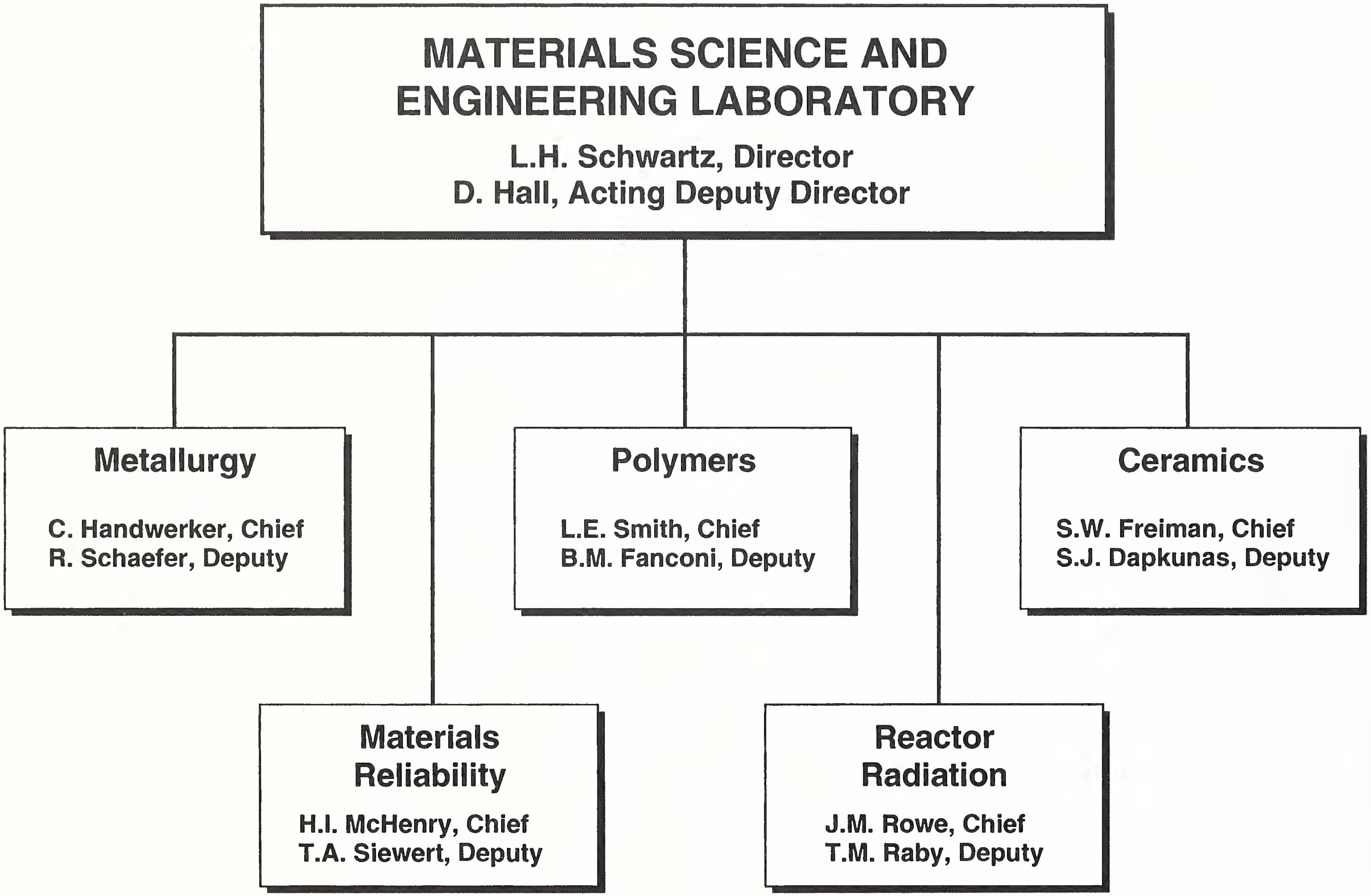
MATERIALS SCIENCE AND ENGINEERING LABORATORY
L.H. Schwartz, Director
D. Hall, Acting Deputy Director
Metallurgy
C. Handwerker, Chief
R. Schaefer, Deputy
Materials Reliability
H.I. McHenry, Chief
T.A. Siewert, Deputy
Polymers
L.E. Smith, Chief
B.M. Fanconi, Deputy
Reactor Radiation
J.M. Rowe, Chief
T.M. Raby, Deputy
Ceramics
S.W. Freiman, Chief
S.J. Dapkunas, Deputy

National Institute of Standards and Technology

Organizational Chart