MATERIALS RELIABILITY

1999 PROGRAMS AND ACCOMPLISHMENTS

MATERIALS SCIENCE AND ENGINEERING LABORATORY

NISTIR 6434

UNITED STATES DEPARTMENT OF COMMERCE

TECHNOLOGY ADMINISTRATION

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

Reliability of Materials: a Critical Issue Throughout the Nation's Infrastructure

The Materials Reliability Division addresses reliability issues across the full range of the national infrastructure, from our previous focus on bridges, railroads, nuclear reactors, and pipelines, to the current focus on materials and related packaging issues in microelectronics, photonics, and other micro-scale structures. In each arena, the Division determines critical materials properties of structures, develops advanced measurement and modeling methods for evaluating failure modes and mechanisms, investigates the basic physics and materials science of failure, and works with appropriate industries to develop solutions leading to enhanced reliability of their products.

UNITED STATES DEPARTMENT OF COMMERCE
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TECHNOLOGY ADMINISTRATION
Dr. Cheryl L. Shavers, Under Secretary of Commerce for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
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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 that enable the producers and users of materials to improve the quality and reliability of their products. Measurement technologies, and the associated materials science base, are developed for materials evaluation to assure reliable performance, for nondestructive evaluation to assure quality of finished materials and products, and for process control to improve the quality and consistency of materials. Within these broad areas of measurement technology, the Division focused its resources in FY1999 on the following three research themes:

Microscale Measurements: These projects develop measurement techniques for evaluating the mechanical, thermal, electrical and magnetic behavior of thin films and coatings at appropriate size scales. The Division had five projects to develop measurements for evaluating the reliability of electronic interconnections. In fiscal year 1999, we continued our interactions with the electronics industry, measuring mechanical properties of thin-film interconnects on increasingly smaller scales and measuring strains in electronic packages. This year also saw the first data on absolute thermal conductivity of thin-films structures and application of infrared microscopy to package analysis. In addition, crystallographic studies with electron microscopy to elucidate the mechanisms of stress voiding were pursued and an electromigration research program initiated.

Microstructure Sensing: In this effort, ultrasonic measurements are applied to characterization of the internal geometry of materials, such as defects, microstructures and lattice distortions. In July of 1999, the Nuclear Regulatory Commission sponsored a workshop in Boulder to focus attention on the international effort to develop nondestructive techniques to characterize embrittlement induced in reactor pressure vessels by long exposure to neutron irradiation. The Division played a major role in the discussions by presenting the results of measurements on 8 of the 11 potentially viable techniques being considered. Also, improvements in our acoustic resonance spectroscopy capabilities enabled us to characterize new materials for microelectronic components, such as crystal oscillators, filters and dielectric resonators.

Process Sensing and Modeling: The projects in this area develop on-line sensors for measuring the materials characteristics and/or processing conditions needed for real-time process control. Fiscal year 1999 was a period of growth, as we expanded our activities into new directions. In welding, our collaboration with the Intelligent Systems Division in Gaithersburg resulted in packaging of our existing weld sensing technology into standalone systems, that were used to demonstrate remote sensing of welding problems over the world-wide web. In high-temperature deformation, the techniques developed during our studies of steels were applied to a study of the formability of aluminum, in a joint project with the Metallurgy Division. In high-energy x-ray diffraction, the techniques developed to monitor the in-situ solidification of turbine blades were applied to the construction of a facility to measure stress and texture in thick structures.

SUMMARIES OF SELECTED ACCOMPLISHMENTS FROM FY1999 RESEARCH

Materials Reliability Series (NIST Technical Notes): Developed at the suggestion of the Assessment Panel in 1998, these reports cover significant research accomplishments of the Division. In FY1999 two new reports were published covering multiyear efforts in modeling constitutive behavior of steels under hot rolling conditions and structure-property relationships of these steels. These join the five reports from last year on thin-film mechanical properties testing, electron-beam moiré microstrain detection (two reports), x-ray monitoring of solidification, and a report to the Nuclear Regulatory Commission on embrittlement detection in reactor steels.

Mechanical Testing of Microelectronic Structures: We continue our interaction with Motorola (AZ) and with Hutchinson Technologies on the measurement of thin film mechanical properties. Work with Motorola (TX) using electron beam moiré techniques on problematic packages showed the source of the failure and led the way to a solution.

Infrared Microscopy of Electronic Packages: In a new project, the infrared microscope was used in conjunction with electron-beam moiré to evaluate failure mechanisms and locations in simulated package components involving organic electronic materials. Failures induced by repeated thermal cycling have been successfully tracked by the two methods.

Test Structure for Interconnect Reliability: Fabrication was completed of a new NIST interconnect test chip (NIST 36) with special structures, such as notched lines, for assessing stress voiding problems as well as the traditional electromigration test structures. The work is done in collaboration with the Semiconductor Electronics Division. Test chips from Sematech and Lucent with very fine damascene copper structures have been investigated by advaneed electron microscopy techniques to determine crystallographic strains in submicrometer grains.

NIST Industrial Fellow: Dr. David Read began a 10 month Industrial Fellowship to the Motorola Advanced Technology Center laboratories in Schaumburg, Illinois where he will pursue research in reliability analysis of microelectronics and gain familiarity with industrial applications of reliability assessment techniques while working in the Design Reliability and Simulation Group. This year he also received the *Measurement Science and Technology* Best Paper Award 1998 for his paper "Young's modulus of thin films by speckle interferometry." The journal is published by the Institute of Physics (UK) and is a leading journal in the instrumentation field.

Nonlinear Acoustics: Techniques for measuring the nonlinear elastic coefficient that controls harmonic generation were developed further by adding a piezoelectric detection technique, by establishing the procedure for correcting second-harmonic measurements for ultrasonic attenuation and by increasing the efficiency of acoustic wave generation through improved comb transducer design and development of a narrowband laser-based technique for excitation of high-amplitude Rayleigh and Lamb waves. These improvements were used to measure the effect of nanometer-sized precipitates on the nonlinear elastic coefficients of steels similar to those used in reactor pressure vessels and to measure nonlinear elastic coefficients outside the laboratory in a field environment.

Steel Embrittlement: Techniques for measuring a magnetostrictive coefficient, accurate shear wave velocities and the transverse incremental permeability on radioactive samples of reactor pressure vessel steels in a hot cell were developed and used to establish empirical relationships between these physical properties and damage by different fluences of neutron irradiation in A533B reactor pressure vessel steel. These three physical properties, as well as several others, were shown to correlate with the hardness of a steel strengthened by formation of the nanometer sized copper-rich precipitates thought to be responsible for embrittlement of reactor steels.

Elastic Coefficients: The Acoustic Resonance Spectroscopy technique was extended by developing the frequency-to-elastic-modulus inversion method to include materials with trigonal symmetry and materials that are piezoelectric. It was then used to measure modulus and damping in: langasite and quartz (trigonal and piezoelectric); potassium dihydrogen phosphate (a low-temperature phase change crystal); metal matrix composites (verification of a general model for composite properties); and layered media (one-dimensional ordered structures).

New Sensors: An automated electromagnetic acoustic transducer (EMAT) probe with a portable instrumentation package was developed and calibrated for measuring plane-stress distributions in metal-plate structural elements such as those found in bridges, buildings, pipelines and earth-moving equipment. It can be viewed as a mobile, noncontacting "strain gage" for measuring the effects of applied loads and residual stresses on structures in the field. Two sensors for inspecting buried gas pipelines from a robot moving inside the pipe are being tested under simulated pipeline conditions. One is a gas coupled ultrasonic transducer for measuring the thickness of the pipeline wall, and the other is an EMAT attached to a conventional magnetic pipeline-inspection vehicle to detect loose coating during the magnetic inspection of a pipeline.

X-ray Patent: On August 12, 1999, Digiray Corporation received the exclusive license to a joint NIST–Digiray invention, "Method and Apparatus for Diffraction Measurement Using a Scanning X-ray Source." Digiray will take over the responsibility for developing the method into commercial products. The NIST inventors are Bill Dubé, Dale Fitting, and Tom Siewert.

Impact Machine Symposium: We helped to organize an international symposium *Pendulum Impact Machines: Procedures and Specimens for Verification*, that was held in conjunction with the May 1999 meeting of ASTM Committee E28 in Seattle, Washington. The 26 papers presented at the symposium are now being reviewed for inclusion in an ASTM Special Technical Publication. NIST contributed to four of the papers, and is assisting with the proceedings.

Stainless Steel Welding Meeting: We hosted the Spring 1999 meeting of Welding Research Council Subcommittee on Welding of Stainless Steel. In conjunction with this meeting, we were able to organize a half-day round robin of leading experts on ferrite measurement in which we looked for the sources of systematic errors in the ferrite number (FN) calibration procedures.

Computers in Welding Conference: We helped to organize the Ninth International Conference on Computer Technology in Welding. NIST staff participated by giving presentations, tutorials, and serving as session chairs. The 58 presentations gave a good summary of the status of the use of modeling, sensing, and control in welding. The proceedings will be published as a NIST Special Publication.

DIVISION CHIEF'S COMMENTARY

The Materials Reliability Division underwent substantial changes in FY1999. Funding reductions resulted in a significant reduction in staff that was effective in June and Harry McHenry, the Division Chief for more than 12 years, retired in July. Furthermore, the focus of the Division has been changing, being directed more into the area of electronic materials research. This has resulted in many staff members working to apply their expertise to new types of materials and problems on a significantly different size scale. With the beginning of the new fiscal year, we are hoping to achieve a more stable environment with less uncertainty for the future.

Fred Fickett
November 7, 1999

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 early 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 advaneed 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 20 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; and electromigration and stress voiding. These projects are conducted in concert with partners from industrial consortia, individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs within government and industry, including the National Semiconductor Metrology Program (NSMP)[3]. The NSMP is a national resource responsible for the development and dissemination of new semiconductor measurement technology.

More information about this program, and other NIST activities in electronic packaging, interconnection and assembly 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.

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[1]*National Technology Roadmap for Semiconductors*, Semiconductor Industry Association, San Jose, California, 1994, 1997; *National Technology Roadmap for Electronic Interconnections*, IPC, Lincolnwood, IL, 1995, 1997; *National Electronics Manufacturing Technology Roadmap*, National Electronics Manufacturing Initiative, Inc., Herndon, Virginia, 1996, 1998.

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

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

Project: MECHANICAL BEHAVIOR OF THIN FILMS

Principal Investigator: D.T. Read

Objectives

The objectives of this project are to develop experimental techniques to: measure the mechanical properties of thin films, including basic tensile properties, fatigue, and fracture resistance, in specimens fabricated and sized like materials used in actual commercial devices; relate thin-film mechanical behavior to microstructure; and extend test techniques from their present level (1 μm thick, and 10 μm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths of 0.1 μm to 1 μm).

Technical Description

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

The main track in the technical approach continues to be to develop tensile test techniques for thin-films 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 framed tensile specimen is one answer. This specimen consists of a frame that carries a tensile coupon. The frame is conveniently handled, for attachment to the grips of the testing device and for alignment. Just before the test, the frame is carefully cut. This strategy can be applied where photolithography and etching techniques allow the definition of the frame geometry. Typically, the frame is patterned in the substrate. The main challenge in specimen fabrication is chemically removing the substrate in the interior of the frame, and especially from underneath the specimen, without damaging the specimen. The classic example is aluminum and copper films on silicon substrates. Most popular etchants for silicon, such as potassium hydroxide, dissolve aluminum rapidly. We have used hydrazine hydrate for etching away silicon under aluminum tensile coupons. The main challenge in conducting the test is cutting the frame without damaging the specimen. With silicon-framed specimens, we have had good results using a dental drill for this purpose.

This year we have tested a series of heat-treated electrodeposited gold films with a thickness of about 5 μm, on copper foil substrates, using the framed tensile specimen approach. These specimens were supplied by Hutchinson Technology Inc. of Northfield, Minnesota. This materials system is used in industry for flexible interconnects. This set of tests brought several firsts for our lab: first gold thin films tested; first series of heat-treated specimens tested; first specimens on copper foil, rather than silicon, frames; and first industrial collaborator in Minnesota. The company made the specimens, and so they handled the challenge of etching the substrate out from under the gold tensile coupons with techniques normally used in the production of interconnects using this materials system. In our attempts to cut the frame, the dental drill grabbed and pulled the copper foil, destroying the specimen; so a new method was developed for cutting the frames. An electric spark drawn from a tungsten tip was used. The tip was repeatedly contacted to the frame, and each spark melted out about 0.1 μm to 0.5 μm of the copper-foil frame. This worked pretty well, the only problem being that the tip occasionally welded to the specimen frame. Digital image correlation was used, rather than speckle interferometry, to obtain displacement from the images obtained during the test. This is experimentally a little more convenient, and allows examination of the specimen behavior as the test goes on.

It is questionable whether the silicon-framed tensile specimen will work for specimens with widths below 10 μm. The specimens may be too delicate. Building on the “skyhook” approach discussed in last year's report, we have developed this year a new conceptual approach to these tests, which we call the force-probe tensile test. The specimen film is deposited on silicon and patterned into a tensile strip, 10 μm wide by 200 μm long at present, surrounded by a frame of the specimen film. The tensile strip is connected to this frame at one end, but the other ends in a tab about 150 μm wide with a 50 μm hole in it. The tab is tethered to the frame with thin strips of the specimen film. The silicon is etched away from under the specimen, and from the surrounding area within the frame, to a depth of about 50 μm, using xenon difluoride. The force probe is similar to a micromanipulator-mounted needle probe used to make electrical contact to thin-film conductors on silicon wafers, with three important differences, the probe tip is reshaped to form a hook, to serve as the “skyhook;” a force sensor is added between the probe tip holder and the micromanipulator, and the micromanipulator is motorized with very accurate positioners on all three axes. Tensile testing is carried out under a microscope by first breaking the tethers to free the specimen and then engaging the specimen tab end with the force-probe's hook and pulling until the specimen fails. The displacement comes from the micromanipulator motor, and is controlled by a computer. The force is measured by the force sensor. The displacement is measured by digital image correlation of a series of typically 100 images obtained during the test.

This year, progress items included development, fabrication, and testing of a set of apparatus for force-probe tensile tests, including the force sensor, a micromanipulator with inchworm-style piezoelectric motors on three axes, and a force pendulum set-up to calibrate the force sensor. In initial tests of aluminum films under the optical microscope, the strength of 1 μm thick films, made in the same way as previously tested films, was similar to the previous results. The forces involved were much smaller, about 2 mg force. However these tests revealed a surprise: the elongation to failure of these 10 μm wide by 200 μm long films was up to 20 %, while that of 200 μm wide by 700 μm long specimens has consistently been around 1 %.

The force-probe rig was adapted to fit inside the chamber of a scanning electron microscope (SEM), and tensile testing was demonstrated. The imaging system needs further development to collect a sufficient number of images for quasi-continuous elongation measurements. The present specimens are near the largest that could be tested in a SEM using the present approach. Magnifications of only a few hundred were needed. But with the high magnification and depth of field of the SEM now available for application in tensile testing, we intend to pursue this technique at aggressively smaller size scales. This will require specimens fabricated by sources outside NIST, such as MOSIS. The force sensor will have to be redesigned, and possibly rethought, for specimens an order of magnitude or more smaller than the present set.

The force-probe approach has scored some successes in experimental tests of some materials that could not be successfully tested by the silicon-frame approach: SiO_2 and intermetallic Al_3Ti. Both of these materials are quite brittle. In both cases the specimens on the wafer failed during the process of etching the silicon away using hydrazine hydrate. When tensile tests were attempted with surviving specimens, the specimens broke during the cutting of the silicon frame. In the new approach, xenon difluoride can be used for the silicon etch, and it is better than hydrazine hytrate on two counts: first, it is a gas, rather than a liquid; second, it is less aggressive than hydrazine hydrate. And, the smaller specimens seem more robust. Some damaged and broken specimens were seen on the wafers after fabrication, but many were intact. A few successful experimental tests were achieved.

External Collaborations

Motorola Advanced Interconnect Systems Laboratory, Tempe, AZ-part of the Motorola Semiconductor Products Sector, has built a microtensile tester based on the NIST design. D.T. Read visited and provided revised test control software. They had a troublesome set of specimens that were very delicate. D.T. Read tested some for comparison with the Motorola results. A written report was provided to the industry collaborator. This set of specimens, and other discussions elsewhere in this report, highlight a fact of life in tensile testing: the marginal materials are the most difficult to test. And yet, these are the ones where testing is most needed. A good rule in such cases would be that if a material is too fragile to test, it is too fragile to use. As yet, the silicon-frame tensile specimen technique is not sufficiently well accepted to make such a rule practical.

Hutchinson Technology, Northfield Minnesota—testing of heat-treated electrodeposited gold thin films. Described in detail above.

Intel, Santa Clara— suggested a few years ago that we test Al_3Ti. Our inability to test this material using the silicon-frame tensile specimen helped to drive us to develop the force-probe technique. As noted above, it now appears that we will be able to test this material. There are complications in the creation of this material. It is made by heating a multilayer Al/Ti film to 450 °C. But if the films are in contact with silicon, it will diffuse into the film, creating an unwanted ternary alloy. Furthermore, hot Ti is very reactive with its ambient atmosphere, so the AlTi multilayer has to be made with Al on the outside everywhere.

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 present technique, framed tensile specimens tested in the piezo-actuated microtensile tester, 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 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 has started an ASTM activity on mechanical behavior of micromechanical structures and materials. An abstract is in work with Hutchinson Technologies to present the work on electrodeposited gold at a symposium sponsored by this group. This will provide an opportunity to assess the level of interest in creating a formal standard for this test method.

Accomplishments

The force-probe approach to tensile testing of thin films was developed and demonstrated in both the optical and the scanning electron microscopes.

A series of electrodeposited gold tensile specimens with different heat treatments, produced by an industry collaborator, was tested using the framed tensile specimen approach.

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

Publication

D. Josell, D. van Heerden, D. Schectman, and D.T. Read, Mechanical properties of multilayer materials, *NanoStructured Materials* **12**, 405-408 (1999).

Project: EXPERIMENTAL MICROMECHANICS BY ELECTRON-BEAM MOIRÉ

Principal Investigator: E.S. Drexler

Objectives

The objectives are to develop and apply the electron-beam moiré technique to measurement of displacements and observation of deformations at high magnification. The study of finer-scale packaging and on-chip features requires that the technique be modified to utilize the atomic force microscope (AFM). New methods of producing higher-density gratings must be found to meet this objective. 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 a hundred nanometers to tens of nanometers can be measured and strain calculated using the electron-beam moiré technique. Deformations are measured over fields ranging from 50 μm by 50 μm to 500 μm by 500 μm. This is accomplished by preparing the specimen surface with crossed-line gratings at pitches of 180 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 200× to 2000×. Deformations produce changes in the local moiré fringe density. These changes are analyzed to give the complete normal and shear displacements from which strains may be calculated.

The technique is eminently suited to the problems of the microelectronics industry. Our collaboration with Motorola continued in FY1999 with our being asked to study three new package geometries. Two were modifications to the original flip-chip package that we tested in FY1998. It was apparent in the first hour when subjected to a −55 °C thermal load that one-specimen geometry was completely unreliable. Motorola saw similar low-temperature failures in the accelerated lifetime tests conducted at their facility. The second package design had taken into account our findings from the original thermal cycle test. The geometry of the solder balls was changed to retard crack growth around its perimeter, and a more compliant solder composition was used so that the crack did not propagate under the solder ball. The third specimen supplied to us was a sequential build-up substrate or high-density interconnect (HDI) for a flip-chip package. The multiple layers containing vias and microvias, and the flip-chip solder balls, underfill, polyimide, and solder mask of the standard flip-chip packages created a complex strain field and new failure mechanisms.

As the industry continues the trend toward smaller and less expensive units, further development of the technique is necessary to maintain relevance. Toward that end we are investigating two new methods for making ultra-fine pitched gratings. In collaboration with researchers at Colorado State University (CSU), self-assembling block co-polymers are being assessed. Dr. Menoni of CSU believes that by using precise processing and etching procedures, a regular grating array with a pitch of 10 nm can be produced. Dr. Winningham and Dr. Douglas of the University of Colorado are conducting research on the outer crystalline shell of the *Sulfolobus acidocaldarius* bacteria. The bacteria produce an outer shell with a two-dimensional hexagonal array with a 22-nm pitch lattice constant. We have proven that is it possible to produce the moiré effect from the interference between this grating and the rastering of the tip in the atomic force microscope (AFM). We will pursue this collaboration as we investigate the feasibility of using this grating for atomic-force moiré on minute on-chip interconnects and vias.

External Collaborations

R. Munroe, Motorola—studies of failure mechanisms in Motorola package designs including flip-chip plastic ball grid array (PBGA) and sequential build-up substrate (or HDI).

Dr. C. Menoni and Dr. B. Parkinson, Colorado State University—collaboration under a Colorado Advanced Materials Institute grant to find new methods to make the moiré grating using self-segregating block copolymers.

Dr. Andrew Winningham and Dr. Ken Douglas, University of Colorado—collaboration to develop an extremely fine-pitched source for moiré gratings using the outer crystalline shell of bacteria.

Planned Outcome

Successful development of a new method of producing moiré gratings for the electron-beam moiré technique will permit measurement of displacements of less than 10 nm. Measurement 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

Two new designs for the flip-chip PBGA specimens were received from Motorola, prepared, and tested. The tests used electron-beam moiré and the specimens were thermally cycled between −55 and 125 °C.

The tests were analyzed and the results reported to Motorola directly in a plant visit that took place in February. The results were welcomed by the Motorola staff and generated much discussion and interaction.

Motorola again requested our help identifying failure modes in the latest product they are attempting to qualify for low-temperature applications. The specimens were received, prepared, and preliminary tests made late in FY1999.

The moiré effect was observed in the AFM using the crystalline outer shell of the *Sulfolobus acidocaldarius* bacteria on a Si substrate obtained from Dr. Winningham of the University of Colorado. This was our first proof that it is possible to scale-down the moiré technique to the AFM, offering us the potential to measure displacements of on-chip features.

We researched, evaluated, and purchased a new image acquisition system that will collect and store high-resolution images from the SEM. The improved resolution will allow us to identify moiré fringe centers with greater confidence.

Publications

E.S. Drexler and J.R. Berger, Mechanical deformation in conductive adhesives as measured with electron-beam moiré, *Journal of Electronic Packaging* **121,** 69–74 (1999).

E.S. Drexler, Reliability of a flip-chip package thermally loaded between −55 and 125 °C, *Journal of Electronic Materials* **28**, 1150–1157 (1999).

E.S. Drexler, Strain measurements in a thermally-cycled flip-chip-on-board solderball, *Materials Reliability in Microelectronics IX*, D.D. Brown, A.H. Verbruggen, and C.A. Volkert, eds., to be published.

E.S. Drexler, Plastic strain in thermally cycled flip-chip PBGA solder balls, *IEEE Transactions on Advanced Packaging*, submitted.

.**Project: STRESS VOIDING AND ELECTROMIGRATION**

Principal Investigators: R.R. Keller, D.T. Carpenter, C.E. Kalnas, J.M. Phelps, F.R. Fickett, J.A. Nucci*

Objectives

The main objective is to develop a mechanistic understanding of the microstructural processes controlling stress voiding (SV), electromigration (EM), and residual mechanical stresses in interconnects. Supporting objectives are: assessment and, when necessary, modification of microstructurally based models that sufficiently describe similar behavior in bulk metals; development, through knowledge of microstructural mechanisms, of test structures for assessing stress voiding reliability and the interactions of stress voiding with electromigration; and investigation of the effects of modification of the metallization/passivation interface on resistance to stress voiding and electromigration.

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, substrate and rigid passivation overlayer, or from atomic flux divergences due to strongly nonuniform 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. Complicating matters is a new issue associated with the integration of copper into chip-level interconnects, namely the development of residual stresses that vary with time. Such stresses are believed to originate from the presence of impurities introduced during the electrodeposition process, and may play an important role in both the measurement and control of SV and EM reliability. Interconnects become less homogeneous as dimensions scale downward, since the structures then comprise individual grains extending through the film thickness and across the line width. Behavior also becomes less homogeneous, and even small variations in microstructure can have detrimental effects on reliability. Understanding and solving the problems of void formation and growth at the microstructural level are essential to the continued development of metallizations on a submicron scale, as identified in the 1997 SIA Roadmap. Knowledge of microstructural effects on interconnect reliability allows for the design and fabrication of test structures that 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.

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*Max Planck Institut für Metallforschung (guest)

One approach to controlling interconnect reliability centers on the fact that stress 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. Interface 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. Interface modification is accomplished by controllably depositing photoresist onto patterned metal lines prior to passivation deposition. Accelerated testing then reveals how voiding has been changed from nonmodified structures. Another novel approach to interconnect reliability control focuses on the measurement of microstructure distributions within thin films, and the concurrent determination of property distributions. The complete structure of grain boundaries can be measured by automated methods and analyzed in a statistical manner. Individual boundary structures are then related to specific properties such as diffusivities. This allows for the determination of a property map of thin-film microstructure, which could potentially be incorporated into interconnect design rules for improved SV and EM reliability.

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.

Electrical evaluation of electromigration test samples under conditions similar to those used in reliability testing is used to further understand the failure modes.

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

T. Marieb, Intel Corp.— 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.

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

M. Gross, Lucent Technologies—studies of microstructure development and residual stresses in damascene copper interconnects.

J. Nucci, C. Volkert, and S. Krämer, Max Planck Insitut für Metallforschung—studies of residual stresses and electromigration in narrow interconnects.

Planned Outcome

Success in meeting the project objectives will result in identification of detailed microstructure-based processes 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. It will allow measurement of complete distributions of structure and properties in thin metal films used for interconnects. The electron microscopy-based characterization techniques developed for routine assessment of interconnect reliability on a microstructural level will be transferred to the microelectronics community.

Accomplishments

Completed fabrication of the NIST 36 test chip for assessing stress voiding and electromigration in two level interconnects with vias, in collaboration with H. Schafft of EEEL/Semiconductor Electronics Division. The structures are used to assess effects of linewidth and lithographic defects on void formation. Included are intentionally patterned notches to create local stress concentrations.

Completed EM and SV testing on passivated aluminum alloy interconnects containing modified interfaces. Observations indicate that void densities are increased in modified regions, presumably due to changes in void nucleation energies. Potential competition between decreased nucleation energies and kinetically favorable sites for rapid void growth depends upon the distribution of grain-boundary structures within the unmodified regions.

Developed methodology for performing convergent-beam electron-diffraction measurements of lattice constants in damascene copper interconnects. Preliminary results indicate that elastic lattice strains in the range 0.1 % to 0.3 % can be routinely detected. Have laid out the framework for automating the process, as well as for improving strain resolution.

Designed and constructed an electromigration test facility containing state-of-the-art probing and high-temperature measurement capability. Made initial measurements of ac versus dc electrical failure modes on a NIST test chip.

Publications

C.E. Kalnas, R.R. Keller, J.M. Phelps, and T.N. Marieb, Effect of modified metal/passivation interfaces on stress voiding in interconnects, *Materials Research Society Symposium Proceedings: Materials Reliability in Microelectronics*, in press (1999).

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Project: THERMAL CONDUCTIVITY OF THIN FILMS

Principal Investigator: D.R. Smith

Objectives

The immediate objective is to develop an apparatus for measuring, *by absolute steady-state methods*, thermal conductivity of thin metallic films of the dimensions typically used (about one micrometer) in modern large-scale integrated circuits (ICs) and their packages, for both in-plane and out-of-plane thermal transport. The long-term objective is to 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 on-chip elements are now significantly less than 1 mm. The advantages of reduced length scales are necessarily accompanied by greater packing density, with attendant increased generation of heat. Unless this heat can be efficiently removed, the lifetime-to-failure of critical elements of the package may be severely compromised due to thermo-mechanical effects, diffusive degradation of elements, or outright catastrophic failure.

Another issue relates to the fact that device elements are approaching a size where the classical physics of transport of electrons and phonons begins to break down. For example, the greatly reduced size of the metallization traces leads to much greater influence of surface effects that could previously be ignored. The surfaces where new behavior is expected are the free surfaces of the semiconductor or metallization, as well as the interfacial boundaries. These 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 bulk metals and semiconductors, this work is often 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. In the case of thermal conductivity, accurate measurement 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, especially when applied to complex microelectronic structures. Evaluation of the individual accuracies of such methods is one of the major objectives of the present work, in order to inform the users of data obtained by transient methods as to whether the conductivities obtained are sufficiently reliable for their purposes.

The specimen design used here 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.

External Collaborations

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

Al Feldman, Ceramics Division—devise a common experimental structure to compare our measurement techniques with his thermal-wave (transient) measurement system.

Planned Outcome

Benefits that would result from successful completion of the project include: transient methods for determining thermal conductivity of thin films could be critically compared to the absolute value obtained from the steady-state method; a modular structure for determining thermal conductivity of in situ thin-film structures would be developed that could be easily incorporated into production wafers for IC devices; and the method developed here would be easily modifiable for incorporation into other electronic and photonic packages.

Accomplishments

Last year a compact vacuum chamber was constructed for the infrared IR microscope. It allows removal of 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 was built to allow the vacuum chamber, and contained specimens to be properly oriented for observation by the IR microscope. This year, the apparatus was put into service.

- Six thermal-conductivity bridge structures of various dimensions and combinations of layers were measured in the evacuated test chamber at four to seven input power values, giving a unique temperature distribution with position for each power.

- Further development of the IR microscope system allowed us to obtain false-color images of the complete heated bridge structure; line-cursor registration of pixel-by-pixel temperatures along the bridge, with the temperature distribution graphed in real-time; and spot-meter registration of temperatures on the bridge at 10 positions.

- The temperature distributions for each bridge and power setting were fitted to parabolic curves, in agreement with the analytical model for the temperature distribution. Two deviations from the model were accounted for on physical and design principles, and corrected temperature-distribution curves were obtained.

- Analysis of the data was completed and a draft of a publication for submission to the *NIST Journal of Research* titled "Thermal conductivity of thin integrated-circuit layers by a Kohlrausch technique" is in preparation.

Project: **X-RAY DIFFRACTION STUDIES OF ELECTRONIC MATERIALS**

Principal Investigators: T.A. Siewert, F.R. Fickett, D. Balzar*

Objective

The objective is to conduct x-ray diffraction (XRD) studies of ferroelectric, photovoltaic, magnetoresistive, and other materials relevant to microelectronics. We focus on microstructural properties, especially strain/stress, crystalline defects, and texture.

Technical Description

Many macroscopic materials properties depend on internal stress, texture, and defect concentration. Because of their influence on diffraction of x-rays and neutrons, the analysis of diffraction patterns is the basis for their determination. For instance, the nonrandom orientation of grains in a polycrystalline aggregate will change relative intensities of diffraction lines, the homogeneous change of interplanar spacing will shift diffraction lines, and crystalline defects, such as dislocations, point, and planar defects, will locally change the interplanar spacing, which will result in diffraction-line broadening. We develop methods to obtain this information from diffraction measurements and apply it to different materials of interest, in particular materials for microelectronics uses. Diffraction is complemented with other methods, such as atomic-force microscopy (AFM) and small-angle-neutron scattering (SANS). Some of the more interesting studies were as follows:

Changes of residual stress and defects that occur upon poling of ferroelectric $BaTiO_3$ polycrystals were studied by high-resolution synchrotron-radiation diffraction. Diffraction-line-broadening analysis shows large microstructural changes, especially along the direction of spontaneous-polarization and poling-field vector. The inhomogeneous strain upon poling is about the same order of magnitude as the strain caused by electrostriction during poling and indicates a substantial increase of the dislocation density. This implies that the application of an external poling field generates defects in the structure and increases the internal stress. The increase of internal stress influences the ferroelectric and phase-transition temperatures. Moreover, the increase of both internal stress and defect concentration may have adverse consequences on both polycrystalline and epitaxial thin films through the accelerated degradation of dielectric properties. Because some dislocation reactions lead to microcracking, this may even result in mechanical failure.

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* Guest Researcher from the Department of Physics, University of Colorado at Boulder.

XRD, AFM, and dielectric-properties measurements were used to study oxide ferroelectric thin films for frequency-tunable microwave devices. Single-phase epitaxial thin films of $Ba_{0.6}Sr_{0.4}TiO_3$ and $KTaO_3$, for applications at 300 K and 77 K respectively, were deposited by pulsed laser deposition. AFM images indicated relatively smooth surface for both films with the rms surface roughness values less than 20 nm. XRD azimuthal scans showed a good epitaxy with the substrate. For $KTaO_3$ film, there is a small number of domains oriented at 45° relative to the <0*k*0> of MgO substrate ([110]). Rocking curves show significantly larger spread of grains around <*h*00> (perpendicular to the film surface) for $KTaO_3$ than for $Ba_{0.6}Sr._{0.4}TiO_3$. Furthermore, $Ba_{0.6}Sr_{0.4}TiO_3$ film exhibited low microwave losses. Because extrinsic losses depend on lattice defects, grain boundaries, and internal stresses, current studies focus on line-broadening analysis of these effects.

Another topic of interest is the accurate determination of strain/stress and texture. The complete texture information allows calculations of orientation-distribution function (ODF) and consequent weighting of monocrystal elastic constants. The polycrystalline elastic constants are needed to calculate accurate elastic stresses from the measured elastic strains. A simultaneous determination of complete strain tensor and texture is possible by Rietveld refinement of diffraction measurements collected at different specimen orientations. Therefore, all the strain and texture parameters can be obtained, along with structural, microstructural, compositional, and other information for all the crystalline phases. The method is generally applicable and especially suitable for determination of stress and texture in multiphase materials by time-of-flight (TOF) neutron and energy-dispersive x-ray diffraction.

A particular problem in low-alloyed *bcc* steels used for reactor-pressure vessels is embrittlement caused by formation of copper-rich precipitates (CRP). The formation of small (1 nm to 2 nm) CRPs is greatly facilitated by large neutron-irradiation damage. Because the CRPs are so small, it is difficult to monitor the kinetics of their formation and growth directly. However, small CRPs are coherent with the *bcc* matrix, which causes local matrix strain and interaction with the dislocation strain fields, thus impeding the dislocation mobility. As CRPs grow, coherency strain and dislocation density and their arrangement change. At some critical size of CRPs, the bulk crystal structure (*fcc*) of copper is achieved and CRPs are no longer coherent with the matrix, which relieves the matrix strain. In a simple isotropic elastic-continuum misfit-sphere approximation, precipitates cause an overall expansion of the host lattice, which changes the lattice parameters. Additionally, the effect of local distortion of the host lattice in the vicinity of precipitates is an inhomogeneous (local) strain that causes diffraction-line broadening. We analyzed XRD line broadening of seven steel specimens with different state of CRPs, as determined by SANS. Measured strain correlates with hardness and fits the model very well if strain from lattice dislocations is accounted for.

External Collaborations

Allen M. Hermann and Badri Veeraraghavan, Department of Physics, University of Colorado at Boulder—nonlinear dielectric thin films, photovoltaics.

Jules L. Routbort and Eun Tae Park, Energy Technology Division, Argonne National Laboratory, Illinois—ferroelectrics.

Robert B. Von Dreele and Kristin Bennett, Los Alamos Neutron Scattering Center, Los Alamos National Laboratory, New Mexico—neutron-diffraction studies.

Peter W. Stephens, Physics Department, SUNY at Stony Brook, New York—synchrotron-radiation diffraction.

Charles Glinka and James Barker, NIST Center for Neutron Research, Gaithersburg—SANS studies.

Planned Outcome

Our XRD and related studies focus on important physical parameters that govern behavior and properties of materials and devices used in microelectronics, nuclear power plants, and elsewhere. It is our intent to establish a causal relationship between microstructure and properties. Thus, the materials features, for instance dielectric and mechanic properties, can be correlated with and understood in terms of microstructural quantities. Moreover, in this way, the behavior of materials under working conditions can potentially be predicted, and possible failure avoided.

Accomplishments

- Currently, we are improving our diffraction facilities for studies of thin films. We obtained Philips thin-film diffractometer and will optimize recently acquired high-energy diffractometer for microdiffraction.

- We studied several ferroelectric materials: $BaTiO_3$, $Ba_{0.6}Sr_{0.4}TiO_3$, and $KTaO_3$. In bulk $BaTiO_3$, we focused on changes in dislocation density upon poling, and in $Ba_{0.6}Sr_{0.4}TiO_3$ and $KTaO_3$ thin films we studied structure and epitaxy by XRD and morphology by AFM.

- We proposed a method for the determination of a complete strain tensor by simultaneous analysis of several tens of diffraction patterns collected at different specimen orientations. The method is generally applicable and especially suitable for determination of stress and texture in multiphase materials by TOF neutron and energy-dispersive x-ray diffraction.

- We completed the analysis of embrittlement in copper-bearing reactor pressure-vessel steels. Small-angle neutron scattering results proved existence and growth of small CRPs as a function of aging time. Correlation between internal strain (caused by CRP and dislocations) and mechanical properties (hardness) was successfully modeled.

- Davor Balzar was elected as a U.S. member of the Commission on Powder Diffraction of the International Union of Crystallography and a member of the Instrument Advisory Team (IAT) for the Engineering Strain Diffractometer, to be constructed at the Spallation Neutron Source (SNS) in Oak Ridge, Tennessee.

Publications

G. Alers, D. Balzar, D.C. Hurley, B. Igarashi, W. Johnson, S. Kim, H. Ledbetter, and P.T. Purtscher, Nondestructive detection of embrittlement by copper in steel, *Proceedings of the Ninth International Symposium on Nondestructive Characterization of Materials* (Sydney, Australia, June 1999).

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Project: INTERFACIAL EVALUATION IN ELECTRONIC PACKAGING MATERIALS USING ELECTRON-BEAM MOIRÉ AND INFRARED MICROSCOPY

Principal Investigators: E.S. Drexler, A.J. Slifka

Objectives

The objectives of this new project are to examine interfaces in electronic packaging materials and elucidate the damage mechanisms at these interfaces by measuring mechanical strain and heat flow at the same positions and on similar size scales. We use electron-beam moiré to measure mechanical strain under thermal loading, at different temperatures, to see the effects of differential thermal contraction and expansion. We use infrared microscopy to see heat flow across the various material interfaces.

Technical Description

As electronic packages become denser and demands for circuit speed increase, the heat loads on these packages increase. Economic and performance issues demand that these packages be made of disparate materials. Due to thermal expansion mismatch and bonding mismatch, failures occur at interfaces of these materials. An understanding of the mechanisms and development of damage at electronic material interfaces is critical to the optimization and enhancement of electronic packages and their reliability.

Electron-beam moiré has very good spatial resolution, as small as 50 nm. Measurements are made at the endpoints of thermal cycling from –55 °C to 125 °C and typically at 25 °C. The room temperature measurements provide a baseline and a direct comparison to the infrared microscopy measurements, which are typically done near room temperature. Large displacements seen at temperature extremes are frequently nucleation sites for cracks. Repeated thermal cycling can be tracked with electron-beam moiré to show how damage evolves and accumulates, leading to failure, which almost always occurs at material interfaces.

Infrared microscopy has a spatial resolution of about 10 μm, which is adequate for many electronic packaging structures. An electronic packaging structure can be heated either with a laser, or self-heated electrically. The heat flow across an interface can be scattered due to bonding mismatch, defects, or cracks. We are interested in how the heat flow changes as the materials undergo thermal cycling. As permanent damage increases, the thermal resistance of interfaces increases. Infrared microscopy can measure changes in interfacial thermal resistance that occurs prior to the evidence of damage at the surface of a specimen as well as changes that occur after damage is readily apparent.

The use of these two techniques as complementary is the most useful aspect of this approach. We use two techniques that measure different properties to examine the same issue, in the same specimen, and in the same area of that specimen.

External Collaborations

Integral passive devices were obtained from Polymore Corporation, but no formal agreements exist at this time.

Planned Outcome

This project will provide industry with tools and information that can uniquely aid them in assessing reliability and give them the information required to design more economical and higher performance electronic packages to meet future needs. Embedded and integral passive devices will be a crucial part of reducing the size of devices in handheld and wireless technologies. We are concentrating our efforts on the reliability issues of the materials used in these applications.

Accomplishments

We acquired conductive inks and adhesives commonly used in current industrial packaging applications. We produced a specimen of appropriate size for both measurement techniques that has three material interfaces of industrial importance. We have instrumented the specimen with grids for use in electron-beam moiré. The grids have proven to remain usable through thermal cycling and infrared microscopy measurement.

We have measured a region of this specimen with both experimental techniques prior to thermal cycling and after 3 and 13 thermal cycles. There was no permanent damage evident on the surface as viewed by optical and electron microscopies, but infrared microscopy showed a change in interfacial thermal resistance at one of the interfaces after only 3 thermal cycles. Electron-beam moiré images showed large strains at extreme temperatures in the polymeric materials. After 13 cycles, a crack had developed near the interface between the two conductive adhesives used to produce the specimen. Infrared microscopy continued to show a growing interfacial thermal resistance at this location. A few more thermal cycles were done in the SEM, to allow moiré imaging as the crack grew. The moiré images showed a marked change in the displacements of material at extreme temperatures as another crack nucleated at the edge of the specimen and grew to meet the first crack that had formed. The two experimental techniques have proven to be complementary and result in a more complete understanding of damage evolution in electronic packaging materials.

ULTRASONIC CHARACTERIZATION OF MATERIALS

The Program on Ultrasonic Characterization of Materials is directed toward development of model based methods of physical measurements that characterize the internal geometries of materials with size scales that extend from atomic arrangements and lattice distortions to grains, reinforcements and surface layers. Our goal is to convert these measurement methods into sensor systems suitable for production line and in-service monitoring of material quality and serviceability.

A primary focus of this program is the characterization of microelectronic structures such as interconnects and dielectric layers as well as the microstructural features of metals, alloys, composites and engineered surfaces. The idea is to establish models that relate microstructure to physical measurements so that by measuring appropriate physical properties, the salient microstructural features can be ascertained. For example, measurements of sound velocity and ultrasonic wave attenuation can be related to elastic properties and mechanical relaxation processes that can be used to characterize fiber-orientation distributions in composites or the texture in polycrystalline metals as well as the dynamics of atomic diffusion and magnetic moment redistributions. These model-based measurements enable industry to replace microscopy with nondestructive methods for the microstructural characterizations needed to assure the quality of advanced electronic and structural materials.

The Ultrasonic Characterization Program is making significant contributions to measurement technology and materials modeling. We have worked with industry to commercialize noncontact ultrasonics, wave-form-based acoustic emission, magnetostrictive transduction and nonlinear ultrasonics. Modeling advances include Green's-function methods for rapid solution of static and dynamic elasticity problems, including wave propagation in layered, anisotropic materials, analysis procedures for deducing all the elastic modulus tensor elements from resonant ultrasonic spectroscopy (RUS) data and separation of internal friction values from ultrasonic attenuation measurements that are dominated by losses from scattering and beam diffraction.

Project: ELASTIC COEFFICIENTS AND RELATED PHYSICAL PROPERTIES

Principal Investigators: H. Ledbetter, 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.

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 K 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.

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.

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* Visiting scientist from University of Colorado, Mechanical Engineering.

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

Accomplishments

We measured elastic properties of two polycrystalline mullite ($3\ Al_2O_3 - 2\ SiO_2$) specimens: commercial and precursor. The C_{ij}'s were corrected for void-free state. For monocrystal $2\ Al_2O_3 - SiO_2$ mullite, we used both Acoustic-Resonance-Spectroscopy and Electromagnetic-Acoustic-Resonance methods to obtain the C_{ij}'s. Mullite has orthorhombic symmetry, hence the nine independent C_{ij}'s.

We studied the effects of piezoelectric and elastic coefficients of $LiNbO_3$, langasite, and quartz. These materials have trigonal symmetry. We determined the elastic constants from resonant frequencies and dimensions. Further study will include determining piezoelectric coefficients simultaneously. We also studied piezoelectric compounds KH_2PO_4 and KD_2PO_4 that undergo a phase transition at low temperature. C_{ij}'s of KD_2PO_4 have been determined for tetragonal symmetry with $C_{12} < 0$ at room temperature. Low-temperature x-ray diffraction and acoustic-resonance-spectroscopy measurements are in progress.

Among studies of metal-matrix composites, we determined C_{ij}'s of short and continuous fiber composites of Al_2O_3/Cu. Further studies will include Q_{ij}^{-1}. In spinnel/Al composites, we studied effects on Q_{ij}^{-1} of different fiber diameters with the same volume fraction. We expect that C_{ij}'s remain unchanged with only effects on Q_{ij}^{-1}. On crossply SiC_f/Ti composites, we focused on thermal stress and interfacial bond that both influence C_{ij}'s.

Studies of diffusion-bonded layered materials included several combinations of specimens: A/B/A, A/B/C, etc. We successfully calculated expected resonant frequencies on A/B/A material and we shall extend the theory to A/B/C materials (three layers). As an example of one-dimensional ordering, we studied textured wires. We successfully determined C_{ij}'s of Cr-Ni-Mo-alloy (MP35N) wire and of a few wires of a different diameter. For wires with diameters smaller than 0.5 mm, work is still in progress.

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Project: NONLINEAR ULTRASONICS FOR MATERIALS CHARACTERIZATION

Principal Investigators: D.C. Hurley, D. Balzar, P.T. Purtscher

Objectives

We aim to develop nondestructive methods to evaluate the mechanical/physical properties of materials using nonlinear ultrasonics. We plan to use the techniques to investigate specific industrial materials such as engineered surfaces or precipitate-hardened alloys. The experimental results will be quantitatively interpreted with the aid of microstructural models.

Technical Description

Many tools for nondestructive materials evaluation emphasize defect detection. In contrast, in this project we aim to quantitatively assess the material's physical properties. We are developing nonlinear ultrasonic techniques to understand relationships between a material's microstructure and its mechanical properties. Nonlinear ultrasonics is an emerging technique that may probe certain properties (such as strain or film adhesion) more sensitively than standard linear ultrasonic methods. The technical approach involves development of sensitive and accurate measurement methods. A unique capability is a laser-based Michelson interferometer system developed at NIST. This method enables direct measurement of absolute ultrasonic displacements, which is essential for nonlinear studies and also useful in linear applications. Other advantages include wide (from DC to greater than 20 MHz) bandwidth, micrometer-scale resolution, an inherently unintrusive nature, and scanning/imaging capabilities.

With this approach, we are using nonlinear bulk waves to investigate several systems. Harmonic-generation experiments are performed to determine a material's nonlinearity parameter. Using microstructural models, relationships are established between the nonlinearity parameter and key properties such as strain and dislocations. We are also adapting our methods to enable nonlinear measurements on surfaces. The interferometric detection scheme described above is suitable for surface waves; however, methods to generate finite-amplitude surface waves with a narrow frequency content are needed. For detailed interpretation, both bulk and surface nonlinear studies require analytical models and measurements by complementary methods.

External Collaborations

D. Barnard, Iowa State University—piezoelectric techniques for nonlinear experiments.
E. Danicki, IPPT Polish Academy of Sciences—comb transducers for narrowband surface waves.
D. Price & A. Richards, CSIRO-TIP Australia—surface wave and nonlinear interface experiments.
RITEC, Inc.—specialized electronics for nonlinear measurements.

Planned Outcome

We are developing new measurement methods to quantitatively assess the mechanical and physical properties of industrial materials. Through this project, we hope to achieve techniques for the nondestructive evaluation of such performance-related properties as adhesion, hardness, or fatigue. In the long term, this work should lead to the development of tools that utilize nonlinear ultrasonics to characterize microstructure and relate it to performance or lifetime.

Accomplishments

A piezoelectric detection technique for nonlinear measurements was successfully implemented. Piezoelectric detection is less straightforward than interferometry, but is more adaptable to conditions outside the laboratory. Development of piezoelectric methods led to a better understanding of an effect first noticed at Iowa State University, which had been thought to represent a new physical phenomenon. Our data indicated that it was more likely an artifact related to transducer operation at resonance. We also developed an apparatus for nonlinear measurements outside the laboratory, and performed experiments with ruggedized single-crystal $LiNbO_3$ transducers to make the apparatus feasible for hot-cell experiments on irradiated steel samples. The basic apparatus works well, but more transducer development is needed.

An apparatus was developed to measure ultrasonic attenuation over a wide frequency range (5 MHz to 25 MHz). The method compares three diffraction-corrected echoes in a pulse-echo immersion setup. Besides being a characterization tool in its own right, it provides attenuation measurements that are needed to correctly calculate the nonlinear acoustic parameter.

We investigated the effect of precipitate strain on harmonic generation using steel samples with copper-rich precipitates. Attenuation-corrected values for the nonlinearity parameter were measured for steel samples whose hardness was determined by heat treatment. X-ray diffraction line broadening measurements showed that the hardness was related to the amount of coherency strain due to the precipitates in the samples. Our experiments showed a correlation between the nonlinearity parameter and hardness, and hence the precipitate strain. We interpreted our results using a model for harmonic generation by precipitate-pinned dislocations; the model's basic features agree with experimental results. Small-angle neutron scattering experiments provided additional information about the precipitate's properties. This information permitted us to analyze our nonlinear results in more detail than had been presented in the literature.

We sought to improve the performance of comb transducers for generating nonlinear surface waves. We devised two new designs based on an analytical model for comb transduction under development by a collaborator. The new combs were much thicker (several tens of acoustic wavelengths) than earlier designs, were much larger in cross-section than the excitation transducer, had a slanted top surface, and could be bolted directly to the sample. Design changes were intended to reduce extraneous signals, improve bonding repeatability, and increase directionality and conversion efficiency. Experiments show that these designs generate somewhat larger displacements with more repeatability and greater ease of use.

We also investigated laser-based approaches for generating nonlinear surface waves. Unlike combs, laser-based generation is noncontacting and is not limited to a single frequency. We identified a "spatial interference fringe" method to generate narrowband surface waves. This method also enables precise measurements of the dispersion (velocity versus frequency) relation, which can be used to determine second-order elastic properties. We designed a surface-wave system to probe thin, sub-micrometer coatings that will be delivered in early FY2000. We gained practical experience with the fringe method during a visit to CSIRO-TIP. There, we developed an optical setup to generate surface waves using the fringe method and detected the waves with a Michelson interferometer similar to ours. In a proof-of-principle demonstration, we optically generated and detected surface waves containing more than 10 cycles at 15 MHz to 20 MHz on an aluminum plate. This work will facilitate fast implementation of our new system in FY2000.

We performed experiments to determine the effective radius of contact transducers using a Michelson interferometer. The experimental results were compared successfully with the predictions of a numerical model. This approach appears to offer very high precision.

Outputs

Publications

D.C. Hurley, D. Balzar, and P.T. Purtscher, Nonlinear ultrasonic assessment of precipitation hardening in A710 steels, *Journal of Material Research,* submitted.

D.C. Hurley, Nonlinear propagation of narrow-band Rayleigh waves excited by a comb transducer, *Journal of the Acoustical Society of America*, **106**, 1782 (1999).

D.C. Hurley, D. Balzar, and P.T. Purtscher, Nonlinear ultrasonic parameter in precipitate-hardened steels, in *Nondestructive Characterization of Materials IX*, R.E. Green, Jr., ed. (Plenum Press, New York, 2000), in press.

Presentations

D.C. Hurley, D. Balzar, and P.T. Purtscher, "Ultrasonic nonlinearity parameter in precipitate-hardened steels," Workshop on Nondestructive Characterization of Embrittlement in Reactor Pressure Vessel Steels (Boulder, July 1999).

D.C. Hurley, D. Balzar, and P.T. Purtscher, "Ultrasonic nonlinearity parameter in precipitate-hardened steels," 9th International Symposium on Nondestructive Characterization of Materials (Sydney, June 1999).

D.C. Hurley, "Nonlinear ultrasonics for materials characterization," IPPT Colloquium, Polish Academy of Sciences (Warsaw, March 1999).

D.C. Hurley and T.P. Lerch, "Characterization of ultrasonic contact transducers using a Michelson interferometer," Acoustical Society of America/European Acoustics Association/German Acoustics Association Conference (Berlin, March 1999).

D.C. Hurley, "Breaking Hooke's law: nonlinear ultrasonics for materials characterization," Physics Department Colloquium, University of Nevada (Reno, October 1998).

Project: INTERNAL FRICTION STUDIES OF DISLOCATION DYNAMICS

Principal Investigators: W. Johnson, P.T. Purtscher, G.A. Alers

Objectives

The objectives of this research project are to develop ultrasonic techniques for measuring changes in ultrasonic internal friction that result from annealing, loading, or irradiating structural alloys and to develop theoretical models that relate these measurements to changes in dislocation network structure and/or pinning. The accomplishment of these objectives will enable real-time evaluation of dislocation dynamics, providing fundamental information on deformation processes. It also will provide a basis for nondestructive ultrasonic evaluation of structural material integrity during production or service.

Technical Description

Ultrasonic measurements can be highly sensitive to the density, symmetry, and pinning of dislocations in crystals, because the anelastic response of dislocations causes damping and slowing of acoustic waves. Dislocation damping has been studied extensively in relatively high-purity metals, and, for these materials, existing theory can explain most observed effects. However, relatively few studies have been performed on impure alloys, and dislocation damping in these technologically important materials is poorly understood.

The experimental approach used in this project employs a unique laboratory system to perform measurements of the anelastic ultrasonic properties of dislocations in commercial alloys 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. 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 resonant damping and frequency. After dislocations break away from pinning points under an applied load, the dynamics of repinning under constant load are reflected in the time dependence of the damping and resonant frequency.

Planned Outcome

Experimental and theoretical results obtained in this project will provide fundamental information on dislocation dynamics, which are directly related to the bulk engineering properties of structural alloys. They are also intended to provide a basis for ultrasonic techniques for nondestructively evaluating the integrity of alloys in production and service. For example, the dependence of dislocation damping on applied stress may provide the basis for determining the state of fatigue or plastic deformation of steel well before visible cracks develop.

Accomplishments

In 1999, an extensive series of measurements on carbon and interstitial-free steels was completed. This work showed that plastic deformation induces an increase in the damping that partially recovers with time. Plastic deformation also permanently reduces the dislocation pinning such that the damping is changed with even small “elastic” loading.

Theoretical analysis in 1999 focused on the time-dependent recovery immediately following deformation. The frequency dependence and relative magnitudes of the damping and velocity changes were found to be inconsistent with the usual forms of established dislocation models.

The experimental apparatus was redesigned and rebuilt to provide more accurate temperature control and the capability of in situ annealing to 300 °C. The greater temperature control is critical for providing accurate velocity measurements needed for establishing theoretical models.

The focus of the research was shifted from commercial steels to binary aluminum alloys in order to explore differences between the dislocation dynamics in *fcc* and *bcc* materials. Binary alloys also are expected to provide a simpler system for developing models. Initial measurements have been performed on polycrystalline Al-0.2 %Zn subjected to elastic loads at several temperatures.

Publications

W. Johnson, G.A. Alers, and B.A. Auld, Acoustic resonator for measuring force, U.S. Patent No. 5813280 (1998).

W. Johnson, G.A. Alers, and B.A. Auld, Electromagnetic acoustic transducer and methods of determining physical properties of cylindrical bodies using an electromagnetic acoustic transducer, U.S. Patent No. 5895856 (1999).

W. Johnson, Dislocation damping after plastic deformation in interstitial-free and carbon steels, *Journal of Alloys and Compounds*, to be published.

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* (Plenum, New York, 1999), p 1863.

Project: **ULTRASONIC MEASUREMENT OF STRESS**

Principal Investigators: A.V. Clark, K.J. Coakley*

Objective

The objective of this program is to develop a nondestructive and mobile "strain gage" that can measure the level of stress in a large structural element without permanently attaching the gage to the part. Such a device could also be used to measure residual stresses left by manufacturing processes such as welding, heat treating and machining.

Technical Description

We exploit the acoustoelastic effect (i.e., the change in ultrasonic wave velocity caused by a stress) to infer stress from precise measurements of the time-of-flight of shear waves propagating through the thickness of common structural elements formed by aluminum and steel plates. The technical challenge is to take into account and correct for the small anisotropy in the shear modulus left in these materials by their fabrication process, which is most often done by rolling or extrusion. We typically find a faster velocity for a shear wave polarized in the rolling direction (RD) and a slower velocity for waves polarized in the transverse direction (TD). When a residual or applied stress is present, the polarization direction for the fast and slow waves is rotated away from the RD and TD and the velocities of the waves are changed in proportion to the magnitude of the stress. Therefore, in order to measure the stress we must not only make accurate measurements of the velocities of the fast and slow shear waves but also the orientation of their polarization vectors relative to RD and TD. To accomplish this, we have constructed a motorized rotating electromagnetic acoustic transducer (EMAT) to automatically determine the orientation of the acoustic axes and to measure very accurately the velocities of the fast and slow shear waves as they propagate over the thickness dimension. Since EMATs require no couplant, rotation can be rapid and no corrections due to variation in couplant thickness need to be introduced. Furthermore, EMATs can be scanned over an area to produce a map of the stress distribution without making direct physical contact with the material.

External Collaborations

Caterpillar, Inc.—to measure the residual stress distributions near welds in order to maximize the fatigue life of their products.

Gas Research Institute—to measure the stresses in buried gas pipelines that have been bent by earth movements.

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*NIST, Statistical Engineering Division

Institute of Gas Technology—to measure the stresses in buried, small diameter cast iron pipes.

Federal Railroad Administration—to detect the thermal stresses built up by temperature fluctuations in long stretches of welded rail.

Planned Outcome

The rotating EMAT and its portable instrumentation can be viewed as a mobile, noncontacting strain gage. It is intended for use on large structures in the field or at structural steel fabrication shops to measure the stress distributions established by loads on the structure or the residual stresses established by the welding processes. In the former case, dangerous conditions imposed on civil structures by earthquakes, avalanches and floods can be recognized and corrective actions taken to prevent catastrophic failures. In the latter case, residual stresses that can shorten the fatigue life of a structure can be detected and rectified.

Accomplishments

1. Experimental Verification: To verify the validity of our approach to rapid mapping of stress distributions in structural elements assembled from thick plates, we measured the stress distribution resulting from the assembly of aluminum and steel shrink-fit specimens. These specimens were prepared by forcing a circular plug into a smaller hole in an annular ring. The plug was cooled and the annulus heated to allow enough clearance for insertion of the plug into the annulus. On reaching ambient temperature, a radial compressive stress and a tensile hoop stress were developed in the annulus. We measured the through thickness times-of-flight (TOF) at four locations and along three scan lines 45° apart prior to assembly using the rotating EMAT and a phase-sensitive detector. After assembly, data were again collected at each measurement location with the same rotating EMAT and instrumentation. Once the acoustic axes were determined by analysis of the phase data and a mathematical model, the EMAT was oriented along these directions and the corresponding TOF data were collected. We used a matrix equation $(\Delta\sigma) = [K] (\Delta\iota)$ to compute the change in plane stress state $(\Delta\sigma)$ from the measured change in TOFs, $\Delta\iota$, between the stressed and unstressed states. The coefficients of the [K] matrix are the stress-acoustic constants of the material and were determined by separate experiments on specimens cut from the original plate stock and subjected to uniaxial tension. The ultrasonic-stress data were found to be in excellent agreement with strain-gage data and with neutron-diffraction measurements made at the NIST Center for Neutron Research.

2. Development of a Portable Instrument for Field Tests: Based on these experiments and on previous field tests on bridges, a simpler, portable stress measurement unit was obtained by special order from a vendor of EMAT systems. It consisted of a lunchbox computer with an on-board digitizer, a compact chassis box containing a powerful tone-burst generator and signal amplifier plus a sensor unit that contained the EMAT, its rotating motor, a diplexer and a preamplifier. Under computer control, the EMAT rotates and its orientation and TOF measurement 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. We tested the portable system by measuring the stress-acoustic constants on several specimens of steel with different heat treatments cut at 0° and 90° to the RD. A consistency check was then performed on specimens cut at intermediate angles. The predicted variation of weighted TOFs with applied stress was in good agreement with the measured values on these specimens. We also found that the stress-acoustic constants were changed somewhat by heat-treatment.

3. Application to Acoustic Microscopy: Recently, Dr. E. Krasika of the Materials Structure and Characterization Division in Gaithersburg proposed a technique for measuring stress with the focused piezoelectric transducer of an acoustic microscope. It operates on the principle of mode conversion at a water-specimen interface to generate the fast and slow shear waves needed to produce the stress measurement conditions described above. A theoretical model for producing an image of a stress distribution in a plate by an acoustic microscope system has been developed by Geoffrey McFadden in Gaithersburg but its comparison with actual measurements requires separate determinations of the third-order elastic constants (TOEC) of the plate. We are using our portable instrument and the rotating EMAT to measure the TOEC of an aluminum plate so that this theory can be verified.

Impacts

- A contract with the Gas Research Institute was recently signed to use the stress distribution mapping capability of the portable unit and rotating EMAT to measure the distribution of stress on a 15 ft long, 24 in diameter sample of gas line pipe while being subjected to three point bending in one million pound mechanical testing machine.

- A contract with the Institute of Gas Technology is intended to demonstrate the feasibility of using the EMAT system to determine bending stresses in the 4-6 in diameter cast iron pipes used for gas distribution systems throughout the world.

- The FRA is funding an SBIR study to demonstrate the use of EMATs for measuring the thermal stress in long sections of welded rail.

Outputs

Publications

A.V. Clark, C.S. Hehman, and T.N. Nguyen, Measurement of stress using a rotating EMAT, *Journal of Nondestructive Evaluation*, submitted.

A.V. Clark, D. Gallagher, C.S. Hehman, P.A. Fuchs, and M.G. Lozev, Ultrasonic measurement of stress in pin and hanger connections, *NISTIR 5080* and *Research in Nondestructive Evaluation*, submitted.

A.V. Clark, K. Coakley, C.S. Hehman and P.A. Fuchs, The effect of material inhomogeniety on ultrasonic stress measurements using a small-aperture EMAT, *Research in Nondestructive Evaluation,* submitted.

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.

Patent Application

A.V. Clark, C.S. Hehman, T.N. Nguyen, and G.A. Alers, Ultrasonic strain gage using a motorized EMAT, U.S. Patent Application No. 09/274,538. NIST #98-020US (1999).

Project: **WAVEFORM-BASED ACOUSTIC EMISSION**

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

Objectives

The first objective is to develop wideband, high-fidelity acoustic-emission methods, including necessary measurement methods, instrumentation, and computational analysis methodologies, for source location and damage characterization. Secondly, we are developing an understanding of acoustic-emission phenomena in order to facilitate discrimination of real acoustic-emission events from extraneous noise (e.g., in bridge steels).

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result of local transient energy releases in a material. Monitoring these waves can provide fundamental information about the location and mechanism of the transient-energy release. Often the energy release is due to a local micro-damage process. The technical approach, which is beyond that currently commercially offered for either resonant or waveform-based AE technology, follows a multifaceted development of all the key components that are relevant to a wideband application of AE technology. These components include development of wideband high-sensitivity sensor/preamplifiers; high-speed wide-dynamic-range digital recording data-gathering systems; finite-element modeling to predict far-field displacement waves from relevant AE sources, wideband experimental AE displacement waveforms from materials of interest; signal-processing techniques to accurately identify source locations and types; and experimental studies of simulated AE wave propagation. The scope in FY1999 covered two phases: (1) finite-element modeling of buried dipole sources in the near and far fields of thick plate; and (2) wideband waveform characterization of AE during fatigue growth as contrasted to the old AE technology using resonant sensors. Special equipment and facilities include a specially modified hydraulic materials testing system, multiple coupled waveform recorders (12-bit, 10 million samples per second digitization rate), and specially developed high-sensitivity, wideband AE sensor/preamplifiers. A commercially available waveform-based AE recording system is being evaluated for use in this program, as well.

External Collaborations

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

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* NIST, Applied and Computational Mathematics Division

Government Agency: The Federal Highway Administration sponsored the development of waveform-based AE for the NDE of bridges. NIST conducted studies on a variety of bridge steels so that the detectability of slow crack growth using AE could be determined. NIST is providing technical expertise in developing specifications for the Federal Highway Administration for an advanced high-speed waveform-based AE 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 AE researchers at NASA Langley to study wave propagation and accuracy of source location.

Planned Outcome

The anticipated outcome of this AE 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 (e.g., bridges) to applied loads. Further, the accurate location of concentrated micro-damage in materials by AE methods will be enhanced, and the ability to separate extraneous noise from damage-related AE 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 AE systems.

Accomplishments

Extensive data analysis of our AE waveform database gathered during monitoring of fatigue crack growth in various bridge steels and on aluminum alloy was carried out. The results were summarized in a paper that has been accepted for publication by the ASNT journal called *Materials Evaluation*. The differences in crack-generated AE at the two crack-growth rates were related to microstructural differences in the various materials as well as the mechanics of the AE source generation and the associated wave propagation.

Final results for the validation of a finite element AE source modeling code were completed. Numerical values were determined for key parameters to ensure that modeled results are correct and converged. These numerical values were based upon the minimum wavelength of interest. A paper summarizing these results has been prepared for submission to an appropriate journal. A series of three-dimensional finite-element runs have been completed for the five independent types of fundamental buried-dipole sources. So far the runs have been completed for two different source depths in a steel plate 25 μm thick. A fixed rise time has been used for the point sources. Results are available at distance up to 15 plate thicknesses for one full quadrant of the radiation pattern.

Publications

M.A. Hamstad and J.D. McColskey, Detectability of slow crack growth in bridge steels by acoustic emission, *Materials Evaluation,* in press.

Project: ULTRASONIC STANDARDS FOR MATERIALS CHARACTERIZATION

Principal Investigators: T. P. Lerch, G.A. Alers, T.A. Siewert

Objectives

The project objectives are to 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; and 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 %. Ultrasonic wave attenuation is well known as a sensitive indicator of microstructural features but there is a lack of consensus within the scientific community on how to define, much less measure, attenuation. There are additional physical properties that can be measured with ultrasonics that give additional information on the microstructures of commercial materials and support the development of models that relate microstructure to measurements. These include the third order elastic constants (TOEC), the orientation-distribution coefficients (ODC) of polycrystals and the magnetostriction coefficients of ferromagnetic alloys. Measurements of these latter properties are discussed elsewhere in this report, but the emphasis is on making the measurements as accurately as possible while the development of standards is put off to a later exercise.

External Collaborations

German Federal Institute for Materials Research and Testing (BAM), Berlin
American Society for Testing of Materials (ASTM)
International Institute of Welding (IIW)
American Society for Nondestructive Testing (ASNT)
Centers for Nondestructive Evaluation at Iowa State University and Johns Hopkins University

Planned Outcome

In the initial stages of this program, emphasis will be placed on establishing standard materials and standard measurement techniques for the velocity of sound in commercial steel and aluminum alloys. Such a goal is fundamental to the use of sound velocity and dynamic elastic modulus measurements for quality assurance in fabrication mills and heat-treatment foundries as well as for calibration of ultrasonic nondestructive inspection instruments in the field. Once these standards are in place, microstructure-based models can be developed to use these accurately measured properties to infer the state of the microstructure and to predict such important engineering parameters as the yield strength or the hardness without performing expensive destructive tests. As standards for the sound velocity are developed, a similar program will be directed toward ultrasonic attenuation so that it, too, can become an important quality assurance tool.

Accomplishments

Standardization of IIW Calibration Blocks: Prior to FY1999, 18 IIW-type calibration blocks were sent to NIST from a variety of sources throughout the world. Well characterized electronic pulsers and receivers were assembled and both commercial "search units" and unbacked piezoelectric discs were obtained from commercial sources. Modern digital oscilloscopes with digitization rates exceeding 100 MHz were procured for the transit time measurements. During FY1999, this equipment was used to make a thorough investigation of the various techniques available for measuring the velocity of longitudinal waves in the thickness dimension of the blocks. For shear waves, both electromagnetic acoustic transducers (EMATs) and lithium niobate single-crystal transducers were used. Ultrasonic attenuation measurements were deduced from the ratio of the amplitude of the second and fifth back surface echoes. Since these blocks are used primarily by NDT inspectors to standardize their search units before performing weld inspections, careful measurements of the "exit point" and the angle of refraction were made using commercial plastic wedge transducers operating at their nominal refraction angles of 45°, 60°, and 70°. The objective of making these angle-beam measurements was to assess the errors that an NDT inspector might encounter because of the variations in the velocities of longitudinal and shear waves in the calibration blocks. The results of this extensive study showed that the main sources of error in the longitudinal wave velocity measurements were the diffraction and bonding corrections which combined to give a total uncertainty of ±0.4 %. Since the bonding error for shear waves using EMATs is very small, the total uncertainty in the shear wave velocity was estimated to be ±0.04 %. These errors are much smaller than the spread in wave velocity values observed among the 18 blocks examined in this project. For the longitudinal velocity, this spread was ±0.6 % and for the shear waves, ±0.9 %. It was concluded that these variations should not affect the primary standardization purposes of the blocks. However, they do show that a proposed European Standard that requires an uncertainty of less than ±0.1 % in the ultrasonic velocities of IIW blocks 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.

International Workshop on NDT Reliability: In 1997, NIST cooperated with several European organizations to organize a workshop entitled "European-American Workshop on Determination of Reliability and Validation Methods on NDE." It was held in Berlin, Germany and had nearly 100 participants. One of its most important outputs was an empirical "formula" to describe the reliability of an NDE system and allow a quantitative measure of its ability to detect and size defects. In FY1999, NIST joined with the same organizations to host, in Boulder, a second workshop to develop this "formula" further. Over 40 people attended with half of them coming from Europe. The output of this workshop was the development of a consensus on the definition of the terms in the formula and examples of how it was being applied in various industries. Thus, the attendees will now be able to apply more standard criteria to assessing the reliability of a particular NDE system and to better focus on the individual elements that are limiting its reliability.

Impacts

- The uncertainty that can be expected for measurements of the velocity of ultrasonic waves in steel calibration blocks using commercially available equipment and transducers is ±0.4 % for longitudinal waves and ±0.04 % for shear waves.

- A proposed European Standard that requires an uncertainty of less than ±0.1 % in the ultrasonic velocities of IIW blocks cannot be met using typical ultrasonic equipment and measurement methods.

- A workshop on the reliability of nondestructive inspection systems was held at NIST in Boulder. Half of the more than 40 attendees came from Europe. A more quantitative approach to assessing and comparing the inspection reliability of NDT systems was developed.

Outputs

Publications

T.P. Lerch, M.C. Renken, and C.M. Fortunko, Variability of ultrasonic properties measured in IIW-type reference blocks, *Document No. V-1133-99, International Institute of Welding* (1999). And *Materials Evaluation*, submitted as two papers.

Project: GREEN'S FUNCTION METHOD FOR MATERIALS SCIENCE

Principal Investigator: V.K. Tewary

Technical Objective

The objective is to develop computationally efficient methods for calculating elastic and lattice Green's functions for advanced materials and apply them to different problems in materials science.

Technical Description

Green's functions can be static or dynamic. They 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 exerts the force that causes lattice distortion. The lattice static Green's function 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. Lattice static Green's functions depend upon interatomic potentials and detailed lattice structure. The lattice Green's functions for crystals containing point defects can be obtained by solving the Dyson's equation using the matrix partitioning techniques. This technique does not work for extended defects such as dislocations. We have developed a lattice statics model that allows us to calculate the Green's functions for dislocations to a desired degree of approximation.

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, fracture properties, etc. The elastodynamic Green's function is used for calculating elastic wave propagation in solids.

We calculate the elastodynamic Green's functions by using a delta-function representation that we had developed earlier. In the delta-function representation, we use a linear combination of the space and time variables instead of using the two separately. This representation has been found to be computationally very efficient for elastostatic as well as elastodynamic problems. We have used the delta-function representation to calculate the elastic wave propagation due to a line force such as generated by electromagnetic-acoustic transducers (EMATs) in isotropic and anisotropic solids.

We have applied the delta-function representation to calculate the elastic wave propagation in a layered semi-infinite solid and a finite plate. The problem is that the delta function representation itself does not satisfy the boundary conditions for a layered solid or a plate. In such cases we need to use the Laplace representation of the Green's function. The complex exponential dependence of the Green's function on the Laplace variable causes problems of numerical convergence, especially for anisotropic solids. We are in the process of developing appropriate methods for calculating the inverse Laplace transform of the Green's function for anisotropic solids.

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. A project on setting up of a library of Green's functions on WWW with Internet access was sponsored by the Systems Integration for Manufacturing Applications Program (SIMA). This project has now been completed.

External Collaborations

Dr. Adam Powell, Massachusetts Institute of Technology—development of Green's Functions library-generation II.

Prof. Laura Bartolo, Kent State University—An interactive web site for elastostatic Green's functions.

Prof. John Berger, Colorado school of Mines—Green's functions and boundary element calculations for the SIMA project.

Prof. Frank Rizzo, Iowa State University—Green's functions and boundary element calculations for the SIMA project.

Dr. Laocet Ayari, Ball Aerospace—application of Green's function and boundary element method to industrial problems on stress analysis of solids.

Planned Outcome

The anisotropic elastodynamic Green's functions will be used to interpret measurements on elastic wave propagation characteristics of electronic materials. The lattice static Green's function method will help in modeling 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 will be very useful 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 Internet for industrial as well as research and teaching applications.

Accomplishments

- A successful workshop on the use of the library of Green's functions was organized in which researchers from universities, industry, and NIST participated. Industrial applications of the Green's function library were demonstrated and discussed in detail.

- An interactive web site has been created that serves as a virtual meeting place and provides a platform for discussions amongst researchers and teachers involved in work on Green's functions. The software for calculating Green's functions is now available for download from the web site. The web site also serves as a problem bank and software bank. The URL of the web site is http://nistgf.kent.edu.

- An analytical solution of the 3-D anisotropic Boussinesq problem has been obtained by using the delta-function representation of the elastostatic Green's function for a semi-infinite solid.

- A lattice statics model has been developed for a semi-analytical calculation of the Green's function for a lattice containing a straight dislocation. The Green's function can be calculated with relatively small CPU effort even for a million-atom model to a desired degree of approximation.

- A computationally convenient delta-function representation has been obtained for displacement fields and velocities of elastic waves due to a line force on the surface of a semi-infinite anisotropic solid. Exact expressions have been obtained for Rayleigh as well as bulk waves in anisotropic solids. The method has been extended to an array of pulsed as well as vibrating line forces.

- Elastodynamic Green's functions have been calculated for a pulsed line source on the surface of an isotropic, layered semi-infinite solid.

Impacts

Industry has shown a strong interest in using the Green's function library software available from the NIST Green's function web site. Ball Aerospace has identified specific problems on stress analysis for which they are exploring the use of Green's function library. A consortium led by NIST has been formed that consists of scientists, engineers, and teachers from universities, industry, and other research institutions involved in work on Green's functions. The consortium interacts through the NIST Green's function web site for discussing research, industrial, and teaching applications of the Green's function library. The NIST Green's function web site will also be used for teaching and as a problem and software bank that will help students to choose industrially relevant problems for their graduate and postgraduate research and in exchanging software.

Outputs

Publications

V.K. Tewary, Green's-function solution of the Boussinesq problem for 3D anisotropic solids, *Journal of Materials Research,* submitted.

V.K. Tewary, Lattice statics model for edge dislocations in crystals, *Philosophical Magazine,* submitted.

Presentations

“Green's Functions Library and its Industrial Applications” at the GF Workshop at NIST, Gaithersburg, October 1998.

“Green's functions method for modeling of lattice defects” at the NRC/NIST Workshop at NIST, Boulder, July 1999.

Project: NONDESTRUCTIVE CHARACTERIZATION OF STEEL EMBRITTLEMENT

Principal Investigators: G.A. Alers, B. Igarashi,* S. Kim, H. Ledbetter, H. Ogi,** P. T. Purtscher

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 measurements of magnetic, eddy-current and ultrasonic properties using sensors that can operate on materials with a variety of shapes and in nonlaboratory environments. Furthermore, models that relate some of these properties to microstructures have been formulated in order to establish a scientific basis for the property/microstructure relationships. During FY1999, effort was concentrated on steel alloys that are embrittled by the formation of nanometer-sized copper-rich precipitates to see if ultrasonic or magnetic measurements could detect these microstructural features that profoundly affect the strength of steel alloys that contain small concentrations of copper. The various measurements used in this extensive program are briefly described below.

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* Dr. B. Igarashi was an NRC Post Doctoral Fellow for 1995-97 and continued his research on magnetostrictive ultrasonic transducers into early FY1999 as part of the Nuclear Regulatory Commission program.

** Dr. H. Ogi, Mechanical Science Division of the Graduate School of Engineering Science of Osaka University, Osaka, Japan, studied the interaction of carbon and copper atoms by low-frequency internal-friction measurements.

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 excite ultrasonic waves by producing the magnetic field with a coil of wire carrying an RF current at the frequency of the desired ultrasonic wave. The amplitude of the ultrasonic wave produced under these conditions can be used to measure the value of the magnetostrictive coefficients of the particular ferromagnetic material involved. In order to use such measurements as a nondestructive materials characterization tool, we have focused our research on verification of theoretical models relating the measured ultrasonic wave amplitudes to the magnetostriction coefficient and then on demonstrating the existence of correlations between the coefficient and precipitation hardening mechanisms in steels. During FY1999, 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.

Measurement of Magnetic Permeability: An important parameter in the equations that relate ultrasonic wave amplitude to the magnetostrictive coefficients is the electromagnetic skin depth because it determines the thickness of the layer in which the mechanical forces act to generate the ultrasonic wave. This parameter, in turn, depends on the frequency of the electromagnetic field, the magnetic permeability of the material and its electrical conductivity. Recent studies of eddy current NDE probes at Iowa State University showed that the inductance of an eddy current probe would not change as it moved toward the surface of a magnetic material if it was operating at a particular frequency. This frequency was directly related to the permeability. We used this technique to measure the relative transverse incremental permeability of a series of A710 steel samples that had been heat treated to various hardness levels by the formation of copper-rich precipitates (CRPs). We found that permeability could be correlated with hardness.

Ultrasonic Shear Wave Velocity: To use ultrasonics for measurement of stress in plates, an EMAT probe and instrumentation to measure the time-of-flight of shear waves across the thickness dimension of a plate were developed. We used this system to measure the velocity of shear waves in plates of A710 steel heat treated to different levels of hardness and in irradiated compact tension specimens of A533B reactor pressure vessel steels. An attempt to demonstrate a clear linear correlation between the shear-wave velocity and the hardness or degree of irradiation was not successful.

Elastic Moduli and Internal Friction: Resonant Ultrasonic Spectroscopy (RUS) is a powerful tool for making accurate measurements of all the elastic modulus tensor elements and their imaginary counterparts, the internal friction or damping capacity. It is a destructive technique in that it requires preparation of specimens with a regular geometry such as a sphere, cylinder or parallelepiped, but its results can be used to interpret any nondestructive measurement of an ultrasonic wave velocity or attenuation. We used it to investigate the effect of CRPs in the heat-treated A710 steel on all the elastic modulus tensor elements and their associated Q^{-1} values. It was found that the moduli increased by about 1 % as the isothermal annealing time increased and no extrema could be associated with the hardness maximum of peak aged condition. The internal friction, Q^{-1}, generally decreased as the annealing time increased.

Low-Frequency Internal Friction: Early in FY1999, a commercial torsion pendulum apparatus for measuring internal friction over the frequency range from 10^{-3} to 10 Hz was delivered and put into operation. Samples of the heat-treated A710 alloy were shaped to fit in this apparatus. The Snoek peak that arises from carbon atoms jumping between interstitial sites in the bcc iron lattice was studied as its shape and position changed following a treatment designed to put the carbon atoms in solution at the start of the experiment. As the hardness increased, the recovery rate of the relaxation strength increased and the activation energy of the relaxation process decreased, suggesting that the copper precipitation changes the kinetics of interstitial carbon diffusion. The results can be interpreted in terms of a 1970 theory of interstitial/substitutional–solute interaction and a Cottrell–Bilby recovery model.

External Collaborations

Professor G.R. Odette, UC Santa Barbara—guidance concerning the mechanism of embrittlement in reactor pressure vessel steels and in the A710 alloy steel.

Planned Outcome

This program, funded by the Nuclear Regulatory Commission, represented an opportunity to demonstrate the value of developing nondestructive evaluation techniques that can predict the strength of materials without performing destructive tests. As reactor pressure vessels age and are embrittled by radiation, their anticipated safe life must be predicted from tests performed on the vessel itself during routine maintenance operations. The materials-characterization studies in this project have revealed several new nondestructive techniques that can be applied to a pressure vessel in the field to predict its hardness a quantity known to be related to the ductile-to-brittle transition temperature used by regulatory agencies as a measure of embrittlement.

Accomplishments

- A noncontact ultrasonic technique for measuring the magnetostriction coefficient of a ferromagnetic material has been developed.

- The magnetostrictive coefficient of irradiated and copper-bearing steels has been shown to be correlated with the hardness (and embrittlement) of these materials.

- The transverse incremental permeability of a magnetic material can be deduced from nondestructive measurements of the inductance of a coil held against the material .

- The transverse incremental permeability can be correlated with the hardness of steel alloys hardened by formation of copper-rich precipitates.

- The Snoek effect can be used to study the kinetics of diffusion of carbon atoms in the vicinity of copper-rich precipitates.

Impacts

- An international workshop on Nondestructive Characterization of Embrittlement in Reactor Pressure Vessel Steels was hosted by NIST in Boulder, Colorado in July 1999.

- Experiments to establish improved models for describing the relationship between nondestructive ultrasonic measurements and the formation of copper-rich precipitates are being developed with the University of California in Santa Barbara.

- An ingenious scheme has been demonstrated for measuring the magnetostriction in the A533B steel under the stainless steel cladding inside a reactor pressure vessel.

Outputs

Publications

G.A. Alers and H.I. McHenry, Nondestructive characterization of reactor pressure vessel steels: A feasibility study, *NIST Technical Note 1500-4* (1999).

G.A. Alers, D. Hurley, B. Igarashi, et al., Nondestructive detection of dopper embrittlement of steel, *Proceedings of the 9th International Symposium on Nondestructive Characterization of Materials* (Sidney, Australia, June 1999).

H. Ogi, H. Ledbetter, and S. Kim, Snoek relaxation in copper-precipitated alloy steel, *Proc. Int. Conf.on Internal Friction and Ultrasonic Attenuation in Solida (ICIFUAS-12)* (Buenos Aires, July 1999).

Presentations

G.A. Alers et al. "Nondestructive characterization of RPV steels: A feasibility study," International Group on Radiation Damage in Pressure Vessel Steels, Nashville, Tennessee, January 1999.

G.A. Alers and H. McHenry, "Ultrasonic and Magnetic Sensing of radiation Embrittlement," SmiRT 15, University of Tokyo, August 1999.

D. Balzar, D. Hurley, B. Igarashi, et al., "Embrittlement in Copper-Bearing Steels by Coherent Precipitates: Characterization by SANS, XRD, Ultrasonic and magnetic Methods," RTAF Meeting, Charleston, South Carolina, September 1999.

Project: SENSORS FOR INDUSTRIAL NDE

Principal Investigators: G.A. Alers, A.V. Clark, B. Igarashi, R.L. Santoyo

Objective

Producing industrial quantities of materials with specified properties requires sensors that can survive in the mill environment and monitor the key variables during critical processing steps. In addition, rugged and reliable sensors are needed after the material is manufactured and put into service in order to make sure that there has been no degradation of important mechanical properties during service. The objectives of this program are 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 such as on composites, structural steels, plastics or ceramics. Furthermore, the sensors often need to be designed to meet the requirements of the environments in which they will be used such as in a buried gas pipeline, a radioactive reactor pressure vessel or on a rolling mill. During this year, five industrial sensor problems have received attention. First, the gas-coupled ultrasonic transducer was tested for its capabilities as an ultrasonic thickness gage for measuring corrosion damage in buried natural-gas pipelines from a fast moving, internal robot called a pig. The second sensor was used in a feasibility study sponsored by the Gas Research Institute and Battelle Memorial Institute to see if ultrasonic measurements of pipeline coating integrity could be added to a pig system designed to detect corrosion damage by the magnetic flux-leakage technique. Third, a rotating EMAT coupled to the new portable ultrasonic inspection unit was tested as a mobile strain gage to measure stress distributions in the beams of large structures as well as the residual stresses near welded joints. Its calibration is described in the project entitled "Ultrasonic Measurement of Stress". Lastly, two new sensors were developed in order to meet the needs of the Nuclear Regulatory Commission's program to develop nondestructive techniques to monitor radiation induced embrittlement of in-service nuclear reactor pressure vessels. One exploited the use of magnetostriction as a physical property related to the microstructural changes that increase the yield strength and cause embrittlement of the steels used in pressure vessels. The other was an eddy current sensor that measures a dynamic magnetic permeability that can also be related to embrittlement. Details concerning the construction and calibration of these sensors are described in the project entitled "Nondestructive Characterization of Steel Embrittlement."

External Collaborations

Southwest Research Institute—gas coupled ultrasonic inspection of buried gas pipelines.
Caterpillar Inc.—measurement of residual stresses near welded joints.
Battelle Memorial Institute—measurements of coating integrity on buried gas pipelines.
University of California in Santa Barbara—collaboration in the study of radiation induced embrittlement.
Gas Research Institute—inspection of buried gas pipelines.
Nuclear Regulatory Commission—inspection techniques applicable to detecting embrittlement in nuclear reactor pressure vessels.

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 problems in flaw detection and materials characterization that have eluded conventional NDE methods.

Accomplishments

Air-Coupled Transducers for Gas Pipeline Inspection: The gas industry needs better methods for inspecting their buried 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. However, their use for gas-pipeline inspection has been impeded by the difficult problem of providing a suitable means to couple the ultrasound from the transducer into the pipe wall. In the past, NIST demonstrated the feasibility of using the compressed gas itself as the ultrasonic couplant and a cooperative research program with the Gas Research Institute and Southwest Research Institute was initiated 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 with 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 by 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 gauging technology currently being used in liquid-filled pipelines could be implemented in a gas pipeline by using properly designed gas-coupled transducers. Several commercial manufacturers of custom transducers were asked to supply piezoelectric transducers with the required bandwidth and dynamic range. Unfortunately, tests performed in the NIST high-pressure chamber using the NIST custom pulser/receiver electronics showed that extraneous vibrational modes in the piezoelectric disc obscured the key reflection from the OD of the pipe wall from which the wall thickness is deduced. By using an angle-beam pitch-catch configuration, the transmitter and receiver transducers could be separated and the arrival time of the key reflection from the OD surface could be resolved. Tests of this configuration were carried out both by computer simulation and by experiments in a water bath. The results showed that more conventional ultrasonic transducers should suffice, and experiments in the high-pressure gas chamber are currently underway. These tests are designed to demonstrate that the remaining wall thickness in a corroded sample of pipeline steel can be measured with the required accuracy by using the high-pressure gas as the ultrasonic wave-coupling medium.

Detection of Loose Coating on a Buried Gas Pipeline with a Magnetic Pig: The most common pig used for inspection of buried gas pipelines throughout the world applies a strong magnetic field to the pipe wall and detects anomalies in the tangential component of this field at the inner surface of the pipe caused by corrosion-induced pipe wall thinning. Since the inspection method is based on the magnetic properties of steel, this type of pig cannot sense any properties of the nonmagnetic coating used to protect the pipe from external corrosion. Ultrasonic waves in the pipe wall, on the other hand, are very sensitive to the integrity of the coating. NIST designed and built an EMAT that would use the magnetic field produced by a magnetic-inspection pig to excite and detect ultrasonic waves in the pipe wall. By properly choosing the frequency of operation and the type of ultrasonic Lamb wave used, this EMAT was able to locate regions of disbonded coating in a 20-foot-long pipe sample maintained for coating studies at Battelle's Pipeline Simulation Facility in Ohio. An enabling technology for the success of this project was the availability of compact instrumentation that could be attached to the magnetic pig and operated inside the pipe as the pig moved it. This instrumentation is described in the paragraph below.

Portable Instrumentation for Measuring Stress in Structural Beams: Late in FY1998, Sonic Sensors, Inc. provided NIST with a rugged portable computer, a compact transmitter/receiver electronic package and a motor-driven EMAT designed to automatically measure ultrasonic birefringence in metal plates. During FY1999, the system was developed further by the addition of software and hardware to measure the transit times of the fast and slow shear waves used by the birefringence technique more accurately and rapidly. Furthermore, the orientation of the principal stress axes relative to the texture axes in rolled plate could also be measured. Details of the system's use are described in the project entitled "Ultrasonic Measurement of Stress."

Impacts

- An ultrasonic thickness gage that uses the high-pressure gas as a coupling medium was demonstrated in the laboratory for measuring the remaining wall thickness in a corroded region of a buried gas pipeline.

- An ultrasonic sensor attached to a conventional magnetic-inspection pig was able to add the detection of disbonded coating to the detection of corrosion pits in the steel walls of buried gas pipelines.

- Portable instrumentation attached to a rotating EMAT is now available for measuring stress distributions from applied loads and residual stresses near welds in the beams used for large structures such as bridges, buildings and construction machinery.

- Noncontact ultrasonic and eddy-current sensors have been developed for detecting the microscopic precipitates that embrittle steels containing small amounts of copper, as are found in reactor pressure vessels and some other structural applications.

Outputs

Publications

M. Renken and C.M. Fortunko, Development of gas-coupled ultrasonic inspection for pipelines—Phase II, *Reports to the Gas Research Institute.*

G.A. Alers, H. McHenry, D. Hurley, et.al., Nondestructive characterization of RPV steels—Phase II, *Nuclear Regulatory Commission Final report on NRC Program* RES-97-005.

Presentations

J. Bruce Nestleroth, Detection of loose coating from an MFL pig, GRI/PRC NDT Committee Meetings in Seattle, Washington (May 1999) and Colorado Springs, Colorado (August 1999).

Patent Application

A.V. Clark, C.S. Hehman, T.N. Nguyen, and G.A. Alers, Ultrasonic strain gage using a motorized EMAT, U.S. Patent Application No. 09/274,538. NIST #98-020US (1999).

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 strongly on the conditions under which the materials are fabricated. These distributions are crucial in determining properties such as strength, ductility, homogeneity, conductivity, and others which are 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 powder metallurgy, electroplating, electronics and aerospace 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. Through studies of the mechanistic, chemical and process variables controlling the structure/properties of electrodeposited coatings and thin films, rational approaches are being developed for the optimization of electrochemical processing conditions to obtain the desired deposit characteristics.

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

- Measurements are made under dynamic conditions to monitor, in real time, materials characteristics during metals processing operations. Special fast-response sensors, simulations and imaging techniques have been developed for application to powder atomization, thermal spray, and welding processes. These sensors are used in conjunction with dynamic models of the process both for design of manufacturing procedures and for applications of real time feedback and control;

- The physical characteristics of electrodeposits are related to electrolyte chemistry and deposition conditions. This work includes measurements of the deposition characteristics of corrosion-preventing aluminum alloys deposited from room temperature molten salts, measurements of the mechanical properties of chromium alloys deposited from environmentally friendly trivalent chromium electrolytes, and measurement of the effects of organic additives on recrystallization of electrodeposited copper used in on-chip interconnects;

- Understanding of metals processing relies on models which relate the behavior of the metal to physical property data. Models are developed describing solidification and solid-state transformations in terms of basic thermodynamic and kinetic properties of industrially important alloy systems.

Project: WELD PROCESS SENSING, MODELING, AND CONTROL

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

Technical Objectives

The objectives of this project are to develop a better understanding of the underlying physics governing arc-welding processes through advanced instrumentation and data analysis techniques; develop simple, nonintrusive, and robust sensors that provide meaningful information about the status of the welding process for real-time monitoring and control; develop theoretical models for GMAW to understand the relationship between input parameters and the resulting process; assist in the development of industry standards for information exchange between intelligent components in a robotic arc-welding cell (for example, robot controller, welding power source, etc., and support other government agencies and U.S. industry with welding expertise.

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. Many of our sensing schemes rely on using model-based algorithms that use the inherent process variables: the current and the voltage; it is sometimes called "through-the-arc sensing." The principal advantage of this sensing scheme is that the external sensors can be placed at the power supply, well away from the robot and torch. We have developed the arc-sensor module (ASM) which makes use of simple voltage and current sensing to provide quality information about the welding process. The ASM outputs seven different quality measures thirty-two times per second. Based on the quality measures, the ASM decides whether a defect has occurred.

Welding engineers who design the welding processes 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 engineering consulting firms for small companies). If a problem occurs, the engineer is summoned while the production line is (expensively) idle. One 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. Therefore, methods need to be developed to deliver sensor data remotely. We have developed methods to handle large amounts of data and deliver them to the remote welding engineer. We demonstrated these concepts by modifying the ASM.

Our welding laboratory includes the following special equipment:

- Four personal computers outfitted with high-resolution, high-speed data collection boards;
- Extensive analysis software and software-development tools;
- Analog signal conditioning and isolation manifolds;
- Current, voltage, wire feed speed, and light-intensity transducers;
- oscilloscope, function generator, and electronic signal spectrum analyzer;
- Optical spectrum analyzer (200 nm to 1000 nm range, 0.01 nm resolution);
- High-speed video system with laser back-lighting;
- Standard video cameras and VCRs;
- Video frame-grabber and image processing systems;
- Six-axis robotic manipulator, three track-type manipulators;
- Conventional SCR, inverter, and transistorized welding power sources;
- Four-roll and capstan wire-feed units.

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

Another significant problem, especially for gas metal arc welding (GMAW) of aluminum, is feeding the wire. Inconsistent feeding causes poor quality welds. Our models have shown that aluminum is very sensitive to variations in wire-feed speed. The mechanical wire-feeding technology has remained basically unchanged for many years and is not fully understood. We are developing models of the feeding process from the pinch rollers in the wire feeder to the exit at the contact tube.

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.

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 data output by the sensors, the weld cell status, and the path information of the robot controller. 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

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

Dave Farson, Professor of Welding Engineering, Ohio State University—works with Timothy Quinn to verify the models of GMAW

Dave Munoz, Professor of Engineering, Colorado School of Mines—works with Timothy Quinn on guiding a CSM Student, Toby Padilla, on a study of the wire feeding in GMAW.

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

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.

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

Accomplishments

We developed a data-management system for the ASM based on Internet protocols. The sensor reports its data over the Internet (or an intranet) to a program that buffers the data, then places it in a database. The database is accessed with a standard database protocol (Open DataBase Connectivity, ODBC). Weld engineers can use a visualization program that we wrote to access the database through the ODBC protocol. The engineers can then monitor and troubleshoot the welding station. The visualization program is written in a generic programming language that can run on almost any platform. The large amounts of data are managed easily using generic or specific scripts developed for databases.

In collaboration with researchers at the Colorado School of Mines, we have developed a model of the wire as it is being pulled through the conduit and can predict the friction between the wire and the liner. The model is now being extended to cases where the wire is pushed through the conduit (the wire buckles in this case).

A coupled model of gas metal arc welding was developed and implemented using a commercial finite-element code. In the GMAW process, a metal electrode carries a large current (e.g., 300 A). An arc is created between the end of the electrode and the work piece in the inert shielding gas. The electrode melts and droplets detach and travel into the molten welding pool. The important physics of the process were identified and the resulting set of partial differential equations were solved with a eombination of commercial software and software that we wrote. The solution was tested for convergence, mesh size, and sensitivity to important parameters.

To spread the knowledge of recent developments in welding technology, we helped to organize the Ninth International Conference on Computer Technology in Welding, held September 28 to 30, 1999 in Detroit, Michigan. NIST staff participated by giving presentations, giving tutorials, and serving as session chairs. The 58 presentations gave a good summary of the status of the use of modeling, sensing, and control in welding. The proceedings will be published as an NIST Special Publication.

General knowledge of our work in welding often brings request for help on welding and general materials problems at other government agencies. This year we published some recent studies on the failure of a chemical blender (at the request of OSHA) and the failure of reinforcing wires in large water siphons (at the request of the Department of the Interior). In addition, we developed weld repair procedures for cracks in the dome of the U.S. Capitol.

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

J. Gilsinn, W. Rippey, J. Falco, T.P. Quinn, B. Russell, and K. Stouffer, A welding cell that supports remote collaboration. *Ninth International Conference on Computer Technology in Welding Proceedings*, T.A. Siewert, ed. (1999).

T.P. Quinn, C. Smith, C.N. McCowan, E. Blachowiac, and R.B. Madigan, Arc sensing for defects in constant-voltage gas-metal-arc welding, *Welding Journal*, **78** 322s–328s (1999).

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T.A. Siewert, IIW Commission V—Quality Control and Quality Assurance of Welded Products: Annual Report 1998/99, *NISTIR 5081, IIW Doc. V-1123-99* (January 1999).

T.A. Siewert, The International Standards System and NDE, *Review of Progress in Quantitative Nondestructive Evaluation, 18B*, Plenum Press, 1217–1224 (1999).

T.A. Siewert, The International Institute of Welding: Report on 1998 Actions, *Materials Evaluation,* **57** (5), 494–498 (May 1999).

C.N. McCowan and T.A. Siewert, Failure analysis of a large blender in a chemical plant, *Microstructural Science,* **26**, 115–122 (1999).

C.N. McCowan and T.A. Siewert, Metallurgical evaluation of prestressed wire failures, *Microstructural Science*, **26**, 127–136 (1999).

T.A. Siewert, C.N. McCowan, and R.A. Bushey, Development of weld repair options for the U.S. Capitol dome, a report to the Architect of the Capitol, Washington, D.C., (1999).

T.A. Siewert, T.P. Quinn, W. Rippey, and L.A. Flitter, Communication between components in a robotic weld cell, a poster on remote monitoring of welding that won an Honorable Mention Award at the 1999 American Welding Society Meeting, April 1999.

Project: THERMOMECHANICAL PROCESSING

Principal Investigators: Y.-W. Cheng, P.T. Purtscher

Objectives

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

Technical Description

Our research interest centers on development of quantitative relationships between the mechanical properties of steel, steel chemistry, and the thermomechanical processing parameters used during steel rolling and forging. Emphasis is on the characterization of the kinetics of microstructural evolution during hot working. Several important factors are included, namely, precipitation effects (carbides, nitrides, and carbonitrides), grain growth and recrystallization (static and dynamic recrystallization), and austenite decomposition. Specifically, constitutive equations under hot-rolling conditions are being developed and composition-structure-property relationships at ambient temperature are being characterized for specific plain-carbon and microalloyed steels.

External Collaborations

University of British Columbia (UBC), Vancouver, British Columbia—The Center for Metallurgical Process Engineering 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.

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.

Planned Outcome

This project develops mathematical models that describe the constitutive behavior under hot-rolling conditions and the structure-property relationships at ambient temperature of steels. These models are incorporated into integrated process models that simulate steel rolling processes and predict mechanical properties of hot-rolled products.

Accomplishments

Research activities on the project of microstructural engineering in hot-strip mills were completed in 1998. In 1999, we completed two NIST Technical Notes based on the research results. The accomplishments for the research included the development of constitutive-behavior models that are suitable for predicting stress-stain behaviors of the following seven steels: A36, DQSK, 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 °C to 1300 °C), strain rates (1 to 400 s^{-1}), and austenite grain sizes (20 μm to 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 whieh 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.

Equations for predicting mechanical properties of a steel from its composition and microstructure at ambient temperature were also developed and validated for plain-carbon and high-strength and low-alloy steels produced on hot-strip mills. 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.

Impacts

The equations developed for the constitutive behavior and the structure-property relationships were successfully incorporated into a process model that was 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 were disseminated to AISI member companies through project-review meetings and workshops.

With the successful completion of this project, the Timken Company, one of the AISI member companies, is planning to extend the technology of thermomechanical processing from flat rolling to tubes and pipes manufacturing. For that purpose, the Timken Company, with some input from NIST, submitted a proposal, "Controlled Thermomechanical Processing of Tubes and Pipes for Enhanced Manufacturing and Performance," to the Office of Industrial Technologies, Department of Energy. The proposal was recently funded.

Publications

Y.-W. Cheng, R.L. Tobler, B.J. Filla, K.J. Coakley, Constitutive behavior modeling of steels under hot-rolling conditions, *NIST Technical Note 1500-6,* (April 1999).

P.T. Purtscher, Y.-W. Cheng, C.N. McCowan, Structure-property relationships in steel produced in hot-strip mills, *NIST Technical Note 1500-7,* (August 1999).

Project: FORMING OF ALUMINUM ALLOYS

Principal Investigator: Y.-W. Cheng, J.D. McColskey

Objective

The objective is to develop test specimens from which data compatible with finite-element formability analyses can be generated.

Technical Description

One of the problems identified in using aluminum sheets for automotive applications is the inability to efficiently produce components within dimensional tolerances. This results in the need to physically redesign and rework forming dies, which is both costly and time-consuming.

Finite-element simulation of the forming operations is one way to reduce the cost and time in redesign and rework of the forming dies and to efficiently improve the forming operations. However, current test methods such as tensile and formability tests do not provide the necessary measurement data for accurate finite-element analyses. This project will develop test specimens from which the needed data compatible with finite-element analyses can be generated.

The test specimens should be simple uniaxial tensile panels that during a test produce a uniform multiaxial strain state that simulates the majority of the aluminum forming operations. To fully characterize the strain-state and plastic-flow behavior of the specimen during test requires a full-field strain measurement.

The proposed technique for achieving the full-field strain measurement is the digital-image correlation technique. During a test, images of the specimen surface are constantly acquired. From the images, full-field displacement and strain are determined. The determined strain field with the loading information will provide not only the mechanical properties of the material such as yield stress, ultimate tensile strength, and percent elongation, but also the full-field strain pattern and the development of Luder's bands. This information can be fed into finite-element code for simulation of aluminum forming operations.

External Collaborations

Brian Armstrong, University of Colorado—collaboration on digital-image-correlation technique.

Planned Outcome

Simple tensile test specimens of aluminum alloys are expected to be developed, from which the needed data for input to finite-element modeling of forming operations will be generated.

Accomplishments

The apparatus for loading tensile panels with the capability of constant image and load acquisitions has been set up. A software that performs digital-image-correlation analysis was acquired, installed and tested. The apparatus and the software are being used to characterize the strain pattern of tensile panels of the 2024-T3 aluminum alloy. Initial results of testing 88.9 mm wide tensile panels showed that specimens were under predominately plane-strain condition. Mechanical properties of Young's modulus, yield stress, and ultimate tensile strength determined from the tests were consistent with values cited in the literature. Results were sent to Metallurgy Division for incorporation into the finite-element analysis.

The digital-image-correlation software acquired for this project is also being used to analyze the images that are acquired during testing microelectronic materials with the microtensile testing apparatus.

Project: **HIGH-ENERGY X-RAY DIFFRACTION STUDIES OF MATERIALS PROPERTIES**

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

Objective

The project objective is to demonstrate that high-energy x-ray diffraction (XRD) can be used for nondestructive characterization of microstructure, especially the texture and stress state of polycrystalline materials.

Technical Description

In addition to our project that uses conventional low-energy x-ray diffraction for characterizing materials, we have been developing procedures that demonstrate the benefits of high-energy x-ray diffraction. The greatest advantage of high-energy x-ray diffraction is the rapid increase in penetrating capability as the x-ray tube potential increases. For instance, the effective penetration depth for nickel is 450 times larger for 320 keV than for 8 keV x-rays, (a commonly used copper K-alpha line radiation). In addition, the x-rays are able to penetrate the containers used in industrial processing, such as a casting mold and other furnace components.

As members of the research team of the NIST Consortium on Casting of Aerospace Alloys during the past few years, 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 keV to 320 keV) to penetrate through material surrounding the casting and to produce a distinctive diffraction pattern that 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.

The success of the XRD solidification sensing technology has led us to investigate 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. Along these lines, we have discovered that a scanning x-ray source has special advantage in some diffraction applications.

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* Guest Researcher from the Department of Physics, University of Colorado at Boulder.

External Collaborations

Digiray Corporation—collaboration to demonstrate the particular utility of a scanning x-ray source in rapidly characterizing the structure of materials.

Alumax Corporation—has furnished aluminum castings produced by their semisolid castings process, and which were rejected during final inspection. We are investigating the capabilities of the diffraction technique to identify regions of the castings in which the semi-solid mixture separated into eutectic and proeutectic-rich areas.

Planned Outcome

We intend to demonstrate that high-energy x-rays can characterize the texture and stress state of bulk polycrystalline materials, now usually obtained by neutron and high-energy synchrotron diffraction.

Accomplishments

We completed reporting on our technique to study the solidification of a single-crystal turbine blade casting within its mold. In particular, we reported the ability of a properly oriented and narrow beam of x-rays to diffract from the individual dendrites in the solid-liquid region of a directionally solidified casting, producing a topographic slice of the dendrites. The interpretation of the internal structure (intensity) of the diffraction spot as being due to dendrites was confirmed by observing corresponding changes in the pattern as the specimen was rotated.

On 12 August 1999, Digiray Corporation received the exclusive license to a joint NIST–Digiray invention, "Method and Apparatus for Diffraction Measurement Using a Scanning X-ray Source." Digiray will take over the responsibility for developing the method into commercial products. The NIST inventors were Bill Dubé, Dale Fitting, and Tom Siewert.

Based on the success of transmission x-ray diffraction to study the solidification of a single-crystal turbine blade casting within its mold, we have completed installation of a new laboratory for high-energy x-ray research. It is designed for characterizing the texture, strain, and other microstructural properties of polycrystalline materials. Furthermore, we plan to extend capabilities to microstructural characterization of polycrystalline and epitaxial thin films and devices. The facility is equipped with several x-ray sources, a four-circle goniometer, a real-time x-ray imaging system, and an energy-sensitive solid-state detector. Please refer to our web site for current information on our research in high-energy x-ray diffraction:

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

Publications

D.W. Fitting, W.P. Dubé, and T.A. Siewert, High-energy x-rays monitor solidification, *Advanced Materials and Processing*, **155**, 43–45 (February 1999).

CERAMIC COATINGS

The Ceramic Coatings Program addresses the development of measurement and characterization methods that will improve processing reproducibility and performance prediction of ceramic coatings. The program addresses plasma spray deposited and physical vapor deposited ceramic thermal barrier coatings (TBCs) 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 nearly one billion dollar North American ceramic coatings market. Collaborations have been established with industrial organizations to enable research on relevant materials and to transfer results to users. Collaborators include Pratt and Whitney, General Electric, Caterpillar, METCO, Praxair Coating Technologies, 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 Aerospace Laboratory, in Japan, to examine functionally gradient materials. Collaborations are also underway with Bundesanstalt fur Materialfurschung und -prufung) (BAM) and Deutsche Forschungsanstalt für Luft-und Raumfahrt (DLR) , both in Germany, for the development of characterization techniques for thin, hard coatings and TBCs.

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

The approach taken in the 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 plasma-spray (PS) powder;

- Development of neutron 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;

- Development of techniques for accurate measurement of the thermal conductivity of PS and physical vapor deposition (PVD) coatings, by use of the guarded hot plate technique suitable for incorporation in ASTM standards, as well as development of monolithic, polycrystalline zirconia Standard Reference Materials, and by the pulsed laser heating technique, to provide a method for comparison with routine industrial techniques;

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

Research on chemical mapping of powders and microstructures is conducted in the Microanalysis Division of the Chemical Science and Technology Laboratory. Thermal property research is conducted in the Materials Reliability and Metallurgy Divisions. The NIST Center for Neutron Research participates in phase analysis, elastic properties determination, 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-microstructure-property relationships.

Project: THERMAL CONDUCTIVITY OF THERMAL-BARRIER COATINGS

Principal Investigator: A.J. Slifka

Objectives

The objectives are to develop methods to measure the thermal conductivity of ceramic coatings and to relate their thermal performance to the microstructure of the coatings. Measurements are made of the thermal conductivity of representative thermal barrier coatings, substrate materials, and monolithics to determine bulk values and interfacial resistances for the coating systems. Evaluations are made of the applicability of microscopy and spectroscopy techniques to the various microstructural analyses. Observations of the effects of varying processing parameters and processing techniques are related to microstructural features and bulk thermal conductivity. The bulk thermal conductivity and microstructural information can be used in conjunction with processing parameters to develop a model that generates desired bulk thermal conductivity given appropriate processing parameters. Interactions with the coatings industry provide a supply of state-of-the-art samples. Development of steady-state measurement apparatus and techniques, appropriate reference materials and documentation to allow comparison with, and calibration of, transient measurement techniques to transfer to industry.

Technical Description

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

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

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 has been constructed for these measurements. Infrared microscopy is used to monitor heat flow on the micron scale and to measure the thermal conductivity of coatings and the thermal resistance between interfaces. Successful measurements of these constituent features with the infrared microscope will allow modeling of the heat flow in coatings that generates bulk thermal conductivity values as obtained from the guarded hot plate.

Thermal conductivity measurements of two ceramic thermal barrier coatings and two monolithic ceramic systems were completed this year using the guarded hot plate. Twenty-four physical-vapor-deposited thermal barrier coatings were measured using the infrared microscope system.

Special facilities used include an infrared microscope system capable of 5 μm spatial resolution from room temperature to 500 K and the world's only guarded-hot-plate thermal conductivity apparatus operating from 400 K to 1300 K.

External Collaborations

DLR (Germany)—collaborating with U. Leushake, U. Steinhauser and W.A. Kaysser of the German Aerospace Research Establishment in determining bulk and microscale thermal conductivity of developmental coatings and FGMs.

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

Pratt & Whitney—completed measurements of thermal conductivities of very thin monolithic coatings under a CRADA.

University of Virginia—collaborating with Derek D. Hass to develop 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 conductivity seen in industrial thermal barrier coatings. Ultimately this will enhance the competitiveness of U.S. industry by providing accurate measurement techniques and standards as well as reliable data on state-of-the-art thermal barrier coatings. This will lead to better material evaluation and the ability to design higher performance systems.

Accomplishments

Measurements of both 5.3 wt. % and 9.1 wt. % yttria-stabilized zirconias were completed. These materials are predominantly tetragonal and cubic, respectively, and are candidates for high-temperature thermal-conductivity standard reference materials (SRMs). Measurements show that both materials provide results with very good repeatability. Due to the difference in phase composition, the thermal conductivities of these two materials are different. This is significant and useful for industry, as the use of different processing parameters results in different phase compositions of ceramic thermal-barrier coatings. Through a collaboration with J.K. Stalick of NIST, Gaithersburg, high-temperature annealing and neutron-diffraction measurements have shown that the 9.1 wt. % material has good high-temperature phase stability. Measurements of electron-beam physical-vapor-deposition (EB-PVD) coatings from Pratt & Whitney were completed 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. Two functionally graded coatings from NAL/KRC, Japan were measured. The collaboration with the Japanese includes thermal shock experiments to determine how increases in microcrack density affect thermal conductivity. Two thicknesses of a plasma-sprayed mullite material from DLR in Germany were measured.

The infrared microscope system for doing comparative, steady-state measurements of thermal conductivity of thermal barrier coatings has measured 24 coatings this year. The EB-DVD coatings from the University of Virginia have controlled microstructures designed to enhance phonon scattering. We have been correlating the various process parameters used to generate coatings with the lowering of 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 for a number of specimens. X-ray and neutron diffraction were used to determine phase content in monolithic ceramics and a ceramic coating.

We are continuing development of an infrared microscopy system to measure interfacial thermal resistance at ceramic/metal boundaries. These boundaries constitute a significant thermal barrier, and happen to be the location of primary failure in thermal barrier coatings. We are pursuing a collaboration with Dr. F. Schmidtz of the Technical University in Aachen on this subject.

Publications

A.J. Slifka, A comparative, steady-state apparatus for measurement of thermal conductivity of thermal barrier coatings, *Surface and Coatings Technology*, submitted.

A.J. Slifka, B.J. Filla, J.K. Stalick, Thermal conductivity of two compositions of yttria-stabilized-zirconia, *Thermal Conductivity 25,* submitted.

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 only obtainable by the use of unique measurement tools at NIST like the neutron diffraction facilities at NCNR, or the magneto-optic indicator film apparatus for observation of magnetic domain motion. Of particular interest is understanding the magnetic behavior of low-dimensional systems, in which one or more characteristic dimensions have been reduced to nanometer sizes. For these new materials, however, it is not known whether their exciting novel behavior is due to new physics or to a logical extension of large-size behavior to small dimensions. Consequently, implementation of this new type of material into marketable products is significantly delayed. NIST is providing the measurement science to address this critical unknown.

Areas of present study include the following:

- Preparation and measurement of "spintronic" systems wherein spin-dependent magnetic devices are integrated directly onto semiconductor chips;

- 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, ion-implanted steel surfaces, and weld metal ferrite standards;

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

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

- Development of magnetic sensors of mechanical properties for incorporation as in situ controls in a steel mill;

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

- Preparation of magnetic measurement standards.

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 Investigators: F.R. Fickett, W.P. Dubé

Objectives

The project objectives are to apply existing magnetic techniques to microscale characterization of films, coatings, engineered surfaces, and bulk materials and to develop new measurement methods allowing improved detection capability or increased portability. Currently available techniques include: force microscopy (magnetic, electric, atomic) and magnetometry using vibrating sample, Hall effect, and superconducting quantum interference device (SQUID) systems. A new method, the magnetoresistive microscope, is under development with the objective of creating an inexpensive magnetic scanning system that operates on the micrometer scale.

Technical Description

Our work in FY1999 involved experimental work on three projects. The first is magnetic evaluation, primarily using the magnetic force microscope (MFM), of ion implanted surfaces on nonmagnetic ferrous alloys, which have both interesting physics of thin magnetic films and potential value as an evaluation technique for surface integrity. The second effort is development of a magnetoresistive microscope (MRM) primarily for application in electronic packaging to detection of current flow in interconnect lines, although it has the potential to be a general purpose, and inexpensive, field detection device. Applied in microelectronics, it offers potential for assessment of line quality and the presence of defects, a problem that is becoming critical as line sizes reach the submicron range. The final project is development of new methods for measuring ferrite content and structure in weld-metal 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 surface condition 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 such as surface integrity, chemical resistance, hardness, or friction coefficient that are of interest in applications.

The MRM work uses microscopic magnetoresistive field sensors to map fields from regions in the 1 μm to 100 μm size range. In concept, these devices can be configured as large-area scanning systems. Because they are not especially sensitive to environment and are relatively inexpensive and robust, they have potential for application in manufacturing environments. They are prepared by conventional deposition and lithography techniques; packaging methods and control electronics are well in hand for many (but not all) applications. The main effort in FY1999 was in developing a technique for removing the temperature sensitivity of the probes and a proof-of-concept experiment to map operating high-frequency microcircuits.

Weld-metal 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 eontent 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. In FY1999 we evaluated a prototype test device based on electrical excitation and field measurement.

External collaborations

P. Wilbur and D. Williamson, Colorado State University/Colorado School of Mines—continuing collaboration on magnetic measurements of ion-implanted stainless steel.

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, Hall, etc.) 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 method for calibrating secondary standards.

Accomplishments

An unimplanted sample was imaged with the force microscope in atomic force mode (AFM) over several regions, then imaged in the scanning electron microscope (SEM) fitted with an orientation imaging microscope (OIM) attachment, that allows identification of the complete crystal orientation of the grains. The sample was then implanted and the MFM image made. Comparison of the images allowed correlations to be made between grain orientation and domain structure.

The MRM work last year predicted the temperature independence of the second-harmonic signal. Investigation, primarily in FY1999, showed that, if we used ac current to drive the head, and detected at the second harmonic of the drive frequency, the temperature signal was eliminated.

This discovery resulted in the paper referenced below. A single series of measurements was made on a line carrying high-frequency current that demonstrated the potential of the system.

A prototype magnetizer/demagnetizer/Hall-probe device was used to measure a set of the weld ferrite standard blocks. The detector was several times larger than optimum, but the data indicated good agreement with the traditional measurement methods. Magnetic force microscope images for a complete set of standard blocks were acquired and processed. We plan to publish a catalog of these images in association with other work on the standards.

Publication

W.P. Dubé, F.R. Fickett, A.B. Kos, and T.J. Silva, Removing temperature sensitivity of magnetoresistive elements for DC field sensing, *Review of Scientific Instrumentation,* submitted.

STANDARD REFERENCE MATERIALS

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

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

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

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

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

Project: CHARPY IMPACT MACHINE VERIFICATION

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

Objectives

The project objectives are to 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; and to 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, an 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 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 J 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 FY1999, a gradual increase from the customer base of a few years ago. The great majority of these machines were within tolerances required by ASTM Standard E23, 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 650 faxes and 2000 phone calls. In our laboratory, we tested the 1250 specimens necessary to confirm that 14 new lots of reference specimens were suitable to go into the SRM inventory. This year, we implemented a new procedure to contact every customer that fails to meet the verification criteria. In this contact (by phone, mail, or fax), we suggest corrective measures.

We helped to organize an international symposium, Pendulum Impact Machines: Procedures and Specimens for Verification, that was held in conjunction with the May 1999 meeting of ASTM Committee E28 in Seattle, Washington. The 26 papers presented at the symposium are now being reviewed for inclusion in an ASTM Special Technical Publication. NIST authors contributed to four of the papers.

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 FY2000, and we plan to produce a report that summarizes all the work. A new workshop is tentatively scheduled for FY2000 or 2001.

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 round robin evaluation to the task group. We consider that this data will be used to produce plastic SRMs, as needed. We have also just completed our part of a round-robin evaluation of subsized metal specimens, that might be used to evaluate the performance of the machines that test the subsized specimens used to monitor power-plant steels.

Chris McCowan serves as the Chairman and U.S. Delegate to ISO TC164 SC4 P, on pendulum impact. Chris McCowan also 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 (fraction 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.

Publications

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D.P. Vigliotti, C.N. McCowan, and T.A. Siewert, Maintaining the accuracy of Charpy impact machines, *Pendulum Impact Testing: A Century of Progress, ASTM STP 1380,* T.A. Siewert and M.P. Manahan, Sr., eds. (American Society for Testing and Materials, West Conshohocken, Pennsylvania, 1999) submitted.

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Project: FERRITE IN STAINLESS STEEL WELDS

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

Objectives

The project objectives are to maintain the system to assign ferrite numbers (FNs) to stainless weld metal specimens, so that the standards sold by NIST will be consistent with previous sets in use around the world; and to 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 stainless steel samples. In section 3.2, the standard describes the importance of secondary standards as: the only way of calibrating instruments for which no primary calibration method exists; the most appropriate standard for in-process checks; and being much more durable than the primary standards.

The secondary standards are arranged in two sets, a lower range set (RM 8480) with eight specimens that are distributed over the range of 0 to 30 FN and a higher-range set (RM 8481) with eight specimens that are distributed over the range of 30 to 100 FN.

In FY1999, we worked with SRMP to finish the certificate and reports of type 1 and type 2 errors, so that the 30 sets of secondary standards that were measured in FY1998 could be delivered and enter SRMP inventory. By the end of the fiscal year, several of these sets had been sold.

In addition, we responded to the international committee that oversees the procedures by procuring sufficient raw material for 55 additional sets. So far, we have assigned reference values to 14 more sets of RM 8480 and are working on the rest.

Our measurements identified several previously overlooked variables in the procedures and the instruments used to make the FN measurements. We have made the appropriate changes to the previous calibration procedure for the assignment of certified FN values to the standards. In addition, our measurements showed several ways in which the procedures in Standard AWS 4.2 might be improved. This data was presented to the national and international oversight committees this year. We presented this data to the Welding Research Council Subcommittee on Welding of Stainless Steel on May 11 (hosted by NIST in Boulder) and to Commission II of the International Institute of Welding (IIW) on July 22, 1999. A public report was given at the April 1999 Annual Meeting of the American Welding Society. In conjunction with the meeting of the WRC subcommittee, we were able to organize a half-day round robin of the leading experts on ferrite measurement in which we looked for the sources of systematic errors in the FN calibration procedures.

In FY2000, we would like to develop a primary calibration system that 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. 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 is to develop 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 that the users group will be ready to adopt this primary calibration technique when it is ready. Actual construction and deployment of primary calibration devices will be in later years.

Planned Outcome

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

Accomplishments

- Prepared the certificate and report of errors so that 30 sets of secondary FN standards could enter the SRM inventory.

- Procured 56 additional sets, and began to assign reference values.

- Prepared reports that document our measurement procedures, summarize the results of measurements on the secondary standards, and provide the raw measurement data for archival purposes.

- Hosted the meetings of the WRC High Alloy Committee, and its Subcommittees on Hardfacing and Welding of Stainless Steel, May 10 to 12, 1999.

Publications

C.N. McCowan, T.A. Siewert, and D.P. Vigliotti, Stainless steel FN secondary standards: gage calibration and assignment of values, *International Institute of Welding, Documents II-1373-99 and IIC-160-99*, July 1999, available from Secretariat of the American Council of the IIW, American Welding Society, Miami, Florida.

C.N. McCowan, T.A. Siewert, and D.P. Vigliotti, Stainless steel FN secondary standards: gage calibration and assignment of values, a full report of the FN program, in preparation as an NIST Technical Note.

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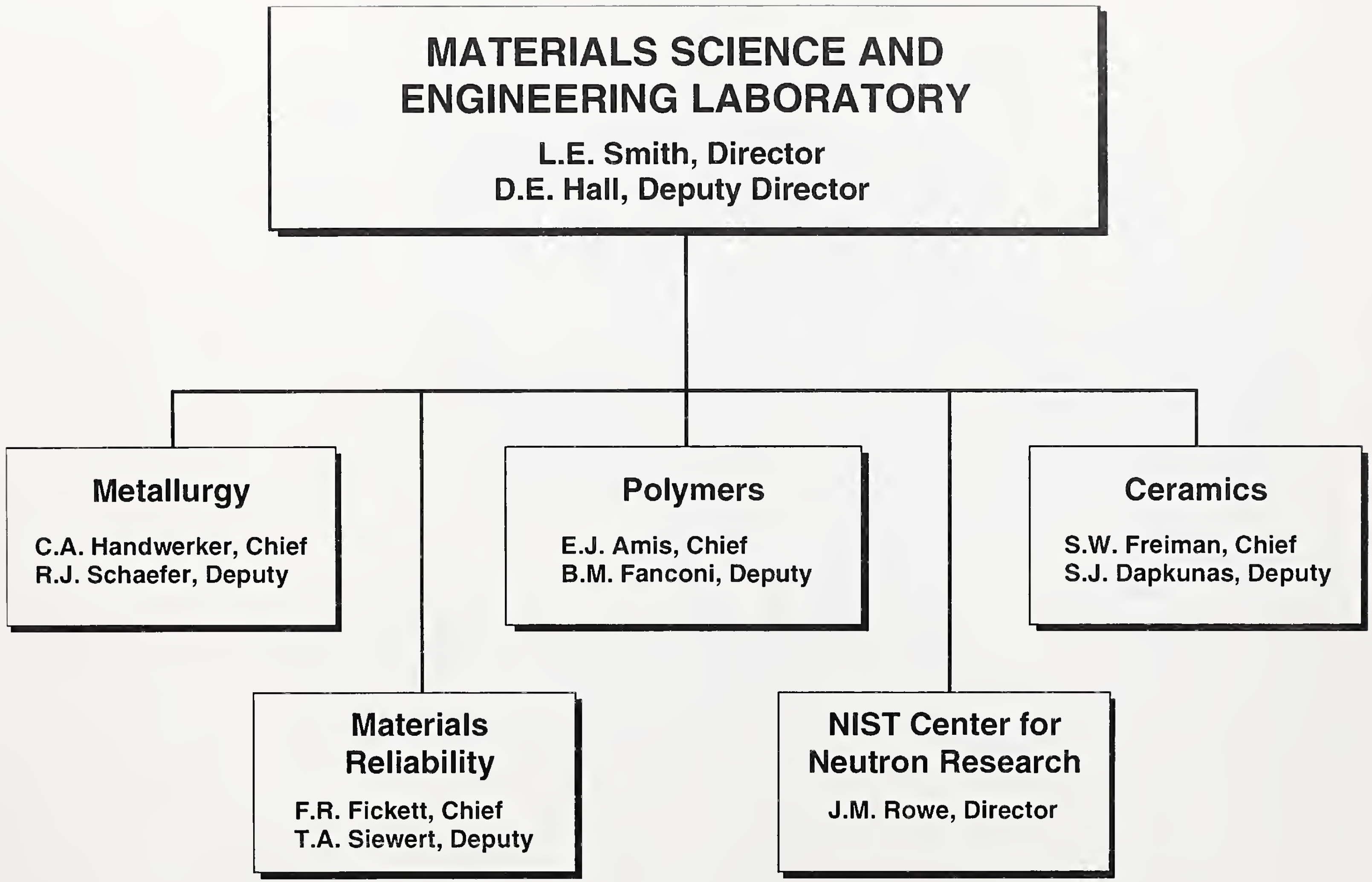
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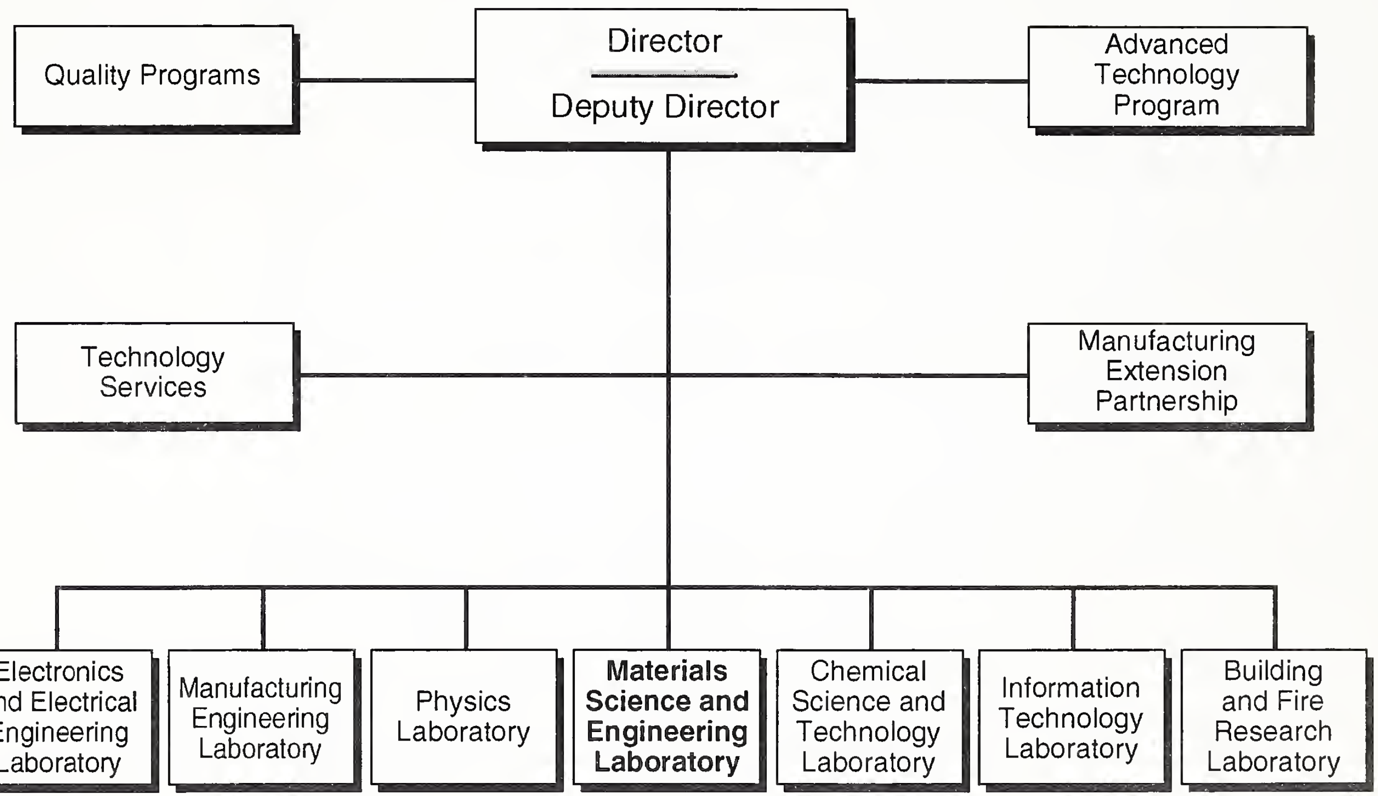

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