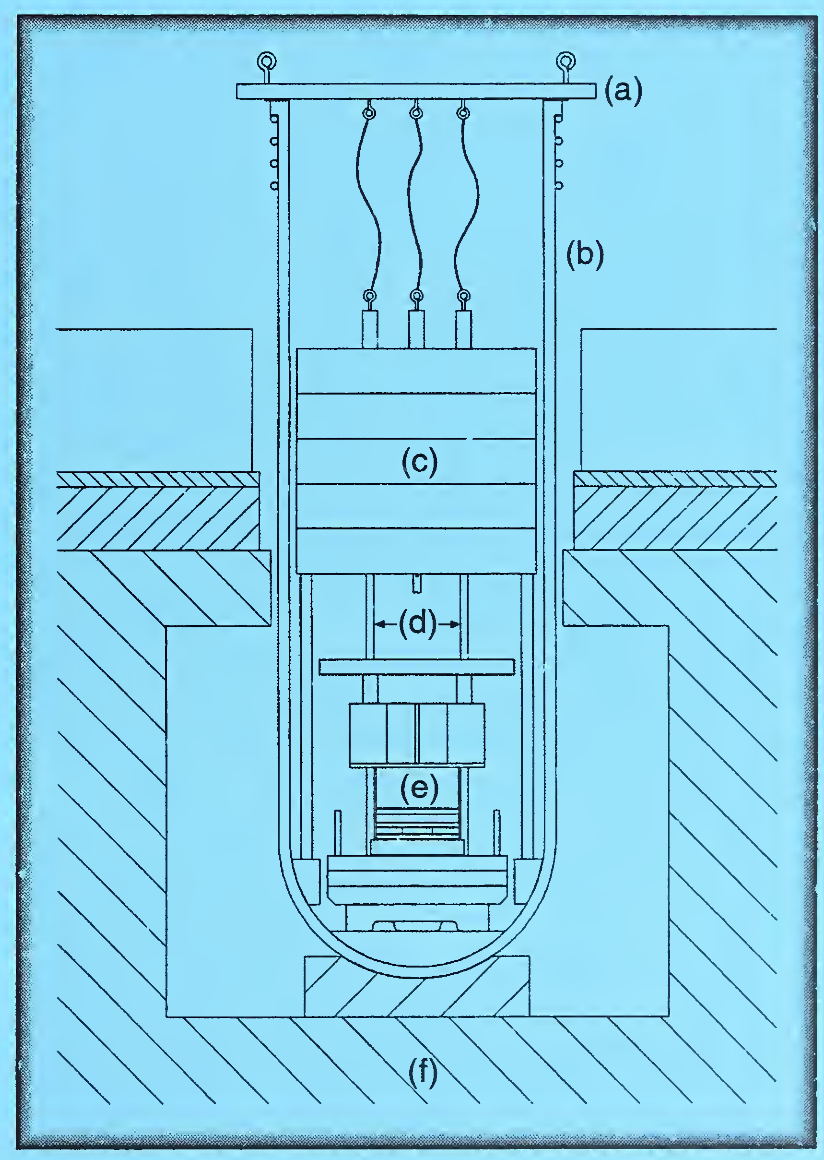
MSEL

Materials Science and Engineering Laboratory

MATERIALS RELIABILITY



NISTIR 6064
U.S. Department of Commerce
Technology Administration
National Institute of Standards
and Technology

Technical Activities
1997

High Temperature Guarded Hot Plate

The high temperature guarded hot plate depicted on the cover is used to measure the thermal conductivity of ceramics and ceramic coatings. With the collaboration of industrial partners in the gas turbine and aerospace industries, this unique apparatus is used to provide baseline measurements on state-of-the-art thermal barrier coatings and to develop Standard Reference Materials (SRMs). Features of the apparatus are as follows: (a) cover plate, (b) ceramic retort, (c) insulation plug, (d) alumina support rods, (e) main stack, and (f) furnace.

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

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U.S. DEPARTMENT OF COMMERCE
William M. Daley, Secretary
TECHNOLOGY ADMINISTRATION
Gary Bachula, Acting Under Secretary for Technology
NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Robert E. Hebner, Acting 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: 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.

The Division works closely with industry and standards bodies to identify the measurements and standards needs of the materials community. In FY97, we placed increased emphasis on disseminating the results of our research in the form of standards. This represents a change from prior years when we emphasized the transfer of our measurement technologies to specific CRADA (Cooperative R&D Agreements) partners.

For ultrasonic characterization of materials, we are working with the American Society for Testing Materials and International Standards Organization to provide traceability to NIST for the ultrasonic test blocks currently used to calibrate inspection instruments. For welding process control, a Standard Reference Materials (SRM) project was initiated for NIST to take responsibility for the stainless steel weld metal secondary standards used worldwide. For electronics packaging, test structures are being developed in collaboration with the Electronics and Electrical Engineering Laboratory for electromigration, microelectromechanical systems and thermal conductivity measurements.

During FY97, the Division cosponsored several meetings intended to assess the measurements and standards needs of the materials community:

1. Workshop on NDE Standards and Modeling, with the Center for Nondestructive Evaluation, Iowa State University, in Ames Iowa, April 1997.

2. European-American Workshop in Reliability and Validation of NDE Methods, with the German Federal Institute for Materials Testing (BAM), in Berlin, June 1997.

3. International Symposium on Nondestructive Characterization of Materials, with the Center for Nondestructive Characterization, Johns Hopkins University, in Boulder, Colorado, June 1997.

4. International Conference on Computer Technology in Welding, with the International Institute of Welding, in San Francisco, July 1997.

An opportunity to apply the Division's measurement capabilities to the solution of an important national problem came in FY97. Shirley Jackson, Chairman of the Nuclear Regulatory Commission (NRC), wants to directly measure radiation damage in reactor pressure vessels instead of monitoring damage with surveillance specimens. The NRC initiated a study by the Division to assess the feasibility of using ultrasonic and magnetic measurements to sense the microstructural changes that cause radiation embrittlement. The feasibility study started in May 1997 and we expect to follow it with a multiyear measurements and standards program intended to enable reactor-life extensions worth billions of dollars.

This report summarizes the technical activities of the Materials Reliability Division during fiscal year 1997. The technical activities are organized according to programs which have been defined and organized by MSEL. The Division has twenty research projects, which contribute to five 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 Measurement 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 two projects that are part of the NIST Standard Reference Materials (SRM) Program.

Selected accomplishments are listed below to highlight the technical activities of the Materials Reliability Division.

Intelligent Welding: As part of a CRADA with Tower Automotive, we evaluated the applicability of the NIST arc sensor module to production welds. The sensors record from 82 production welds (containing 4 defective welds) were used to tune the sensor quality thresholds. Another 444 welds were monitored using these thresholds. In total, the sensor flagged 5 of the 6 defective welds produced during these trials, and it did not flag any defect-free welds.

Directional Solidification Sensor: Working with the Aerospace Casting Consortium, we measured the width of the mushy zone during directional solidification of turbine-blade castings. The width varied from 10 to 20 mm for the casting conditions evaluated. The results are being used for refining and validating the casting process model.

Thermomechanical Processing: The American Iron and Steel Institute is a sponsoring research project entitled, "Microstructural Engineering for Hot-Strip Mills." As a part of this project, structural-property relationships for hot-rolled steels have been incorporated into the process-model for hot-strip rolling being developed with the University of British Columbia. The equations and the process model have been disseminated to AISI member companies for evaluation.

Ultrasonic Sensing of Dislocation Dynamics: A noncontacting ultrasonic resonance system was incorporated with a tensile testing machine, enabling real-time high-resolution measurements of damping and velocity changes produced by static and dynamic loading of cylindrical test specimens. This system has been used to study changes in dislocation pinning that result from cyclic fatigue and heat treatment of steels.

Nondestructive Characterization of Embrittlement: The Nuclear Regulatory Commission initiated study to assess the feasibility of using physical-property measurements for evaluating radiation embrittlement in reactor-pressure vessel (RPV) steels. Nonlinear ultrasonics, magnetostriction and incremental permeability were found to be the most promising measures of copper-precipitation hardening, the likely cause of radiation embrittlement.

Ultrasonic Standards: The ASTM Committee on Nondestructive Testing requested NIST for help in revising ASTM E-164 "Standard Practice for Ultrasonic Contact Examination of Weldments." In response, we developed an ultrasonic velocity measurement procedure with a 0.1% uncertainty, and we initiated an evaluation of ultrasonic test blocks provided by industrial participants on the ASTM committee.

Electronic Interconnects: The reliability of on-chip interconnects is often limited by stress voiding that arises from relaxation of fabrication stresses due to thermal expansion mismatch. A standard test structure, NIST 34, has been designed in collaboration with the Semiconductor Electronics Division to evaluate the effects of line width, pitch and stress concentrations on stress voiding.

Printed Wiring Boards: We participated in the Improved Registration Project of the Interconnection Technology Research Institute (ITRI). This project made a significant contribution to eliminating $70 million lost annually in scrap, due to product rejection, by identifying design features that do and do not affect printed wiring board reliability. The savings will result from a better alignment between real reliability issues and inspection standards. We assisted in planning of the experiments and analysis of the data, coordinated writing of the final report, and presented the results at an ITRI meeting.

Thermal Conductivity Standard: We completed measurement and data analysis on Pyroceram 9606 for use as a standard reference material for high temperature thermal conductivity. The NIST high temperature guarded hot plate apparatus allows absolute thermal conductivity to be determined from 100°C to 800°C and is currently the only such apparatus in the world. As part of the work, NIST staff participated in an international round robin on this material in which other laboratories determined the thermal conductivity by a variety of less direct measurements, mostly using transient techniques.

Ferrite Standards for Stainless Steel Welds: The susceptibility of stainless steel welds and castings to hot cracking is reduced if the microstructure contains ferrite. Thus, ferrite levels are specified, and measured, to ensure structural integrity. At the request of the International Institute of Welding, NIST has become the source of ferrite standards.

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 these 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, sense microstructural changes in real time, and use a model-based control strategy to achieve the desired microstructure in the finished product. Microstructural consistency is essential to the commercialization of advanced materials because it assures reliable properties and performance of the material.

The IPM Program 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 that has its roots in sensor development for nondestructive evaluation of materials. Unique MSEL capabilities are being used to measure thermophysical properties at elevated temperatures; these data are combined with model enhancements and then incorporated in industrial software for metal casting. In addition, specialized measurement capabilities such as nuclear magnetic resonance and small-angle neutron scattering are used to understand the evolution of microstructure and morphology in ceramics and polymers.

Project: WELD ARC SENSING

MSEL Program: Intelligent Processing of Materials

Principal Investigators: R.B. Madigan
T.P. Quinn, T.A. Siewert, B.J. Filla, 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 ultraviolet light, smoke, molten metal spatter, and electronic noise generated by the welding arc. To gain acceptance by manufacturers, sensors must be physically small if they are attached to the torch, so as not to affect the path of the 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: current and 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 measure arc-light emissions with higher resolution. 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 generator.

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 analytical 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 backlighting
- 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, works with Bruce Madigan to determine the best arc-sensing techniques for fabrication of heavy equipment structures.

Colorado School of Mines. Steve Liu, Professor of Metallurgy, used 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 the 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 and used the NIST high-speed video unit to confirm the sensor output.

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

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.

Caterpillar works with us to learn what new sensing strategies might be applied to improve their production.

Oakley Tube works with us to apply through-the-arc sensing strategies to pulsed gas-tungsten-arc welding in the manufacture of stainless steel tubing.

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

Planned Outcome

Develop weld sensors that provide more information about the quality of the welding conditions, leading to the development of more intelligent weld controllers. Work with sensor developers and end-users to refine and implement these concepts in production weld cells.

Accomplishments

As we finish a CRADA with Tower Automotive on arc sensing, we are testing the range of applicability of our arc sensor module to high-volume production welds. The sensor records from 82 production welds (containing 4 defective welds) were used to tune our sensor quality thresholds. Another 444 welds (during three production shifts) were monitored using these settings. In total, the sensor flagged 5 of the 6 defective welds produced during these trials, and did not flag any defect-free welds.

In conjunction with Oakley Tube, our arc sensor module, originally designed for use in gas-metal-arc welding, was applied to pulsed gas-tungsten-arc welding of a continuous-production, stainless-steel tube rolling mill. Data were taken during welding on the factory floor and subsequently analyzed. Packaging debris was intentionally placed on some of the rolled tube just prior to welding, causing small (2-10 mm) defects (lack of fusion). The sensor was able to detect all of the defects by monitoring when the output from the algorithm exceeded a threshold. The threshold was set so that no warning was given when no defect occurred. The next step will be to determine whether this technology should be incorporated into production.

We are finishing a CRADA with the Delphi Chassis Division of General Motors. After a training and evaluation period in our laboratory, we shifted to a mode where they are evaluating this technology in their weld laboratory and making some production trials this year. Recent data indicates that our sensor module is useful for benchmarking production weld cells, evaluating new products (quantitative evaluation of electrode and power source performance) and developing control charts.

We worked with researchers at Colorado School of Mines on investigating some of the problems specific to the gas-metal-arc welding of aluminum. This small project supported the development of a full proposal to NSF.

Publications

1. Sensing of Gas Metal Arc Welds, T.A. Siewert, R. B. Madigan, and T.P. Quinn, Proceedings of Revitalization-Diagnostic International Conference, Dubrovnik, Croatia, October, 1996.

2. Trends in Welding Software, Siewert, T.A.; Proc., 7th Intl. Conf. on Computer Technology in Welding, NISTSP 923, ed. T. A. Siewert, in press.

3. Welding Software Aids Engineers, T.A. Siewert, Welding Design and Fabrication, pp. 21-25, November 1996.

4. The International Institute of Welding: Report on 1997 actions, T.A. Siewert, Mater. Eval. 56 (2), pp. 153-159, February 1998.

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).
- Develop theoretical models for GMAW to understand the relationship between input parameters and the resulting process.
- Develop and test arc sensing and control algorithms and transfer them to American industry.

Technical Description

Theoretical 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 audible and visual feedback.) The models of the complex welding processes lead to better understanding by welding engineers, and to 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: current and 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.

Academic: Ben-Gurion University. Dr. M. Szanto works with Timothy Quinn to develop a fully coupled model of gas-metal-arc welding.

Accomplishments

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 their characteristics lie 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. With the sensor implemented on a PC-style computer with a sophisticated graphical user interface, tests were conducted on a factory floor during production. The constants used to tune the algorithm were found from the first 82 welds that included 4 defective welds. Another 444 welds were monitored with 2 more defective welds. The sensor flagged 5 of the 6 defective welds and did not flag any defect-free welds.

A model of the electrode extension in GMAW was completed for aluminum. The model predicts the amount of electrode that extends beyond the contact tube before it melts, the electrode extension. The electrode extension determines the arc length for a given contact tube-to-work-distance. The arc length controls the heat-input to the system and hence the final quality of the weld. This work is an extension of the heat-transfer model developed previously for steel electrodes. Fitting the model to experiments identified a single unknown parameter in the model. For a given electrode extension the model could predict the current within an rms value of 3 A for the ER-1100 electrode used. The model predicts that aluminum is much more sensitive to changes in the operating parameters than is steel (Table 1), confirming the practical observation that aluminum is more difficult to weld with a gas-metal arc.

Table 1: The change in current necessary to change the electrode extension 8 mm for the given mean wire feed speed (WFS). The speeds given approximate the range of practical welding conditions

Steel		**Aluminum**	
WFS	ΔI	WFS	ΔI
100 mm/s	28 A	80 mm/s	1.0 A
125	42	100	1.6
150	57	125	2.3

In conjunction with Ben-Gurion University (Israel), work has begun on a fully coupled model of GMAW. The model predicts the heat transfer, fluid flow, moving boundaries, and phase changes in the electrode, plasma, and weld pool. The model uses the finite-element method to solve the highly nonlinear set of partial differential equations. Calculations using constant-property data and estimated boundary conditions have qualitatively the same behavior.

Planned Outcome

Develop models and control techniques that will make robotic welding systems more intelligent, leading to higher productivity and reduced scrap.

Publications

1. Weld Defect Decisions using Through-the-arc Sensor Data, Quinn, T.P.; Madigan, R.B.[853]; Smith, C.B.[Tower Automotive]; Blachowiak, E.G.[A.O.Smith]; Proc., 7th Intl. Conf. on Computer Technology in Welding, NISTSP 923.

Project: THERMOMECHANICAL PROCESSING

MSEL Program: Intelligent Processing of Materials

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

Objectives

The objectives of this program are to develop, improve, and validate models that are needed to characterize the thermomechanical processing of steels. With accurate process models, steel products can be manufactured with greater economy 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, steel chemistry, and the thermomechanical processing parameters used during steel rolling and forging. Emphasis is on the characterization of the kinetics of microstructural evolution during hot working. Several important factors are included, namely, precipitation effects (carbides, nitrides, and carbonitrides), grain growth and recrystallization (static and dynamic recrystallization), and austenite decomposition. In addition, constitutive equations are developed and composition-structure-property relationships are characterized. The structure-property relationships at ambient temperature and the constitutive behavior for hot-rolling conditions at elevated temperatures are being determined for specific plain-carbon and microalloyed steels. Mathematical equations are developed to describe the structure-property relationships and the constitutive behavior based on physical-metallurgical principles coupled with experimental observations. These equations are being coded into computer programs that can be used to calculate a material's mechanical properties at ambient temperature or its stress-strain curves under hot-rolling conditions. The computer programs can also be incorporated into process models to simulate the steel rolling conditions in hot-strip mills and to predict the mechanical properties of hot-rolled products.

External Collaborations

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

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

Industrial. United States Steel Corporation. U.S. Steel is the industrial sponsor for the AISI project conducted by UBC and NIST.

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

Accomplishments

In 1997, we continue our research effort for the American Iron and Steel Institute (AISI) on microstructural engineering in hot-strip mills. The objective of this research 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 KBall Incorporated (Pittsburgh). Major portions of the research are being done at NIST and UBC. UBC conducts research on heat transfer, quantitative characterization of the kinetics of microstructural evolution, and model verification with plant and pilot plant trials. NIST's efforts focus on studies of constitutive behavior under hot-rolling conditions, and on structure-composition-property relationships at ambient temperature.

One goal of studying constitutive behavior is to predict the flow behavior of a given steel under various processing conditions. Knowledge of 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.

Accomplishments for 1997 build on earlier work from the two previous years. In 1995 and 1996, models were developed for two plain carbon steels, and one niobium-treated high strength low-alloy steel (HSLA-Nb). These 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 important parameters characterizing the stress-strain curve, including the 0.2% yield stress, the steady-state stress before dynamic recrystallization, the strain at which dynamic recrystallization occurs, and the steady-state stress after dynamic recrystallization has taken place. This year, the model for the HSLA (Nb steel) was refined and improved. We then obtained data for three additional Nb-microalloyed steels (Stelmax 80, 52 ksi HSLA, and Nb-IF). The models for the three new steels were compared and contrasted with that for the HSLA-Nb steel.

The mechanical properties of a steel at ambient temperature are closely related to composition and microstructure. In 1995, the structure-property relationships for plain-carbon steels were validated. In 1996, the strengthening effects due to the precipitates VC, VN, and V(CN) were quantified and described using a Shercliff-Ashby model, which considers the effects of solid-solution hardening in addition to precipitation strengthening. Traditionally, only the effect of precipitation is considered. Industrial practice treats the increment of strengthening due to precipitation as a function of coiling temperature. In 1997, this model was extended to include Nb-alloyed and Nb-Ti-alloyed steels.

The equations developed for the structure-property relationships have been successfully incorporated into a process model which is being developed at UBC-Canada. The process model simulates the processes involved in rolling flat steel slabs in hot-strip mills, and it 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, Journal of Testing and Evaluation, ASTM, to be published.

2. "Modeling of the Microstructure and Mechanical Properties of Steels during Thermomechanical Processing", R. Kuziak, Y-W. Cheng, M. Glowacki, and M. Pietrzyk, NIST Technical Note 1393, National Institute of Standards and Technology, Boulder, CO, 1997.

3. "Modeling of Microstructure and Properties in HSLA Steels", M. Militzer, W.P. Sun, W.J. Poole, and P.T. Purtscher, THERMEC '97, The Metallurgical Society of AIME, Warrendale, PA, to be published.

Project: X-RAY DIFFRACTION SENSING OF A LIQUID-SOLID INTERFACE

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

Objectives

- Develop high-energy, transmission x-ray diffraction (XRD) as a nondestructive means for locating, characterizing, and following the liquid-dendrite-solid region in an investment casting during directional solidification.

- Map isocontours of solid fraction 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 solidification models for investment casting.

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). Therefore, we are developing a noninvasive x-ray diffraction (XRD) technique to monitor the position, shape, and extent of the liquid-dendrite-solid region (mushy zone) 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 can be used to determine the extent and shape of the mushy zone in a nickel superalloy casting. An optimal x-ray energy (often between 100 and 320 keV) capable of penetrating through the furnace, mold walls, and casting has been chosen using our analytical model for the transmission XRD process.

Experiments and modeling have proven conclusively that the x-ray diffraction sensor can detect the regions of liquid and solid in a casting. The high-energy x-ray diffraction technique also appears appropriate for investigating the region of dendritic solidification in alloy castings. We are optimizing the configuration of the solidification sensor on a resistively heated directional solidification furnace.

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.

Planned Outcome

Our intent is to develop a noncontact x-ray sensor to monitor the solidification of single-crystal turbine-blade castings and transfer this technology to the aerospace casting industry. Knowledge of the location and shape of the solidification front would allow the casting process to be optimized (through confirmation of solidification models), reducing scrap and increasing the production rate and yield.

Accomplishments

We have begun to probe the liquid-dendrite-solid region of a single-crystal, nickel alloy casting using our transmission x-ray diffraction technique. Real-time XRD patterns were recorded as the x-ray beam was scanned vertically in the casting. Intensity of the diffraction spot is high when the probing x-ray beam is directed into the crystalline solid and decreases as the x-ray beam is moved into the region of the casting containing only molten alloy. Plots of diffraction-spot intensity versus vertical position (increasing temperature) in the casting seem to correlate well with model predictions of fraction solid versus temperature. Under very specific conditions we have visualized dendrites in the mushy zone of the casting, using a white-beam topographic method. An estimate of dendrite width is possible at various vertical positions in the region of solidification.

Although we have been using primarily transmission XRD to sense the physical state (liquid, solid) of areas in a single-crystal casting, the orientation of the growing crystal is also easily measurable. We have combined information from an x-ray imager and an energy-sensitive sensor to determine, in real time, crystalline orientation.

Publications

1. T.A. Siewert, W.P. Dubè, and D.W. Fitting, *US Patent 5,589,690*, "Apparatus and Method For Monitoring Casting Process", issued 12/31/96.

2. D.W. Fitting, W.P. Dubé, and T.A. Siewert, "High energy x-ray diffraction technique for monitoring solidification of single crystal castings" *Proceedings of the Eighth International Symposium on Nondestructive Materials Characterization*, (Plenum Press, New York, 1997).

3. D.W. Fitting, W.P. Dubè, and T.A. Siewert, "Real-time monitoring of turbine blade solidification using x-ray diffraction techniques," *Annual Report of the NIST Consortium on Casting of Aerospace Alloys*, pp. 60-71, 30 May 1997, and the *Semi-Annual Report of the NIST Consortium on Casting of Aerospace Alloys,* October 1997 (available to Consortium members).

ULTRASONIC CHARACTERIZATION OF MATERIALS

The Program on Ultrasonic Characterization of Materials is directed to the development of model-based methods of physical measurement that 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, composites, nondestructive evaluation 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 COEFFICIENTS AND RELATED PHYSICAL PROPERTIES

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

Objectives

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.

Measurements and modeling to understand physical effects, especially microstructural, that influence diffraction (x-ray and neutron) in condensed matter.

Technical Description

Our research emphasizes measurements and modeling of elastic coefficients 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 coefficients, 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 coefficients with specific heat, thermal expansivity, atomic volume, the Debye temperature, and the Grüneisen parameter.

Many microstructural features, most notably defects, define a material's diffraction properties. We are interested much less in traditional diffraction studies, such as crystal structure, and more in different materials-science related applications: crystal-defect structure, strain, texture, and similar. We make measurements with laboratory x-ray sources and at large national user facilities using synchrotron x-ray and neutron diffraction.

* Visiting scientist from Ruđer Bošković Institute, Zagreb.
** NRC-NIST Postdoctoral Research Associate.
*** Visiting scientist from University of Colorado.

Planned Outcomes

Consequences of our studies must be awaited. We intend that our studies provide at least the following:

1. better, or new, measured values of basic physical properties;
2. measurement-method improvements;
3. critical observation-theory comparison;
4. novel theoretical approaches;
5. relationships among physical properties;
6. occasional small discoveries of unexpected behavior.

See brief description under Objectives.

Accomplishments

The following publication list reveals most of our recent achievements. Beside these we progressed significantly on other topics:

1a. Reactor-pressure-vessel steels. For two alloys with seven heat treatments, we measured elastic-stiffness coefficients and internal frictions. We consider the relationships between these two properties and mechanical-deformation properties such as hardness, yield strength, ultimate strength, and ductile-brittle-transition temperature.

1b. Reactor-pressure-vessel steels. By analyzing diffraction-line broadening, we determined inhomogeneous strains in specimens with different heat treatment. Strain is attributed to changes in coherency of copper-rich precipitates and their interaction with dislocations. It correlates with mechanical (hardness and strength) and acoustic (nonlinearity parameter β) properties.

2. Covalent materials. For Ge and Si, we measured the monocrystal elastic-stiffness tensor C_{ij} and the internal-friction tensor Q_{ij}^{-1}. We interpreted our results using the Koehler-Granato-Lücke theory of vibrating dislocations. Thus, we measured also the frequency and strain dependences of Q^{-1}.

3. Yttrium boride, YB_{66}. We measured the monocrystal elastic stiffnesses of this compound, the only known cubic-symmetry boron compound. We hope to probe the subtleties and complexities of boron's chemical bonding. Further measurements will include low temperatures and internal friction.

4. Polymer-matrix composites. In these materials, attenuation, high scattering, and usually noncrystalline complex molecular structure present formidable measurement problems. We considered five measurement methods and concluded that impulse-excitation is the best choice. Now, we extend existing isotropic models to the anisotropic case.

5. Hardness. For many elements and compounds, we considered relationships between hardness and the elastic-stiffness tensor. We found strong correlations between hardness and the shear modulus. Essential to this research is the ability to calculate elastic coefficients from basic theory for both real and imagined (possible-high-hardness) compounds. The theoretical approach consisted mainly of *ab initio* all-electron density-functional theory.

6. High-T_c Oxide superconductors. For $(La\text{-}M)_2CuO_4$ with M = Ca, Sr, Ba, we measured the low-temperature elastic coefficients. Further efforts will include internal friction and relationship of results to the Landau theory of second-order phase transitions. Also, our efforts continue to relate Q^{-1} (T) to orbital symmetry of the superconducting pairing functions.

7. Glasses. In fused silica and GeO_2-SiO_2 glass, we measured C_{ij} and Q^{-1}. Further studies will include temperature effects and relaxation peaks.

8. Indium. Between ambient and liquid-helium temperature (4 K), we measured indium's polycrystal elastic stiffnesses. During cooling, the Young modulus increases about 55%, compared with a 12% increase for copper. Our study also led to the Debye characteristic temperature and the Grüneisen parameter.

9. Ceramic-ceramic composites. From U. Karlsruhe, we obtained unusually well-made well-characterized specimens of Al_2O_3/ZrO_2 over the entire 0 – 100% composition region. We measured the C_{ij} and Q_{ij}^{-1} and modeled the C_{ij} successfully using the Ledbetter-Datta scattered-plane-wave ensemble-average theory.

10. Strontium titanate, $SrTiO_3$. Our low-temperature C_{ij} measurements led to a new interpretation of transitions below 108 K. Prevailing views invoke a Müller phase (the same Müller who recently received the Nobel physics prize). We believe that the transitions are simply dynamic instabilities caused by soft optical-phonon modes.

11. Zirconia-yttria, ZrO_2 – Y_2O_3, monocrystal. For various compositions, we measured the C_{ij}. We shall try to explain the results by fundamental calculations using *ab initio* all-electron density-functional theory.

12. Carbon steel. For a 4340 steel, we measured C_{ij} and Q_{ij}^{-1} and found good correlations with hardness, which was varied by thermal treatment.

13. Creep in torsion. We modified our existing shear-mode creep-recovery apparatus to a different deformation mode: torsion. We studied several materials, especially lead and polyethylene.

14. Strain-time/Q^{-1} – frequency relationship. We developed methods for converting creep-recovery strain-time measurements to internal-friction/frequency diagrams. Results for indium and lead show a strong low-frequency $Q^{-1}(f)$ peak.

15. Reinforced glass. From U. Tokyo, we obtained glass specimens reinforced with various volume fractions of silicon carbide. Except for higher SiC volume fractions, measurement-modeling agreement is good. The small discrepancies may arise from the material's anisotropy. For the modeling, we used the Ledbetter-Datta scattered-plane-wave ensemble-average theory.

16. Thermal-expansivity α. We began to assemble an apparatus to measure thermal expansivity. We detect displacement with a laser interferometer with 10-nm sensitivity. Initially we shall measure up to 100-200°C. Later, we shall modify the apparatus for low-temperature measurements. In principle, for typical measurement conditions, the expected measurement uncertainty $\Delta\alpha/\alpha$ equals about 0.001.

17. Iron-chromium-nickel alloys. From IZFP (Saarbrücken), we obtained well-characterized specimens of 308 austenitic steel. C_{ij} measurements revealed a strong texture and a macroscopic symmetry less than orthotropic. Microstructure and preliminary x-ray diffraction confirm the lower symmetry. Thus, our results dispute the usual assumption of transverse-isotropic symmetry.

18. Martensitic steels. Here, diffraction-line broadening is caused by both carbon interstitials and high densities of dislocations. It increases with increasing carbon content and also correlates with hardness and the acoustic nonlinearity parameter β.

19. Strain and texture determination. Based on measurements made at pulsed-neutron sources at Los Alamos and Argonne National Laboratories, we proposed a novel method to determine the complete strain tensor in multiphase materials. The strain tensor is obtained simultaneously with texture, structural, and microstructural properties for each crystalline phase. This method can be applied to both neutron and x-ray measurements, and it is particularly useful for energy-dispersive measurements.

20. Metal-matrix composites. We studied residual strain and texture in several composites: Al/SiC (whisker), Al/alumina-mullite (particle), and Cu/diamond (nanoparticle) composites. Bulk values were obtained from neutron-diffraction measurements by application of the novel method mentioned in item 19.

21. Poled $BaTiO_3$ polycrystals. We studied changes upon poling of $BaTiO_3$ ceramics by using x-ray laboratory and synchrotron sources. Line-broadening analysis showed that poling multiplies defects in the structure, whereas there was no measurable change in elastic strain.

22. D. Balzar, invited as a member of International Centre for Diffraction Data (ICDD), Newtown Square, Pennsylvania. The ICDD is a not-for-profit scientific organization that collects, edits, publishes, and distributes powder-diffraction data for the identification of crystalline materials in the forms of the Powder Diffraction File. Approximately 100 scientists from around the world compose the active membership. The membership is not solely honorary because the ICDD draws its Board of Directors, committees, and subcommittees from it.

23. D. Balzar was selected as a member of the International Program Committee of the

24. 6th European Powder Diffraction Conference (EPDIC-6), to be held in Budapest, 22-25 August 1998. EPDIC is the largest European meeting in this field.

25. D. Balzar acted as an instructor on General Structure Analysis System at the workshop of the 2nd LANSCE User Group Meeting, Los Alamos National Laboratory, New Mexico, 6-8 August 1997.

26. H. Ledbetter signed an official agreement with the Mechanical Engineering Laboratory (MITI) in Tsukuba, Japan to study cluster-diamond-reinforced copper. This material may provide higher stiffness along with improved friction-wear properties.

Publications

1. D. Balzar, Diffraction Line Broadening — Nuisance or Lattice-Imperfections Fingerprints, *Croatica Chemica Acta* 69 (1996) 1069-1115.

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8. D. Balzar, P.W. Stephens, H. Ledbetter, J. Li, M.L. Dunn, Synchrotron X-ray Diffraction Study of the Surface Layer in Poled Ceramic $BaTiO_3$, *Materials Research Society Symposium Proceedings Volume 453*, Edited by P.K. Davies, A.J. Jacobson, C.C. Torardi, T.A. Vanderah, Materials Research Society, Pittsburgh, Pennsylvania, (1997) 715-720.

9. D. Balzar, T. Ungár, H. Ledbetter, Residual Broadening in NIST SRM 660 LaB_6, *J. Appl. Cryst.*, in preparation.

10. D. Balzar, R.B. Von Dreele, K. Bennett, H. Ledbetter, The Complete Elastic-Strain Tensor by Rietveld Refinement of Diffraction Measurements, *J. Appl. Phys.*, submitted.

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12. D. Clerc, Mechanical hardness and elastic stiffness of alloys: semiempirical models, J. Phys. Chem. Solids, submitted.

13. D. Clerc, <u>Ab initio</u> elastic properties of diamond-like materials: Electronic factors that determine a high bulk modulus, J. Phys. Chem. Solids, submitted.

14. D. Clerc, Atomic-level mechanical-hardness calculations: Diamond-like materials, J. Phys. Chem. Solids, submitted.

15. D. Clerc and H. Ledbetter, Mechanical Hardness: a new theory based on screened electrostatics and elastic shear, J. Phys. Chem. Solids, forthcoming.

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17. D.C. Hurley, P.T. Purtscher, D. Balzar, K.W. Hollman, C.M. Fortunko, Nonlinear Ultrasonic Properties of As-Quenched Steels, *Review of Progress in Quantitative NDE* (1997), in press.

18. D.C. Hurley, D. Balzar, P.T. Purtscher, K.W. Hollman, Nonlinear Ultrasonic Properties of Quenched Martensitic Steels, in preparation.

19. S. Kim and H. Ledbetter, Indium's low-temperature elastic coefficients, J. Mater. Sci. Eng., forthcoming.

20. M. Lei, A. Migliori, H. Ledbetter, Acoustic evidence against BCS behavior in monocrystal $YBa_2Cu_3O_7$, Physica C (1997) 1077-1078.

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Project: NONLINEAR ULTRASONICS FOR MATERIALS CHARACTERIZATION

Principal Investigator: D. C. Hurley
D. Balzar, C. M. Fortunko, P. T. Purtscher

Objectives

- Identify quantitative relationships between nonlinear-ultrasonic measurables and the mechanical/physical properties of materials. Develop and evaluate appropriate experimental techniques. Develop analytical models to predict and explain experimental results.

- Use nonlinear ultrasonic methods to characterize the microstructure of specific systems such as engineered surfaces or precipitate-hardened alloys.

Technical Description

We are investigating the utility of nonlinear ultrasonic techniques to understand relationships between microstructure and mechanical/physical properties. Nonlinear ultrasonics can, in principle, probe certain microstructural properties more sensitively than conventional (linear) ultrasonics. For instance, standard 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.

The technical approach is based on our development of new sensitive and accurate nonlinear ultrasonic techniques. Our principal experimental tool is a Michelson interferometer incorporating an infrared laser. The interferometric system represents a unique measurement capability and was developed at NIST. An interferometer enables direct measurement of absolute ultrasonic displacements, which measurement is essential for nonlinear studies. Unlike other techniques used for nonlinear experiments, interferometric detection possesses micrometer-scale resolution, is inherently nonintrusive, and has the potential to scan large, curved surfaces. Our system possesses a remarkable combination of bandwidth and sensitivity: displacements as low as 5×10^{-12} m can be detected over a 20 MHz bandwidth, with a spatial resolution of approximately 20 nm. Last year, this experimental approach was validated through baseline measurements and quantitative comparison with established methods.

Using this approach, applications to microstructural characterization are now underway. Studies of idealized or "model" bulk systems have begun. In a parallel effort, instrumentation and methods are being 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 must be developed to predict and explain experimental results. Mechanical measurements will also be used to interpret results. In this way, quantitative relationships between nonlinear measurables and microstructural properties can be established. Finally, we will examine the feasibility of our methods for industrial materials such as diamond-coated tools or ion-implanted parts. Obstacles to success will be identified, and ideas for practical solutions will be developed.

External Collaborations

Industrial. Our CRADA with RITEC, Inc. continued in FY97 for development of prototype electronics for nonlinear ultrasonic measurements. The instrument, which is the first of its kind and not a turnkey system, represents a unique instrumentation capability. Modifications based on our experimental evaluation of the prototype are being incorporated into a second version, which is scheduled for early FY98. The instrument's value can be judged by the fact that several laboratories worldwide have already placed orders for a commercial version.

ATP. Initiated contact with various ATP participants to obtain specimens, including staff at Los Alamos National Laboratory, General Motors, and Empire Hard Chrome, Inc. As a result of these discussions, Empire Hard Chrome agreed to ion-implant several steel samples of a suitable size for surface-wave experiments.

International. Established a collaboration with Prof. E. J. Danicki (Polish National Academy of Sciences, Warsaw) and jointly prepared a proposal for the Marie Sklodowska-Curie Joint Poland-America Fund. The project was funded and will begin in 1998. This project addresses a fundamental obstacle to nonlinear surface-wave experiments, namely how to generate surface waves with the desired amplitude and sufficient spectral purity. Work will focus on modeling and experimental verification of ultrasonic wave generation by different devices.

Academic. Developed collaborative ties with several academic groups, including Prof. O. Buck at Iowa State University. Of particular interest are nonlinear experiments in the model system of copper with substitutional aluminum prepared by hot isostatic pressing methods, and a quantitative comparison of the ISU and NIST measurement methods.

Planned Outcome

In industry, methods are needed to facilitate the assessment of mechanical and physical properties of materials in a quantitative manner. We are working towards this goal, and anticipate that new measurement approaches for microstructural characterization will result. In particular, we hope to achieve techniques for nondestructive assessment of adhesion, hardness, fatigue, and the like. In the long term, our work should lead to the development of tools that utilize nonlinear ultrasonics to characterize microstructure and relate it to performance or lifetime. Publications, conference presentations, and personal contacts throughout the process will keep industry informed of, and interested in, our work.

Accomplishments

Last year, created a nonlinear ultrasonic measurement system incorporating the Michelson interferometer and specialized electronics discussed above. Having developed the necessary measurement techniques and demonstrated sufficient sensitivity, we have now begun to apply the techniques to the characterization of bulk and surface microstructure.

In FY97, performed bulk nonlinear experiments to assess their potential for microstructural characterization. Measured the nonlinear ultrasonic parameter β in a series of quenched martensite specimens with varying carbon content. Specimen-to-specimen variations in β were significantly greater than the relative experimental uncertainty of ±2%, and a monotonic increase in β with carbon content was observed. In contrast, high-precision (±0.1%) velocity measurements showed virtually no change with carbon content, indicating that nonlinear methods were more sensitive to microstructural differences in the samples. The increase in β is believed due to harmonic generation by dislocations created by quenching. X-ray diffraction experiments on the same specimens revealed that the dislocation density was extremely large ($\sim 10^{12}/cm^2$), and increased monotonically with carbon content. This evidence is consistent with a previously developed model predicting a linear increase in β with dislocation density.

Participated in a Nuclear Regulatory Commission feasibility study for steel mechanical-property measurements. Performed bulk nonlinear ultrasonic experiments on steels with copper-rich precipitates. In steels with 1.13 mass percent copper, the nonlinear parameter β increased by 11% for a hardness increase of 10% (53.2-58.5 HRA). Results are consistent with a microstructural model in which the strain is associated with the relative coherency of the precipitates. Nonlinear ultrasonic experiments are sensitive to the average bulk strain; therefore, they should be sensitive to the relative state of precipitate coherency. X-ray diffraction line broadening measurements of the microstrain support this hypothesis and show an excellent correlation between the experimental values for strain and β. Additional experiments are planned to investigate this behavior in more detail.

Adapted laboratory apparatus to meet surface-wave requirements. Possible surface-wave generation methods were evaluated: wedge, edge, EMAT, integral grooves, and comb. Methods were evaluated based on such characteristics as ease of use, spectral purity, relative efficiency, and amplitude of generated waves. Of the methods evaluated, the comb currently appears to have the greatest potential. Demonstrated the ability to produce surface waves of sufficient amplitude and spectral purity with combs in the frequency range 1-10 MHz. Observed second harmonic generation of surface waves on aluminum using a comb. Since it was unknown whether any method would prove suitable in practice on nonpiezolectric materials, this experimental demonstration was critical. Interpreted theoretical expressions in the literature to show that results agreed with theoretical predictions, if diffraction effects were included. Analyzed expressions to show that diffraction effects significantly complicated data interpretation. Conceived and designed a modified comb structure to minimize diffraction effects; testing of this structure is currently in progress. With the measurement system developed over the last two years, we are now able to investigate nonlinear ultrasonic effects of surface microstructure with outstanding precision and spatial resolution.

Publications

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2. D. C. Hurley, P. T. Purtscher, K. W. Hollman, and C. M. Fortunko, "Nonlinear ultrasonic properties of as-quenched steels," in *Proc. 8th Intl. Symp. Nondes. Char. Mat.*, edited by R. E. Green, Jr. (Plenum Press, New York, in press).

3. D. C. Hurley, P. T. Purtscher, D. Balzar, K. W. Hollman, and C. M. Fortunko, "Nonlinear ultrasonic properties of as-quenched steels," in *Review of Progress in Quantitative NDE Vol. 17*, edited by D. O. Thompson and D. E. Chimenti (Plenum Press, New York, in press).

4. D. C. Hurley, D. Balzar, P. T. Purtscher, and K. W. Hollman, "Nonlinear ultrasonic parameter β of quenched martensitic steels," submitted for publication.

Project: INTERNAL FRICTION MEASUREMENTS FOR MONITORING MICROSTRUCTURAL EVOLUTION

Principal Investigator: W. Johnson
George Alers, Steve Schaps, Tony Sinclair*

Objectives

Develop ultrasonic techniques for sensing and characterizing changes in dislocation structure and/or pinning that result from annealing, loading, or irradiating structural alloys. Develop models that relate the ultrasonic measurements to changes in bulk mechanical properties, enabling nondestructive evaluation of materials during production or service.

Technical Description

Ultrasonic measurements can be highly sensitive to the density, symmetry, and pinning of dislocations in crystals, because the nonelastic response of dislocations results in damping and slowing of acoustic waves. This phenomenon, which has been studied extensively in basic research, is being pursued here as a tool for the nondestructive evaluation of commercial structural alloys.

In 1997, the focus of research has been the effect of static and dynamic loading on the ultrasonic damping in steel. Two projects have been pursued using similar experimental techniques. In the first, funded by the Federal Highway Administration, the goal is to nondestructively detect early fatigue damage (prior to crack formation) in bridge steels, such as A36. In the second, funded by the Nuclear Regulatory Commission, the goal is to characterize the state of irradiation-induced embrittlement in A533B steel reactor pressure vessels. The principle approach in both studies is to use resonant ultrasonics to sense the degree to which dislocations break away from pinning points when specimens are subjected to loads in a tensile testing machine. This may provide information on the strength of dislocation pinning, which determines the inclination of fatigued or irradiated material to crack.

Planned Outcome

The aging of our country's bridges has become a major issue in recent years, because the structural integrity of many bridges is unknown and the cost of their replacement is tremendous. The successful implementation of an ultrasonic sensor for early detection of fatigue damage in bridge steels would have a large impact, because it would provide unique nondestructive information on the structural integrity.

* Visiting Scientist from the University of Toronto

A similar situation exists for aging nuclear reactors. The accumulation of irradiation damage in steel containment vessels must be evaluated to predict the inclination of the material to fail in an emergency situation, or the vessels must be replaced. The economic advantage of effective nondestructive evaluation of the material versus replacement of a vessel is great, if the vessel is, in fact, structurally sound.

Accomplishments

A unique resonant ultrasonic system was implemented for studying properties of specimens undergoing static or dynamic loading in a tensile testing machine. Highly accurate measurements of damping and velocity are achieved with a specimen geometry that has resonant vibrational modes localized in a central portion of the specimen and, thus, has insignificant energy losses through the testing machine grips. In addition, energy losses through transducer coupling are essentially eliminated by employing noncontacting electromagnetic-acoustic transducers. (The specimen configuration and transducers are similar to those used previously to measure applied force with vibrational spectroscopy [1].) Because of the high resolution and the frequencies employed (typically, 0.5-3 MHz), this system achieves unprecedented sensitivity to the anelastic properties of dislocations.

To study the effect of fatigue on ultrasonic properties, several specimens A36 steel were subjected to 5 Hz cyclic loading until they failed, with maximum loads typically in the range of 70-110% of yield. The fatigue cycling was periodically stopped to allow performance of two types of ultrasonic tests as a function of quasistatic load. In the first test, the resonant frequency and damping were measured as a function of slowly varying load from near zero to a significant fraction of yield. In the second test, the same ultrasonic parameters were measured as the load was (1) stepped rapidly from near zero to a significant fraction of yield, (2) held at the high load for a time on the order of one hour, and (3) reduced back to the minimum and held for many hours. The motivation for exploring relative tests of this type is that they offer greater potential for practical implementation in the field, where accurate absolute meaurements of damping would be very difficult, if not impossible. The results of both these tests show systematic changes in damping as a function of the number of fatigue cycles. Perhaps the most interesting result is that static loading for an extended period (the second test) induces a change in damping that recovers with time after the loading, and this recovery varies as fatigue damage accumulates. The change in damping is apparently associated with the breakaway of dislocations under load followed by thermally activated repinning involving migration of point defects. The dependence of the magnitude of this change on the number of fatigue cycles is qualitatively explained in terms of the multiplication of dislocations in early stages of fatigue and the subsequent development of tangled (relatively immobile) dislocation structures.

The potential for ultrasonic sensing of irradiation embrittlement was explored in A710 and A533B steels with various thermal treatments used to alter mechanical properties. The experimental system described above was enhanced by providing the capability to impose a static magnetic field and vary the temperature in the range -100°C to 70°C. The purpose of the magnetic field was to reduce magnetic contributions to the damping (thus, increasing the sensitivity to direct dislocation effects). The purpose of the temperature control was to explore the temperature dependence of dislocation pinning and repinning and relate this to the ductile-to-brittle transition temperature, which is the mechanical property of ultimate interest to the nuclear industry. Recovery of damping after a load was measured in a manner similar to that used for the fatigue specimens. The magnitude and time-dependence of this recovery varied with temperature in a manner consistent with the fact that, at lower temperatures, dislocations are more strongly pinned and, once depinned by an applied load, are repinned more slowly. Thus, the results indicate a sensitivity to material parameters that affect the transition from ductile to brittle behavior. Unfortunately, recovery behavior of this type was not observed until the material was plastically deformed to increase the number of sufficiently mobile dislocations; so, the test cannot be performed nondestructively. There may still be hope for a nondestructive ultrasonic technique, since preliminary measurements of the equilibrium (constant load) background damping of several A533B specimens show differences with some correlation to the ductile-to-brittle transition temperature measured in a Charpy test.

Publications

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Project: ULTRASONIC MEASUREMENTS OF STRESS

Program: Ultrasonic Characterization of Materials

Project Leader: A.V. Clark
G.A. Alers, C.S. Hehman*, T. Nguyen*

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 steel bridges and buildings.

Technical Description

Since stress changes the velocity of sound in the stressed materials, measurement of the sound velocity can be used to determine stress in a variety of materials and structures. Most applications to date have been limited to laboratory settings because the effect is small, requiring care with transducer fixturing and a high degree of operator skill. Furthermore, variations in microstructure have a competing effect. Our approach is to use noncontacting electromagnetic-acoustic transducers (EMATs) which require no couplant to generate sound in the structure and are easily fixtured. We are currently evaluating a method which has potential to suppress the effect of microstructure variation and which gives all the components of the plane stress tensor.

External Collaborations

Industry. Caterpillar Inc; Ultrasonic measurement of residual stresses in welded structures. Sonic Force Corp; Ultrasonic measurement of stress in steel buildings.

Government agencies/laboratories; Virginia Transportation Research Council and the Federal Highway Administration; ultrasonic measurement of stress in steel bridges.

Planned Outcome

We expect to develop the measurement technology for an ultrasonic strain gage and commercialize the technology with the help of CRADA partners.

* Student at the University of Colorado

Accomplishments

Separation of Microstructure and Stress Effects

Most techniques for measurement of ultrasonic stress determine some combination of the principal stresses. Furthermore, they require some means of correcting for the effect of microstructure on sound speed. We are investigating a method which will determine absolute (as opposed to relative) magnituded of all components of plane stress. Furthermore, the method offers the possibility of suppressing the effect of microstructure variation. Primary interest is focused on measuring stress on structural components such as rolled plate and I-beams. We use EMATs to generate and receive shear waves propagating through the material thickness dimension. In the absence of stress, we typically find that the wave velocity is greatest when the shear wave is polarized in the rolling direction and smallest when polarized in the transverse direction. When the polarization is oriented at any arbitrary angle between these directions, the wave splits into components polarized along these acoustic axes, and interference effects can be observed as the polarization direction is rotated. If a shear stress exists, these acoustic axes will be rotated away from the rolling and transverse directions.

During FY97, a theory to model the interference between the fast and slow waves was developed. Furthermore, an EMAT which is rotated by a motor at constant angular velocity was assembled. By integrating this transducer with a commercial instrument which measures the phase of the combined fast and slow wave as the EMAT is rotated, the orientation of the fast and slow axes as well as the two velocities can be determined very quickly. This information is being used in different applications to measure stress.

First, to determine residual stress, we deduce the shear stress from the orientation of the acoustic axes and the velocities. Then, the equations of stress equilibrium are used to determine the other stresses. The accuracy of this procedure was established by measuring the residual stress in a specimen which had a known stress distribution.

Second, to use the EMAT as an ultrasonic strain gauge, the times-of-flight of both shear waves were measured at locations of interest prior to the application of a stress. After the specimen was stressed, the orientation of the acoustic axes and the times of flight were remeasured at the same locations. Because the EMAT requires no couplant and minimal fixturing, these measurements could be made very rapidly. Our interactive algorithm for determination of acoustic axes and velocities requires minimal operator skill, and the measurements are less labor-intensive than installation of strain gauges.

Interaction with Industrial Partners

A collaboration with researchers from Virginia Transportation Research Council and the Federal Highway Administration was formed in previous years to characterize pin-and-hanger connections. These connections are used to suspend an interior span from the outer spans of a bridge in order to compensate for the effects of thermal expansion. If corrosion at the pin occurs, it can cause torsion in the pin and bending of the hanger. It is anticipated that ultrasonic measurements of the bending stress in the hanger will be a simple and robust way to determine the status of the connection. For simulating measurements on a hanger with a "bad" connection, we have constructed a cantilever section to fit in our mechanical testing machine. Measurement were made to simulate 3 scenarios: (1) continuous monitoring with an EMAT left *in situ* to act as a stress sensor; (2) intermittent monitoring, in which measurements are made at the time of hanger installation and then again later in order to determine any changes in stress; and (3) measurements with no *a priori* information. For all three scenarios, ultrasonic measurements were compared to data taken with strain gauges. For the continuous monitoring-scenario, excellent agreement was obtained between the EMAT and the strain gages as the machine applied various loads. In the intermittent monitoring scenario, the ultrasonic-strain-gauge approach was used to determine the repeatability in measurements with the transducer being replaced many times at the same location before and after application of stress. The rms error for this procedure was less than 7 MPa (1 ksi) in steel. The third scenario essentially represents measuring a residual stress state. In this case, the error was larger but still only 11 MPa (1.5 ksi). For many practical applications these errors will be quite acceptable.

As part of a CRADA with Sonic Force Corporation, NIST has trained their personnel and helped modify their instrumentation to perform shear wave velocity measurements on structural steel I-beams. As a result, they have been able to measure stress in floor beams at construction sites in order to determine the changes in beam loading during the pouring of concrete to form the floor. In their normal course of business, Sonic Force has assembled a Technical Advisory Board consisting of internationally recognized experts in bridges, cranes, and railroad rails. With guidance from these experts and technical support from NIST, they are attempting to determine whether ultrasonics can be used to measure the (small) changes in stress in building support structures that result from earthquake damage.

Plans are underway to use the ultrasonic-strain-gauge approach to monitor the development of residual stresses in box beams as they are fabricated by welding the corners together. This effort supports an ATP project at Caterpillar, Inc. and will be carried out during the spring of FY98.

Publications

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

2. A.V. Clark, T.L. Anderson, M.G. Lozev, and P.A. Fuchs, “Quantitative Safety Analysis Using Fracture Mechanics and Ultrasonic Stress Measurements,” ibid.

3. A.V. Clark, P.A. Fuchs, M.G. Lozev, D. Gallagher, C.S. Hehman, “Ultrasonic Measurement Of Stress In Bridges”, Proc. 8th International Conference on Nondestructive Characterization of Materials, Boulder, CO (June, 1997).

4. A.V. Clark, M.G. Lozev and P.A. Fuchs, “Field Tests of The Ultrasonic Stress Measurement Technique On Two Bridges,” submitted to J. Nondest. Eval.

5. A.V. Clark, S.R. Schaps, “An Acoustoelastic Determination of Residual Stress by Measurement Of Resonance Peaks And Phase Shifts,” submitted to J. Nondest. Eval.

6. P.A. Fuchs and A.V. Clark, “An Ultrasonic Instrumentation For Measuring Applied Stress In Bridges,” submitted to to J. Nondest. Eval.

7. A.V. Clark, C.S. Hehman, D. Gallagher M.G. Lozev, and P.A. Fuchs, “Ultrasonic Measurement Of Stress In Pin And Hanger Connections,” to be published in Proc. Structural Reliability Technology Conf., San Antonio, TX (March, 1998)

Project: WAVEFORM-BASED ACOUSTIC EMISSION

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

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 (e.g. bridge steels).

- Investigate applicability of above methods for use in thick-walled fiber-composite structures (e.g. offshore technology applications and composite bridges).

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 technical approach, which is beyond that currently commercially offered for either resonant or waveform-based acoustic-emission technology, follows a multifaceted development of all the key components that are relevant to a wideband application of acoustic emission technology. These components include development of wideband high-sensitivity sensor/preamplifiers, high-speed wide-dynamic-range digital recording data-gathering systems, finite-element modeling of the predicted far-field displacement waves from relevant acoustic-emission sources, wideband experimental acoustic-emission displacement waveforms from materials of interest, the development of signal-processing techniques to accurately indentify source locations and types, and experimental studies of simulated acoustic-emission wave propagation. The scope in FY97 covered three phases: (1) finite-element modeling of buried dipole sources in the near- and far-field of thick plates; (2) wideband waveform characterization of acoustic emission during fatigue crack growth as contrasted to the old acoustic-emission technology using resonant sensors; and (3) studies of the characteristics of wave propagation in thick-walled fiber composite shells. The third phase is in support of an ATP related program, "Composite Production Risers". Special equipment and facilities include a specially modified

* NIST, Applied and Computational Mathematics Division

hydraulic materials testing system, multiple coupled waveform recorders (12-bit, 10 million samples per second digitization rate), and specially developed high-sensitivity, wideband acoustic-emission sensor/preamplifiers. A commercially available waveform-based acoustic-emission recording system was evaluated for use in this program, as well.

External Collaborations

Academic. Collaborating with University of California, Los Angeles, on modeling near- and far-field displacement results, by independent methods, using a buried acoustic-emission "point-source".

Private Laboratory. Relationship of the physical mechanism of an acoustic-emission crack source in steel to the mathematical moment-tensor acoustic-emission characterization of the same source with SRI International. Provided NIST-developed sensor and amplifier to Lockheed-Martin for evaluation of their acoustic emission program on the X-33. Also collaborating with Lincoln Composites and Stress Engineering Services in studying mechanical-physical properties of fiber reinforced composites.

Government Agency. The Federal Highway Administration sponsored the development of waveform-based acoustic emission for the NDE of bridges. NIST provided technical expertise in developing specifications and in evaluating proposals for the Federal Highway Administration for an SBIR for an advanced high-speed waveform-based acoustic-emission system. NIST evaluated proposals and monitored phase I of this SBIR and evaluated Phase II proposals and is currently involved in monitoring phase II. Transducer technology developed under this program is used in other NIST programs to facilitate accurate determination of ultrasonic attenuation (e.g. Nuclear Regulatory Commission program).

Commercial Companies. Digital Wave Corporation collaborated with NIST in evaluating waveform-based equipment for use in monitoring fatigue-crack growth. Developed interactions with Grand Junction Steel for welding of steel test samples, per typical bridge specifications for future acoustic-emission testing.

Planned Outcome

The anticipated outcome of this acoustic-emission effort will be fundamental information about micro-deformation and defect response of various materials to applied stress. This information will provide unique input to basic studies (e.g. damage mechanics) by materials scientists and enhance the application of nondestructive characterization of the response of structures (e.g. bridges) to applied loads. Further, the accurate location of concentrated micro-damage in materials by acoustic-emission methods will be enhanced and the ability to separate extraneous noise from damage-related acoustic emission will be significantly upgraded. Finally, the exchange of technical ideas and establishment of collaborations, with various vendors, will establish the viability of third-generation acoustic emission systems. To that end, NIST will establish a laboratory that can be used as a test bed for the evaluation of advanced acoustic-emission systems.

The anticipated outcome of the ATP supported project is two-fold. First, techniques to determine, as a result of acoustic-emission monitoring of a proof test, the presence and location of any concentrated damage in a composite structure with thick walls will be developed. Second, a means will be established to relate detected damage to macroscopic degradation in properties of a composite structure.

Accomplishments

Validated the finite-element modeling code for the fundamental buried-dipole acoustic-emission source. Validation was accomplished by comparison with other independent results (published and unpublished). Studied finite-element parameter relationships, such as resolution and source size necessary to create a finite-element-based buried "point source". Also examined effects of plate thickness on far-field out-of-plane displacements.

In support of industry and ASTM, NIST is investigating the microstructural, geometrical, and pulse-shape factors that can cause apparent variations in velocity and signal amplitude. Also NIST is developing a model that will enable sensitivity studies to establish rational tolerances for inclusion in future documents. Finite-element approaches developed for acoustic emission studies were used to model relevant ultrasonic wave propagation.

NIST is taking a leading role in assisting in organizing the first unified international meeting of the two major and oldest technical groups from different countries dedicated to acoustic-emission research and applications. The "International Acoustic Emission Conference" will provide an international forum for participants to present and discuss industrial and research applications of the science and technology of acoustic-emission. In addition, the conference will provide a forum to compare and contrast the acoustic-emission technologies used in the past with more recently developed approaches.

Completed paper on extraneous-noise acoustic-emission waveforms generated during fatigue cycling of both thick and thin steel samples. Compared waveforms from commercial resonant sensors and wideband sensors. Demonstrated that standard fixed threshold approaches, as currently practiced with resonant sensors, do not eliminate the misidentification of extraneous acoustic-emission as real crack-based acoustic emission. Showed that wideband waveform approaches allow classification of various types of extraneous acoustic emission generated during fatigue cycling. Gathered an acoustic-waveform database at low crack growth rates for various bridge steels as contrasted to an aluminum alloy.

Attended Acoustic Emission Working Group Meeting in Chicago, where M.A. Hamstad delivered the Gold Medal Speech. Hamstad also presented two papers on acoustic emission at this conference.

Publications

1. M.A. Hamstad, J. Gary, A. O'Gallagher, "Far-field Acoustic emission Waves by Three-Dimensional Finite Element Modeling of Pencil Breaks on a Thick Plate", Journal of Acoustic emission, Vol. 14, No. 2, 1996, pp. 103-114.

2. M.A. Hamstad and J.D. McColskey, "Wideband and Narrowband Acoustic Emission Waveforms from Extraneous Noise During Fatigue of Steel Samples," Accepted for publication by the Journal of Acoustic Emission.

Project: ULTRASONIC STANDARDS FOR MATERIALS CHARACTERIZATION

Project Leader: C.M. Fortunko
T.P. Lerch, K.H. Hollman, M.C. Renken, S.A. Kim, and P. Heyliger

Objectives

Develop a system of ultrasonic methods for characterization of microstructures of advanced materials. Disseminate the results through technical publications, participation in standards activities, and collaborations with other laboratories and industry.

Technical Description

To use ultrasonics for material microstructure characterization, mechanical-property and internal-strain determination, and validation of micro-mechanical models, absolute elastic-wave velocities must often be known with an uncertainty of less than 0.1%. We can now achieve this level of performance in our laboratory in the 100kHz - 10MHz region. However, accuracy of typical laboratory measurements is of the order of 1%, and larger. Accuracies of elastic-wave velocities calculated from mechanical tests are typically of the order of 5%, and may be larger.

Another quantity, attenuation, must also be measured with high accuracy. However, current experimental methods are generally inadequate and should be improved and harmonized. We plan to address this in the future.

We have already demonstrated the feasibility of high-accuracy measurements of elastic-wave velocities and their utility in microstructure characterization and validation of micro-mechanical models. We now use such methods, including Resonance Ultrasonic Spectroscopy (RUS) and certain short-pulse, time-of-flight methods for routine, high-accuracy determination of elastic-stiffness coefficients of materials with complex microstructures and single crystals. We are also improving our experimental capabilities and developing measurement models and prototype artifact materials. Potential candidates for reference materials include high-quality single crystals of fused silica and silicon.

Our current effort also includes development of appropriate contacts with the scientific and industrial communities that rely on accurate velocity measurements in quality control. To help accomplish this goal, we have developed collaborations with several leading laboratories (Los Alamos, (German) Federal Institute for Materials Research and Testing (BAM)) and industry-consensus organizations, such as the American Society for Testing of Materials (ASTM) and International Institute of Welding (IIW). Discussions are now under way with another government agency to explore the use of our measurement techniques for characterization of single crystals used in frequency-control applications.

In FY 1997, we completed an experimental study leading to the quantification of uncertainties of the RUS, which we use routinely to determine elastic-stiffness constants. As a consequence, we now have a good understanding of the physical factors that affect accuracy and resolution of this method. The study was conducted using specimens made of high-quality fused silica and single-crystal silicon. We are now conducting a similar study for our short-pulse, time-of-flight methods. As a result of this study, we expect to be able to model and correct for the effects of several physical phenomena that affect our measurements, including diffraction, sampling-rate and digital quantization effects, time-window effects, transducer thickness, temperature, and air loading. This study should be completed in FY 1999.

We have already had the opportunity to apply the results of our work to an industrial standards program that has international implications. In March 1997, ASTM Committee E-7 on Nondestructive Testing approached NIST for help in resolving known problems with ASTM E-164 "Standard Practice for Ultrasonic Contact Examination of Weldments". This document includes an Annex describing the US design of the International Institute of Welding (IIW) calibration block. Currently, the document does not stipulate the requirements and tolerances for the speed of sound and attenuation, due to the lack of a consensus. Yet, the calibration block is used in the US and elsewhere to establish critical factors, including location of ultrasonic beam exit point, angle of refraction, and important search-unit settings, such as gain. NIST agreed to work with ASTM towards a long-term solution to the problem.

In FY 1997, we have characterized three IIW-type calibration blocks by accurately measuring ultrasonic velocities along the three principal directions of propagation. As a result, we were able to identify many of the microstructure-related factors that might explain the block-to-block variability that was observed in the US and elsewhere. We are also developing a computer model that will enable us to perform the sensitivity analyses needed to identify and quantify the critical parameters. We have established a collaborative effort with the (German) Federal Institute for Materials Research and Testing (BAM), which is conducting a similar study.

A current European proposal calls for the ultrasonic velocities of an IIW-type block to be specified with an absolute accuracy of 0.1%. However, it is generally believed that this specification cannot be met and may not even be necessary. We believe that our work will lead to the establishment of a rational specification that can be incorporated in future ASTM and ISO documents. A report summarizing the FY 1997 NIST activity will be given at the January 1998 ASTM meeting.

M. Renken is working on a Ph.D. thesis at the University of Colorado at Boulder. The thesis title is Time/Frequency Analysis of Dispersive Elastic Waves. The goal of this work is to develop quantitative measurement models that are needed to account for time and frequency-domain effects, as well as digitization errors that have been observed in our short-pulse, time-of-flight measurement methods.

External Collaborations: (German) Federal Institute for Materials Research and Testing (BAM), Los Alamos National Laboratory, ASTM, Iowa State University Center for Nondestructive Evaluation. C.M. Fortunko is a member of the Editorial Board for the Journal of Measurement Science and Technology, a publication of the Institute of Physics.

Planned Outcome

Improved US design of an ASTM ultrasonic test block for calibration of ultrasonic instruments. Improved ISO version of the above calibration block. Understanding of the physical factors that affect the accuracy of Resonant Ultrasonic Spectroscopy and development of appropriate reference materials with accurately known elastic constants.

Accomplishments

Demonstrated that the Resonant Ultrasonic Spectroscopy system can be used to determine the elastic constants of homogeneous isotropic and anisotropic materials with an accuracy of at least 0.1%.

Quantified the accuracies of several short-pulse, time-of-flight methods for determining the elastic-wave velocities in homogeneous, isotropic materials.

Improved a short-pulse, time-of-flight method for determination of ultrasonic velocities in isotropic and anisotropic material. The new method will likely lead to more accurate reporting of elastic-constant data.

Identified some of the microstructural effects that affect the performance of IIW-type ultrasonic calibration blocks. Developed collaboration with BAM to address future revisions of ASTM and ISO specifications for IIW-type ultrasonic calibration blocks.

NIST and BAM organized the European-American Workshop on Determination of Reliability and Validation Methods of NDE. The workshop was held on June 18-20 at BAM in Berlin, Germany. Effects of microstructure on NDE measurements were identified as an important parameter by the participants of the consensus session at the end of the Workshop. There were nearly 100 participants representing 19 countries.

NIST was an active participant in a focused workshop on Resonant Ultrasonic Spectroscopy. The Office of Naval Research sponsored the workshop at the Asilomar Conference Center on May 11-15, 1997. In our presentation, we compared RUS measurements with conventional methods.

Publications

1. M.C. Renken, S. Kim, K.W. Hollman, and C.M. Fortunko, "Accuracy of Resonant Ultrasound Spectroscopy: an Experimental Study", in Proc. 1997 IEEE Ultrasonics Symposium, in press.

2. C.M. Fortunko, M.C. Renken, K.W. Hollman, and S. Kim, " An Experimental Evaluation of the Resonant Ultrasonic Spectroscopy Method", in Rev. Prog. QNDE, Vol. 17, D.O. Thompson and D.E. Chimenti Eds. (Plenum, New York, 1997), in press.

3. K.W. Hollman, P.T. Purtcher, and C.M. Fortunko, "Characterizing Martensitic Steel with Measurements of Ultrasonic Velocity", in Nondestructive Characterization of Materials VIII, R.E. Green, Jr., et al. Eds. (Plenum, New York, 1997), in press.

Project: GREEN'S FUNCTION METHODS FOR MATERIALS SCIENCE

Principal Investigator: V.K. Tewary

Objectives

- To develop computationally efficient methods for calculating thermoelastic, elastostatic, and elastodynamic Green's functions for bounded solids.
- To use the linear Green's functions to calculate nonlinear waveforms in anisotropic solids with prescribed boundary conditions.
- To apply the Green's functions in the development of a boundary-element formulation for stress analysis and propagation and scattering of elastic waves in anisotropic solids.

Technical Description

Green's function gives the response of a solid to a localized probe. It gives the solution of a set of operator equations for prescribed boundary conditions and an arbitrary integrable source function. For example, if the probe is a mechanical force and the operator equations are space-time dependent Christoffel equations, the Green's function will give the elastodynamic response of the solid. Since it gives the response of the whole solid, it provides a convenient mathematical technique to model various physical properties of anisotropic solids. Combined with the boundary-element analysis, the Green's function method provides a powerful numerical tool for stress analysis and elastic-wave scattering calculations in engineering materials of different geometrical shapes. Our interest is in solving separate equations of elastic equilibrium separately as well as coupled with equations of thermal diffusion.

Whereas the free-space Green's function can be calculated relatively easily, complications arise in obtaining solutions that satisfy prescribed boundary conditions. We use a virtual-force method for this purpose. In this method, we apply a distribution of virtual forces just outside the boundary that gives a solution of the homogeneous equations. For simple geometrical shapes the virtual forces can be determined analytically. For irregular geometrical shapes, the virtual forces have to be determined numerically and the method becomes similar to the boundary-element method.

In the nonlinear case, first we transform the problem of homogeneous equations with inhomogeneous boundary conditions to the problem of inhomogeneous equations with homogeneous boundary conditions. The inhomogeneity in the equation is treated as a source term. We then use the corresponding Green's function for the homogeneous boundary conditions to solve the inhomogeneous equation. The solution requires solving an integral equation, which we do by using the Born approximation.

The Green's function 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 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. We have a project on setting up of a library of Green's functions on WWW with Internet access, which exploits this characteristic of the Green's functions. This project has been sponsored by the NIST-wide program entitled, "Systems Integration for Manufacturing Applications", (SIMA). Work on this project is being carried out in collaboration with Colorado School of Mines and Iowa State University. The analytical calculation of Green's functions is done at NIST, whereas the boundary-element analysis of elastostatic problems is carried out at Colorado School of Mines and elastodynamic problems at the Iowa State University. The final results for Green's functions for some specific material shapes of industrial interest will be put on the WWW and will be accessible through the Internet.

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 for elastostatics as well as elastodynamics problems. Using the calculated Green's functions, we have developed boundary-element formulations for interface cracks and analysis of moiré fields in anisotropic materials.

External Collaborations

1. Collaboration with Prof. John Berger of Colorado School of Mines on elastostatic calculations on ansiotropic materials for the SIMA project.

2. Collaboration with Prof. Frank Rizzo of Iowa State University on elastodynamic problems for the SIMA project.

3. Collaborations with Martin Marietta on the application of the Green's function/boundary element method to industrial problems.

Planned Outcome

Green's functions would provide an efficient method for modeling and calculating physical characteristics of solids. We plan to develop the method further to include lattice characteristics in the Green's function method that will enable us to study the microstructure of solids.
The Green's-function method combined with the boundary-element analysis makes it suitable for industrial applications. The work on SIMA project will result into a widespread availability of Green's functions on WWW, which will be very useful for industrial applications.

Accomplishments

Elastic waveforms were calculated for nonlinear bulk waves in cubic solids as function of frequency. At large frequencies in the range of experimental interest the results are very close to those reported in the literature using crude approximations. Similarly, a perturbation calculation of the nonlinear Rayleigh waves reproduces qualitatively the same result as reported in the literature.

A boundary-element formulation for the anisotropic crack problems based upon a Green's function has been developed. The traction free-boundary conditions on the crack faces are satisfied exactly with the Green's function using virtual forces so that no discretization of the crack surfaces is necessary. The Green's function contains both the inverse square root and oscillatory singularities associated with the elastic anisotropic interface crack problem. Results are obtained for interface cracking in a copper-nickel bimaterial.

A procedure for analyzing moiré fringe patterns using boundary elements was developed. The kernels of the boundary integrals are based on anisotropic elastic Green's functions calculated earlier for bimaterial problems. The kernels are appropriate for homogeneous problems as well as degenerate isotropic problems. The moiré fringe data provide full-field displacement information and are analyzed in a least squares' sense. The numerical procedure is shown to be a logical extension of the local collocation method developed for linear elastic-fracture problems. It is found that moiré fields associated with both displacement components are needed for an accurate analysis.

An exact transient thermoelastic Green's function has been derived for an anisotropic bimaterial. The analysis is based upon an extension of the Stroh formalism for anisotropic elasticity to the anisotropic thermoelastic problem. The application of the exact Green's function to boundary-integral analysis of thermal-barrier coatings is being studied.

Subcontracts for doing some specific work in the SIMA project were formulated, negotiated, and awarded to Prof. John Berger of Colorado School of Mines and Prof. Frank Rizzo of Iowa State University. In the SIMA project, Green's function for an anisotropic bimaterial composite solid with arbitrary fiber orientation has been calculated and introduced into a boundary-element method (BEM) program. This gives us a general anisotropic, bimaterial boundary-element program that would enable us to analyze materials with general anisotropy. This has direct bearing on the Green's-function library as one of the demonstration problems concerning stress analysis in composite materials. The boundary-element program will be used to generate the discretized Green's functions needed in the library for composite materials with general fiber orientation.

The Green's function for an anisotropic bimaterial composite solid with arbitrary fiber orientation under thermal loading has been calculated. This will allow for thermomechanical analysis of the library entry for composite materials. Improvements to the BEM code used to generate the discretized Green's functions have been initiated. At present, the variation of boundary quantities is taken as piecewise constant. The improved code will allow for piecewise linear variations in the boundary quantities. This will allow for more accurate calculation of discretized Green's functions. Initial graphical user interfaces have been developed and some results have been placed on the WWW.

Publications

1. 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. **19**, 41 (1996).

2. J. Berger and V.K. Tewary, "Boundary-integral equation formulation for interface cracks in anisotropic materials," Comp. Mech. **20**, 261 (1997).

3. J. Berger and V.K. Tewary, "Boundary element analysis of moire fields in anisotropic materials," Engineering analysis with boundary elements, **18,** 317 (1996).

4. 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).

5. V.K. Tewary, "Inversion of elastic waveform data for anisotropic solids using the delta-function representation of the Green's function," Submitted to the Journal of the Acoustic Society of America.

Project: NONDESTRUCTIVE CHARACTERIZATION OF STEEL EMBRITTLEMENT

Project Leader: G.A. Alers
B. Igarashi

Technical Objective

The objective of this program is to relate physical properties that can be measured nondestructively to the mechanical strength properties of materials that can be measured only by destructive mechanical tests. Such relationships are known to exist since empirical correlations between ultrasonic and magnetic measurements and hardness or yield strength can be found in the literature. It is anticipated that the establishment of a scientific basis for these correlations will demand development of models that relate the measurable physical properties to the microstructure and then models that relate the microstructure to the mechanical properties.

Technical Description

During the past few years, NIST has established unique capabilities for making nondestructive magnetic, eddy-current and ultrasonic property measurements with sensors that can operate on materials with a variety of shapes and in non-laboratory environments. Furthermore, models that relate some of these properties with microstructures have been formulated in order to establish a scientific basis for the relationships. During FY97, work on relating Lamb wave velocities in thin sheets of copper, brass and bronze with the rolling and recrystallization textures produce by cold working and annealing these alloys was completed. For applications in steel, work was begun on making a large number of magnetic and ultrasonic property measurements on several types of steel in order to develop a large list of measureables to relate to the large number of microstructures that give steel its broad range of mechanical properties.

External Collaborations

The Nuclear Regulatory Commission sponsored research for development of nondestructive techniques to detect radiation embrittlement in reactor pressure vessels.

Planned Outcome

Nondestructive measurements of physical properties will be related to microstructures in commercial metals so that mechanical strength can be predicted based on known relationships between strength and microstructure. This can greatly reduce or eliminate the need for making destructive strength tests by sampling techniques in production facilities; it could also enable the prediction of remaining life in structural elements that have been exposed to hostile environments in industrial settings.

Accomplishments

Measurement of Magnetostriction. In ferromagnetic materials and, in particular, steel, the application of a magnetic field will change dimensions. This phenomenon, called magnetostriction, can excite ultrasonic waves by producing the magnetic field with a coil of wire carrying an RF current at the frequency of the desired wave. The amplitude of the ultrasonic wave produced under these conditions can measure the value the magnetostrictive coefficients of the particular ferromagnetic material involved. A quantitative model for the efficiency of ultrasonic transducers that operate by magnetostriction was published in the late 1970's. During FY96, NIST made a concerted effort to verify the theoretical model and to establish relationships between the magnetostrictive coefficients and the microstructure of several steels. The latter effort was motivated by the desire to use measurements of the magnetostriction coefficient as a nondestructive materials characterization tool. Early in FY97, noncontact measurements of magnetostrictive ultrasonic transducer efficiency and certain features in the magnetic field dependence of the magnetostriction coefficients deduced from these measurements were correlated with precipitation-hardening mechanisms in ULC and HSLA steels. Later in the year, the techniques were refined and applied to steels of interest to the Nuclear Regulatory Commission as part of a new program to investigate nondestructive methods for monitoring the development of radiation-induced embrittlement of reactor pressure vessels. One of the steels studied was an alloy of iron containing 1.3% copper. This alloy could be strengthened by a heat treatment that allowed coherent copper rich precipitates (CRPs) to form prior to the formation of the equilibrium ϵ phase of copper in iron at room temperature. This CRP phase hardens dilute copper alloys of iron and is believed to form by radiation-enhanced diffusion of impurity levels of copper in reactor pressure vessels. Its detection by nondestructive methods could form the basis for predicting the onset of embrittlement in operating nuclear reactors by a nondestructive test of the vessel. It was found that the magnetostriction coefficient at high fields could be correlated with the Rockwell A hardness of the Fe-1.3%Cu alloy. An attempt to correlate the temperature dependence of the magnetostriction with the ductile-to-brittle transition in A533B reactor pressure vessel steels was initiated at the end of FY97.

Nondestructive Prediction of Strength Parameters. In May of 1997, The Nuclear Regulatory Commission awarded a contract to NIST to investigate several physical property measurements that might be used to monitor the changes in strength of A533B steel as it is irradiated as the wall of a reactor pressure vessel. The measurements would ultimately be applied to the inside of the pressure vessel that is covered with a 10 mm thick layer of 308 stainless steel. Thus, only those techniques that would reflect the properties of the underlying A533B should be considered. NIST, therefore, focused its attention on ultrasonic and magnetic methods that would penetrate the coating. In order to avoid dealing with radioactive samples during the initial feasibility studies, an iron-copper alloy known to simulate radiation hardening by precipitation of fine copper rich clusters was investigated. The properties measured were: the elastic modulus tensor elements, C_{ij}; the intrinsic damping or internal friction as measured by the Q of a vibrational mode of the sample, or by the logarithmic decrement, d, of the decay of resonant vibrations; the non-linear acoustic parameter, b, that controls second-harmonic generation in a propagating ultrasonic wave of finite amplitude; the magnetostrictive coefficient, l; the differential permeability, m; the magnetic coercive force, Hc, and the saturation magnetization, Ms. All of these properties were measured with as high an accuracy as possible on samples of Fe-1.3%Cu that had been quenched and tempered to exhibit a range in values of yield strength, hardness, elongation, and ultimate tensile strength. Linear correlations between the strength parameters and the physical properties m, l and b were observed, while Q and d proved to be sensitive to the state of the copper clusters but were not linearly correlated with the strength. Values of C_{ij} and Hc were more sensitive to the formation of equilibrium ϵ phase copper precipitates than to the copper clusters that control the mechanical properties. During FY98, the physical property measurements will be extended to the A533B alloy and the temperature dependence of some properties will be measured in order to investigate possible relationships with the ductile-to-brittle transition temperature.

Publications

1. G.A. Alers, P.T. Purtscher, J.F. Breedis and F.N. Mandigo, "Monitoring of Texture Development of Copper Alloy Sheet," Proceedings of the 8th International Symposium on Nondestructive Characterization of Materials, Boulder, Colorado, June, 1997.

2. B. Igarashi, G.A. Alers and P.T. Purtscher, "Correlation of Magnetostrictively-generated EMAT Waves with Strengthening Precipitation in Steels," Proc. 1996 IEEE Ultrasonics Symposium, IEEE Cat. No. 96CH35993, Issn 1051-0117, San Antonio, TX.

3. B. Igarashi, G.A. Alers and P.T. Purtscher, "Magnetostrictive EMAT Efficiency as an NDE Tool," Proceedings of the 8th International Symposium on Nondestructive Characterization of Materials, Boulder, Colorado, June, 1997.

4. B.Igarashi,G.A. Alers and P.T. Purtscher. "An Ultrasonic Measurement of Magnetostriction," Proc. 1997 Ultrasonics Symposium, in press.

5. P.T. Purtscher, B. Igarashi, D.C. Hurley, K.W. Hollman, D. Balzar and S. Kim, "Microstructural Characterization of Hardened Steel by Nondestructive Methods," Proc. 39th Mechanical Working and Steel Processing Conference, Iron and Steel Society, Warrendale, PA, Oct. 1997, Indianapolis, IN.

6. G.A. Alers and H.I. McHenry, "Physical Property Measurements for Characterization of RPV Steels," Proc. of EPRI Workshop on NDE for Damage Assessment, La Jolla, CA, Oct. 1997.

Project: **SENSORS FOR INDUSTRIAL NDE**

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

Technical Objectives

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. In addition, reliable sensors are needed after the material is manufactured and put in use in order to make sure that no defects have developed during service. The objective of this program is to develop sensors that can both survive in a mass-production environment and produce outputs useful for process control but can also monitor the degradation of parts as they are used in the hostile environments for which they were designed.

Technical Description

Most sensors must be specialized to the materials on which they are to be used. Furthermore, they 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 this year, three industrial sensor problems have received attention. First, the gas-coupled ultrasonic transducer was improved in order to enhance its reliability for detection of cracking in buried gas pipelines. Second, a portable inspection unit was assembled to make quantitative measurements of the residual stress level in railroad wheel rims as these stresses develop during service on railroad cars. Third, a novel ultrasonic transducer was developed to inspect cables for corrosion damage that can appear after prolonged usage in harsh chemical environments. Fourth, an automatic system for the continuous monitoring of the internal friction and the resonant frequency of cylindrical materials was developed and used to address the old problem of early detection of fatigue damage.

External Collaborations

Industrial

IEM, Inc. for EMAT inspection of railroad rails.
Southwest Research Institute for gas-coupled ultrasonic inspection of gas pipelines

Academic

Colorado School of Mines, Engineering Division for development of a copper cable inspection device.

Sponsors

Gas Research Institute for inspection of buried gas pipelines.
FHWA for early detection of fatigue damage in bridge steels.
FRA for a residual-stress measuring device for railroad wheels.

Planned Outcome

This project provides a major opportunity for technology transfer in that it provides industry and other research institutions with innovative sensors designed to solve flaw detection and materials characterization problems that have eluded conventional NDE methods.

Accomplishments

Air Gas-Coupled Transducers for Gas Pipeline Inspection. The gas industry needs better methods for inspecting 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 demonstrated the feasibility of using the compressed gas itself as the ultrasonic couplant; A cooperative research program with the Gas Research Institute and Southwest Research Institute was initiated in order to show that the gas-coupled approach could achieve the sensitivity necessary to detect stress-corrosion cracking (SCC) in a pressurized-natural-gas transmission pipeline. To achieve this goal, NIST assembled a special pressure vessel, custom pulser/receiver electronics, and unique transducers to demonstrate that crack-like artificial flaws could be detected in smooth, flat test plates when the ultrasonic beam entered the pipe wall at a small angle. During FY97, the work was extended to include more realistic conditions such as curved, somewhat rough pipe surfaces and actual cracks which were very irregular in shape. Mathematical models were developed to improve our understanding of the underlying physical phenomena and improvements were incorporated in the transducer-scanning fixture for the pressure vessel as well as in our pulser/receiver electronics and transducers. As a result, it was determined that the condition of the inner surface of the pipe, along with internal reverberations in the transducers, could be major impediments to crack detectability. By briefing the industry on the objectives and status of the project, it was decided to shift the emphasis away from crack detection towards the characterization of corrosion damage. Southwest Research and the industry are now collecting and characterizing a library of specimens containing examples of common corrosion type defects while NIST is optimizing the transducer designs for minimizing reverberations and for directing the sound waves perpendicular to the pipe wall.

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.

Early in FY97, the FRA 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 rail car. A compact, portable computer with space for adding a few circuit boards was purchased, and software to control the measurement process was written for it. A powerful signal-generating board and a fast signal-digitizing board were obtained from commercial vendors and installed inside the computer. The EMAT sensor used in previous years for feasibility demonstrations was remounted in a probe unit suitable for repeated use on wheels in the field. A compact preamplifier unit was added to the computer case and a very sturdy cable was constructed to connect the probe to the portable computer. Tests of the final system showed that it performed as well as the old laboratory version that had been used to develop the stress-measuring technique. By the end of FY97, several demonstrations of the portable unit had been performed for FRA and AAR representatives at the Transportation Test Center in Pueblo, Colorado.

During FY97, the Air Force awarded a Phase I SBIR program to International Electronic Machines Corporation (IEM) of Albany, New York to develop a technique for measuring the longitudinal stress in the rails of a rocket-testing track. The stress to be measured is produced by a tensioning mechanism designed to guarantee that the each rail is perfectly straight over a long distance. Thermal stresses produced by changes in temperature during the day and night can modify the tension so that the actual stress distribution along the track can become different from what is required for safe operation of the facility. Since NIST has had long experience with ultrasonic techniques for stress measurement, IEM asked NIST for assistance in reviewing a variety of possible approaches and in performing of some preliminary tests on short pieces of rail mounted in the mechanical testing machines available in Boulder.

Flaw Detection in Cables. The Center for Advanced Control of Energy and Power Systems at the Colorado School of Mines was presented with the problem of detecting corrosion in the buried copper network that provides the ground plane for electrical utility substations. Electrical resistance methods become useful only after the corrosion has become very serious. Ultrasonic methods require introducing the acoustic energy through copper cables made of many individual wires twisted together. This presents an irregular cylindrical surface to any contacting transducer and the individual wires tend to break up the wave fronts and attenuate the signals. By designing an EMAT that coupled to the entire circumference and generated ultrasonic waves with wave lengths that were long compared to the diameter of the individual strands, it was possible to excite a torsional mode of vibration that could propagate over long distances and that would reflect from broken strands or corroded regions that changed the gross dimensions of the cable. A student from CSM assembled the EMAT according to NIST specifications and used NIST instrumentation to characterize the torsional wave mode it produced. By building a receiver EMAT and by optimizing the transmitter/receiver pair for pulse-echo operation, it was possible to demonstrate the detection of flaws in the cable several feet away from the transducers even when the cable was buried in the ground. Off-site demonstrations and field trials of the system are planned for early in FY 98.

Resonant-Body Sensors. Measurement of the free vibrational modes of spheres, cubes and cylinders has become a very useful tool for ultrasonic materials characterization because the elastic moduli can be measured very accurately, and the intrinsic damping capacity or internal friction can be determined without having to make large corrections for the measurement method employed. Unfortunately, application of these techniques to industrial sensing problems is difficult because the data reduction programs apply only to very specific shapes and the specimen must be supported and its vibrations sensed by procedures that do not influence the vibrations. 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.

During FY97, a program funded by the Federal Highway Administration was initiated to see whether resonant ultrasonic spectroscopy (RUS) could be used to detect fatigue damage in steel taken from old bridges. The original plan was to machine cylindrical specimens from the bridge steels and from new steels of the same composition, and then to compare their RUS responses. A far more controlled study was then developed in which the new steels were damaged in laboratory fatigue tests, and RUS was used to monitor changes in the elastic constants and the damping as the fatigue damage developed. This approach addressed the much broader question of finding techniques to detect fatigue damage in its early stages when detectable cracks have not yet appeared. To produce cylindrical samples with controlled amounts of fatigue damage, round-bar tensile-test specimens were machined from thick plates of the steel commonly used for bridge construction. The gage section of these specimen was made long enough to accommodate a central region with a slightly larger diameter. The dimensions of this central section compared to the overall length of the gage section were carefully chosen by a theoretical analysis designed to insure that certain torsional waves propagating along the axis of the cylinder would be completely reflected at the diameter change. This would insure that the wave's vibrational energy would be trapped in the central region of large diameter. By placing a special EMAT around the central region, the specific torsional waves could be excited without making physical contact to the specimen. By driving the EMAT for a long time (several milliseconds), the energy in the torsional waves could be increased until the central region became a vibrating mechanical resonator. When the EMAT excitation was removed and the EMAT switched to a preamplifier, the decay of the resonant vibrations could be monitored. The rate of decay of the vibrations measures the intrinsic damping capacity or internal friction of the material, and the frequency of the vibrations measures the elastic modulus that controls the torsional wave mode. Both the damping and the modulus are physical properties that are very sensitive to the microstructure of the material and, in particular, reflect some aspects of the dislocation substructure. Detailed descriptions of the application of this resonant-body sensor to detection of fatigue damage and to radiation-induced embrittlement can be found in the section entitled "Internal Friction Measurements for Monitoring Microstructural Evolution".

Microstructure Development in Copper and Brass Sheet. For the past several years, NIST has had 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 to 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. 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 to deduce three of the Orientation Distribution Coefficients (ODCs) used to describe the anisotropic physical properties of rolled sheet metal. If these three coefficients are used as the coordinates of a three-dimensional graph, it is possible to make a graphical display that exposes how different textures emerge from different rolling and annealing procedures. Since the desirable mechanical properties of these alloys are influenced by texture, this graph makes it possible to define the most efficient path to achieve a texture with particular desirable properties.

Publications

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2. R.A. Livingston, W.Johnson, D. McColskey, C.M. Fortunko and G.A. Alers, "Measurement of Material Properties for Early Detection of Fatigue Damage in Highway Bridge Steels", Proceedings of the 8th International Symposium on Nondestructive Characterization of Materials, Boulder, Colorado (June, 1997).

3. Mike Chenoweth, "Development of Advanced Sensor and Diagnostic Techniques for Health Assessment of Ground Risers", ACEPS Progress Report, Colorado School of Mines, R. Shoureshi, Director (1997) .

Awards

1. 1997 ASNT Outstanding Paper, "Ultrasonic Measurement of Residual Stress in Rims of Inductively Heated Railroad Wheels", published in Materials Evaluation, Vol. 54(8), pp. 929-934 (1996).

ELECTRONIC PACKAGING, INTERCONNECTION, AND ASSEMBLY

Today's U.S. microelectronics and supporting infrastructure industries are in fierce international competition to design and produce new smaller, lighter, faster, more functional electronics products more quickly and economically than ever before.

Recognizing this trend, in 1994 the NIST Materials Science and Engineering Laboratory (MSEL) began working very closely with the U.S. semiconductor packaging, electronic interconnection, assembly, and materials supply industries. These earlier efforts led to the development of an interdivisional MSEL program committed to addressing industry's most pressing materials measurement and standards issues central to the development and utilization of advanced materials and material processes within new product technologies, as outlined within leading industry roadmaps[1]. The vision that accompanies this program — to be the key resource within the Federal Government for materials metrology development for commercial microelectronics manufacturing — may be realized through the following objectives:

- develop and deliver standard measurements and data
- develop and apply *in situ* measurements on materials and material assemblies having micrometer- and submicrometer-scale dimensions
- quantify and record 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

With these objectives in mind, the program presently consists of nearly twenty separate projects that examine key materials-related issues, such as electrical, thermal, and mechanical characteristics of polymer and metal thin films; solders, solderability and solder joint design[2]; interfaces and adhesion; electromigration and stress voidage; and built-up stress and moisture in plastic packages. These projects are always conducted in concert with partners from industrial consortia, individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs within government and industry, including the National Semiconductor Metrology Program (NSMP)[3]. The NSMP is a national resource responsible for the development and dissemination of new semiconductor measurement technology.

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*, NISTIR5817 (http://www.msel.nist.gov/epia1996/contents.htm). Copies may be obtained by contacting Michael Schen at (301) 975-6741 or michael.schen@nist.gov.

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[1] *National Technology Roadmap for Semiconductors*, Semiconductor Industry Association, San Jose, CA, 1994, 1997 (in draft); *National Technology Roadmap for Electronic Interconnections*, Institute for Interconnecting and Packaging Electronic Circuits, Lincolnwood, IL, 1995, 1997 (in draft); *National Electronics Manufacturing Technology Roadmap*, National Electronics Manufacturing Initiative, Inc., Herndon, VA, 1996.

[2] http://www.ctcms.nist.gov/programs/solder

[3] http://www.eeel.nist.gov/810.01/index.html

Project: MECHANICAL BEHAVIOR OF THIN FILMS

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

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 0.1 to 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 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 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 millinewtons, and the specimens cannot be handled directly.

In addition to metallic thin films, nonmetallic thin films of different types are now being used in advanced commercial products. For example, polycrystalline silicon is a common structural material in MEMS (micro-electro-mechanical systems) such as pressure sensors and accelerometers. Many of these devices have mechanical functions, and so their mechanical properties are of interest. 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. Similarly, low-dielectric-constant (low-k) films are now a subject of intense research by the semiconductor chip makers and little is known about their mechanical properties. Because the multilayer interconnect structure on advanced integrated-circuit chips includes several different materials and is subject to thermal stresses, the mechanical behavior of novel dielectric films is important. Specimen designs, fabrication techniques, and measurement techniques are being developed to extend the metal-film measurements to nonmetals.

The main track in the technical approach continues be to develop thin-film tensile test techniques 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 frame is cut leaving the tensils coupon intact. We have had good results using a dental drill to cut the silicon.

The tester's instrumentation produces data on force and grip displacement; however 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 novel technique for reducing the ESPI data was needed because the high optical magnification used in imaging the specimen produce a much lower density of laser speckles than in conventional, low-magnification applications. This development was completed this year. Displacement in the tensile direction is measured at potentially every pixel on the specimen and typically, 10,000 or more out of the 50,000 possible pixels produce useable data.

It is questionable whether the silicon-framed tensile specimen will work for specimens with widths below 10 μm. The specimens may be too delicate. We have developed a new conceptual approach to these tests. Last year a skyhook probe approach which tests the film *in situ* on the wafer was explored with some success for "large" specimens of polysilicon, which had widths down to 10 μm. The "skyhook" reaches in and hooks to the free end of the specimen, then pulls it to failure. A newly proposed approach to smaller specimens is similar. The specimen is in the shape of a "dogbone" coupon, with one end firmly attached to the substrate and the other end, which has a hole in it, free. The proposal is to use our atomic force microscope (AFM) tip as a hook to engage the hole in the free end of the tensile specimen, and then to stretch the specimen to failure while acquiring force and displacement data with the instrumentation of the AFM.

Another disadvantage of the present technique is the use of hydrazine hydrate as the etchant to complete the silicon-framed tensile specimen. This etchant removes silicon from under the gauge section of the tensile coupons. This chemical is the only liquid etchant that we have found that removes silicon but not aluminum. However, it is hazardous, and it has failed to produce useable specimens of SiO_2. We recently became aware of a new and exotic etchant for silicon, xenon difluoride. This material is actually commercially available, for tens of dollars per gram. It has been reported in the literature to be a useful etchant for silicon, and to leave aluminum unetched. It has the appearance of a white salt, and is used in a gas-phase reaction vessel. It sublimates and dissociates on the silicon surface, where released fluorine reacts with silicon to produce the volatile gas SiF_4. This etchant should be useful for: thin-film materials that are incompatible with hydrazine hydrate, such as SiO_2; fabrication of silicon-framed tensile specimens by industrial facilities where hydrazine hydrate is not allowed; and etching specimens so delicate that immersion in liquid may be damaging. The proposed AFM tensile specimens are thought to be in the last category.

External Collaborations

D. T. Read participated in the Improved Registration Project of the Interconnection Technology Research Institute, a consortium of leading US manufacturers of printed-wiring boards. This project made a significant contribution to eliminating $70 million lost annually in scrap due to product rejection, by identifying design features that do and do not affect the reliability of printed-wiring boards. The savings will result from a better alignment between real reliability issues and inspection standards. Read assisted in planning of the experiments and analysis of the data, coordinated the writing of the final report, and presented the results of the project orally in a well-received talk at an ITRI meeting.

Motorola Advanced Interconnect Systems Laboratory, Tempe, AZ — is planning to build a microtensile tester based on the NIST design. I have supplied them with complete information, including mechanical drawings, photographs, assembly instructions, *etc.* Motorola representatives visited NIST Boulder to discuss the collaboration with Division management and to work with the NIST apparatus. Motorola fabricated several silicon wafers with aluminum, copper, and nickel tensile specimens. Testing is in progress on these materials.

Intel, Santa Clara — suggested that we measure the mechanical properties of $TiAl_3$. This is an intermetallic compound which may be formed during the manufacture of advanced integrated circuit chips; however, they wanted us to make the specimens. One attempt was made to produce a layer of $TiAl_3$ on a silicon wafer. X-ray diffraction and transmission electron microscopy (TEM) data are inconclusive regarding the molecular state of the film, so we do not know how much $TiAl_3$ was actually produced. We are continuing with this study.

Dow Corning Corp. — Dr. Hui Liou visited NIST and gave a seminar describing a spin-on glass that forms a low-k dielectric. He suggested that we collaborate to perform tensile tests on this material. So far we have supplied him with two iterations of silicon wafers, and he has prepared films of their product and returned the wafers for testing. We have attempted to make silicon-framed tensile specimens from these wafers. The chemistry of this material is sufficiently different from that of SiO_2 that adaptation of the fabrication process is necessary.

University of Colorado — D. T. Read served on the Ph.D. committee of Wan Suwito. Mr. Suwito completed his thesis, which was based in part on experiments in a microtensile tester built at the university according to the NIST design.

Planned Outcomes

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. Two highlights of FY97 were the development of the speckle interferometry method for displacement measurement for thin film tensile tests, and Motorola's decision to implement their own microtensile tester. Standard test methods remain in the future. The first milestone 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 the prospects are good. A more distant prospect is a standard test method based on tensile testing using the AFM.

Accomplishments

Completion of development of microscopic speckle interferometry technique for displacement measurement. The new data reduction technique makes possible the use of speckle interferometry at high optical magnifications. A paper on the technique was prepared for *Measurement Science and Technology*. Speckle interferometry results confirmed the low values for Young's modulus previously seen in copper thin-film specimens measured by other techniques. We have examined the crystallographic texture of some of our specimens by X-ray diffraction, and found that it is strongly <111> as expected. This does not explain the Young's modulus results, but it does quantify an important variable. Work with W. Suwito of the University of Colorado on epitaxial silicon layers 15 μm thick included Young's modulus measurements by speckle interferometry. The important result was that Young's modulus values in agreement with bulk values were obtained for the <100> and <110> directions. The technique was also used to measure Young's modulus on additional specimens of previously tested materials such as titanium-aluminum multilayers.

Tensile tests were conducted samples of electrodeposited nickel and sputtered aluminum from Motorola. As mentioned above, we supplied them with the necessary information to allow them to build their own microtensile tester based on the NIST design.

The xenon difluoride reaction etch system was built and a series of attempts were made to etch all the way through silicon wafers. Eventually, it was demonstrated that this relatively innocuous etchant can be used to produce silicon-framed tensile specimens.

Data on correlation between microtensile and nanoindentation measurements of thin film mechanical properties were reported at two conferences. Our results, and the general opinion of researchers in the field, is that these correlations are now at best qualitative.

Final forms of manuscripts "Tension-tension fatigue of copper thin films" and "Piezo-actuated microtensile tester" were completed as was "PWB Hole-to-Land Misregistration: Causes and Reliability, ITRI Project Final Report," a collaboration with industry.

Organization of the symposium, "Application of Fracture Mechanics in Electronic Packaging Materials" for the 1997 ASME International Mechanicanical Engineering Congress and Exposition, November 17-21, 1995, Dallas, Texas.

David Read continues as Associate Technical Editor of the *Journal of Electronic Packaging*.

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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. "Young's Modulus of Thin Films by Speckle Interferometry," by D. T. Read, *Measurement Science and Technology* (accepted for publication).

4. "PWB Hole-to-Land Misregistration: Causes and Reliability, ITRI Project Final Report," by David Read (report coordinator), David Arivett, Roger Jones, Bob Holmes, Tom Paur, Don Pucci, Gary Roper, Richard Snogren, Jack Fisher, Fred Hickman, John Phinney, and Marshall Andrews, Interconnection Technology Research Institute, Austin, Texas.

5. "Opportunities and Needs for Interfacial Fracture Mechanics in Microelectronic Packaging Industry," William T. Chen, David Questad, David Read, and Bhagat Sammakia, in Application of Fracture Mechanics in Electronic Packaging, proceedings of ASME IMECE, 1997, edited by D. T. Read and W. T. Chen.

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Project: EXPERIMENTAL MICROMECHANICS BY e-BEAM MOIRÉ

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

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. 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. Demonstrate the capabilities of the e-beam moiré measurement technique to potential industrial users interested in displacements introduced into electronic packaging and interconnect structures by mismatch in thermal expansion.

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 to 500 by 500 μm. This is accomplished by preparing the specimen surface with crossed-line gratings at pitches of 350 nm to 1 μm and dot-array grating at pitches of 100 nm to 200 nm, using electron-beam lithography, and observing them in the scanning electron microscope at magnifications from 200x to 2000x. Deformations produce changes in the local moiré fringe density. These changes are analyzed to give the complete normal and shear deformations.

External Collaboration

3M — The electron-beam moiré technique is being used to evaluate a series of chip-on-glass specimens, assembled with different curing procedures. The specimens are loaded thermally and the quality of the anisotropic conductive adhesive cures is compared using electron-beam moiré.

Intel — We are participating in a comparison of nanoscale displacement measurement techniques sponsored by Intel, Chandler, AZ. Intel provides specimens of multi-chip module substrate (FR4) material to be thermally loaded. Displacements will be calculated and compared with other techniques. The goal of this exercise is for Intel to determine which technique best meets their analytical needs.

Intel — We have received specimens containing layered interconnect structures from Intel, Hillsboro, OR. Intel is seeking thermally-induced displacement data to support their modeling effort. The on-chip features and displacements are very small, requiring further development of the technique in order to make the measurements.

Colorado School of Mines — Collaboration with Prof. J. R. Berger on a study to apply improved electron-beam moiré techniques and advanced modeling to determination of deformation modes of conductive adhesives.

Planned Outcome

Continued successful development of the e-beam moiré technique will permit measurement of displacements of less than 10 nm. Displacement capabilities of this order of magnitude are necessary to measure displacements likely to occur within the on-chip interconnects now used in the industry. Development of durable, high-contrast gratings will be the first step in the study of thermal fatigue of electronic packages. Once we have the capability of measuring displacements during thermal fatigue, it then becomes likely that we will be able to identify potential failure sites and observe how failures occur. Transfer of the measurement technology to the industry is a continuing element of the project.

Accomplishments

Dot arrays were produced with pitches of approximately 100 and 200 nm. The arrays were thermally loaded, and data collected and analyzed for the first time for complete normal and shear deformations at this spatial resolution.

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 behavior for the different locations was analyzed.

Participation at the request of Intel (AZ) in an evaluation of nanoscale displacement measurement techniques and at the request of Intel (OR), use of the e-beam moiré to attempt to measure displacements within on-chip layered interconnect structures.

Publications

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

2. "Electron-Beam Moiré Study of Local Deformation in Conductive Adhesives," E. S. Drexler, and J. R. Berger, 1997, Symposium Proceedings of the Fall 1996 Meeting of the Materials Research Society: *Electronic Packaging Materials Science IX*, Vol. 445, S. K. Groothuis, P. S. Ho, K. Ishida, and T. Wu, eds. Materials Research Society, Pittsburgh, PA) pp. 75-80.

3. "Thermal and Mechanical Deformation Measurements of Conductive Adhesives Using Electron-Beam Moiré," Drexler, E. S., M.S. Thesis (Colorado School of Mines, CO, 1996).

4. "Boundary Element Analysis of Moire Fields in Anisotropic Materials," *Engineering Analysis with Boundary Elements*, J. R. Berger and V. K. Tewary, Vol. 18, No. 4, pp. 317-325, 1996.

Project: STRESS VOIDING AND ELECTROMIGRATION

Principal Investigator: R. R. Keller
C. E. Kalnas, J. M. Phelps

Objectives

Develop a mechanistic understanding of the microstructural processes controlling stress voiding (SV) and electromigration (EM) in interconnect structures. Assess and, when necessary, modify microstructurally based models that sufficiently describe similar behavior in bulk metals. Develop, through knowledge of microstructural mechanisms, standard test structures for assessing stress voiding reliability, and the interactions of stress voiding with electromigration. Investigate the effects of metallization/passivation interface modification on the resistance to stress voiding and electromigration. Determine the effects of physical vapor deposition (PVD) target microstructures on thin film microstructures and interconnect reliability.

Technical Description

Stress voiding and electromigration are failure phenomena that presently limit the reliability of narrow interconnects. They occur during thermal- and electric current-induced stressing, respectively. The end result is the formation and growth of voids in the metal due to the development of severe tensile stresses. Such stresses result from differential thermal expansion among the metal, substrate and rigid passivation overlayer, or from atomic flux divergences due to strongly non-uniform local diffusion during electrical current stressing. The impact of SV and EM 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 are essential to the continued development of metallizations on a submicron scale, as specifically identified in the 1997 SIA Roadmap. Knowledge of microstructural effects on interconnect reliability allows for the design and fabrication of standard test structures for assessing the susceptibility of interconnects to stress voiding and electromigration. Included in such structures is the possibility for studying the interactions between stress voiding and electromigration.

One approach to controlling interconnect reliability involves variations in PVD target materials. Changes in target microstructure are suspected to lead to the development of different interconnect microstructures, and hence offer a possible means for controlling interconnect reliability. Target materials of the same nominal chemistry are fabricated by different methods to produce different microstructures and composition distributions; thin films are then deposited using the various targets and characterized subsequently. Films will also be fabricated into narrow lines for determination of SV and EM behavior. A second approach to controlling interconnect reliability centers on the fact that stress voids typically nucleate at intersections of metallization grain boundaries with the passivation/interconnect interface. The adhesion between interconnect metallizations and surrounding passivation will be controlled through various interface modification schemes in an attempt to reduce the occurrence of stress voids.

Electron microscopy and, in particular, electron diffraction techniques are used to quantitatively characterize on a local scale the microstructures of films and narrow metallizations for interconnects. Backscatter Kikuchi diffraction, orientation mapping, and transmission electron microscopy 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, and correlated to interconnect reliability.

External Collaboration

TexSEM Laboratories — Collaboration with D. Field in a study of stress voiding and thin-film microstructures in films used as conductors in microelectronic structures.

Tosoh SMD — Collaboration with A. Bolcavage and C. Wickersham in a study of sputtering target metallurgical effects on thin-film microstructures and reliability.

Intel Corporation — Collaboration with T. Marieb in a study of stress voiding and electromigration in aluminum lines for microelectronic structures and in a study of the effects of global interfacial contamination on stress voiding.

Cornell University — Collaboration with J. Nucci in a study of stress voiding and thin-film microstructures in copper films used as conductors in microelectronic structures.

University of Michigan — Collaboration with J. Sanchez, Jr. and C. Wauchope in studies of microstructure and diffusion in films used as conductors in microelectronic structures.

Planned Outcomes

Identification of microstructural information associated specifically with void formation and growth in interconnects subjected to stress voiding and electromigration, including variations in grain-boundary structures and residual strains and stresses. Development of electron microscopy-based characterization techniques for routine assessment of interconnect reliability on a microstructural level. Design and fabrication of standard test structures for assessing stress voiding and electromigration.

Accomplishments

Finished 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. Discussed possible implications of structures that lead to stress voiding on subsequent electromigration behavior. On a local scale, grain-boundary triple junctions that favor rapid growth of stress void, may not necessarily favor rapid growth of electromigration void. Conversely, junctions favoring rapid growth EM voids may not necessarily have been favorable for growth of SV void.

Used orientation mapping techniques to characterize bulk sputtering target materials, nominally composed of Al-0.5Cu, but fabricated by different means. Targets consisting of AlCu precipitates, typical of targets found commercially, showed much larger grain sizes than targets of more homogeneous composition, but similar (100) texture strengths. Studies of the corresponding differences in film microstructures are underway. This effort is a collaboration with H. Schafft of the EEEL Semiconductor Electronics Division.

Designed first stress voiding test structures, for incorporation onto the NIST34 standard test chip, in collaboration with H. Schafft of the EEEL Semiconductor Electronics Division; anticipate fabrication to be completed by spring 1998. These are geometrically simple structures intended for assessing effects of linewidth and serpentine meandering on void densities, as well as for measuring biaxial film stresses by X-ray diffraction. Began design of structures for the next generation of stress voiding testing.

R. R. Keller co-organized and co-edited the symposium "Materials Reliability in Microelectronics VII," at the 1997 Materials Research Society Spring Meeting.

Publications

1. "Grain Boundary Misorientation Angles and Stress-InducedVoiding in Oxide Passivated Copper Interconnects," J. A. Nucci, R. R. Keller, D. P. Field, and Y. Shacham-Diamand, *Applied Physics Letters* 70, 1242-1244 (1997).

2. "Local Textures and Grain Boundary Structure Characterization in Voided Copper Interconnects," R. R. Keller, J. A. Nucci, and D. P. Field, *Journal of Electronic Materials* 26, 996-1001 (1997).

3. "Materials Reliability in Microelectronics VII," J. J. Clement, R. R. Keller, K. S. Krisch, J. E. Sanchez, Jr., and Z. Suo, editors, *Materials Research Society Symposium Proceedings,* v 473 (1997).

4. Effects of Local Crystallography on Stress-Induced Voiding in Passivated Copper Interconnects," R. R. Keller and J. A. Nucci, *NATO Advanced Study Institute Series: Electron Crystallography*, in press (1997).

Project: THERMAL CONDUCTIVITY OF THIN FILMS

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

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) and their packages, for both in-plane and out-of-plane thermal transport. Acquire the requisite technology for manufacturing suitable specimens of polysilicon, silica glass, and metallizations for measuring their heat transport at length scales of the order of about one micrometer. Measure metallization traces, polysilicon and silica glass, 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. Ultimately we will produce thin-film test specimens of thermal conductivity for evaluation and use by industry.

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. Dr. Gordon Moore, chairman emeritus of Intel Corp., predicts that the minimum dimension of lithographic patterning on ICs will soon be only 0.18 μm ("Moore's Law Extended: The Return of Cleverness", Industry Insights, *Solid State Technology*, p. 364, July 1997) . The advantages of reduced length scales are necessarily accompanied by greater packing density of the individual devices, with attendant increase in density of generation of heat (Moore: "If you keep scaling, power becomes an increasingly important problem", *ibid.*). Unless this increased heat production can be efficiently removed from the IC, the lifetime-to-failure of critical elements of the package may be severely compromised due to diffusive degradation of elements within ICs, or the element 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, this work 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. Long measurement times are required by absolute steady-state techniques. 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, convection and radiation. Transient methods determine the thermal diffusivity, from which the conductivity may be calculated, if the density and specific heat capacity are also known. There is on-going debate as to the accuracy of transient methods. 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.

The anticipated outcome during FY98 is that the ongoing exploratory measurements will be completed. What is learned from these studies will form the basis for design of the more sophisticated second round of devices. During this second phase, the measurement system will be improved, and specimen design features needing improvement will be identified and carefully studied. This work will provide experimental data, and measurement systems and standards for thermal measurements of thin films will be developed.

The specimen technology that we are now developing is a restricted technology adopted temporarily for achieving a match between some of the present practices of micromechanical engineering and the needs of this investigation. The preliminary technology being used for the first cycle of specimen manufacture is simpler and less expensive, while adequate for the initial work. A more sophisticated technology, permitting a greater range of choice in semiconductor and metallization layers will be used to complete the final target measurement system and standard test structures.

External Collaboration

A new ASTM subcommittee, E-37.05, on thermal properties of thin films is being formed. At a recent (late October 1997) joint conference on thermal conductivity and thermal expansion, where E-37.05 was being organized, participation by us in this subcommittee was offered.

Planned Outcome

Benefits that will result from successful completion of the project will include: (1) transient methods for determining thermal conductivity of thin films will be critically compared to the steady-state method being developed here; (2) a modular structure for determining thermal conductivity of in-situ thin-film structures will be developed that will be easily incorporated into production wafers for IC devices; (3) the method developed here will be easily modifiable, if necessary, for incorporation into other IC packages of custom design.

Accomplishments

Device designs were submitted to MCNC on 11 November 1996 for their MUMPs-16 device manufacturing run and were received in March of 1997. During the wait for the return of these specimens, other design ideas were developed and submitted to MCNC for the MUMPs-19 run of May 1997, and these devices were received in August 1997. Also during the waiting period, hardware (computer, current source, digital voltmeter and scanner, and breadboard), was assembled and software was written for efficient mounting and measuring of the electrical and thermal properties of the thermal-conductivity devices. Further software is being developed to select those portions of data needed for specific analysis, such as electrical-property data and, separately, thermal-property data of the circuits so that layer-specific parameters may be calculated. This will give us the separate electrical and thermal conductances of the metallization and polysilicon layers.

For the MUMPs-19 run, some innovative designs were developed that (if the manufacturing processes work as planned, delivering good adhesion between polysilicon layers) will also allow inter-layer contact resistance between polysilicon layers to be determined. Other designs were developed that will use resistance elements as both heaters and resistance thermometers. This will allow temperature measurement by IR microscopy and by either resistance thermometry (or, if desired, thin-film thermocouples) to be compared, and the more reliable method selected.

Publications

Earlier work in the field of thermal conductivity resulted in FY97 publication of:

1. D. Smith "Thermal Conductivity of Fibrous Glass Board: An Intercomparison for Guarded Hot Plates and Heat Flow Meters," *NIST Tech. Note* 1391 (May 1997)

2. D. Smith, "Thermal Conductivity of Fibrous Glass Board by Guarded Hot Plates and Heat Flow Meters: An International Round Robin," *Int. J. Thermophysics*, 18 (6), 1557 - 1573 (1997)

CERAMIC COATINGS

The Ceramic Coatings Program is a measurement and characterization effort which addresses the processing reproducibility and performance prediction issues that are primarily associated with 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 more than 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. Collaboration is also underway with BAM (Germany) for the development of characterization techniques for thin, hard films. Research is conducted on the processing and properties of Physical Vapor Deposited (PVD) ceramic coatings in collaboration with Praxair, an Advanced Technology Program (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 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 and tungsten-carbide 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 that 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-microstructural-properties relationships.

Project: THERMAL CONDUCTIVITY OF THERMAL-BARRIER COATINGS

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

Objectives

Develop methods to measure the thermal conductivity of ceramic coatings and to relate their thermal performance to the microstructure of the coatings. Measure the thermal conductivity of representative thermal-barrier coatings, substrate materials, and monolithics to determine bulk values and interfacial resistances for the coating systems. Evaluate microscopic and spectroscopic 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 are being developed that relate the thermal conductivity of various classes of ceramics and 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. 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 between constituent interfaces. The heat flow in coatings will be modeled using input from the infrared microscope measurements that generates bulk thermal conductivity values as obtained from the guarded hot plate.

Thermal conductivity measurements of three ceramic thermal-barrier coating and three monolithic ceramic systems were completed this year using the guarded hot plate. A functionally graded coating and a physical-vapor-deposited (PVD) thermal-barrier coating were measured using the infrared microscope system.

Special facilities used include an infrared microscope system capable of 5 micrometer spatial resolution from room temperature to 500 K and a guarded-hot-plate thermal-conductivity apparatus operating from 400 to 1300 K.

External Collaborations

DLR (Germany) — 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.

NAL/KRC (Japan) — Collaborating with Dr. A. Kumakawa by evaluating microstructure and microchemical composition and measuring thermal conductivity of FGMs prepared by Nippon Steel.

Pratt & Whitney — Measure thermal conductivity of very thin monolithic coatings and layered coatings under a CRADA.

Idaho National Engineering Laboratory — Collaborate with B.H. Rabin in determining microscale thermal conductivity of developmental FGMs.

Caterpillar — Collaborating with Brad Beardsley on the measurement of the thermal conductivity of FGMs and monolithic coatings using our absolute, steady-state technique and comparing with a transient laser-flash method.

Planned Outcome

This project will provide industry with standard reference materials that have been measured using both steady-state and transient methods, so that the necessary correlation exists between the fast, user-friendly methods used by industry and the slower, but more accurate and reliable steady-state methods. Standard reference materials are being developed that cover the range of thermal conductivity seen in industrial thermal-barrier coatings. Ultimately this will enhance the competitiveness of U.S. industry by providing accurate measurement techniques and standards as well as reliable data on state-of-the-art thermal-barrier coatings. This will lead to better material evaluation and the ability to design higher performance systems.

Accomplishments

Completed testing on MgO, which is a good model material for high thermal conductivity. This monolithic material demonstrated that the performance of the bulk thermal conductivity apparatus at the high-thermal-conductivity end is adequate for testing of superalloy and stainless steel substrate materials. Measured Haynes 230, a nickel-based superalloy substrate material. Measured one FGM and one monolithic thermal-barrier coating from Caterpillar. Measured an electron-beam, PVD thermal-barrier coating from Pratt & Whitney, in the as-received condition. Unfortunately, these specimens were too rough in this condition to give reliable results, and will be polished and re-tested. Measurements of Pyroceram were completed and a final report sent to the NIST Office of Standard Reference Materials (OSRM), which will result in a new high-temperature thermal conductivity SRM. Measured polycrystalline tetragonally-stabilized zirconia particles (TZP), a monolithic ceramic with higher temperature capability and slightly lower thermal conductivity than Pyroceram. TZP is our next candidate SRM material. Tested a model FGM specimen from INEL, using the infrared microscope system. This measurement demonstrated the feasibility of the micrometer-scale steady-state measurement technique using the infrared microscope. Began testing on a PVD thermal barrier coating from DLR, Germany and tested a thin version of the coating using the infrared microscope system.

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. Quantitative measurements of thermal conductivity are being performed and methods of reducing thermal noise are being explored to obtain the necessary repeatability and accuracy typical of a steady-state thermal-conductivity measurement.

Publications

1. A. J. Slifka, B. J. Filla, J. M. Phelps, A. Kumakawa, and N. Shimoda, "The Effect of Thermal Shock on the Thermal Conductivity of a Functionally Graded Material," *Functionally Graded Materials* 1996, Elsevier Science B.V.(1997), pp. 425-432.

2. B. J. Filla, "A Steady-State High-Temperature Apparatus for Measuring Thermal Conductivity of Ceramics," *Review of Scientific Instruments,* 68(7), (1997), pp. 2822-2829.

3. A. J. Slifka, B. J. Filla, and J. M. Phelps, "Absolute, Steady-State Measurement of the Thermal Conductivity of Magnesium Oxide," submitted to the *Journal of Heat and Mass Transfer.*

4. B. J. Filla and A. J. Slifka, "Thermal Conductivity Measurements of Pyroceram Using a High-Temperature Guarded-Hot-Plate," to be published in *Thermal* Conductivity, 24, 1998.

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, in terms of both material properties and performance. This is the focus of the Magnetic Materials Program. Proper measurements of key magnetic properties, 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 NBS 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 effect
- observation and micromagnetic modeling of magnetic domains for understanding magnetization statics and dynamics in advanced and conventional materials
- measurement and characterization of nanoscale magnetic interactions in multilayers, nanocomposites, and low-dimensional systems, needed for understanding and applying the new physics of these materials
- measurement and modeling of the enhanced magnetocaloric effect in nanocomposites
- structure and magnetic characterization of new superconducting materials

- nanotribology of magnetic hard disks, measurement of stiction, friction, and wear at the nanometer scale

- measurement and understanding the origin of magnetic exchange bias in conventional and advanced magnetic structures and devices

- development of magnetic sensors of mechanical properties for incorporation as *in situ* controls in a steel mill (see description above)

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

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

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

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, engineered surfaces, and bulk materials. 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 magnetic materials and engineered surfaces. Develop a scanning system (magnetoresistive microscope - MRM) using these sensors. Evaluate the potential for application in evaluation of current-flow-path problems in microelectronic interconnects. In all of these areas, evaluate application of magnetic-force microscopy (MFM), atomic-force microscopy (AFM), and related measurements to magnetic evaluations on yet smaller scales.

Technical Description

Our work in FY97 has expanded the scope of the project significantly. At present three major efforts are underway. The first is the magnetic evaluation of ion implanted surfaces, primarily on nonmagnetic ferrous alloys, which offer both interesting physics of thin magnetic films and potential value as an evaluation technique for surface integrity. This work is primarily carried out with the MFM/AFM system, but the MRM operating in dc mode also has some promise in this area. The second effort is the application of the MRM to electronic packaging evaluation of ac current flow in microscopic lines associated with off- and on-chip interconnects. The device offers a potential for assessment of line quality and the presence of defects, a problem that is becoming more critical as the line sizes head to the submicron range. The last effort is associated with the embrittlement of nuclear-reactor pressure-vessel steels. The cause of the embrittlement is proposed to be very small clusters of copper atoms within the ferromagnetic steel. These small defects and their associated strain fields can potentially be detected by magnetic parameters, such as coercivity, that are sensitive to pinning of domain walls by the clusters.

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 on (or in) ferromagnetic substrates, their characterization by magnetic techniques offers promise. In many instances, the magnetic properties of the surface layer may correlate well with the properties of interest in applications such as surface integrity, hardness and friction coefficient.

The MRM effort is concentrating on microscopic magnetoresistive field sensors for mapping of fields from regions in the 1-100 μm range. In concept, these devices can 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. Preliminary work to evaluate their use in magnetic mapping of operating high-frequency microcircuits has been a large part of this year's effort. The small size of these sensors makes them ideal, not only for the applications mentioned above, but also for flaw detection in magnetic media and other structures associated with data storage as well as for more general magnetic microstructure analysis.

The reactor steels part of the effort is relatively new. The work to date has concentrated on determination of conventional hysteresis loops for a variety of steels that may serve as surrogates for the real thing (which is somewhat radioactive). To this end, we have modified an existing vibrating-sample magnetometer to allow measurement of hysteresis loops to applied fields of nearly 400 kA/m, but still with high precision at the very low fields where coercivity measurements are made. A side project is involved with ion implantation of copper into steel in an attempt to create a better, albeit small, model material suitable for analysis with the MFM.

Our scanning probe microscope (SPM) was delivered in late October. Several months were required to get it properly set up and to become proficient in its operation. The main application in this project has been in magnetic-force microscopy and associated atomic-force imaging, but it is also a Division resource for AFM, including potentially serving as a test bed for several new experiments in the electronic packaging and interconnects effort. The instrument is also capable of scanning tunneling microscopy (STM), and a number of more exotic modes, such as electric force microscopy.

External Collaborations

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. We are still awaiting implanted samples of chrome-plated steel made by the participants. Measurements of similar samples from a different project at Los Alamos show interesting behavior of the chrome coating under implantation.

A collaboration on magnetic measurements of ion-implanted stainless steel with Colorado State University and Colorado School of Mines has been very active. Many MFM measurements have been made on a large number of samples. An attempt to understand the results in light of our data and data on the same samples from Mössbauer and traditional magnetic-parameter measurements is continuing.

Planned Outcome

This project is anticipated to result in a wide-ranging micromagnetic sensing capability for the Division. The microscale 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 data-storage systems. The larger scale magnetometry systems (vibrating sample magnetometer, B-H loop generator, etc.) also will be employed in evaluation of large samples to determine the effect of microstructure on material magnetic properties.

Accomplishments

Ion implantation studies have measured the surface magnetic signatures of four stainless steel compositions with various surface preparations after implantation with nitrogen ions at temperatures from 325°C to 475°C, which covers the range of formation of all of the magnetic surface-layer types. Wide variations in magnetics are seen depending on the composition. Surface damage from polishing creates its own magnetic signature and removal of that layer prior to implantation has been the topic of many of our measurements. When the implanted region forms it is tough and very thin, only a micrometer or so, with a magnetic signature much like that of magnetic garnet, in which the moments are perpendicular to the surface.

Continuing experience with the computer-controlled scanning magnetoresistive microscope has made progress on several fronts. For one thing, we have pretty much learned how to mount the recording head units without destroying them with static discharges. The most dramatic success this year was a demonstration of high-frequency imaging of relatively small conducting lines, including a clear image of a defect purposely induced in one of the lines. A series of tests on thermal properties of the heads and their field sensitivity led to a major success in the dc field measurement application of the device; in place of the normal dc bias current, the detector is driven by ac and the second harmonic signal is detected. The success came in predicting and then demonstrating that the second harmonic signal would be temperature independent and, thus, heroic efforts are not needed to control the device temperature. This is a major advance in the effort to apply the MRM to dc field measurement problems.

Eight different reactor steels or surrogate materials were measured on the vibrating-sample magnetometer and the data analyzed to give accurate values for saturation magnetization and coercivity. There is a clear correlation with defect structure, but thus far it is not clear that the measurement will be capable of discriminating the effect of the small cluster defects from the many other defects and precipitates in real-world steel.

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 MACHINE VERIFICATION

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

Objectives

- Provide rapid, accurate assessment of test data generated by our customers on the SRMs, and, where merited, certify the conformance of Charpy impact test machines 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.

Planned Outcome

Furnish users and owners of Charpy impact machines with the best possible verification services.

Accomplishments

We had about 800 customers for this service in FY97, a number similar to those 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 over 500 faxes and 1200 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.

We continue to evaluate the need for new impact verification services. In 1995, we expanded on several years of informal interaction with ASTM Committee D 20 on plastics by forming and leading a new task group, X-10-279, to investigate the procedures and materials needed to verify the performance of plastics impact machines. Since then, we have prepared a series of candidate metal specimens (from alloys that should avoid the aging problems common to plastics) and plastic specimens (of compositions that are commonly tested on these machines). The data from this round robin are now under review by this task group.

Chris McCowan continues as the Chairman of ASTM Subcommittee E28-07 on impact testing. We continue to use these ASTM meetings as a forum to discuss the statistical trends from our customer evaluations (percentages of machines that meet the requirements and the distribution of data around the mean). The technical committee members have been quite pleased with our openness in sharing this data, and we are preparing a technical paper on this data. We have scheduled an international symposium on Charpy impact testing for the May 1999 meeting of this ASTM Committee, and are soliciting contributions. Dan Vigliotti was given a certificate of appreciation by Pratt and Whitney for helping them to achieve certification to ISO 9000.

Publications

1. T.A. Siewert, D.P. Vigliotti, L.B. Dirling, and C.N. McCowan, "Performance Verification of Impact Machines for Testing Plastics," submitted to Journal of Testing and Evaluation.

Project: FERRITE IN STAINLESS STEEL WELDS

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

Objectives

- Design and implement a system to assign ferrite numbers to stainless weld metal specimens, so that the standards sold by NIST will be consistent with those now used around the world.

- Develop a new calibration system based on primary units, such as magnetic field strength.

Technical Description

Austenitic weld metals usually contain a small amount of ferrite to reduce the tendency for cracking during solidification. The quantitative measurement of this ferrite is important commercially, as it is commonly specified in contracts and production standards. The amount of ferrite is measured magnetically following industry standards. In the United States, this standard is American Welding Society Standard A4.2-91 *Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic and Duplex Austenitic-Ferritic Stainless Steel Weld Metal.*

This standard specifies both primary and secondary calibration of the instruments. Primary calibration is based on the NIST coating thickness standards, such as SRMs 1323 and 1363, while secondary calibration is based on calibrated weld samples. In section 3.2, the standard describes the importance of secondary standards as: the only way of calibrating instruments for which no primary calibration method exists, the most appropriate standard for in-process checks, and being much more durable than the primary standards. At least three different companies have produced and sold sets of secondary standards during the past 30 years, but this is only a sideline for these companies. The assumption of this responsibility by NIST offers a stable source of supply to the world, and clearly complements our present role in providing the present primary standards.

The current specimens are arranged in two sets: a lower range set with 8 specimens that are well distributed (logarithmically) over the range of ferrite numbers (FN) of 0 to 30 FN, and a higher range set of 8 specimens that are well distributed over the range of 30 to 100 FN. At the request of Commission II of the International Institute of Welding (IIW), we have spent the last few months in procuring 30 sets (15 sets in a low range and 15 sets in a high range) of uncalibrated ferrite specimens from Russia, developing internal calibration procedures so these could become secondary standards for ferrite in stainless steel welds, and using these procedures to finish assigning reference values to these 30 sets.

In addition to the calibration of these sets as secondary standards according to the internationally recognized procedure (AWS A4.2, based on NIST coating thickness standards), we plan to develop a primary calibration system which will be traceable to primary electrical quantities. The most likely basis for the system will be dc magnetic measurements. Initial work will determine the actual magnetic properties of the existing secondary standard materials at both the macro- and micro-magnetic levels. Conventional metallography will play a significant role in this phase of the work. Magnetic force microscopy and vibrating-sample magnetometry will be used along with superconducting quantum-interference-device (SQUID) magnetometry as necessary to characterize the ferrite magnetics. The ultimate goal for FY99 will be development of a portable, easily used, standard magnetic measurement device suitable for accurate determination of ferrite concentration. This standards development activity will occur parallel to the assignment of values according to the existing standard, and will be performed in close collaboration with IIW Commission II experts, so the users group will be ready to adopt this primary calibration technique when it is ready. Actual construction and deployment of primary calibration devices will be in later years under WCF.

Planned Outcome

Put stainless-steel weld standards into SRM inventory and replace this inventory as sold. Develop a fundamental calibration strategy, to replace the current qualitative procedure.

Accomplishments

1. We have purchased 15 sets of specimens in the low-FN range and 15 in the high-FN range. These specimens have been marked with unique identification numbers using a mechanical indentor, and are being prepared for SRM inventory.

2. Microstructural analysis of several specimens is revealing how the characteristic distances in the structure determine the standard deviation in the magnetic force measurements.

Publications

1. Presentation to International Institute of Welding, Subcommission II-C on our initial calibration work, July 17, 1997.

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

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

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- Acoustic emission
- Composite materials
- Mechanical testing

McCowan, Chris N.
- Welding metallurgy
- Charpy impact testing
- Metallography and fractography

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- Fracture mechanics
- Materials processing
- Fracture control

Phelps, John M.
- Electron microscopy
- Chemical microanalysis
- Compositional mapping

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- Fracture properties of metals
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Read, David T.
- Electronic packaging
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Schaps, Stephen R.
- Electrical design
- Nondestructive evaluation
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- Ultrasonic NDE of welds
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- Welding metallurgy of steel
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- Thermal barrier coatings
- Surface characterization

Smith, David R.
- Thermal conductivity
- Thermal expansion
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Tewary, Vinod K.
- Solid state physics
- Green's function methods
- Elastic wave propagation

Tobler, Ralph L.
- Fracture mechanics
- Material properties at low temperatures
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Vigliotti, Daniel P.
- Charpy impact testing
- Standard reference materials
- Fabrication technology

Yukawa, Sumio
- Fracture mechanics
- Codes and standards
- Structural safety

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
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      - M.C. Renken
      - R.L. Santoyo
      - V.K. Tewary
      - D. Balzar (GR)
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    - 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
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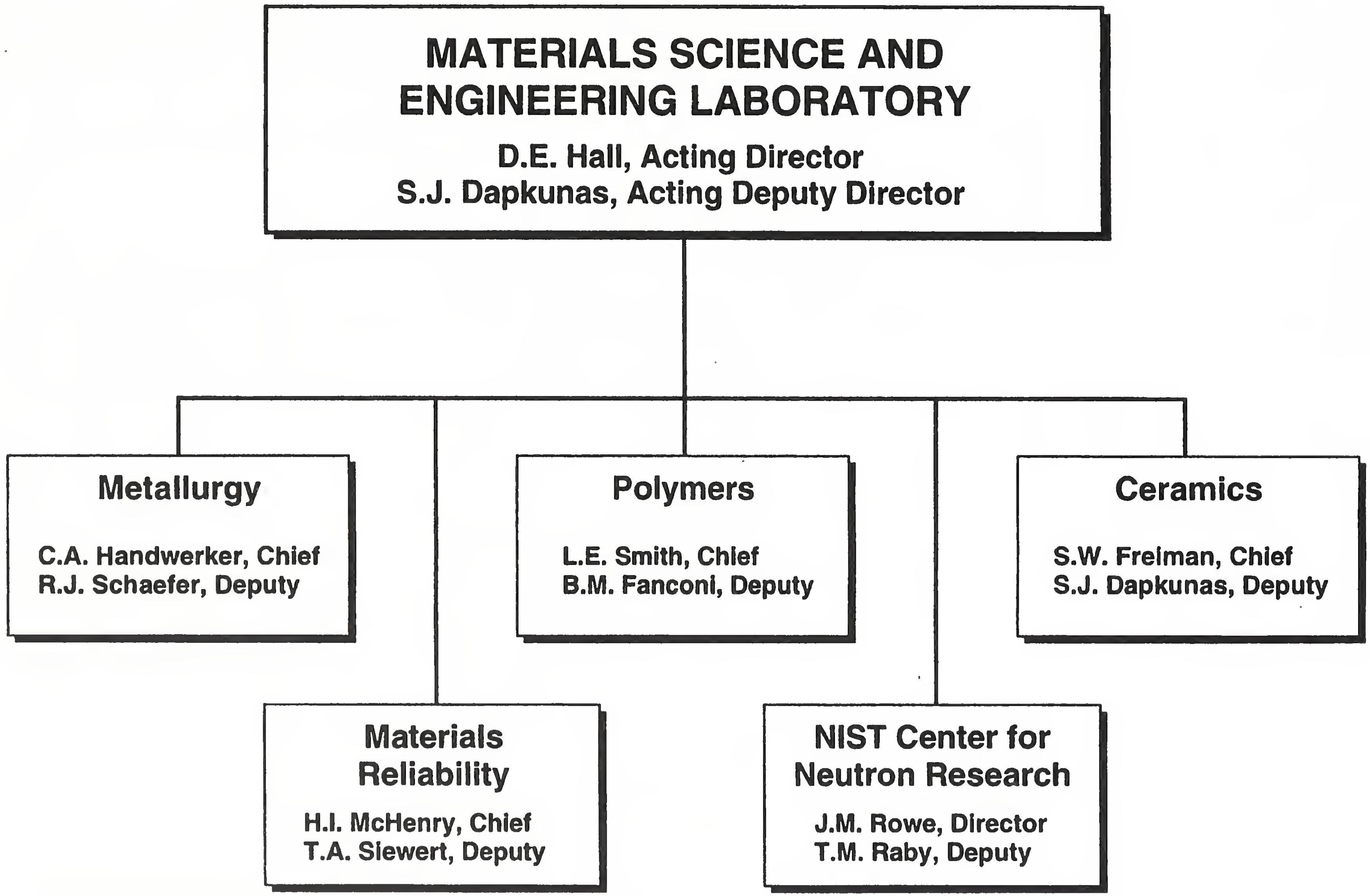
    - V.E. Ciaranello
      - W.P. Dube
      - D.W. Fitting
      - B.J. Filla
      - R.B. Madigan
      - C.N. McCowan
      - T.P. Quinn
      - D.P. Vigliotti
      - S. Yukawa (INT)
  - **.08 Materials Evaluation** — F.R. Fickett
    - S.J. Barnhill
      - E.S. Drexler
      - C.E. Kalnas
      - R.R. Keller
      - J.M. Phelps
      - D.T. Read
      - A.J. Slifka
      - D.R. Smith


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

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Organizational Chart