MATERIALS RELIABILITY

1998 PROGRAMS AND ACCOMPLISHMENTS

MATERIALS SCIENCE AND ENGINEERING LABORATORY

NISTIR 6248

UNITED STATES DEPARTMENT OF COMMERCE

TECHNOLOGY ADMINISTRATION

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

Smaller Structures and Higher Speeds: Materials Reliability in Microelectronics

The reliability of modern electronics, from on-chip structures to the lines and layer interconnects within the packages, is a matter of increasing concern. Continuing reductions in scale change the effective material properties while tighter packing of components greatly increases the potential for wide thermal excursions with their attendant adverse effects.

Additionally, higher CPU speeds require that new materials be added to an already complex mix. The Materials Reliability Division is assuming an increasing role in the evaluation of materials for microelectronics and associated packaging by determining the critical materials properties of actual structures and providing advanced measurement methods for reliability assessment.

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MATERIALS SCIENCE AND ENGINEERING LABORATORY

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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 geometry 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.

Intelligent Processing of Materials: Fiscal year 1998 was a critical year for Intelligent Processing of Materials because each project completed major, multiyear commitments and new directions were initiated. Welding CRADAs (Cooperative Research and Development Agreements) with automotive parts manufacturers were completed and a project on robotic arc welding was initiated in collaboration with the Intelligent Systems Division. Research on x-ray diffraction sensing of the liquid-solid interface for the Aerospace Casting Consortium was completed and facilities and equipment were purchased to follow it with a project to develop high-energy transmission x-ray diffraction as a materials characterization tool. The microstructural engineering project sponsored by the American Iron and Steel Institute was completed and research was started on the formability of aluminum alloys in collaboration with the Metallurgy Division.

Ultrasonic Characterization of Materials: In fiscal year 1998, a multidisciplinary effort involving ten staff members demonstrated that ultrasonic and micromagnetic measurements could detect embrittlement in reactor pressure vessel steels. The Nuclear Regulatory Commission provided additional funding to make measurements on irradiated steels within a hot-cell at the University of Michigan. In addition, we demonstrated that magnetostriction measurements could be made through 1 cm thick stainless steel cladding on full-thickness sections of a decommissioned reactor pressure vessel.

Micrometer-Scale Measurements: The Division has four projects to develop measurements for evaluating the reliability of electronic interconnections. In fiscal year 1998, we transferred measurement technology to the electronics industry for measuring the mechanical properties of thin films and for strain measurements in electronic packaging. In addition, test structures were developed for evaluating the susceptibility of interconnections to stress voiding and for measuring the thermal conductivity of thin metallic films.

A new approach to reporting research results was initiated in fiscal year 1998. The Materials Reliability Series of NIST Technical Notes are reports covering significant research accomplishments of the Division. Five reports were published covering multiyear efforts in thin film testing, electron-beam moire, and x-ray monitoring of solidification. We also included our report to the Nuclear Regulatory Commission on embrittlement detection in the series.

The Division suffered a major loss of technical leadership when Chris Fortunko died on June 27, 1998 due to a heart attack. Chris is remembered for his remarkable scientific knowledge and his broad technical experience. He used these skills to provide leadership for the Division and for the worldwide nondestructive evaluation (NDE) community. His unique contribution was to bring the discipline and rigor of measurement science to the less orderly world of material testing and NDE.

This report summarizes the technical activities in the research projects of the Materials Reliability Division during fiscal year 1998. The Division's projects are organized according to programs that have been defined and organized by the Materials Science and Engineering Laboratory (MSEL). The Division has eighteen research projects that contribute to five MSEL Programs. 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.

Robotic Arc Welding: With the Intelligent Systems Division, we developed a robotic welding cell for NIST's Manufacturing Engineering Laboratory and demonstrated use of the internet for remote communication, monitoring and control of arc welding within the cell.

Directional Solidification Sensor: We completed the development of an x-ray diffraction technique to monitor the solidification of single crystal turbine blade castings and transferred the technology to the Aerospace Casting Consortium. The technique was patented (U.S. 5,589,690) and received the Federal Laboratory Consortium's 1998 Award for Excellence in Technology Transfer.

Microstructural Engineering: We completed the research project entitled "Microstructural Engineering in Hot-Strip mills" for the American iron and Steel Institute. We developed and validated models to predict the stress-strain behavior of steels under hot-rolling conditions and to relate the mechanical properties of steels to their chemical composition, microstructures and processing. The models are used in a Hot-Strip Mill Model which was delivered to the steel industry by our research partner, the University of British Columbia.

Nondestructive Characterization of Embrittlement: Experimental techniques were developed to perform ultrasonic and magnetostriction measurements on irradiated steels in a "hot cell" at the Phoenix Reactor of the University of Michigan. The magnetostriction measurements are particularly promising because they correlate with embrittlement and can be performed through stainless steel cladding in the 200 mm thick plates used in reactor pressure vessels.

Green's Function Library: A project to set up a library of Green's functions on the world wide web with internet access is being carried out in collaboration with the Colorado School of Mines and Iowa State University. Web pages to view the library have been created at Iowa State and at NIST.

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.

Mechanical Testing of Thin Films: We provided Motorola with the design and specifications to allow them to build a microtensile test machine based on the NIST design. Once the parts were obtained, we assisted Motorola in final assembly, calibration and initial application of the test machine.

Test Structure for Interconnect Reliability: Fabrication was completed of the NIST 34 test chip for assessing stress voiding and electromigration in single-level interconnects, in collaboration with the Semiconductor Electronics Division. The geometrically simple structures are used to assess effects of linewidth and serpentine meandering on void densities.

Thermal Conductivity of Ceramic Coatings: The infrared microscope was validated as a method for doing comparative, steady-state measurements of thermal conductivity on thermal barrier coatings. The validation was done using a plasma-spray coating measured previously in the guarded-hot-plate system.

METALS PROCESSING

The properties of metals and their alloys depend strongly on their processing history. For example, the distributions of phases, grain structure, alloy compositional segregation, and defects in final commercial products depend not only on their compositions but also on the conditions under which their materials are fabricated. These distributions are crucial in determining the alloy strength, ductility, homogeneity, and other properties important for industrial applications. The Metals Processing Program focuses on measurements and predictive models needed by industry to design improved processing methods, provide better process control, develop improved alloy and coating properties, and reduce costs.

Major successes in applying measurements and modeling to processing applications have been achieved through NIST's interactions with the aerospace, powder metallurgy, electroplating and electronics industries. Predictive models developed at NIST for solidification and microstructural evolution during processing have been incorporated by industry into design systems for casting of aerospace alloys and production of defect-free electronic materials, helping to reduce rejection rates arising from defective parts. Cooperative research and development projects with industry have resulted in significant improvements in process control for welding and for atomization of steel and superalloy powders. Standard Reference Materials, certified for coating thickness, microhardness, or chemical composition, are being fabricated by electrodeposition techniques and powder metallurgy. Data and understanding concerning mechanistic, chemical, and process variables controlling the structure/properties of coatings and thin films produced by electrodeposition are being developed to take further advantage of this electrochemical process, which does not require high purity starting materials and is readily adaptable to large-scale production.

Measurements and predictive models for processing are being developed to aid industry in tailoring materials properties for particular applications. Intelligent processing of materials through *in situ* property measurements combined with control systems based on process models is being pursued. Specifically:

- Measurements and models are developed to help design materials production processes. This work includes measurements and thermochemical evaluations to provide alloy phase diagrams, which are the roadmaps alloy designers use to predict the alloy phases that can be produced under specific processing conditions. These evaluations are playing key roles in NIST collaborations with industrial companies on electronic solders and casting of superalloys for aerospace applications.

- Measurements are made under dynamic conditions to monitor, in real time, properties of materials while they are actually being produced and to determine difficult-to-measure process parameters while the process is occurring. Special fast-response sensors, simulations and imaging techniques have been developed for application to powder atomization and thermal spray processes, and workshops have been held to transfer these techniques to industry. Here, dynamic models of the process are important both for design of manufacturing procedures and for applications of real time feedback and control.

- To evaluate the adequacy of process models, it is important to measure the properties of the final materials and relate them to the process conditions. Work in this respect includes evaluation of methods used to optimize properties of electrodeposited coatings, corrosion resistance of rapidly solidified nitrogenated steels, and liquid-phase bonding of alloys.

In all of this work, the goal is to help U.S. industry apply measurements and predictive modeling to produce improved materials at reduced cost.

Project: WELD PROCESS SENSING, MODELING, AND CONTROL

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

Technical Objectives

- Develop a better understanding of the underlying physics governing arc-welding processes through advanced instrumentation and data analysis techniques.

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

- 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.

- Assist in the development of industrial standards for exchange of information between intelligent components in a robotic arc-welding cell (for example, robot controller, welding power source, etc.).

Technical Description

Sensing

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. Our sensing scheme, through-the-arc sensing, relies on using model-based algorithms that use the inherent process variables: the current and the voltage. 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 was refined through feedback from our CRADA partners. Several of them are presently working to incorporate the ASM concepts into their welding production lines. The ASM was developed with various compositions of steel electrodes. We are now investigating its application to aluminum compositions.

Our Welding Laboratory includes the following special equipment:

- four 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 back-lighting
- standard video cameras and VCRs
- video frame-grabber and image-processing system
- 6-axis robotic manipulator, three track-type manipulators
- conventional SCR, inverter, and transistorized welding power sources
- 4-roll and capstan wire feed units

Models and Controls

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 audio and visual feedback.) The models of the complex welding processes lead to better understanding by welding engineers, improved welding sensors, and can lead to feedback-control schemes.

We are investigating the use of arc-light emissions for process monitoring and control. Using a simple photodiode and broad-band optical interference filters, we developed sensors for measuring arc length and droplet frequency. A high-speed video system with laser backlighting provides reference measurements of arc length and droplet frequency and is used to calibrate each sensor. A more sophisticated approach involves using a monochromator to allow higher-wavelength resolution measurements of arc-light emissions. With this device we intend to further exploit specific portions of the emission spectra for process monitoring and control. We have also been investigating weld spatter. Using voltage, current and arc-light measurements in conjunction with the high-speed video system, we are studying mechanisms of spatter generation. 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 major contributors to generation of spatter.

Standards

Most robotic welding cells use proprietary interfaces between the various components. Once the system is in place, it is difficult for the end user to change components because of the lack of standard interfaces and data-exchange methods. In addition, sensor manufactures must customize their sensors for each system.

Welding engineers who design the welding process used in a production robotic welding cell (and later troubleshoot them) are usually grouped in a central location (a corporate engineering group for large companies, or consulting firms for small companies). If a problem occurs, the engineer is summoned while the production line is (expensively) idle. A highly desirable solution is for remote staff to monitor the welding cell, foresee problems before they occur, and collaborate in fixing the problems without physically being present.

The AWS A-9 Committee (Computerization in Welding) is developing standards for the interfaces among cell components. An intelligent, robotic arc-welding cell developed at NIST in collaboration with the Intelligent Systems Division is being used to test and demonstrate the standards by adapting NIST welding sensors and robot controllers to the standard interfaces. Controller designs and interfaces are based on the Integrated Systems Architecture for Manufacturing (ISAM) methodology.

The NIST welding cell is integrated into an intranet to demonstrate the utility of remote collaboration, programming, and control. Information that is available includes the quality of data output by the sensors, the status of the weld cell, and the information of the robot controller's path. Microphones and cameras in the weld cell allow the arc to be viewed remotely and allow for post-weld inspection and communication with the operators.

External Collaborations

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

Ohio State University. Dick Richardson, Professor of Welding Engineering, works with Timothy Quinn to determine the best arc-sensing techniques for fabrication of heavy equipment structures.

Colorado School of Mines. Dave Munoz, Professor of Engineering, works with Tim Quinn on guiding a CSM Student, Toby Padilla, on a study of the interactions between the contact tube and the electrode.

Industrial. The American Welding Society Committee A9 is working with us to develop industrial standards for data collection and equipment interfaces.

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.

Accomplishments

Our accomplishments fell into three major categories: National Advanced Manufacturing Testbed (NAMT, our collaboration with industry and MEL), research in support of ATP thrusts, and general collaborations with the welding industry and other research organizations.

The NAMT intelligent welding cell is online with open interface technologies. The ASM (our sensor technology) has been integrated into the cell. The American Welding Society A-9 committee has agreed that the cell can be used as a national testbed to develop and demonstrate the standard that they are writing for interface standards in robotic arc-welding cells. Internet technologies make the data gathered by the sensors in the cell available remotely. Cameras and videoconferencing tools connect the operators of the cell with remote collaborators. The cell has been demonstrated on several occasions to visiting industry as a model of future monitoring on the factory floor.

In support of ATP thrusts, we developed several artificial-intelligence models that improve the identification of welding problems from the weld electrical signals. Traditional neural networks, fuzzy inference systems, and adaptive neuro-fuzzy systems have all demonstrated an ability to distinguish between good and bad weldments, with (Mandani) fuzzy systems showing the highest degree of recognition. The weld problems that we can currently recognize are: loss of shielding, workpiece contamination, and weld burnthrough.

We completed our cooperative research and development agreements (CRADAs) with A.O. Smith Corporation, Johnson Controls, and the Delphi Chassis Division of General Motors. We learned that our arc-imaging technology (back-lighted laser shadowing) was as useful to them as our weld-sensing technology.

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 sensor has been hardened and long term production line testing has been scheduled.

We are working with researchers at Colorado School of Mines on investigating some of the problems specific to wire feeding in GMAW. Our models have shown aluminum is very sensitive to variations in wire feed speed. Practically, aluminum GMAW is very difficult to do. The mechanical wire feeding technology has remained basically unchanged for many years and is not fully understood. Working with researchers at the Colorado School of Mines, we are starting to develop physics-based models of the wire from when it exits the pinch rollers of the wire feeder to when it exits the contact tube. The first model developed is for the friction on the wire as it is fed through the conduit.

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 as is observed in experiments. The model now couples the fluid calculations with the Maxwell's equations, calculates the position of the moving boundary of the droplets as they fall through the arc, and uses a simple model to calculate the heat flow across the interface between the plasma and the electrodes. Experiments to verify the model are continuing.

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.

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

Publications

1. "Trends in Welding Software, Siewert, T.A.; Proc.," 7th Intl. Conf. on Computer Technology in Welding, NISTSP 923, ed. T. A. Siewert, Nov. 1997.

2. "Weld Defect Decisions Using Through-the-Arc Sensor Data," T.P. Quinn, R.B. Madigan, C.B. Smith, and E.G. Blachowiak, Proc., 7th Intl. Conf. on Computer Technology in Welding, NISTSP 923, ed. T. A. Siewert, Nov. 1997.

3. "IIW Commission V - Quality Control and Quality Assurance of Welded Products: Annual Report 1997/98," T.A. Siewert, NISTIR 5069, IIW Doc. V-1100-98, January 1998.

4. "The International Institute of Welding: Report on 1997 Actions," T.A. Siewert, Materials Evaluation, 56 (2) , pp. 153-159, February 1998.

5. "IIW Commission V - Quality Control and Quality Assurance of Welded Products: Annual Report 1998/99," T.A. Siewert, NISTIR 5081, IIW Doc. V-1123-99, in press.

6. "The International Institute of Welding: Report on 1998 actions," T.A. Siewert, Mater. Eval., submitted.

Project: THERMOMECHANICAL PROCESSING

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 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 relationships between composition, structure and property 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 and 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 (UBC), Vancouver, British Columbia. The Center for Metallurgical Process Engineering at UBC and NIST are co-investigators for an AISI project entitled, "Microstructural Engineering for Hot-Strip Mills". UBC's principal investigators are Professors E. B. Hawbolt and I. V.Samarasekera.

Industrial. United States Steel Corporation (U.S. Steel) and the member companies of American Iron and Steel Institute (AISI). U.S. Steel is the industrial sponsor for the AISI project conducted by UBC and NIST.

Accomplishments

In 1998, 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 relationships between structure, composition and property 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 forms is essential to fully exploit the potential of numerical techniques, such as the finite element method, for analyzing and simulating hot rolling or forging.

We have completed the AISI project in 1998. Accomplishments for the constitutive-behavior modeling include the development of models that are suitable for predicting stress-stain behaviors of the following eight steels: A36, DQSK, HSLA-V, HSLA-Nb, HSLA-50/Ti-Nb, HSLA-80/Ti-Nb, and two interstitial-free (IF) grades. These models calculate a full stress-strain curve for a wide range of temperatures (800-1300°C), strain rates (1-400 s^{-1}) 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. The models have been used in the Sims equation to calculate the rolling forces in strip and plate rolling.

The mechanical properties of a steel at ambient temperature are closely related to composition and microstructure. Equations have been developed and validated for plain C and HSLA steels produced on the hot-strip mill. The lower yield strength, ultimate strength, and percent total elongation are the main outputs from the equations. For thicker, plate-type products, the ductile-to-brittle transition temperature can also be predicted. A process model developed at UBC allows for considerable flexibility and detail in the setup for the hot-strip mill and still provides accurate predictions for yield and ultimate strength, within about +/-35 MPa. The two sets of equations developed for the structure-property relationships are applicable to a wider range in chemistry than just the eight grades in the program. Base strength equations are considered applicable to grades where the microstructure is predominantly polygonal ferrite; prediction of precipitation strengthening (using Shercliff-Ashby model) is applicable to steels with Nb contents from 0.02 to 0.09 %, 0.08 % V, and excess Ti levels up to 0.025 %.

The equations developed for the constitutive behavior and the structure-property relationships have been successfully incorporated into a process model which has been 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. "Microstructural Engineering in Hot-Strip Mills," Y. W. Cheng and P. T. Purtscher, final report submitted to AISI, September 1998.

2. "Design and Performance of an Automated Test Facility for Hot-Compression Testing," Y. W. Cheng, B. J. Filla, and R. L. Tobler, Journal of Testing and Evaluation, Vol. 26, No. 1, January 1998, pp. 38-44.

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, 1998.

Project: **X-RAY DIFFRACTION SENSING OF SOLIDIFICATION IN NICKEL SUPERALLOY CASTINGS**

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

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.

- 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

As members of the research team of the NIST Consortium on Casting of Aerospace Alloys, we developed a technique based on transmission x-ray diffraction (XRD) to study the solidification of a single-crystal turbine blade casting within its mold. In our research, we used the ability of high-energy x-rays (150 to 320 keV) to penetrate through material surrounding the casting and to produce a distinctive diffraction pattern which clearly indicates whether the sampled region is liquid or solid. A real-time transmission Laue x-ray image of the casting shows an ordered pattern of x-ray scattering (diffraction spots) from the solid and a diffuse ring of scattering from the liquid. The dramatically different spatial patterns provide a high-contrast, unequivocal spatial discrimination of the physical state of the alloy.

Low-energy x-ray diffraction systems are very common in research laboratories, so the advantages of high-energy x-ray diffraction are often overlooked. The greatest advantage is the rapid increase in penetrating capability as the tube potential increases. Raising the x-ray energy increases the penetration so rapidly that a 320 keV beam can penetrate 450 times more nickel than can the 8 keV radiation from a diffraction system using copper characteristic-line radiation. In addition, the x-rays are able to penetrate the casting mold and reasonable thicknesses of other furnace components

For a thick, single-crystal specimen, transmission diffraction efficiency increases as the x-ray energy increases, peaks, and then decreases for energies higher than several hundred kiloelectronvolts. At low x-ray energies, the atomic scattering factor is quite large, with much of the energy being diffracted to large scattering angles, both forward and backward. However, for high x-ray energies, the diffraction becomes increasingly forward-directed. The atomic scattering factor which is large in the forward direction, the enormous number of unit cells along the path of the primary beam (all with the same crystalline structure and orientation), in addition to the substantial intensity of high-energy x-rays which penetrate through a mold and specimen, all account for the efficiency of high-energy transmission diffraction. An optimal x-ray energy (often between 100 and 320 keV) capable of penetrating through the furnace, mold walls, and casting was chosen using an analytical model we developed for the transmission XRD process.

External Collaborations

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. We have interacted with Consortium members (American Foundrymen's Society, Auburn University, GE Aircraft Engines, Howmet Corporation, PCC Airfoils, UES Software, University of Arizona, and University of Illinois) to identify sensor needs for investment casting and to direct our XRD research to solving these problems.

Industrial. Howmet Corporation has been an active collaborator in developing the XRD casting 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, and (4) assisted during metal casting experiments.

Planned Outcome

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

Accomplishments

We developed a technique based on transmission XRD to study the solidification of a single-crystal turbine blade casting within its mold. High-energy x-rays (100 to 320 keV) penetrate through material surrounding the casting and produce a distinctive diffraction pattern which clearly indicates whether the sampled region is liquid or solid. We validated the method, first on pure metals and thereafter on nickel-alloy castings within a mold in an industrial directional-solidification furnace. The high energies permitted transmission XRD to be performed on a nickel-alloy specimen 17 mm thick. XRD images were obtained even though the x-ray path through the furnace included glass furnace ports, molybdenum furnace windings, aluminum-oxide hot-zone coil support, and refractory-oxide mold material.

Plots of diffraction-spot intensity versus vertical position (increasing temperature) in the casting correlate well with lever-law model predictions of fraction solid versus temperature. We were able to map isocontours of fraction solid in the mushy zone of a superalloy casting and verified solidification models for investment casting. We also found that with precise alignment of the x-ray beam with respect to the growing crystal, we could observe a topographic image of the dendrites solidifying within the casting mold.

Members of the XRD research team were awarded the Federal Laboratory Consortium's 1998 Award for Excellence in Technology Transfer "For the Development and Transfer of Modeling Tools and Sensors for the Design of Complex Components of Aircraft Engines".

NIST filed a patent application for the x-ray diffraction solidification-sensing technique and was awarded US Patent 5,589,690.

The success of the XRD solidification sensing technology has lead us to begin investigating other uses for high-energy x-ray diffraction such as characterizing the texture and stress state of polycrystalline structural components fabricated by casting or welding. We have also completed installation of a new laboratory for x-ray research. The facility is equipped with several x-ray sources: a 4-circle specimen manipulation goniometer, a real-time x-ray imaging system, and energy-sensitive x-ray detectors. Please refer to our web site for the current direction of our research into high-energy x-ray diffraction:

(http://www.boulder.nist.gov/div853/balzar/highenergyx.htm).

Publications

1. D.W. Fitting, W.P. Dubè, and T.A. Siewert, "High-Energy, Transmission X-Ray Diffraction for Monitoring Turbine-Blade Solidification." National Institute of Standards and Technology Technical Note 1500-3 (Materials Reliability Series) 150 pp. June 1998.

2. 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,* 27-28 May 1998, and the *Final Report of the NIST Consortium on Casting of Aerospace Alloys* (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 which characterize the internal geometries of materials, such as defects, microstructures, and lattice distortions. Our goal is to convert these measurement methods into sensors suited for production line and in-service measurements of materials quality and serviceability.

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

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

Project: ELASTIC COEFFICIENTS AND RELATED PHYSICAL PROPERTIES

Research Leader: H. Ledbetter
D. Balzar*, M. Dunn, S. Kim, H. Ogi*****

Objectives

Understand, through measurements and modeling-theory, the elastic properties of solids that possess high scientific or 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, the Grüneisen parameter, and many other properties, including practical properties such as hardness.

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 such as crystal-defect structure, strain, and texture. We make measurements with laboratory x-ray sources and at large national user facilities using synchrotron x-ray and neutron diffraction.

* Visiting scientist from University of Colorado, Physics.
** Visiting scientist from University of Colorado, Mechanical Engineering.
*** Visiting scientist from Osaka University, Mechanical Science Division, Graduate School of Engineering Science.

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 comparisons;
4. novel theoretical approaches;
5. relationships among physical properties;
6. occasional small discoveries of unexpected behavior, those hard unexplained facts that require new critical thinking.

See brief description under Objectives.

Accomplishments

Torsion pendulum. We installed a mechanical-spectroscopy apparatus that measures the internal friction Q_{ij}^{-1} very accurately. To begin, we shall focus on the Snoek peak caused by interstitial atoms in b.c.c. metals, especially iron.

The publication list given below shows many of our recent achievements. Beside these we progressed significantly on other topics:

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

2. 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. Strain correlates with mechanical (hardness and strength) and acoustic (nonlinearity parameter β) properties.

3. 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}.

4. Covalent materials. Studying high-quality (low-defect-density, low-chemical-impurity) silicon monocrystals, we looked for evidence of dislocation-kink motion. Preliminary results look positive.

5. 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.

6. Al/Li alloys. We completed an ab initio quantum-mechanical calculation that explains (for the first time) the remarkable elastic-stiffness increase upon adding Li to Al.

7. 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. We extended existing isotropic models and we wrote two manuscripts on this isotropic-anisotropic extension.

8. 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.

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

10. 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. We began a manuscript.

11. 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. Measurements above 108 K show acoustic-phonon softening.

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 now 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. Applications to engineering are obvious. Relationship of α to elastic coefficients is clear. For composites, challenging modeling problems arise.

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 and show that the macroscopic symmetry is probably monoclinic.

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. NiTi/Al composites. NiTi is a shape-memory alloy with remarkable mechanical-physical properties. We measured the C_{ij} and Q_{ij}^{-1} down to liquid-nitrogen temperature, through the crystal-structure transformations.

22. TiB_2/NiAl composites. On these, we measured the C_{ij} and the Q_{ij}^{-1}. Successfully, we modeled the results using the Ledbetter-Datta scattered-plane-wave ensemble-average theory. A surprising finding was that the literature (older) values of TiB_2's C_{ij} are quite wrong, leading to misinterpretations of chemical bonding.

23. 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.

24. D. Balzar acted as a member of the International Program Committee of the 6th European Powder Diffraction Conference (EPDIC-6), held in Budapest, 22-25 August 1998. EPDIC is the largest European meeting in this field.

25. 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.

26. Davor Balzar delivered an invited lecture, Texture by Rietveld Refinement, at the 47th Annual Denver X-Ray Conference, Colorado Springs, August 3-8, 1998.

27. Davor Balzar delivered an invited lecture: Line-broadening analysis and standards, at the 6th European Powder Diffraction Conference, Budapest, Hungary, August 22-25, 1998.

28. Davor Balzar was elected as a member of the permanent EPDIC Committee.

29. Davor Balzar was selected to organize and chair a Microsymposium: Applications of Line Broadening at the 18th Congress and General Assembly of the International Union of Crystallography, to be held in Glasgow, Scotland, August 4-13, 1999

30. H. Ledbetter was invited to deliver the keynote address at the APEC Symposium on High-Performance Metal-Matrix Composites (Tsukuba, Japan, 2-4 March, 1999).

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2. D. Balzar, H. Ledbetter, Software for Comparative Analysis of Diffraction-Line Broadening, *Advances in X-ray Analysis* 39 (1996), in press.

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

15. D.C. Hurley, D. Balzar, P.T. Purtscher, K.W. Hollman, Nonlinear Ultrasonic parameters in quenched martenisitic steels, *J. Appl. Phys*. 83 (1998) 4584-4588.

16. S. Kim and H. Ledbetter, Low-temperature elastic coefficients of polycrystalline indium, *J. Mater. Sci*. Eng. A252 (1998) 139-143.

17. H. Ledbetter, D. Balzar, S. Crudele, W. Kriven, Elastic properties of mullite, *J. Am. Ceram*. Soc. 81 (1998) 1025-1028.

18. H. Ledbetter and M. Dunn, Predicted habit planes from two models: invariant-plane-strain and elastic-energy-minimization, in Displacive Phase Transformations and Their Applicaitons in Materials Engineering (TMS, Warrendale, 1998) 341-347.

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Project: NONLINEAR ULTRASONICS FOR MATERIALS CHARACTERIZATION

Principal Investigator: D.C. Hurley

Objectives

- Identify quantitative relationships between nonlinear ultrasonic measurables and the 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 a material's microstructure and its mechanical or 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 or remaining lifetime.

The technical approach is based on development of sensitive and accurate methods for linear and nonlinear ultrasonic measurements. Our primary experimental tool is a laser-based Michelson interferometer system developed at NIST. This system represents a unique capability, since it enables direct measurement of absolute ultrasonic displacements. Measurement of absolute displacements is essential for nonlinear studies and is useful in many linear applications as well. Advantages of interferometric detection include micrometer-scale resolution, an inherently nonintrusive nature, and 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 μm. The scanning capability of the interferometer means that the spatial dependence of the ultrasonic wavefield can also be examined.

Using this approach, microstructural characterization of bulk systems with nonlinear ultrasonics has begun. In a parallel effort, instrumentation and methods are being adapted to enable nonlinear 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 and conventional ultrasonic measurements will also be used to interpret results. In this way, quantitative relationships between nonlinear measurables and microstructural properties can be established.

External Collaborations

Industrial. Our CRADA with RITEC, Inc. continued in FY98 to develop prototype electronics for nonlinear ultrasonic measurements. The instrument's high-power, low-distortion generation electronics and superheterodyne detection circuits represent a unique instrumentation capability. Modifications based on our experimental evaluation of the prototype were incorporated into the commercial version, which was introduced in late FY98. The instrument's value is reflected in the fact that RITEC has already received numerous orders by government, academic, and industrial laboratories worldwide.

ATP. Ion-implanted steel specimens from Empire Hard Chrome, Inc. were obtained in early FY98. The specimens were created to meet our size requirements for surface-wave experiments. Nanohardness measurements of the samples by either NIST-Gaithersburg or commercial vendors were investigated. However, it was decided that the measurements would not produce meaningful information on these particular specimens.

International. In FY98, we began a joint project with E. Danicki (National Academy of Sciences, Poland) funded by the Marie Sklodowska-Curie Joint Poland-America Fund. The project addresses how to generate surface waves with the amplitudes and spectral purity needed for harmonic generation experiments. A research plan was created during Danicki's ten-day visit to NIST in January 1998. Plans include modeling and experimental verification of ultrasonic wave generation by different devices to identify the most effective method. This year, we also established collaborative ties with D. Price and A. Richards (CSIRO, Australia). Areas of common interest and potential collaboration have been identified. For instance, NIST is helping CSIRO to improve the sensitivity of their interferometric methods. Nonlinear experiments to be performed at NIST have been planned. The experiments are designed to validate the theoretical model CSIRO is developing to investigate nonlinear guided waves.

Academic. Collaborative ties with several academic colleagues were continued, including D. Barnard at Iowa State University. Through frequent conversations with Barnard, we have kept informed of his experimental progress and obtained valuable advice for our development of piezoelectric detection methods. Meanwhile, our experiments have provided insight into some details of his experimental methods. Further collaborative effort is planned, including a quantitative comparison of Iowa State's piezoelectric and NIST's interferometric methods.

Planned Outcome

We are working to develop new measurement methods to quantitatively assess the mechanical and physical properties of industrial materials. In particular, we hope to achieve techniques for the nondestructive evaluation of such performance-related properties as adhesion, hardness, and fatigue. In the long term, our work should lead to the development of tools which 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

In FY98, our experimental capabilities in both bulk and surface nonlinear ultrasonic measurements were extended. Previously developed techniques were refined for improved performance, and new methods were implemented. Having validated the accuracy and sensitivity of the new methods, we began to utilize them along with our existing methods to characterize materials microstructure.

The Nuclear Regulatory Commission (NRC) feasibility study begun in FY97 to investigate steel mechanical-property measurements continued into FY98. Last year's experiments on steels with 1.13 mass percent copper indicated a correlation between the bulk nonlinearity parameter β and specimen hardness. Results were consistent with a microstructural model which associated the strain with the relative coherency of copper-rich 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 supported this hypothesis and revealed a correlation between the experimental values for β and strain. This year, further measurements were made with the interferometer on more specimens covering a wider range of hardness and thus more variation in precipitate state. Although data analysis is still in progress, preliminary results reveal variations in β with hardness similar to last year's results.

The NRC program also included work on implementing a piezoelectric detection technique for nonlinear ultrasonic experiments. Measurement of absolute displacements with a piezoelectric transducer is less straightforward than with the interferometer, but appears more easily adaptable to measurements outside the laboratory. While developing the experimental methods to use piezoelectric detectors, we obtained results that shed light on an effect noticed by the Iowa State University group. It had been suggested that an observed variation in β with fundamental amplitude represented a new physical phenomenon. However, our data indicated that it was more likely related to transducer operation at resonance. Further experiments in this area together with Iowa State are planned. To make the piezoelectric method truly field-worthy, we worked with a vendor to fabricate a ruggedized version of single-crystal $LiNbO_3$ transducers. Experiments using these transducers on specimens previously measured by other methods yielded incorrect values for β. It is thought that the front wear plate produced interference effects in the second harmonic signal. Furthermore, the sensitivity of these transducers was sufficiently reduced to prohibit high-precision measurements in attenuative materials such as steels. Therefore, further work is needed to make this a truly practical method.

Experimental methods for nonlinear ultrasonic surface waves were further developed in FY98. A major challenge for nonlinear surface-wave experiments is the ability to generate narrow-frequency, finite-amplitude waves. In FY97, we demonstrated the ability to produce surface waves of sufficient amplitude and spectral purity using a Sokolinskii comb device. Experiments showed that surface waves excited by the comb were strongly diffracted due to finite-aperture effects. In FY98, we performed further experiments with a 10 MHz comb and analyzed the results in greater detail. Using the interferometer, we measured the on-axis amplitudes of the fundamental and second-harmonic wave components as a function of position. A theoretical model for harmonic generation and diffraction by a two-dimensional line source was extended to calculate the displacement amplitudes. Quantitative comparison between model and measurements required only two adjustable parameters: the ultrasonic attenuation α and the Rayleigh-wave nonlinearity parameter β_{11}, which contains a combination of the second- and third-order elastic moduli. The best-fit value for β_{11} was in excellent agreement with values calculated from literature data on third-order elastic moduli. Thus we are confident that the model adequately describes comb behavior on unmodified surfaces. A second, "bullseye" comb with concentric teeth was also tested in FY98. This comb was designed to eliminate the diffraction effects that complicated interpretation of the linear comb data. However, the comb appears impractical for use, since surface-wave amplitudes were too small to observe harmonic generation.

Lastly, in FY98 we extended our experimental capabilities for other ultrasonic measurements. An immersion, pulse-echo technique to measure ultrasonic attenuation in the 5-25 MHz frequency range was developed. This allowed us to obtain attenuation values for the NRC steel specimens and therefore calculate attenuation corrections to β. In addition, the interferometer was used for linear ultrasonic measurements of such properties as surface-wave velocity and spatial diffraction of longitudinal waves. Such measurements provide complementary information to the nonlinear results, and allow comparisons to be drawn between nonlinear and linear approaches.

Publications

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Project: **INTERNAL FRICTION MEASUREMENTS FOR MONITORING MICROSTRUCTURAL EVOLUTION**

Principal Investigator: W. Johnson
George Alers, 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.

The experimental approach used in this project employs a unique laboratory system to perform measurements of the anelastic ultrasonic properties of dislocations during loading in a tensile testing machine. The specimens are designed to have "trapped" resonant vibrational modes localized in a central portion of the specimen and, thus, have insignificant energy losses through the machine grips.[1-3] In addition, energy losses through transducer coupling are essentially eliminated by employing noncontacting electromagnetic-acoustic transducers. Because of these features, this system provides unprecedented resolution and accuracy in measurements of ultrasonic internal friction under applied loads.

The strength of dislocation pinning is reflected in load-induced changes in ultrasonic damping and velocity. The focus of the research is primarily on measurements of relative changes in damping, rather than absolute values, because such measurements are considered to have greater practical potential for implementation in a field environment.

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* Visiting Scientist from the University of Toronto.

Planned Outcome

Experimental and theoretical results obtained in this project are intended to serve as a basis for developing an ultrasonic technique for nondestructively evaluating the integrity of structural alloys in service. For example, the aging of our country's bridges has become a major issue in recent years, because the integrity of many bridges is unknown and the cost of replacement is tremendous; the successful implementation of an ultrasonic sensor for early detection of fatigue damage in bridge steels would greatly aid in determining which bridges should be replaced or repaired.[4] A similar situation exists for a wide variety of structural components that may be unintentionally subjected to large loads that plastically deform the material; the implementation of an ultrasonic technique for detecting prior plastic deformation would enable the identification of compromised sections of material before visible cracks develop.

Accomplishments

Exploratory studies in 1997 showed that fatigue[4] and plastic deformation have significant effects on the equilibrium ultrasonic damping and the time-dependent response of the damping to loading. The extension of this research program in 1998 has sought to provide further fundamental experimental data that will serve to establish a physical model for the observed effects.

An extensive series of measurements were performed of the time-dependent response of ultrasonic properties to the application and release of elevated loads in A710 and A36 carbon steels and interstitial-free steels. These measurements were performed as a function of plastic strain, number of fatigue cycles, fatigue stress, temperature, ultrasonic frequency, magnetic field, duration and magnitude of load, and high-temperature annealing.

The results from all the materials that were studied lead to the same central conclusion: the effects of elevated loads on the damping are strongly dependent on prior plastic deformation, whether such deformation is produced by monotonic or oscillatory stress. In undeformed material, elevated loads below the proportional limit had no measurable effect on the damping. However, in deformed material, even relatively low loads induced an increase in damping followed by recovery, presumably because of a greater short-range dislocation mobility.[5] The results suggest that this behavior may be common to all ferritic steels, and may serve as a basis for nondestructively obtaining information on mechanical history.

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3. W. Johnson, G. A. Alers, and B. A. Auld, "Acoustic resonator for measuring force," U. S. Patent no. 5813280 (issued 9/29/98).

4. R. A. Livingston, W. Johnson, D. McColskey, C. Fortunko, and G. Alers, "Measurement of material properties for early detection of fatigue damage in highway bridge steels," *Nondestructive Characterization of Materials VIII* (Plenum, New York, 1998), p. 475.

5. W. Johnson, A.N. Sinclair, P.T. Purtscher, and G.A. Alers, "Effects of plastic deformation on ultrasonic damping in ferritic steel," *Review of Progress in Quantitative Nondestructive Evaluation, Vol. 18* (proceedings of the conference in Snowbird, UT, July 19-24, 1998)(to be published).

PROJECT: ULTRASONIC MEASUREMENT OF STRESS

Principal Investigators: **A.V. Clark**
C.S. Hehman, K. Coakley*.

Objective:

Exploit the acoustoelastic effect (i.e., changes in stress cause changes in ultrasonic wave velocity) to infer stress from precise measurements of time-of-flight of shear waves propagating through the thickness of common structural materials such as aluminum and steel.

Technical Description

Our interest centers on stress measurement in large structures such as bridges, pipelines, etc. The structural elements here are in the form of rolled products or I-beams. The rolling process creates a small anisotropy in the shear modulus. Consequently we typically find a faster velocity for a shear wave polarized in the rolling direction (RD) and slower velocity in the transverse direction (TD). A wave polarized at an intermediate angle will split into components polarized along the RD and TD. Consequently these directions are pure-mode polarization directions (acoustic axes) for the unstressed state. Because the components have different velocities they will be out of phase with each other and interference will result; the amount of interference varies with transducer orientation.

Application of stress has two effects. First, unless the principal stress direction coincides with the RD and TD, the acoustic axes are rotated. Second, the velocities of waves polarized along the acoustic axes will be changed. Hence our measurement of stress requires locating the acoustic axes and measuring the times of flight of waves polarized in these directions.

External Collaborations

Industry: Caterpillar, Inc. (ATP Project)
Gas Research Institute (Contract)
Sonic Force Corp. (CRADA)

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*NIST, Statistical Engineering Division

Planned Outcome

We have constructed a motorized rotating electromagnetic-acoustic transducer (EMAT) for measurements to determine the orientation of the acoustic axes. Since the EMAT requires no couplant, rotation is rapid and no artifacts occur due to variation in couplant thickness. We used the EMAT in conjunction with a commercial phase-sensitive instrument to collect data as the EMAT rotates.

A theory which models the effect of interference on the phase data has been developed. The model has up to 14 adjustable parameters, which allow for a very accurate fit of the data. However a 4-parameter model appears adequate for most applications. The parameters account for transducer characteristics, as well as inhomogeneity and anisotropy in the velocity and attenuation of the material under test.

Accomplishments

Experimental Verification

To verify the validity of this approach we measured stress changes resulting from the assembly of an aluminum shrink-fit specimen. This consists of an inner plug whose diameter is slightly larger than the inner diameter of an annulus. The plug was cooled and the annulus heated to allow enough clearance for insertion of the plug into the annulus. On reaching ambient temperature there is a radial compressive stress and a tensile hoop stress in the annulus. We measured the times-of-flight (TOF) at 4 locations on 3 scanlines 45° apart prior to assembly. These data were collected with a custom-made gate and a time-interval-averaging counter. After assembly, phase data were collected at each measurement location with the rotating EMAT and the phase-sensitive instrument. Once the acoustic axes were determined (by analysis of phase data with our model) the EMAT was oriented along these directions and corresponding TOF data collected with the counter.

The weighted TOFs in the stress state were computed from these data in the stressed state. For example one such quantity is calculated by: $\iota_1 = T_s \cos^2 \varphi + T_f \sin^2 \varphi$, where φ is the rotation of the acoustic axes caused by stress. We use a matrix equation $(\Delta\sigma) = [K] (\Delta\iota)$ to compute changes in the plane stress state, where the $\Delta\iota$ represent differences in the weighted TOFs between stressed and unstressed state. The coefficients of the [K] matrix are the stress-acoustic constants of the material and are determined from specimens under uniaxial tension..

We compared our ultrasonic-stress data with two other methods: strain-gage data, and neutron-diffraction measurements made at the NIST Center for Neutron Research reactor. In both cases we obtained good agreement.

Development of Portable Instrumentation for Field Tests

Based on these experiments and on previous field tests on bridges, we procured a simpler, portable stress measurement unit. This consists of a lunchbox computer with an on-board digitizer, and a sensor housing which contains the EMAT, motor and preamplifier. Under computer control the EMAT rotates and its orientation and TOF are recorded in a data file. The TOF is determined by digitizing the received waveform and using an interpolation scheme to determine when a selected zero crossing occurs.

This system is simpler since only TOF data are collected and analyzed. (Our previous hybrid system used both a phase-sensitive instrument and a counter.) However using the TOF data to determine the orientation of the acoustic axes required revision of our previous 14-paramenter algorithm.

We tested both the portable system and our revised algorithm by measuring the stress-acoustic constants on specimens of the same grade of steel having different heat-treatment. The constants were determined from measurements on specimens cut at 0 and 90° to the RD. Then a consistency check was done on specimens cut at intermediate angles. The predicted variation of weighted TOFs with applied stress was in good agreement with measured values on these specimens. We also found that the stress-acoustic constants were changed somewhat by heat-treatment.

Interaction with Industrial Partners

Plans are now underway to use the portable instrument to measure stresses in gas pipelines. A 610 mm (24 in) diameter pipe will be supplied to NIST by the Gas Research Institute. The pipe will be subjected to 3-point bending in a test machine with a load capacity of 445 kN (1000 kip). Ultrasonic measurements will be made at different axial and circumferential locations as the pipe is loaded to yield.

Publications

1. A.V. Clark and C.S. Hehman, "Measurement of Plane Stress States Using a Rotating EMAT," Invited Paper, Spring Conference of The American Society for Nondestructive Testing, Anaheim, CA, March 23-27, 1998.

2. A.V. Clark, C.S. Hehman, D. Gallagher, M.G. Lozev, and P.A. Fuchs, "Ultrasonic Measurement of Stress in Pin and Hanger Connections," submitted to *Journal of Nondestructive Evaluation.*

3. A.V. Clark, C.S. Hehman, and T.N. Nguyen, "Ultrasonic Measurement of Stress Using a Rotating EMAT," submitted to *Research in Nondestructive Evaluation*.

4. A.V. Clark and S.R. Schaps, "Acoustoelastic Determination of Residual Stress by Measurement of Resonance Peaks and Phase Shifts," *Journal of Nondestructive Evaluation*, Vol. 17 No. 3, p. 89 (1998).

5. A.V. Clark, C.S. Hehman, K. Coakley, and P.A. Fuchs, "The Effect of Material Inhomogeneity on Ultrasonic Stress Measurement Using a Rotating EMAT," in review process.

6. A.V. Clark and C.S. Hehman, "Algorithm for Determination of Pure-Mode Polarization Directions from Time-of-Flight Data Collected with a Rotating SH-Wave EMAT," in review process.

Project: WAVEFORM-BASED ACOUSTIC EMISSION

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

Objectives

- Develop wideband, high-fidelity acoustic-emission methods, including necessary measurement procedures, 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 (for example, bridge steels).

- Investigate applicability of the 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 releases of transient energy 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 to predict 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 identify source locations and types, and experimental studies of simulated acoustic-emission wave propagation. The scope in FY98 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 with the old acoustic-emission technology using resonant sensors; and (3) studies of the characteristics of propagation of waves from artificial and real sources into thick-walled fiber composite shells, as well as monitoring of stress-corrosion damage in composites. The third phase is in support of an ATP related program, "Composite Production Risers". Special equipment and facilities assembled include a specially modified hydraulic system for materials

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* NIST, Applied and Computational Mathmatics Division

testing , multiple coupled waveform recorders (12-bit, 10 MHz digitization rate), and specially developed high-sensitivity, wideband acoustic-emission sensor/preamplifiers. A commercially available waveform-based acoustic-emission recording system is being 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. Also collaborated with Lincoln Composites 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 is providing technical expertise in developing specifications for the Federal Highway Administration for an SBIR for an advanced high-speed waveform-based acoustic-emission system. Also NIST is currently involved in monitoring Phase II SBIR developments. 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). NIST is also collaborating with acoustic-emission researchers at NASA Langley to study wave propagation and accuracy of source location.

Commercial Companies. Developed interactions with Grand Junction Steel for welding of steel test samples per typical bridge specifications for acoustic-emission testing of welded bridge steels.

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 material scientists and enhance the application of nondestructive characterization of the response of structures (such as 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 (for example, based on stress corrosion damage).

Accomplishments

Validated the finite-element modeling code for the fundamental, buried-dipole, and acoustic-emission point source. Validation was accomplished by comparison with other independent results (published and unpublished) and by convergence studies. Studied finite-element parameter relationships, such as resolution and source size necessary to create a finite-element-based buried "point source". Examined plate thickness and source rise-time effects on far-field out-of-plane displacements. Also studied reflections from plate boundaries by finite-element modeling and validated the results by experiments with a NIST absolutely calibrated sensor.

NIST took a leading role in organizing the first unified international meeting of the two major and oldest technical groups from different countries dedicated to research on and applications of acoustic emissions. The "International Acoustic Emission Conference" (August 1998) provided 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 provided a forum to compare and contrast the acoustic emission technologies used in the past with more recently developed approaches. Attended this meeting and carried out co-chairman role (Hamstad) and presented three papers (McColskey and Hamstad).

Gathered an acoustic-waveform database at two different rates of crack growth for various bridge steels as contrasted to an aluminum alloy. Carried out a microstructure examination for correlation with acoustic emission due to crack growth.

Publications

1. M.A. Hamstad and J.D. McColskey, "Wideband and Narrowband Acoustic Emission Waveforms from Extraneous Sources During Fatigue of Steel Samples," *Journal of Acoustic Emission*, Vol. 15 No. 1-4, 1997, pp. 1-18 (Note: Actual publication date Sept. 1998).

2. W.H. Prosser, M.A. Hamstad, J. Gary and A. O'Gallagher. "Comparison of Finite Element and Plate Theory Methods for Modeling Acoustic Emission Waveforms". Submitted to *Journal of Nondestructive Evaluation*, May 1998.

3. W.H. Prosser, M.A. Hamstad, J. Gary and A. O'Gallagher. "Reflections of AE Waves in Finite Plates: Finite Element Modeling and Experimental Measurements," Submitted to *Journal of Acoustic Emission*, June 1998.

4. M.A. Hamstad, J. Gary and A. O'Gallagher. "Wideband Acoustic Emission Signals as a Function of Source Rise-Time and Plate Thickness," Progress in Acoustic Emission IX, Acoustic Emission Working Group and Acoustic Emission Group, Los Angeles, CA, pp. IV-48 to IV-57, 1998.

Project: ULTRASONIC STANDARDS FOR MATERIALS CHARACTERIZATION

Project Leader: T.P. Lerch
M.C. Renken, G. Alers

Objectives

- Develop accurate ultrasonic methods for characterization of microstructures of commercial alloys and advanced materials.

- Identify and quantify all uncertainties affecting each of the measurement methods.

- Disseminate the results through technical publications, participation in standards activities, and collaborations with other laboratories and industry.

Technical Description

In order to use ultrasonics to characterize material microstructure, absolute elastic-wave velocities must often be known with an uncertainty of less than 0.1%. Determination of mechanical and internal strain properties, as well as validation of micro-mechanical models also require that the elastic-wave velocities be known very accurately. We have achieved this level of performance in our laboratory in the 100 kHz–10 MHz region with Resonance Ultrasonic Spectroscopy (RUS). However, the accuracies of more typical laboratory measurement methods are of the order of 1% or greater. Mechanical testing methods used to determine elastic moduli typically have accuracies on the order of 5% or greater. We would like to increase the accuracies of these more typical methods by studying, and eventually reducing, the amount of uncertainty in each method.

Attenuation is another microstructural characteristic that must be measured to within a very narrow range of uncertainty. Preliminary work has started on improving and refining current experimental methods used to measure this material property. However, there is a lack of consensus among the scientific community on how to define, much less measure, attenuation. This issue must continue to be addressed in the future.

We have already demonstrated the feasibility of high-accuracy measurements of elastic-wave velocities and their utility in microstructural characterization, as well as their use for validation of micro-mechanical models. We now use such methods, RUS, and certain short-pulse, time-of-flight methods for routine, high-accuracy determination of elastic-stiffness coefficients of single crystals and of materials with complex microstructures.

Our current effort also includes the development of appropriate contacts within those segments of the scientific and industrial communities that rely on accurate velocity measurements for quality control. We have developed collaborations with several leading laboratories, including the (German) Federal Institute for Materials Research and Testing (BAM), and industry-consensus organizations, such as the American Society for Testing of Materials (ASTM), and the International Institute of Welding (IIW).

In FY 1998, we began a number of studies designed to identify the advantages and disadvantages of using various elastic-wave velocity measurement techniques, including the First Arrival Superposition Technique (FAST), the Pulse-Echo Overlap (PEO) method, and the Multiple-Echo Zero Crossings (MEZC) technique. A concerted effort was made to identify and quantify the known uncertainties affecting each of these measurement techniques. As a result, a general understanding of the physical factors that affect the accuracy and resolution of the techniques is being developed. Theoretical measurement models are also in the process of being developed. When completed, we expect to be able to model and correct for the effects of several physical phenomena that affect the measurements, including diffraction, bonding issues, transducer characteristics, sampling rate and digital quantization, as well as for time-window effects.

The preliminary results of this research are being applied to an industrial standards program that has international implications. In March of 1997, ASTM Committee E-7 on Nondestructive Testing approached NIST for help in resolving known problems with the ASTM standard E-164 "Standard Practice for Ultrasonic Contact Examination of Weldments". This document includes an annex describing the U.S. 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 in these blocks, due to a lack of consensus. However, these calibration blocks are used in the U.S. and elsewhere to establish critical factors, including location of the ultrasonic beam exit point, angle of refraction, and important search-unit settings, such as gain and time-distance calibrations.

In contrast, a proposed European standard calls for the ultrasonic velocities of an IIW-type block to be specified with an absolute uncertainty of 0.1%. However, our preliminary measurements indicate that this specification cannot be met using typical ultrasonic equipment and measurement methods. We believe that our work will lead to the establishment of a rational specification that can be incorporated in future documents of both ASTM and ISO.

In order to aid the ASTM committee in the revision of their current E-164 standard, we have collected a substantial number of IIW-type blocks and have conducted a series of ultrasonic measurements on them, including the determination of elastic-wave velocities, attenuation, exit point locations, and angles of refraction. Based on these measurements, we are able to report the magnitude of block-to-block variations for these different quantities. Variations in the measurements due to human factors and application factors are also being quantified, allowing us to isolate the microstructural differences among the different blocks. The results of these measurements will be reported at an upcoming ASTM meeting in January of 1999.

M.C. Renken is completing a Ph.D. thesis at the University of Colorado in Boulder on the subject of time/frequency analysis of dispersive elastic waves. The goal of this work is to develop quantitative measurement models that will account for time- and frequency-domain effects, as well as digitization errors that have been observed in our short-pulse, time-of-flight methods.

External Collaborations

The (German) Federal Institute for Materials Research and Testing (BAM), ASTM and Iowa State University's Center for Nondestructive Evaluation.

Planned Outcome

A revision of the ASTM standard for the IIW-type test block based on quantitative measurements. A possible revision of the ISO standard of the same calibration block. An improved design of the U.S. IIW-type calibration block. Identification of the sources of variability in elastic-wave velocity measurements due to material microstructure, equipment, procedures, and human factors.

Accomplishments

Conducted a series of elastic-wave velocity measurements on 18 IIW-type calibration blocks using commercial NDT transducers, single crystal transducers, and electromagnetic acoustic transducers. Determined the range of variability of longitudinal and shear wave velocities in this series of blocks.

Measured ultrasonic beam exit points and angles of refraction for the sample of test blocks. Determined the range of variability of these measurements in the sample of blocks.

Identified and quantified a number of the uncertainties affecting the various techniques for measuring velocity.

Updated and revised the pulse-echo overlap method for determining elastic-wave velocities with the aid of Emmanuel Papadakis, who is an expert on material velocity measurements.

Gave a report summarizing this activity to the ASTM winter meeting in January. Presented preliminary results of our IIW-type calibration block study to the ASTM subcommittee meeting in June. Presented a total of 3 technical papers based on the IIW-type block study: one at the Review of Quantitative NDE in July, the others at the IEEE Ultrasonics Symposium in October.

Assisting in the organization of the European-American Workshop on Determination of Reliability and Validation Methods of NDE. This workshop is sponsored by BAM, NIST, and the American Society for Nondestructive Testing (ASNT). Effects of material microstructure on NDE measurements were identified as an important parameter in the 1997 workshop. NIST will host this workshop in September 1999. We expect nearly 100 participants representing approximately 20 countries.

Publications

1. K.W. Hollman and C.M. Fortunko, "An accurate method for measurement of transverse elastic-wave velocities," *Meas. Sci. Technol.*, 9, 1721-1727, 1998.

2. T.P. Lerch, M.C. Renken, and C.M. Fortunko, "Measurements of longitudinal-wave velocity in IIW-type calibration blocks," in *Rev. Prog. QNDE*, 18, D.O Thompson and D.E. Chimenti Eds. (Plenum, New York, 1999), in press.

3. T.P. Lerch, M.C. Renken, and C.M. Fortunko, "Accurate measurements of longitudinal-wave velocity in IIW-type calibration blocks," in Proc. 1998 IEEE Ultrasonics Symposium, in press.

4. M.C. Renken, C.M. Fortunko, C.T. Mullis, "Effects of signal-processing parameters on phase velocity uncertainty," in Proc. 1998 IEEE Ultrasonics Symposium, in press.

Project: **GREEN'S FUNCTION METHOD FOR MATERIALS SCIENCE**

Principal Investigator: Vinod K. Tewary

Technical Objectives:

- To develop computationally efficient methods for calculating elastostatic and elastodynamic Green's functions for bounded solids.
- To apply the Green's functions in the development of the boundary-element formulation for stress analysis and propagation of elastic waves in anisotropic solids and to interpret the results of measurements on the elastic response of solids.
- To develop lattice static Green's-function method for modeling lattice defects in crystals and to interpret the results of measurements using X-ray diffraction.

Technical Description

Green's functions give the response of a solid to a probe and are also called response functions. Our interest is in the elastic response of a solid to a mechanical force. For time-dependent forces, we use dynamic or time-dependent Green's functions. The static Green's functions are used for time-independent problems. In the case of dynamic Green's functions, our interest is only in the causal Green's functions.

At the atomistic level, the response of a crystal lattice is given by the lattice Green's function that we calculate using the Born-von Karman model of a crystal lattice. A defect or a discontinuity in the lattice may exert the force that distorts the lattice. The lattice static Green's function method is used to calculate the lattice distortion. The physical measurements that are sensitive to lattice distortions are, for example, neutron or X-ray diffraction patterns. Static lattice Green's functions depend upon interatomic potentials and detailed lattice structure.

The bulk properties of solids, such as the elastic constants, can be modeled by assuming the solid to be a continuum of matter. In this case, we use the continuum model. The continuum-model Green's function or the elastic Green's function is defined in terms of the elastic constants of the solid. The elastostatic Green's functions are used to calculate the stress distribution in a solid, fracture properties, etc. The elastodynamic Green's function is used for calculating elastic wave forms in solids and non-destructive characterization of solids.

The continuum or the elastostatic Green's function is the asymptotic limit of the static lattice Green's function. Thus, the Green's-function method provides a convenient framework for an integrated model of a solid or a unified formulation that can model a solid in different physical regions and over different length scales.

A major computational advantage of the Green's function is that it is a characteristic of the material and its geometry, and is independent of the probe. The Green's function can be calculated in steps of increasing geometrical or structural complexities using the previous value as input. It is, therefore, possible to calculate and store the Green's functions for basic geometrical shapes and structures and different material parameters, for use in further calculations. Thus it is useful to set up a library of Green's functions. Static lattice Green's functions for several fcc and bcc solids will be made available soon at the web site of our Division.

SIMA project: A project on setting up of a library of Green's functions on WWW with Internet access has been sponsored by SIMA. Work on this project is being carried out in collaboration with the Colorado School of Mines and the 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 the Colorado School of Mines, and elastodynamic problems at 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.

External Collaborations

1. Collaboration with John Berger of Colorado school of Mines on elastostatic calculations on anisotropic materials for the SIMA project.

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

3. Collaboration with Ball Aerospace on the application of Green's function/boundary element method to industrial problems on stress analysis of solids.

Planned Outcome

The elastodynamic Green's functions will be used in interpreting measurements on elastic wave propagation characteristics of solids. The static lattice Green's function method will enable us to study the microstructure of solids and interpret experimental results on X-ray diffraction from crystals. The elastostatic and elastodynamic Green's-function method, combined with the boundary element analysis, makes it suitable for industrial applications such as stress analysis, fracture analysis, and NDE of solids. The work on SIMA project will result into a widespread availability of Green's functions on the WWW, which will be very useful for industrial applications.

Accomplishments

1. A static lattice Green's function method for crystals containing point and extended defects has been formulated using a model interatomic potential.

2. The Dyson equation for the Green's function for point defects is solved by the method of partitioning of matrices. The method is applied to calculate lattice distortion due to point defects and defect-defect interaction in copper.

3. The Dyson equation for the Green's function for extended defects is solved by an entirely new technique by projecting it into the defect space and using a defect-space Fourier-transform method. The correspondence between continuum and static lattice Green's functions for defects in solids has been established, which is a necessary condition for the reliability of the method. The method has been applied to calculate lattice distortion due to a dislocation and interaction between a point defect and a dislocation in a model cubic lattice. The method will be extended to fcc lattices and will be applied to interpret measurements on X-ray scattering from dislocations in copper.

4. A theoretical design of a possible system for measurement of elastic constants using the response of solids in slowness space has been proposed. Some preliminary calculations have been carried out.

5. A web page has been created describing the work on Green's functions. Computer codes for calculations of Green's functions for solids will be available on this web page.

6. SIMA project: work carried out by the Iowa State University and the Colorado School of Mines in collaboration with MRD.

    - Work on the Green's-Function-Library capability to calculate and visually display far-field ultrasonic data for scattering of ultrasonic waves from buried flaws in linear, isotropic, elastic structures, is completed.

    - A convenient GUI is in place also such that an analyst has the capability to obtain data, in visual form, quickly, and with no computing expertise required.

    - A preliminary web page to view this capability has been created at Iowa State University: **http://www.public.iastate.edu/~shanlu/aladin/nowhere/menu.html**

    - Various solutions for stress and displacement, with graphical displays of the data in the bracket of an elastic "structural bracket" with an internal slot, under an applied bending load, as a function of size, position, and orientation of the slot are now available.

- The graphics for data display, e.g., deformations and peak boundary stresses, have been developed, as have the computing engines for forming and using the discretized Green's functions.

- The GUI for data entry, bracket shape, and slot shape and location choices is well developed.

- Work on our Green's-function-library capability, for the intact and damaged-composite materials models, has been completed and placed, in draft form, on a limited-access web page.

- A research paper on this capability is in preparation which will include an additional, more sophisticated model for interfacial shear stresses than is currently to be found on any web page.

- Analytic determination of derivatives with respect to material orientation in the anisotropic Greens' functions required for Sensitivity Boundary Integral Equations for fiber orientation in anisotropic materials has been completed.

Publications

1. J.R. Berger, J.S. Siklowitz, and V.K. Tewary: "Green's function for steady state heat conduction in a bimaterial composite solid." Accepted for publication *in Quarterly Journal of Mechanics and Applied Mathematics.*

2. J.R. Berger and V.K. Tewary: "BIE Green's functions for interfaces in anisotropic solids." Accepted for publication in *Computational Mechanics*.

3. V.K. Tewary: "Inversion of elastic waveform data in anisotropic solids using the delta-function representation of the Green's function" *J. Acoustic Soc. America*, **104,** 1716 (1998).

4. V.K. Tewary: "Lattice statics Green's function for modeling of dislocations in crystals" *Computational and Mathematical Models of Microstructural Evolution* (Published by Materials Research Society, USA; Editors- J.W. Bullard, L. Chen, R.K. Kalia, and A.M. Stoneham, **529,** 15 (1998).

Project: NONDESTRUCTIVE CHARACTERIZATION OF STEEL EMBRITTLEMENT

Project Leader: G.A. Alers
B. Igarashi, W. Johnson, D. Hurley, H. Ledbetter and P. Purtscher

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 are currently 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 can be achieved with the 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 very accurate, nondestructive physical property measurements of magnetic, eddy-current and ultrasonic properties using 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 property/microstructure relationships. During FY98, effort was concentrated on steel alloys that are embrittled by the formation of nanometer-sized copper-rich precipitates to see whether ultrasonic or magnetic measurements could detect these microstructural features that have such a profound effect on the strength of the alloys. In addition, techniques for deducing the nine elastic constants of plate or sheet products without cutting out specimens with special shapes were developed.

External Collaborations

SPONSORS:

Nuclear Regulatory Commission for development of nondestructive techniques to detect radiation embrittlement in RPV steels.

GUEST WORKERS:

Tony Sinclair was a Guest Scientist working in the Division while on a sabbatical leave from the Faculty of Applied Science and Engineering at the University of Toronto in Canada.

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, or it could enable the prediction of remaining life in structural elements that have been exposed to hostile environments in industrial settings.

Accomplishments

1. Measurement of Magnetostriction: In ferromagnetic materials and in particular steel, the application of a magnetic field changes the dimensions of the sample. This phenomenon, called magnetostriction, can be used to excite ultrasonic waves by producing the magnetic field with a coil of wire carrying an RF current at the frequency of the desired wave. The amplitude of the ultrasonic wave produced under these conditions can be used to measure the value the magnetostrictive coefficients of the particular ferromagnetic material involved. In order to use such measurements of the magnetostriction coefficient as a nondestructive materials characterization tool, we have focused our research on verification of theoretical models that relate measured ultrasonic wave amplitudes to the magnetostriction coefficient and then to demonstrating the existence of correlations between the coefficient and precipitation hardening mechanisms in ULC and HSLA steels. During FY98, the techniques were refined and applied to steels of interest to the Nuclear Regulatory Commission as part of a feasibility study aimed at developing nondestructive methods for monitoring the development of radiation induced embrittlement in reactor pressure vessels. Encouraging results were obtained on a surrogate alloy of iron containing 1.13% copper which could be embrittled by a heat treatment that forms the same copper-rich precipitates that are believed to cause radiation-induced embrittlement. Late in FY 98, the ultrasonic method of measuring the magnetostrictive coefficient was extended to A533B reactor steel samples in the shape of compact tension specimens that had been exposed to neutron irradiation inside nuclear reactors. These materials are radioactive so the measurements to demonstrate a correlation between magnetostriction and radiation damage had to be carried out by manipulators in a hot cell at the Phoenix Reactor of the University of Michigan where a set of specimens with known radiation histories are stored. Preliminary analysis of the data obtained in these tests indicates that there is a linear correlation between the magnetostriction and the neutron fluence to which the specimens were exposed.

2. Nondestructive Prediction of Strength Parameters. As a result of the feasibility study sponsored by the Nuclear Regulatory Commission, several physical properties that could be measured nondestructively were found to correlate linearly with the hardness. Those that showed the greatest promise for predicting values for other strength-related parameters are being studied in more detail on additional samples of copper-bearing steel alloys and on carbon-containing alloys strengthened by conventional heat treatment practices. The results of these studies are presented in other project descriptions in this document. The elastic moduli as derived from ultrasonic wave velocity measurements are well known to be potential candidates for nondestructive prediction of strength although their measurement must be performed with a precision better than ±0.5 %. The main source of error in ultrasonic wave velocity measurements is the coupling medium that transfers the acoustic energy to and from the transducer. By using EMATs operating on metals, this error is eliminated because the sound waves are launched and detected at the free surface of the sample. Following the magnetostriction measurements on the radioactive fracture toughness samples in the hot cell at the University of Michigan, high precision shear and Rayleigh wave velocity measurements were performed on the same samples. The technique used allowed these measurements to be made with a precision of between ±0.1% and 0.01%. Preliminary analysis of the data showed that a total neutron fluence of 2×10^{19} N/cm^2 changed the shear wave velocity by much less than 0.1% while it changed the Rayleigh wave velocity by more than 1%. This can be interpreted as showing that the extensional elastic moduli are far more sensitive to radiation damage than the shear moduli.

3. Microstructure Monitoring in Rolled Plate. For the past several years, NIST has studied the formation of rolling and recrystallization textures in sheet copper, brass and bronze products where the intrinsic elastic anisotropy of these alloys produce large anisotropies in both the elastic and mechanical properties of the final commercial products. In order to relate measurements of ultrasonic wave velocities to the texture and microstructure of not only these materials but also to rolled aluminum and steel products, the FY 98 effort was devoted to the development of ultrasonic measurement techniques that could be performed from one surface of the plate or sheet and not require cutting out any samples with special shapes. By using electromagnetic acoustic transducers (EMATs) for the measurements, a wide variety of wave types could be utilized and the methods could be readily transferred to process-control applications in an operating rolling mill. In general, rolled plate and sheet products must be viewed as materials with orthorhombic symmetry because the rolling, transverse and thickness directions form a coordinate system of three orthogonal axes along which the properties are not equal. For ultrasonic wave propagation, this means that there are nine elastic constants and nine independent wave velocities that must be measured in order to fully characterize the material. However, if the individual grains within the material posess cubic symmetry, as is the case for copper brass, aluminum and steel, there are mathematical relationships that reduce the number of independent elastic constants to only five. To test the validity of all these arguments and to determine the accuracy with which wave velocity measurements could be converted into elastic-modulus values, 3 mm thick plates of copper, brass and bronze with various degrees of texture were investigated using thru-thickness shear and longitudinal waves and in-plane Lamb and shear-horizontal ultrasonic waves in the frequency range between 0.4 and 2 MHz. In all, 13 different wave velocities were measured using techniques that yielded data with accuracies

between 0.1 and 0.5%. Values for the nine elastic moduli were deduced from various combinations of these data and it was concluded that the three shear modili could be determined to an accuracy of ±0.5% or better, the three diagonal stiffness moduli to an accuracy no better than ± 2% and the off diagonal moduli to an accuracy no better than ± 4%. These errors are expected to be reduced considerably by choosing Lamb-wave modes that are particularly sensitive to extensional deformations and are not highly dispersive.

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Project: SENSORS FOR INDUSTRIAL NDE

Project Leader: G. A. Alers
B. Igarashi, R.L. Santoyo, R.E. Schramm and 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, rugged and reliable sensors are needed after the material is manufactured and put into service in order to make sure that no important mechanical properties have degraded during service. The objective of this program is to develop sensors that can produce outputs useful for process control in the environment of a mass-production facility as well as to monitor the degradation of materials as they are used in the hostile environments found in field applications.

Technical Description

Modern sensors are usually specialized for the materials on which they are to be used and they often need to be designed to monitor special processing variables as well as to survive the environments in which the materials will be used. During this year, four industrial sensor problems have received attention. First, the gas-coupled ultrasonic transducer intended for inspection of buried natural-gas pipelines was modified in order to test its capabilities as an ultrasonic thickness gage for monitoring corrosion damage from a "pig" moving through the pipeline. Second, a new portable ultrasonic inspection unit was assembled to make quantitative measurements of the in-plane stress components in plate-like structures under field conditions. Its primary application is described in the project entitled "Ultrasonic Measurement of Stress". Two other transducers that were developed in previous years were put into service. One was used to demonstrate that corrosion in copper grounding cables could be detected by a pulse-echo technique based on the propagation of a low-frequency torsional ultrasonic wave along the length of the buried cable. The other transducer became the basis for a system for continuous monitoring of the internal friction and elastic modulus of tensile test specimens during the accumulation of high-cycle fatigue damage. Its use is described in the Project entitled "Internal Friction Measurements for Monitoring Microstructural Evolution".

External Collaborations

Industrial

Southwest Research Institute for gas coupled ultrasonic inspection of gas pipelines.
Caterpillar Inc. for measurement of residual stresses near welded joints.

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 residual stress measurement in 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

1. Air Coupled Transducers for Gas Pipeline Inspection. The gas industry needs better methods for inspecting their gas-transmission pipelines for flaws such as loss of wall thickness and stress corrosion cracking. Ultrasonic inspection methods have been used successfully in liquid-filled pipelines but their use for 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 and a cooperative research program was initiated with the Gas Research Institute and Southwest Research Institute in order to show that gas-coupled ultrasonic inspection could be performed in a pressurized natural-gas transmission pipeline. In previous years, it was demonstrated that an angle-beam approach could be used to detect small slots in the OD of a pipe from transducers operating in the high-pressure gas on the ID of the pipe. However, the sensitivity to small stress corrosion cracks appeared to be limited by surface roughness and the requirement for precise angular positioning of the transducer relative to the pipe wall. Therefore, a less ambitious goal of measuring the transit time of an ultrasonic wave propagating through the thickness was undertaken in order to demonstrate that the thickness-gaging technology currently used in liquid filled pipelines could be implemented in a gas pipeline by using properly designed gas-coupled transducers. Most of the design requirements were already available in the gas-coupled transducers used for crack detection but the bandwidth needed to be extended and the recovery time after excitation had to be shortened. Several commercial manufacturers of custom transducers were asked to supply piezoelectric transducers with a high damping layer attached to the back side of the piezoelectric element in order to extend its bandwidth. Tests performed in the NIST high pressure chamber using the NIST custom pulser/receiver electronics showed that extraneous vibrational modes in the piezoelectric disc or in its case were large enough to obscure the first reflection from the OD of the pipe wall. Measurement of the arrival time of this first echo is necessary for determination of the remaining wall in a corroded region where multiple echos will be suppressed by the irregular shape of the pitted surface. Composite transducers that

suppress the extraneous modes by surrounding the piezoelectric elements with a damping material are currently being tested but they, too, show unacceptable "ring-down" times. The current plan is to investigate a pitch-catch configuration in which a separate transmitter and receiver are inclined to the ID surface so that they communicate by an angle-beam shear wave reflected at the OD surface. This arrangement will lengthen the time to the arrival of the first echo and will also tolerate a longer recovery time from the excitation pulse.

2. Instrumentation for Measuring Residual Stress in Large Structures. During FY97, the FRA awarded NIST a contract to assemble a compact and portable EMAT based instrument for measuring the residual stress level in railroad-wheel rims while the wheels are in-service on a rail car. By mounting commercially available circuit boards in a compact, portable computer and by assembling an EMAT that could be switched automatically between two shear-wave polarizations, a prototype instrument that output the value of the apparent residual stress in the rim of a railroad wheel was produced. During FY98, this instrument was demonstrated to representatives of the railroad industry at the Transportation Technology Center (TTC) of the Association of American Railroads (AAR) in Pueblo, Colorado, and is available for their use. Based on the experience gained with this prototype on railroad wheel rims, specifications for a more versatile but still portable instrument were drawn up and an RFQ was circulated among commercial manufacturers of EMAT inspection systems. The winning respondent provided NIST with a rugged portable computer and a compact transmitter/receiver package that could drive the EMAT with higher power and deliver signals with a better signal-to-noise ratio than the FRA unit. Its transducer was equipped with a motor and control circuits that would allow automatic collection of data on transit time versus polarization angle instead of the simple pair of readings at two angles separated by 90 °. Because of the additional information contained in the angular dependence data, the orientation of the principal stress axes relative to the texture axes in plate-like materials can now be measured and stress distributions around welds and other joints can be determined by scanning a hand-held probe around the region adjacent to the joint. Details of the system's use are described in the Project entitled "Ultrasonic Measurement of Stress".

3. 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. Conventional ultrasonic methods require coupling a flat piezoelectric transducer to the irregular cylindrical surface presented by a bundle of individual wires twisted into the shape of a cable. By designing an EMAT that would couple to the entire circumference and generate an ultrasonic wave with a wavelength that was long compared to the diameter of the individual wire strands, it was possible to excite a torsional mode of vibration that could propagate over long distances and would reflect from broken strands or corroded regions that changed the gross dimensions of the cable. Because the wave was torsional, it did not couple strongly to the earth surrounding the cable when it was buried, and so the inspection of long distances under ground could be carried out. During FY98, a laboratory prototype was taken into the field and demonstrated to representatives of EPRI and other electric utilities. The student responsible for building and testing the transducers used the system as the basis for his Masters Degree thesis at the School of Mines and an application for a joint patent between NIST and CSM is being filed.

4. Resonant Body Sensors. Measurement of the free vibrational modes of spheres, parallelepipeds and cylinders has become a very useful tool for ultrasonic materials characterization. (See the Project entitled, "Elastic Coefficients and Related Physical Properties".) 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 by procedures that do not influence the vibrations. During the past few years, special EMATs that satisfy the requirement for weak coupling have been developed for operation on cylindrical bodies, and a new technique for trapping the vibrational energy away from attachment points has allowed sturdy mechanical gripping of the specimen without influencing the resonant vibrations.

During FY98, several different configurations of EMAT coils and magnets were investigated and it was demonstrated that breathing modes, torsional modes and generalized flexure modes (axial shear modes) could be excited and detected selectively in the trapping region. This allowed both shear and extensional elastic moduli to be measured along with their associated internal-friction coefficients. Because sturdy mechanical attachment to the sample is possible, large stresses could be applied and the effects of plastic deformation and fatigue damage could be studied. A discussion of the use of the trapped-mode studies made with this transducer system can be found in the Project entitled, "Internal Friction Measurements for Monitoring Microstructural Evolution".

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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,

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[1] *National Technology Roadmap for Semiconductors*, Semiconductor Industry Association, San Jose, CA, 1994, 1997; *National Technology Roadmap for Electronic Interconnections*, Institute for Interconnecting and Packaging Electronic Circuits, Lincolnwood, IL, 1995, 1997; *National Electronics Manufacturing Technology Roadmap*, National Electronics Manufacturing Initiative, Inc., Herndon, VA, 1996, 1998 (in press).
[2]http://www.ctcms.nist.gov/programs/solder
[3]http://www.eeel.nist.gov/810.01/index.html

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 can be found at: (http://www.msel.nist.gov/research.html) or in *Electronics Packaging, Interconnection and Assembly at NIST: Guide and Resources*, NISTIR 5817, copies of which may be obtained by contacting Frank Gayle at (301) 975-6161 or frank.gayle@nist.gov.

Project: MECHANICAL BEHAVIOR OF THIN FILMS

Principal Investigator: D. T. Read

Objectives

Develop experimental techniques to measure the mechanical properties of thin films, including basic tensile properties, fatigue, and fracture resistance. Relate mechanical behavior of thin film to microstructure. Extend test techniques from their present level (1 μm thick, ≥ 10 μm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths 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 interfacial 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 quite different from those of bulk materials of the same chemical composition. While the general principles of conventional mechanical testing are applicable to thin films, conventional test equipment and techniques are not. Because vapor-deposited films are of the order of 1 μm thick, the failure loads are of the order of gram-forces, and the specimens cannot be handled directly.

In addition to metallic thin films, 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) devices 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, but 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 tensile test techniques for thin film that are the same in principle as standard macroscopic tensile tests. The key element with thin films is that the specimens are so small and delicate that they cannot be handled directly. Hence a handling strategy must be utilized. The silicon-framed tensile specimen is the answer. This specimen consists of a silicon frame that carries a tensile coupon. The frame is conveniently handled, for attachment to the grips of the testing device and for alignment; then the silicon is carefully cut. We have had good results using a dental drill for this purpose.

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. There are additional technical reasons to seek a better approach for testing very small specimens. We have developed a new conceptual approach to these tests. Previously, a "skyhook" 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 on to the free end of the specimen, and pulls it to failure. This year a similar approach was attempted with metal specimens with widths in the 10 to 20 μm range. Specimens were designed and fabricated. The silicon was etched away from the specimen using xenon difluoride (see paragraph on etching silicon below). We used the probe tip of our atomic force microscope (AFM) 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. While this experiment was successful in that metal specimens were pulled to failure with the AFM tip, and signals related to force and displacement were recorded, the AFM approach was shelved because of the difficulty of calibrating the AFM tip for in-plane force.

Instead, we developed a specimen stage instrumented for small in-plane forces, mounted this stage atop the moving grip of the piezo-actuated microtensile tester, hooked the free end of the specimen with a tungsten needle probe tip that was bent to form a hook, and used the piezo drive to stretch the specimens to failure. This approach appeared to be experimentally reproducible. The forces were measured using the calibrated in-plane force stage. However, the total displacement includes an unwanted contribution from the deformation of the specimen grip around the hook. Digital image correlation using sequences of images acquired during the test was attempted as a means of measuring displacement. This appears feasible for use in obtaining the yield and ultimate strengths and the extension to failure. It is potentially accurate enough for measurements of Young's modulus if images of high quality can be obtained. Because the magnification used for observing these tests is high already, and will need to be higher for smaller specimens, we are making plans to perform this experiment in the scanning electron microscope, where the depth of focus is higher than in the optical microscope.

Another disadvantage of the present technique is the use of hydrazine hydrate as the etchant to complete the silicon-framed tensile specimen by removing 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 became aware of a new and exotic etchant for silicon, namely, 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 the silicon then reacts with fluorine to produce the 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. We have developed a reactor for etching silicon wafers and chips with xenon difluoride, and used it successfully to etch the next-generation tensile specimens discussed above. A key feature of these specimen designs is that etching fully through the wafer is no longer required. An etch that removes approximately 50 μm of silicon is sufficient.

External Collaborations

Motorola Advanced Interconnect Systems Laboratory, Tempe, AZ has built a microtensile tester based on the NIST design. DTR 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. In July of 1998 DTR visited the Motorola site for a week. We assembled the mechanical and electrical parts of the tester, calibrated it, and successfully conducted two tensile tests of materials made by Motorola.

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. It has become clear that both Ti and Al react with Si during the 450 °C heat treatment necessary to cause Ti and Al to react to form $TiAl_3$. X-ray diffraction revealed the presence of a TiAlSi phase in the reacted specimens. Furthermore, these specimens were extremely brittle, and could not be tested. Intel uses Ti and Al on top of SiO_2 , which (they claim) does not react with Ti and Al at 450 °C. If we were to leave the SiO_2 under our specimens, we would have to test a composite specimen consisting of oxide and metal. Such a test would produce ambiguous data. The problem of producing $TiAl_3$ specimens is exacerbated by the aggressiveness of the hydrazine hydrate etch used in specimen preparation. We plan to attempt to use the next-generation technique to test this material in the current fiscal year.

Semiconductor Research Corporation (SRC) — D.T. Read has joined the SRC Technical Advisory Board (TAB) on Packaging and Interconnect (with SRC's recent reorganization this has become part of Nanoscale Integration Systems (NIS)). He represents MSEL to this organization, and reports to the Electronic Packaging and Interconnect Project in MSEL.

Planned Outcome

There are three general types of anticipated outcomes (multiyear) from this work: data; experimental techniques; and standard test methods. Data have been generated during this and previous reporting years. The apparatus and experimental techniques have been developing. The highlight of FY98 was the transfer of the technology for thin-film tensile testing to Motorola. The present technique is useful and will be kept available. Development efforts are focused on the next-generation technique. 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. An industry-led group is planning to start an ASTM activity on mechanical behavior of micromechanical structures and materials.

Accomplishments

As mentioned above, we supplied Motorola with the necessary information to allow them to build their own microtensile tester based on the NIST design. They accumulated the parts, with some advice from D. T. Read. He visited them in July of 1998 for one week. During this visit, their tester was assembled, calibrated, and used to conduct tensile tests of nickel thin films made by Motorola.

A NIST Technical Note was produced, which summarizes several years of research on tensile testing of thin films. It provides mechanical drawings, computer programs and other information necessary for any interested group to construct and operate a piezo-actuated microtensile tester.

The xenon difluoride reaction etch system was used successfully to produce next-generation metal tensile specimens.

A proof-of-principle experiment was conducted for tensile testing using the atomic force microscope (AFM) as a test apparatus. Although this operation was shown to be possible in principle, it does not seem to be the best way to proceed.

An in-plane force stage was designed, fabricated, and used in next-generation tensile tests of aluminum specimens with an ultimate tensile force of approximately 2 millinewtons (0.2 gram-force). The mechanical behavior, specifically strength and ductility, of electron-beam-evaporated aluminum films appears substantially the same as previous measurements of specimens much larger in width and length.

Digital image correlation was developed to the point where it can be useful in microtensile testing, both optically and at higher magnifications, such as in the SEM. The key to the use of this technique is obtaining images with excellent resolution at the appropriate magnification.

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

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Project: EXPERIMENTAL MICROMECHANICS BY ELECTRON-BEAM MOIRÉ

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

Objectives

Develop and apply the electron-beam (e-beam) moiré technique to measurement of displacements and observation of deformations at high magnification. The technique is eminently suited to the problems of the microelectronics industry. As the industry continues the trend toward smaller and less expensive units, further development of the technique is necessary to maintain relevance. Improving the experimental technique to allow writing of more durable, high-contrast gratings will permit e-beam moiré to be used to characterize the thermal fatigue behavior of electronic packages containing inexpensive, but perhaps less than compatible material systems. We continue to pursue new methods of producing higher density gratings to permit the study of finer-scale packaging and on-chip features. As we apply the technique to the problems and concerns of the microelectronics industry, we use the experimental results to characterize failure modes and to verify various modeling approaches. A final objective is to reach out to industry to make them aware of the technique and its potential to solve some of the reliability problems they have.

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 displacements on the order of hundreds to tens of nanometers can be measured and strains calculated using the e-beam moiré technique. Deformations of packaging elements are measured over fields ranging from 50 x 50 μm to 500 x 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 gratings 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 displacements.

External Collaborations

Motorola — We were approached by Motorola, seeking a source for low-temperature strain/displacement measurements. They were finding that unexpected failures occurred in some flip-chip on-board packages at low temperatures (the same package performed very well at temperatures from room temperature to 125 °C). We were able to make measurements over the full temperature range (-55 to 125 °C) and supply them with the displacement data at locations identified by Motorola and point out areas of unusually high displacements not previously identified as a potential problem.

Intel — We participated in a comparison of a number of nanoscale-displacement measurement techniques sponsored by Intel, Chandler, AZ. Intel's goal in this program was two-fold: (1) To obtain data unavailable to them internally on critical elements in their package; and (2) To learn details about the various techniques to determine which would best meet their in-house needs (i.e., technology transfer). E. Drexler visited Intel and gave a presentation on the technique. Intel then provided us with four specimens of single-chip and multi-chip substrates (FR4), with and without the chip, to be thermally loaded between room temperature and 120 °C. The tests were conducted, images were analyzed, and displacements calculated, and the results transferred to Intel in a timely manner.

Colorado State University — C. Menoni and B. Parkinson are collaborating with us at NIST under a Colorado Advanced Materials Institute grant to find new methods to make the moiré grating. They are investigating the use of self-segregating block copolymers for the task. Using precise processing and etching procedures is expected to result in a regular grating array with a pitch of 10 nm. This would greatly improve the resolution of the e-beam moiré technique, allowing displacement measurements of features not only in smaller packages, but also on the chip.

Colorado School of Mines — Collaboration with J.R. Berger and his graduate student Bob Shepherd on an e-beam moiré study of a metal-metal interface verified advanced modeling approaches to determination of deformation modes

Planned Outcome

Successful development of a new method of producing moiré gratings for 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

Continued objectives of the program are to maintain relevance and to inform industry of the capabilities of the e-beam moiré technique. The accomplishments of this year have made great strides toward that end. Two dominant players in the world of microelectronics approached NIST to learn more about what the technique could do for them. This gave us the unique opportunity to demonstrate the technique on a problem that was of crucial interest to their programs and goals. A comprehensive document on the e-beam moiré technique was prepared for the Materials Reliability Series of NIST Technical Notes. It contains step-by-step instructions and computer programs to enable the reader to conduct e-beam moiré experiments on their own scanning electron microscope. It also contains the entire body of published work by NIST staff members on the e-beam moiré technique.

Tests were conducted at the request of Intel Corporation of Chandler, AZ on vias contained in single-chip and multi-chip substrates. The specimens were thermally loaded from room temperature to 120 °C, data acquired and reduced, and strains calculated and reported to Intel. The information was used by Intel to evaluate potential nanoscale displacement measurement techniques.

Tests were conducted at the request of Motorola of Austin, TX on flip-chip-on-board specimens. The specimens were thermally loaded between -55 and 125 °C, and displacements measured to help Motorola identify potential debonding sites at low temperatures in the package. The data sent to Motorola and they requested NIST to conduct more tests to evaluate package modifications.

Publications

1. "Error Analysis and Thermal Expansion Measurement with Electron-beam Moiré," J.R. Berger, E.S. Drexler, and D.T. Read, Experimental Mechanics 38, No. 3, 167-171 (1998).

2. *Procedures for the Electron-Beam Moiré Technique,* NIST Technical Note 1500-2, Materials Reliability Series. U.S. Government Printing Office, Washington, DC, 134 pp. (1998).

3. *Electron-Beam Moiré Technique: Advances, Verification, Application,* NIST Technical Note 1500-5, Materials Reliability Series, U.S. Government Printing Office, Washington, DC, 72 pp. (1998).

4. "Mechanical Deformation in Conductive Adhesives as Measured with Electron-Beam Moiré," E.S. Drexler and J.R. Berger, Journal of Electronic Packaging, accepted for publication.

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 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 voids can lead to open-circuit failures. Stresses result from differential thermal expansion among the metal and substrate layers and rigid passivation overlayer, or from atomic flux divergences due to strongly non-uniform local diffusion during electrical current stressing. Unless a more complete mechanistic understanding is developed, the impact of SV and EM is projected to worsen as the dimensions of interconnect structures continue to scale downward, and as new materials are introduced into interconnect architectures. Interconnects become less homogeneous as dimensions scale downward, since the structures then comprise individual grains through the film thickness and across the line width. Behavior also becomes less homogeneous, and even small variations in microstructure can detrimentally affect reliability. The introduction of new materials such as electrodeposited copper and low-k dielectrics poses new concerns since their behavior on the interconnect scale is poorly known. 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 test structures which act as a vehicle for the development of standard measurement and characterization methods for stress voiding and electromigration. Included in such structures is the possibility for studying the interactions between stress voiding and electromigration.

One approach to controlling reliability of interconnects centers on the fact that stress voids and electromigration voids typically nucleate at intersections of metallization grain boundaries and at the passivation/interconnect interface. The role of the interface on reliability is complex and not well understood. Interfacial flaws serve to decrease the activation energy for void nucleation according to thermodynamic calculations. In addition, debonded regions can modify the local stresses in the interconnect line. Ultimately, the interface between interconnect metallizations and surrounding passivation can be modified in an attempt to reduce or control the occurrence of voids. Interfaces can be modified by controllably depositing photoresist onto patterned metal lines prior to deposition of passivation. Accelerated testing then reveals how voiding has been changed from non-modified structures. Another approach to controlling interconnect reliability involves studying the effects of variations in PVD target material. Changes in target microstructure are suspected to lead to the development of different interconnect microstructures, and therefore different interconnect behavior. 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 subsequently characterized. Films will also be fabricated into narrow lines for determining SV and EM behavior.

Electron microscopy is 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.

The knowledge gained from the above types of studies is used in the design of test structures in collaboration with Semiconductor Electronics Division (EEEL). These structures provide for the development of standard measurement and characterization methods to assess interconnect reliability.

External Collaborations

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 discontinuous interface contamination on stress voiding.

Lehigh University
Collaboration with S. G. Cargill, III in a study of the effects of modified interfaces on electromigration of aluminum and aluminum-alloy lines.

Tosoh SMD Corporation
Collaboration with A. Bolcavage in a study of sputtering target metallurgy effects on thin film microstructures and reliability.

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.

SEMATECH
Collaboration with V. Blaschke in a study of processing effects on the microstructure of damascene copper interconnects.

TexSEM Laboratories
Collaboration with D. Field in studies of microstructures in thin films for conductors in microelectronic structures.

Planned Outcome

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 characterization techniques for routine assessment of interconnect reliability on a microstructural level based on electron microscopy. Design and fabrication of test structures for assessing stress voiding and electromigration.

Accomplishments

Completed fabrication of the NIST 34 test chip for assessing stress voiding and electromigration in single-level interconnects, in collaboration with H. Schafft of EEEL/Semiconductor Electronics Division. The geometrically simple structures are used to assess effects of linewidth and serpentine meandering on void densities. Submitted designs for test structures to be incorporated onto NIST 36, a test chip for assessing stress voiding and electromigration in two-level interconnects, incorporating via structures. Included are intentionally patterned notches to create local stress concentrations.

Fabricated and began EM and SV testing on passivated aluminum-alloy interconnects containing modified interfaces. Preliminary observations indicate that void densities are increased in modified regions, presumably due to changes in void nucleation energies. Assessment of the potential competition between decreased nucleation energies and kinetically favorable sites for rapid growth of void is underway.

Characterized the microtexture and 0.35 μm wide copper lines fabricated using damascene processing. Found that a low-temperature (50 °C) PVD seed layer resulted in a stronger plated copper texture, as compared to a high-temperature (150 °C) seed. This was due to the fact that the low-temperature seed resulted in a smoother surface from which plated grains could grow by interfacial energy minimization. The rougher surface created by the high-temperature seed did not allow this to happen as easily.

Performed segregation experiments in Al-1Cu blanket films. Found that Al-Cu precipitates formed primarily at grain-boundary triple junctions where the dislocation structures of the three intersecting boundaries did not line up well (U-lines). This created a divergence in diffusive flux at the junction, allowing a buildup of copper during annealing.

Publications

1. "Crystallographic Characterization of Interconnects by Orientation Mapping in the SEM," C.E. Kalnas, R.R. Keller, and D.P. Field, Characterization and Metrology for ULSI Technology: 1998 International Conference, eds. D.G. Seiler, A.C. Diebold, W.M. Bullis, T.J. Shaffner, R. McDonald, and E.J. Walters, American Institute of Physics, 849-853 (1998).

2. "Local Crystallography and Stress-Induced Voiding in Passivated AlSiCu Interconnects," R.R. Keller, C.E. Kalnas, and J.M. Phelps, submitted to Applied Physics Letters.

Project: THERMAL CONDUCTIVITY OF THIN FILMS

Principal Investigator: D. R. Smith

Objectives

Develop an apparatus for measuring, *by absolute steady-state methods*, thermal conductivity of thin metallic films of the dimensions typically used (about 1 μm) 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 the appropriate size scales. 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 absolute steady-state measurements with the results of transient methods to evaluate the precision and uncertainty of the transient methods. Ultimately produce modular thin-film test structures for measuring thermal conductivity; the test structures will be designed for evaluation and use by industry.

Technical Description

The use of, and dependence on, ICs by modern digital electronics, including computers and electronic communication technologies, underlies almost every technological advance of modern society. Great advantages in increased technical efficiency and reliability of ICs, as well as reduced unit cost, are being achieved by reducing the dimensional scales within the electronic package and on the chip. Typical width scales for elements within ICs continue to decrease and are now significantly less than 1 μm. The advantages of reduced length scales are necessarily accompanied by greater packing density of the individual devices, with attendant increased generation of heat. Unless this increased heat 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 effects 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 both metals and semiconductors, this work is not useful in predicting behavior 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. First, long measurement times are required by absolute steady-state techniques. Second, 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 data obtained by transient methods as to whether the conductivities obtained by their methods are reliable for their purposes.

The specimen technology developed for production of microelectromechanical systems (MEMS) that we are now using is a restricted technology adopted temporarily for matching some of the present practices of micromechanical engineering to the needs of this investigation. This preliminary technology being used for the first cycle of specimen manufacture is simple and relatively inexpensive, and adequate for the initial work. The specimen design is an adaptation of the classic Kohlrausch self-heating method for measuring thermal conductivity to a Kelvin bridge circuit for measuring electrical resistance. In this case the Kelvin circuit is a *literal* bridge of narrow thin film bridging the space between two electrical contact pads. The theory of the Kohlrausch method allows the thermal conductivity of the bridge material to be determined from the electrical parameters of the circuit together with the temperature distribution and electrical power dissipation. 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

Contacts have been made with Albert Feldman, of the Ceramics Division of NIST-Gaithersburg, who is developing a thermal-wave (transient) measurement system for thermal diffusivity of thin films. We are exploring the possibility of devising a common experimental structure that could be used to compare our measurement techniques.

We are pursuing formation of a new ASTM subcommittee on measurement of thermal transport properties of thin films.

Planned Outcome

The anticipated outcome during FY99 is that ongoing measurements will be completed and published, along with description of measurement apparatus. What is learned from these studies will form the basis for design of more sophisticated test devices. During the next phase, specimen design features needing improvement will be identified and carefully studied. This work will provide additional experimental data; additional measurement systems and modular standard devices for thermal measurements of thin films will be developed.

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

A compact vacuum chamber has been constructed which is compatible with the IR microscope used to measure the temperature distribution along the thin-film device. The vacuum chamber removes air between the bridge and the substrate, eliminating parasitic conductive heat loss from the bridge device. A complete rotation-translation-tilt mechanism with six degrees of freedom has been provided to allow the vacuum chamber, and contained specimen (actually four specimens per IC chip), to be properly oriented for observation by the IR microscope.

Some innovative designs have been developed that (if the manufacturing processes work as planned, delivering good adhesion between polysilicon layers) will 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 resistance thermometry (or, if desired, thin-film thermocouples) to be compared, and the more reliable method selected for practical applications.

Extensive thermal measurements on van der Pauw and Kelvin bridge structures have been completed, and data analysis systems necessary to translate the IR images to thermal conductivity are nearly complete. Calculation of magnitudes of potential sources of bias, such as radiative heat losses, are also nearing completion.

CERAMIC COATINGS

The Ceramic Coatings Program is a measurement and characterization effort which addresses the processing reproducibility and performance prediction of thermal-spray deposited ceramic coatings. The program addresses plasma-spray-deposited and physical-vapor-deposited ceramic thermal-barrier coatings used in aircraft and land-based turbines and diesel engines and wear-resistant coatings used in many applications. These materials are a significant portion of the one billion-dollar thermal-spray market. Collaborations have been established with industrial organizations including Pratt and Whitney, General Electric, Caterpillar, METCO, Praxair (an ATP awardee), as well as the Thermal Spray Laboratory at the State University of New York at Stony Brook, NASA Lewis Research Center and the Thermal Spray Laboratory at Sandia National Laboratory. The program includes collaboration with the National Mechanical Engineering Laboratory in Japan to examine functionally gradient materials. Collaborations are also underway with Bundesanstalt fur Materialforschung und -prüfung) (BAM) and Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR)–both in Germany–for the development of characterization techniques for thin, hard coatings.

Participants in the NIST program are located in the Ceramics and Materials Reliability Divisions, and NIST Center for Neutron Research of the Materials Science and Engineering Laboratory, as well as in the Chemical Science and Technology Laboratory.

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

- development of techniques for characterization of physical and chemical properties of stabilized zirconia 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 and PVD coatings, by use of the guarded-hot-plate technique suitable for incorporation in ASTM standards and by the pulsed laser heating technique, to provide a method for comparison with routine industrial techniques.

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

Research on chemical mapping of powders and microstructures is conducted in the Microanalysis Division of the Chemical Science and Technology Laboratory. Research on thermal properties is conducted in the Materials Reliability and Metallurgy Divisions. The NIST Center for Neutron Research participates in phase analysis and residual-stress measurement projects. A strong attribute of the coatings research program is the use of common materials for which complementary data can provide a more complete understanding of processing-microstructures-property 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 conductivities of ceramic coatings and to relate their thermal performance to the microstructure of the coatings. Measure the thermal conductivities of representative thermal-barrier coatings, substrate materials, and monolithics to determine bulk values and interfacial resistances for the coating systems. Evaluate microscopy and spectroscopy techniques for the various microstructural analyses. Observe the effects of varying processing parameters and processing techniques on microstructural features and bulk thermal conductivity. Model bulk thermal conductivity using microstructural information and processing parameters, which will result in a model that generates desired bulk thermal conductivity given appropriate processing parameters. Interact with the coatings industry to insure a supply of state-of-the-art samples. Develop measurement apparatus and techniques, appropriate reference materials and documentation to allow comparison with, and perhaps calibration of, measurement techniques used in industry.

Technical Description

Accurate knowledge of the thermal conductivities of thermal barrier coatings and their 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 conductivities of coatings on both the macro- and micro-scale, this project uses advanced techniques of electron microscopy to characterize the microstructure and microchemistry responsible for the bulk thermal performance of coating systems.

Standard reference materials are being developed that relate the thermal conductivities of various classes of ceramics and ceramic coatings to the thermal diffusivities. Industry commonly measures thermal diffusivity because it is a relatively fast, user-friendly measurement.

An absolute, steady-state measurement of thermal conductivity is used in order to obtain the thermal conductivity of coatings with the greatest possible accuracy and reliability. A modified guarded hot plate (GHP) has been constructed for these measurements. Infrared microscopy is used to monitor heat flow on the micron scale and to measure the thermal conductivities of coatings and the thermal resistances between interfaces. Heat flow in coatings will be modeled using input from infrared-microscope measurements combined with bulk thermal conductivity values obtained from the guarded hot plate.

Measurements of thermal conductivity of six new ceramic systems, three thermal barrier coatings and three monolithics, were completed this year using the guarded hot plate. Two plasma-sprayed thermal-barrier coatings and 10 physical-vapor-deposited thermal-barrier coatings were measured using the infrared-microscope system.

Special facilities used include an infrared microscope system capable of a spatial resolution of 5 micrometers from room temperature to 500 K and the world's only 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 functionally graded materials (FGMs).

NAL/KRC (Japan) — Collaborating with A. Kumakawa to evaluate microstructure, microchemical composition, and thermal conductivities of FGMs prepared by Nippon Steel.

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

Caterpillar — Collaborating with Brad Beardsley on the measurement of the thermal conductivities of FGMs and monolithic coatings comparing GHP measurements with the laser-flash method.

University of Virginia — Collaborating with D. Hass on development of electron-beam directed-vapor-deposition (EB-DVD) thermal-barrier coatings.

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 conductivities 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

Tested Pyroceram 9606 as an apparatus calibration after redesigning the bottom heater plate of the GHP and doing routine re-wiring after 5 years of nearly continuous operation. Began measurements of both 5.3 mass % and 9.1 mass % yttria-stabilized zirconias. These materials are predominantly tetragonal and cubic, respectively, and are candidates for high-temperature standard reference materials (SRMs) of thermal conductivity. Preliminary measurements show that both materials provide results with very good repeatablity. Due to the difference in phase composition, the thermal conductivities of these two materials are different. This should be significant and useful for industry, as the use of different processing parameters results in different phase compositions of ceramic thermal-barrier coatings. We measured electron-beam physical-vapor-deposition (EB-PVD) coatings from Pratt & Whitney after polishing of the coatings. Three different thicknesses were measured, with two specimens of each thickness measured to determine repeatability of the EB-PVD process and to ensure reliability of the thermal conductivity results. We measured two functionally graded coatings from NAL/KRC, Japan. The collaboration with the Japanese includes thermal-shock experiments to determine how increases in microcrack density affect thermal conductivity. Also, we measured two thicknesses of an EB-PVD coating with a unique microstructure from DLR in Germany.

The infrared microscope system for doing comparative, steady-state measurements of thermal conductivity of thermal barrier coatings was validated and we have measured 10 coatings this year. The validation was done using a plasma-sprayed coating measured previously in the guarded hot plate. Numerous EB-DVD coatings from the University of Virginia were measured using this system. The coating morphology is varied by varying process parameters to generate coatings with very low thermal conductivity.

We have determined the microstructure of coatings using electron, atomic-force, and magnetic-force microscopy techniques. Energy- and wavelength-dispersive spectroscopies were used to determine microchemical content and distribution of chemical species, and x-ray diffraction was used to determine phase content in monolithic ceramics and in a ceramic coating.

Development of an infrared-microscopy system to measure interfacial thermal resistance at ceramic/metal boundaries is continuing. These boundaries exhibit a significant thermal barrier, and have been shown to be the location of primary failure in thermal-barrier coatings. A collaboration is planned with F. Schmitz of the Technical University in Aachen.

Publications

1. "Absolute, Steady-State Measurement of the Thermal Conductivity of Magnesium Oxide," A.J. Slifka, B.J. Filla, and J.M. Phelps, *Journal of Research of the National Institute of Standards and Technology,* Vol.103, 4, 357-363 (July-August, 1998).

2. "Thermal Conductivity Measurements of Pyroceram Using a High-Temperature Guarded-Hot-Plate," B. J. Filla and A. J. Slifka, *Thermal Conductivity,* Vol.24., 85-96 (1998).

3. "Thermal Conductivity of a Zirconia Thermal Barrier Coating," A.J. Slifka, B.J. Filla, J.M. Phelps, G. Bancke, and C.C. Berndt, *Journal of Thermal Spray Technology,* ASTM, Vol.7, 1, 43-46 (March 1998).

4. "A Comparative, Steady-State Apparatus for Measurement of Thermal Conductivity of Thermal Barier Coatings," A.J. Slifka, submitted to *Surface and Coatings Technology.*

MAGNETIC MATERIALS

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

In the present trend to make devices smaller, thereby reducing weight or increasing storage density, new magnetic materials are constantly being developed. One critical need for implementation of these materials is the development of the measurement science needed for their characterization, 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, analyses 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 neutron diffraction facilities at NCNR, or the magneto-optic indicator film apparatus for observation of motion of magnetic domains. 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 address 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 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 and 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

- 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 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 capability for microstructural analysis not otherwise available, either in terms of detection ability or simplicity of application. Evaluate application of magnetic force microscopy (MFM), atomic force microscopy (AFM), and related measurements to microscale magnetic evaluations focusing on both ion-implanted engineered surfaces and evaluation of electronic interconnects. Investigate creation of a magnetic mapping device using commercial magnetoresistive magnetic recording heads. 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. Apply traditional magnetic measurements such as vibrating sample magnetometry (VSM), Hall-effect field detection, and superconducting quantum interference device (SQUID) magnetometry to unique materials evaluation problems, such as current work on assessment of irradiation-induced damage in reactor steels and new methods of evaluating weld metal standards.

Technical Description

Our work in FY98 continues to cover a wide range of experimental work in different aspects of materials science. Four significant projects were carried out. The first, which is ongoing, 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. The second effort, also ongoing, 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 assessing line quality and the presence of defects, a problem that is becoming more critical as the line sizes head to the submicron range. The third effort, now essentially complete, is associated with the embrittlement of steels used in nuclear reactor pressure vessels. 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. The final project, also continuing, is in the development of new methods for measuring ferrite content and structure in weld ferrite standards.

Ion implantation and ion-beam-assisted deposition techniques are methods frequently used to create engineered surfaces to improve the chemical or mechanical properties of metallic materials. They alter the properties of the substrate surface only to micrometer depths. Measurement of the condition of the surface and uniformity of the preparation is often beyond the capability of traditional characterization methods. 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, chemical resistance, hardness, or friction coefficient.

The MRM effort applies microscopic magnetoresistive field sensors to mapping of fields from regions in the size range from 1 to 100 μm. 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 application in manufacturing environments. They are prepared by conventional deposition and lithographic techniques; packaging methods and control electronics are well in hand for many (but not all) applications. In dc field-sensing applications the temperature sensitivity of the probes is a serious issue and much of the work in FY98 was devoted to this problem. Evaluation of magnetic mapping of operating high-frequency microcircuits continues to be investigated. The small size of these sensors makes them ideal, not only for the applications mentioned above, but also for detection of flaws in magnetic media and other structures associated with data storage as well as for more general magnetic microstructure analysis.

Reactor steel measurements were completed this year. The work concentrated on measurement of hysteresis loops for a variety of steels that we hoped might serve as surrogates for the real thing (which is somewhat radioactive). For this measurement, we use a modified 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. There was reason to suspect that the coercivity should be a measure of the precipitates that cause the embrittlement. In addition a prototype field device to determine a loop by looking at the underlying steel through the thick stainless-steel coating of an actual reactor was developed.

Weld ferrite standards are small blocks of alloy prepared so as to model as-welded metal. These secondary standards are used for field calibration of magnetic instrumentation used to determine ferrite content in stainless-steel weld metals. The most common standard instrument is one in which a magnet of known strength is pulled from the surface of the sample, with the required pulling force being related to the ferrite content. In discussions at NIST it was felt that it would be desirable to have a magnetic measurement standard for this application that was more closely related to fundamental electrical standards. To this end a number of preliminary magnetic measurement experiments were designed using standards which were on hand as well as some Russian material proposed for new standard sets. The measurements involve using the MFM to view the actual ferrite structure within the nonmagnetic austenite matrix and the VSM to investigate the hysteresis loop. Also, we are pursuing design of a prototype standard test device based on electrical excitation and field measurement.

The scanning probe microscope (SPM), housed under this project and used in magnetic-force mode in the ion implantation and weld metal work, continues to be applied to many other problems, primarily in AFM mode in evaluation of thin-film interconnect structures. Application to scanning-probe potentiometry of thin line structures is being developed in collaboration with EEEL and we have been exploring its application (in electric-force mode) to evaluation of domain structures and switching in ferroelectric wireless devices and materials. It was also used as a test bed in initial experiments on sub-micrometer thin-film tensile testing.

External Collaborations

Empire Hard Chrome — a producer of implanted materials in the ATP project *Plasma-Based Processing of Lightweight Materials for Motor-Vehicle Components and Manufacturing Applications* provided samples of chrome plated steel.

Colorado State University and Colorado School of Mines — our collaboration on magnetic measurements of ion-implanted stainless steel continues.

Planned Outcome

This project is anticipated to result in a wide-ranging micromagnetic sensing capability for the Division. The microscale measurement technology (MFM, AFM, MRM, SQUID, etc.) 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 (VSM, B-H loop generator, etc.) also will be employed in evaluation of large samples to determine the effect of microstructure on material magnetic properties. Weld-metal ferrite studies should ultimately result in a new measurement method for calibrating secondary standards.

Accomplishments

Ion implantation work continues to rely on the MFM as the main measurement tool. Last year we measured two sets of plasma-source ion-implanted (PSII) samples (H13 steel and 304SS with various combinations of Cr-plated, unplated, or implanted). Neither set was interesting in its magnetic signature. This year measurements were made on a new set of 316SS PSII samples. Again, the magnetic signature of the bare steel shows the distorted magnetics typical of mechanically polished surfaces, while the plated samples show no magnetism. The chrome coating is thick enough that no significant ion concentration makes it through to the ferromagnetic material below. This view is supported by a calculation using the TRIM code which gives a gaussian distribution of implanted ions centered on a depth of about 64 nm with a half-width of about 27 nm.

For engineered surfaces created with a different set of implantation parameters, however, the situation is quite different as we demonstrated with our FY97 work on 300 series stainless steels. There, we showed excellent magnetic signatures in 304 and 316 samples implanted with lower-energy N ions (~600-700 eV) at elevated temperatures and to doses significantly higher (~10^{20}) than those used in PSII. These parameters are typical of ones commonly used in other surface modification work. Based on this work we have pursued a study of the development of the thin magnetic layer responsible for the enhanced wear and chemical resistance. A total of 14 samples were prepared and 12 of them implanted for times varying from 15 seconds to 240 minutes. The development of the implanted layer and its associated magnetic signature is clearly seen in Fig. 1. It is obvious from these MFM images that the crystal structure of the stainless steel has a significant impact on the properties of the implanted layer: at the least, the magnetic domain structure of the layer is very sensitive in this regard. A new study is now underway to relate the domain structure to the grain orientations as determined by orientation-imaging microscopy.

The MRM system work has made some progress. Our understanding of the mechanical and electrical behavior of the recording heads that serve as detectors in this early work is increasing so that we are destroying them at a decreasing rate. A lengthy series of experiments were carried out to understand the thermal, electrical and magnetic behavior interactions of the heads and to quantify the ac-bias effect discovered last year, when we predicted and then demonstrated the temperature independence of the second-harmonic signal. A paper describing and explaining the effect and its implications is nearing completion.

Reactor steel samples from twelve different materials were measured with the VSM and the data analyzed to give saturation magnetization and coercivity. Coercivity shows a strong variation with material type and heat treatment, but the coercivity data does not appear to track with hardness for these materials. This does not necessarily eliminate H_c as a test, since it is one of the few parameters that can be measured through a nonmagnetic metal cladding. Little effect is seen on the saturation magnetization, as would be expected. The hysteresis curves contain all the data necessary to evaluate various permeabilities and the core loss, but the required analysis is complex and was not done. There is a chance that irradiated samples could be measured by this technique or by SQUID magnetometry. To demonstrate detection through the metal cladding, a prototype Hall detector was built. With it, we demonstrated that it detects hysteretic magnetic properties of the underlying steel layer and that we could approach saturation with this simple device.

Sets of weld ferrite standards and a large piece of Russian centrifugally cast steel were mapped for magnetic structure using a conventional commercial Hall-effect gaussmeter for a rough determination of the surface normal fields. The fields showed a roughly uniform variation with ferrite content, but with some significant deviations. Samples were then cut from select regions of the cast steel piece and measured using the VSM. The resulting hysteresis loops showed significant variations in both coercivity and saturation magnetization across the cast piece, which could indicate variation in the ferrite structure or composition (coercivity) and ferrite content (saturation magnetization) in the various regions. A first try at an electrical measuring system was made and this prototype, while much larger than what we will ultimately need, seems to be giving consistent data on large samples. Application of the MFM in this experiment shows it to be superb for imaging the magnetic ferrite regions. Two blocks, one with low and the other with high ferrite content were prepared for MFM analysis. Surfaces must be carefully prepared to give a good polish without excessive surface damage. The results show the magnetic ferrite regions and their associated domains in a most dramatic manner. A catalog of magnetic images for a complete set of standard blocks is planned.

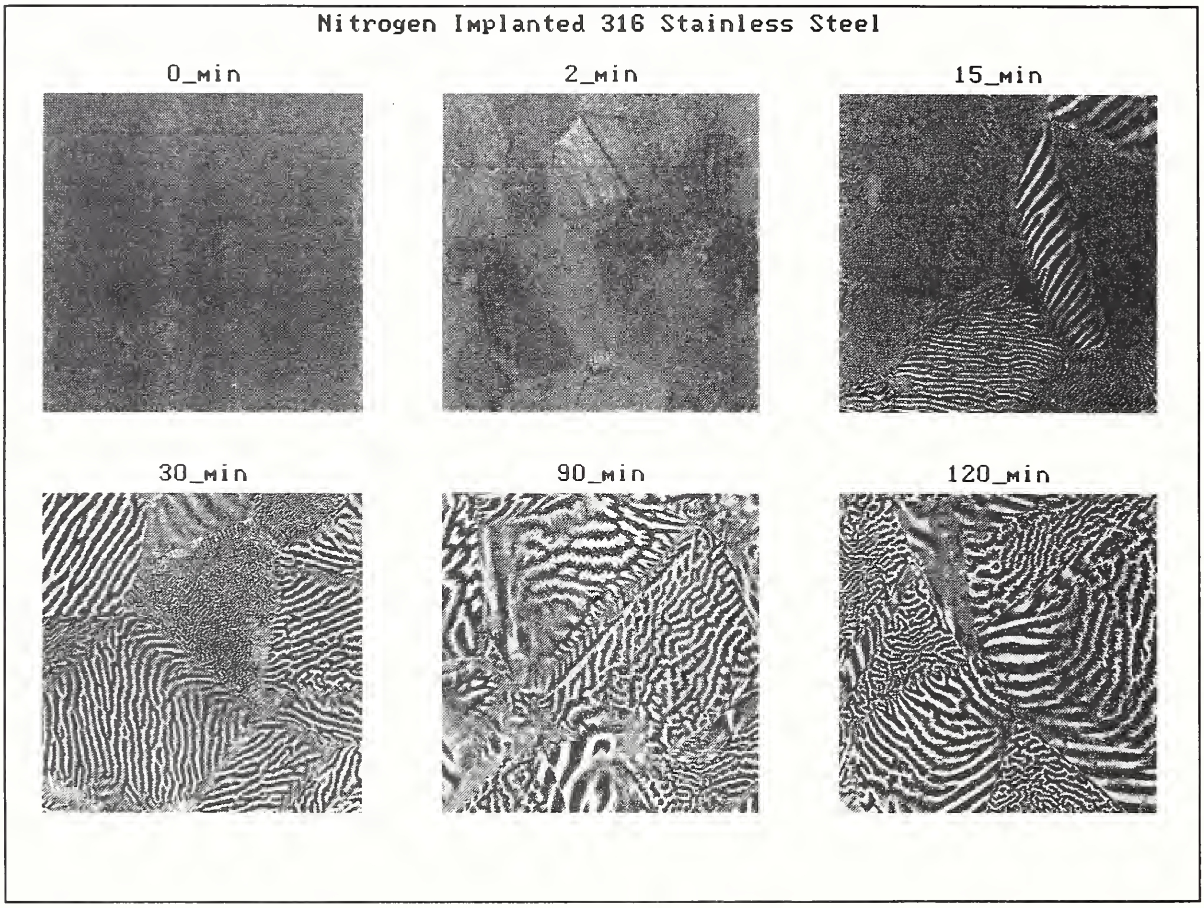

Fig. 1. **Nitrogen Implanted 316 Stainless Steel:** Magnetic scan (MFM) data showing development of the magnetic structure of the very thin layer (~1 μm thick) that forms on the surface of a bulk sample of nonmagnetic stainless steel as it is implanted with nitrogen. The scan size is 50 μm square for all images. The nitrogen expands the austenite lattice to a structure which is ferromagnetic, as shown by the development of the striped domains. Some time is required to reach the maximum concentration of nitrogen in the surface layer. The domain-size variation seen on the grains probably results from the different intrinsic magnetic properties of the expanded lattice in each of these crystal faces.

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 to calibrate measurement systems and to assure the long-term adequacy of measurement-assurance programs. The aim is to assist industry, science, and academia to achieve the level of product conformance and measurement quality required for national and international commerce and trade.

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

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

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

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

Project: CHARPY IMPACT TESTING

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

Technical Objectives

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

- Participate in the activity in ISO Committee TC 164, so our specimens and procedures remain compatible with the associated international standards and other regional standards.

Technical Description

The Charpy impact test uses a swinging hammer to assess the resistance of a material to brittle fracture. The absorbed energy is measured from a calibrated scale, encoder, or an instrumented striker. 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 Measurement Services, which oversees the administrative aspects of the program, and the Materials Reliability Division, which handles the technical and certification aspects.

NIST provides highly characterized Charpy-impact 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 1000 customers for this service in FY98, a significant increase from the 800 that we have averaged in 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 900 specimens necessary to confirm that 11 new lots of reference specimens were suitable to go into the SRM inventory.

In cooperation with the NIST Standard Reference Materials Program and the American Society for Testing and Materials, we had hosted a workshop in Norfolk, Virginia during November 1995. This workshop, Materials and Heat Treatments for ASTM Charpy V-notch Verification Specimens, sought new ideas to improve the verification specimens offered through the Standard Reference Materials Program. The participants identified several multi-year research projects that might improve the performance of the NIST reference materials. Since the workshop, we have been coordinating the machining and heat-treating tasks among the volunteer organizations: Timken, Teledyne-Vasco, Sure Tool, and Thomas Shearer Inc. In addition, Bob Gassner has performed some ultrasonic measurements of the round-robin specimens. These ultrasonic measurements are being cross-correlated to hardness and impact-energy measurements for a better understanding of the microstructural uniformity of the specimens by heat treatment. The final specimens in the research projects are due in early FY-99, and we plan to produce a report that summarizes all the work. A new workshop is tentatively scheduled for FY-00.

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 plastic specimens (of compositions that are commonly tested on these machines) and have presented a report on the evaluation of the round robin to the task group. We consider that this data will be used to produce plastic SRMs, as needed. We are presently participating in a round robin evaluation of sub-size metal specimens.

Chris McCowan continues as the Chairman of ASTM Subcommittee E28-07 on impact testing, and Dan Vigliotti continues as the Chairman of the Task Group that oversees Standard E23, the main standard for Charpy 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 have received 25 abstracts, 5 from authors in our Division.

Publications

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

Project: FERRITE IN STAINLESS STEEL WELDS

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

Technical 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 *Standard Procedures for Calibrating Magnetic Instruments to Measure the Delta Ferrite Content of Austenitic and Duplex Austenitic-Ferritic Stainless Steel Weld Metal.*

The A4.2 standard specifies both primary and secondary calibration procedures for the instruments used to measure ferrite in stainless-steel welds. Primary calibration is based on the NIST coating thickness standards, such as SRMs 1323 and 1363, while secondary calibration is based on certified samples of stainless steel. 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.

The secondary standards are arranged in two sets, a lower-range set with 8 specimens that are distributed over the range of 0 to 30 FN, and a higher-range set of 8 specimens that are distributed over the range of 30 to 100 FN.

In FY98 we completed our measurements and assigned FN values to the specimens in 30 sets of secondary standards. To accomplish this we spent a considerable amount of time evaluating the variables in the procedures and the instruments used to make the FN measurements. From the results of these evaluations we developed an internal calibration procedure for the assignment of certified FN values to the standards. In addition, these results showed several ways in which the procedures in Standard AWS 4.2 might be improved.

Approximately 25,000 individual measurements were made to certify the 30 sets. This data has been analyzed and used to quantify the variation of the secondary standards. The preliminary data was presented to Commission II of the International Institute of Welding (IIW) on July 17, 1997. A full report will be made in April, 1998 at the Annual Welding Convention (submitted for publication in the Welding Journal).

In FY99 we plan to procure additional specimens from Russia for certification as secondary FN standards, and pursue plans 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 occur in later years under WCF.

Planned Outcome

Replace secondary FN standard in inventory as they are sold. Develop a fundamental calibration strategy, to replace the current qualitative procedure.

Accomplishments

1. Completed FN measurements on 30 sets of secondary FN standards and placed standards in SRM inventory.

2. Prepared the certificate for the secondary standards (now in review).

3. Prepared a report for SRM files that documents our measurement procedures, summarizes the results of measurements on the secondary standards, and provides the raw measurement data for archival purposes.

Publications

1. "Stainless Steel FN Secondary Standards: Gage Calibration and Assignment of Values," C.N. McCowan, T.A. Siewert, and D.P. Vigliotti, International Institute of Welding, Document IIC-128-98, September 1998, available from Secretariat of the American Council of the IIW, American Welding Society, Miami, Florida.

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