MATERIALS RELIABILITY DIVISION

FY 2000 PROGRAMS AND ACCOMPLISHMENTS

NIST

National Institute of
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Cover Caption

The Materials Reliability Division develops and applies advanced measurement and modeling methods for evaluating failure modes and mechanisms, investigates the basic physics and materials science of failure, and works with industries in traditional and emerging markets to develop solutions leading to enhanced reliability of their products. Current Division efforts focus on materials and material-related packaging issues in microelectronics, photonics, and other micro-scale structures. Other projects address reliability of large structures, such as bridges, railroads, nuclear reactors, and pipelines.

UNITED STATES DEPARTMENT OF COMMERCE
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MATERIALS SCIENCE AND ENGINEERING LABORATORY

MATERIALS RELIABILITY DIVISION

FY 2000 PROGRAMS AND ACCOMPLISHMENTS

Fred R. Fickett, Chief

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NISTIR 6595

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DISCLAIMER

Certain commercial equipment, instruments, or materials are identified in this report to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Executive Summary

The Materials Reliability Division develops measurement technologies that enable producers and users of materials to improve the quality and reliability of their products and to meet the ever more stringent materials challenges in the microelectronics market. The metrology devices and concepts, and the associated materials science base, cover the range of materials from metals to polymers to ceramics. Specimen dimensions range from the microscale and nanoscale of electronic packages and their components to the massive structures found in gas pipelines and bridges. Many measurement techniques are brought to bear on the problems, ranging from traditional and advanced ultrasonic testing to advanced transmission electron microscopy, scanned-probe microscopy, and new measurements yet to be named. The Division also provides measurements and standards to support the instruments necessary for assuring the accurate determination of impact resistance of structural steels through the standard reference materials (SRM) program. In FY00 the Division focused its resources on the following research areas:

Microscale Measurements: These projects develop measurement techniques for evaluating the mechanical, thermal, electrical and magnetic behavior of thin films and coatings at size scales typical of modern electronic chip and package structures. With our industrial partners, we used crystallographic studies with electron microscopy to clarify the mechanisms of electromigration failure. Concurrent development of a electromigration test facility with both variable frequency and variable temperature further increased our capabilities to study this critical failure mode of modern electronics. Scanned-probe microscopy is being developed as a measurement technique offering the promise of moving to even finer scales in determination of acoustic, thermal, and mechanical properties, with successful demonstrations of all modes in FY00. This year also saw the return from industry of one staff member who had completed a successful term as a NIST Industrial Fellow at Motorola, and the departure of another for a nine-month stay at the Max Planck Institute for Metal Studies.

Microstructure Sensing: In this program, ultrasonic measurements are applied to the characterization of materials on a scale extending from atomic dimensions (lattice defects), through microstructures (grains) to macrostructures (pipelines). During FY00, emphasis shifted from development of ultrasonic techniques applicable to structural steels to similar measurements on the materials used in microelectronic devices. On the nanometer scale, the atomic force microscope was modified to measure the compliance of surfaces at this level of resolution. The laser ultrasonics and acoustic microscope facilities moved into frequencies over 100 MHz, where the acoustic wavelengths better match the dimensions of the structures used in modern microcircuit devices. As a result, we can now measure the elastic moduli of deposited films whose thickness dimensions lie in the range of 0.1 to 10 micrometers and use the results in models to describe the response of the film and substrate to environmental variables such as temperature changes and processing conditions. Also, improvements in our capabilities in acoustic-resonance spectroscopy enabled us to characterize new materials for microelectronic components, such as crystal oscillators, filters and dielectric resonators. Work at the large scale of dimensions continued with development of techniques to detect and measure residual stress and plastic deformation in large structures in the field.

Process Sensing and Modeling: The projects in this area develop measurement technology for determining a material's characteristics and/or implementing real-time process control. FY00 was a period of transition, as we expanded our activities into several new directions. We noted that the material-property data for lead-free solders were widely distributed through the literature and so started a database effort. In high-energy x-ray diffraction, the techniques that we had developed to monitor the in-situ solidification of turbine blades were applied to the detection of brittle intermetallic phases in solder joints. In welding, our collaboration with the Intelligent Systems Division in Gaithersburg resulted in demonstration of remote sensing of welding problems over the world wide web for several automobile suppliers. 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.

Division Chief's Commentary:

FY00 completes the first year of operation of the Materials Reliability Division under new management. The focus of the Division has changed, being directed more into the area of electronic materials research while maintaining a presence in a few of the infrastructure support efforts that had been the main activity of the Division for many years. Many staff members have worked successfully to apply their expertise to new types of materials and problems on a significantly different size scale. This report describes these activities in some detail. During the year, new equipment and facilities were procured and put in place to support our developing research areas with increased capabilities on the smaller scales inherent in our new directions. We anticipate significant accomplishments in these areas in the upcoming year.

Fred Fickett
November 27, 2000

Technical Highlights

The following Technical Highlights section includes expanded descriptions of research projects that have broad applicability and impact. These projects generally continue for several years. The results are the product of the efforts of several individuals.

Development of Advanced Techniques for Evaluation of Interconnect Failure

Void formation in interconnects is a major reliability-limiting failure mode. Voids occur both during processing, due to relaxation of thermal stresses, and in service, due to physical movement of metal atoms under the influence of very high current densities. The consequences for reliability become more serious as interconnect line dimensions become smaller. Understanding of the mechanisms responsible for void formation and the associated effects of interconnect material and crystal structure is critical for developing new and optimizing existing processes for higher reliability of the chip structure. In this ongoing program, techniques are developed for evaluating modes of voiding and for understanding electromigration damage under a wide variety of conditions.

Background

The metallic "wires" that connect the elements of electronic devices, carrying the signals and control currents, are called interconnects. They are ubiquitous in modern electronics, with present-day devices containing several kilometers of interconnects. Failure of just one such segment can cause an entire device to function improperly. Interconnects are almost always thin film structures that are prepared by any of a number of techniques depending on their location and function. The larger interconnects (often up to tens of micrometers in width) connect chips and other components on the circuit board. Typically made from deposited copper, these lines may be either on the surface or embedded in the multilevel circuit board. Failure in such lines is usually caused by flexing of the circuit board or by excess current. At the chip level, the lines are much smaller (presently just a few hundred nanometers wide) and comprise up to seven levels of the structure. They are made of either pure or alloyed aluminum or copper. The classic IBM picture of a six-level copper-line chip is shown in Figure 1.

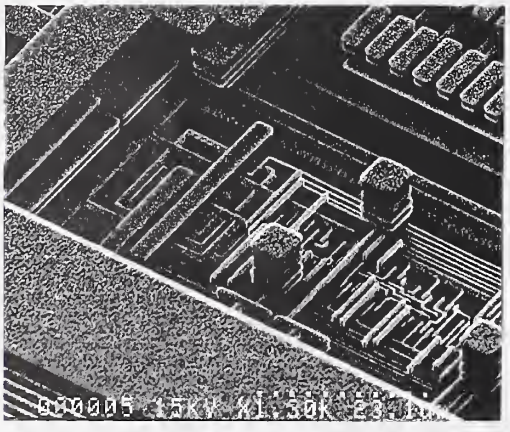

Figure 1. IBM CMOS 75 damascene copper interconnect structure.

Chip-level interconnects are the main focus of this program. Failure of these lines tends to occur when voids form either during processing (stress voiding), or in service when carrying currents that may approach one million amps per square centimeter (electromigration). The Division program started with an emphasis on the stress voiding problem, and has recently changed focus to the study of electromigration failure, looking at the relationships between highly localized microstructure variations in the lines and the formation of voids and hillocks.

Approach

It was clear at the beginning of the program that understanding void creation required two experimental activities. The first was the controlled production and identification of voided regions of interconnects, and the second was measurement and analysis of the crystal and grain boundary structures of such regions and comparison to areas that remained intact. It has long been known that the main regions of void formation are the junctions of three grains or of two grains and a surface. However, why only certain junctions resulted in voiding was unclear until this effort. At this point, the significance of *local* variations in microstructure became apparent. This realization carries over into present efforts.

From the beginning, samples were provided by industry, starting with relatively wide (5 to 10 micrometer) aluminum lines of several years ago, to the sub-micrometer copper lines in use today. In addition, we created some researeh samples of our own in collaboration with the Semiconductor Electronics Division, adding lines modified with notches and bends to their standard electromigration test chips.

As the work has progressed, various measurement methods have been brought to bear on the problems. Our use of electron microscopy to determine localized structure began with electron backscatter diffraction in the scanning electron microscope, and resulted in some early publications on stress voiding in copper lines. With the arrival of damascene processing, it became clear that considerable basic research was needed to attack the electromigration problem from a mechanistic point of view. Rather than refining the subtleties of grain boundary structures, we found that industry needed a more near-term understanding of stresses and strains associated with electromigration. This required that we pursue additional techniques. Development of our capability in convergent-beam electron diffraction (CBED) using our transmission electron microscope (TEM) with its smaller spot

Size (tens of nanometers) led to our current research activities. These measurements, described in the project section of this report, allow the determination of the strain state of single grains, which often are on the order of 100 nanometers in size or even smaller. The pattern analysis required to extract the information remained tedious and an effort was started to develop a unique computer-based analysis scheme that will be made available publicly.

As the project progressed, we realized that we needed electromigration test capability in our own laboratory that would be more versatile than commercial apparatus. This would allow us to systematically determine the effects of various electrical and thermal parameters on the failure modes. To this end, we developed and constructed a test facility allowing both ac and dc testing of lines down to sizes well below one micrometer with variable current, frequency, waveform, and thermal environments.

Accomplishments

The major accomplishments of the program in FY00 are in the application of CBED to evaluation of three-dimensional elastic strains in sub-micrometer grains in interconnects, primarily damascene copper. The work has benefited greatly from collaborations with Max-Planck-Institut (MPI) and Lucent Technologies. We have made preliminary measurements over a wide range of temperatures and the results are consistent with published data. Application of our pattern analysis software to CBED patterns like the one in Figure 2 was successful in determining lattice parameters semi-automatically using optimization algorithms. Further refinements are underway. The preliminary work was done in preparation for a nine-month visit to MPI by Bob Keller during which measurements of *in-situ* electromigration will be performed in the TEM.

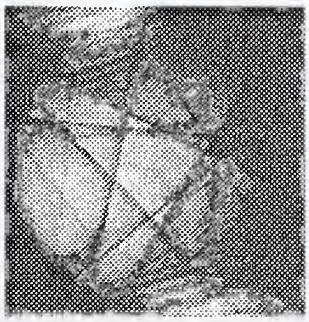

Figure 2. (122) oriented convergent-beam electron diffraction pattern from copper. The line positions allow a determination of the three-dimensional elastic strain state with a precision of approximately 0.01% strain.

The newly constructed electromigration test apparatus was put into service late in the year and initial tests were performed on aluminum lines from an early NIST standard test chip. The apparatus performed flawlessly. Some of the very first test results are shown in Figure 3. The dc electromigration void is shown as forming at a grain boundary intersection as is typical of this failure mode. The ac effect shown in the image on the right is most unusual and, to our knowledge, has never been seen before. This type of failure has been reproduced a number of times in our qualification tests, but we are not yet ready to offer an explanation for the observed structure. This observation has convinced the Max Planck group to pursue ac studies as well, and they are in the process of constructing a similar apparatus.

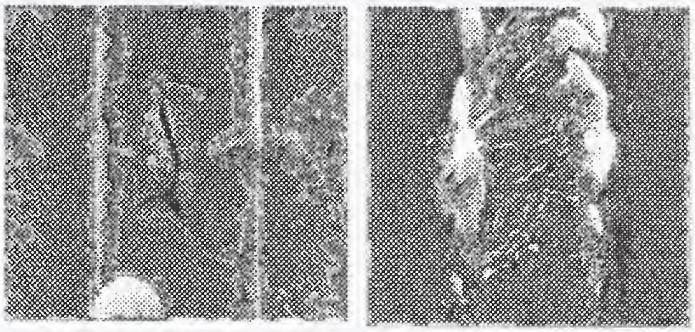

Figure 3. Electromigration failure in aluminum alloy interconnects. The line on the left was carrying dc, while the one on the right was carrying a 100 Hz alternating current. The lines are about two micrometers across and less than one micrometer thick.

Summary

The results from this new program area have been encouraging. The interest shown by industry and by the MPI indicates that we are very much on the right track to developing an expanding program attacking the issues involved in understanding of the mechanisms of electromigration failure, as opposed to contributing to conventional reliability engineering, which is common in industrial design. The measurement techniques, analysis methods, and data developed here not only will be of immediate value, but will provide solutions to many future problems.

For More Information On This Topic

Nucci, J.A., Keller, R.R., Krämer, S., Volkert, C.A., Gross, M.E., "Localized Measurement of Strains in Damascene Copper Interconnects by Convergent-Beam Electron Diffraction," Materials, Technology, and Reliability for Advanced Interconnects and Low-k Dielectrics, Materials Research Society Symposium Proceedings, in press; available online at http://www.mrs.org, for MRS members (2000).

Robert Keller, Fred Fickett

Detection of Intermetallics in Solder Joints by High-energy X-ray Diffraction

We have shown the potential for high-energy x-ray diffraction, as a nondestructive technique, to identify and quantify potentially harmful intermetallic layers in solder joints. The long-term integrity of the joints between solder-ball and copper-pads is crucial to the reliability of printed-circuit boards. Yet, brittle fracture is often observed at the interface of the solder and a buffer layer, that is used as a protective surface finish for the contact pads. The fracture is attributed to the formation of brittle intermetallics.

Background

The quality of the interface between solder and the copper pad (on the circuit board) is a key component in the reliability of printed-circuit boards. The reliability of these joints is becoming even more important with the advent of higher pin counts and finer pitch products, such as ball grid array (BGA) technology and chip-scale packages (CSP). Brittle failure of these joints (and so failure of the entire board or system) is observed in service under conditions of extended thermal cycling, vibration, and thermal or mechanical shock. The brittle fracture often initiates in and propagates through the intermetallics that grow at the interface between the solder and a protective buffer layer on the contact pads. Currently the industry is switching to electroless Ni/Au, which is increasingly used in high-density BGA packages and flip-chip applications, where the problem of intermetallic growth is even more severe. The current interest in lead-free alloys, especially those high in Sn, increases the need for better measurement techniques for these degradation mechanisms.

Currently, the growth of intermetallics is diagnosed destructively; shear testing is used to measure the joint integrity, then the fracture surfaces are examined by light and scanning electron microscopy (SEM). Unfortunately, the SEM's energy-dispersive x-ray capability has some limitations. The technique has a limited penetration depth (only a few micrometers) and then is able to identify only chemical elements and approximate stoichiometry of intermetallic compounds, not the crystallographic phases present at the interface. Perhaps the greatest disadvantage of optical or SEM examination of the intermetallics is that the short penetration depth requires the solder joint to be cross-sectioned.

The x-ray diffraction technique permits direct and unequivocal identification of the crystallographic phases in a solder joint. Furthermore, a high-energy x-ray beam (up to 320 keV) can penetrate a circuit board and the attached devices to the buried intermetallic layer, and therefore nondestructively identify compounds detrimental to the package's reliability. Because the diffraction intensity is proportional to the concentration, the method can also estimate the relative concentration of the intermetallics.

Accomplishments

We prepared bulk intermetallic samples from Cu and Sn powders by solid-state reaction at elevated temperature. The reference diffraction patterns of the Cu-Sn intermetallic phases were then recorded by 8 keV x-rays (Cu-K_α characteristic radiation), and found to be close to that of the $Cu_{6.26}Sn_5$ structure. Furthermore, we made samples with an intermetallic layer by prolonged aging of soldered copper boards at elevated temperature. We used lead-free solders with the composition Sn-3.7 % Ag. The existence of the intermetallic layer was confirmed by both light microscopy and SEM. Figure 1 shows an optical micrograph with intermetallic layer clearly visible. Its average thickness is estimated at 5 μm, and the approximate composition, as determined from the line EDS scan, is somewhat richer in tin than that of the $Cu_{6.26}Sn_5$ structure.

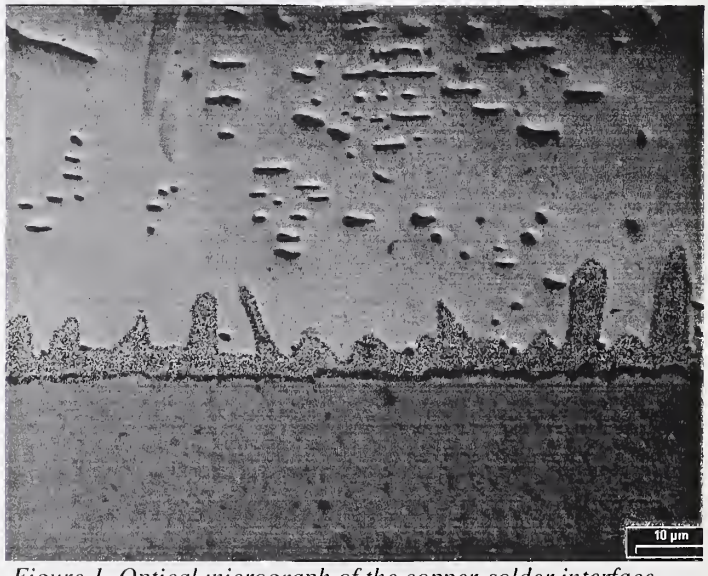

Figure 1. Optical micrograph of the copper-solder interface. Adjacent to the copper (at the bottom) is a 1 μm thin darker region, most likely Cu-rich-Sn solid solution, followed by the irregularly shaped Cu-Sn intermetallic layer, which ranges between 5 and 10μm thick.

The 8 keV x-rays cannot penetrate more than 10 μm of Cu and much less of Pb. Therefore, we used a high-voltage (up to 320 kV) industrial x-ray tube with a W target to try to nondestructively identify buried intermetallic layers. A W target produces a characteristic K_α energy of 59 keV, which extends the penetration depth in most materials from the micrometer to the millimeter range. In this particular case, diffraction patterns can be recorded in both transmission and reflection geometries. We determined that the latter yields both better resolution and higher intensity. However, because at high energy Bragg reflections move to smaller diffraction angles, the diffraction pattern becomes even more cluttered, which makes the detection of intermetallics diffraction lines even more challenging. Figure 2 presents the calculated diffraction pattern at 59 keV, as given by four phases: Cu, Sn, Pb, and intermetallic $Cu_{6.26}Sn_5$. We can conclude that an irrefutable confirmation of this phase would be difficult and dependent on the instrument resolution and diffraction-line broadening.

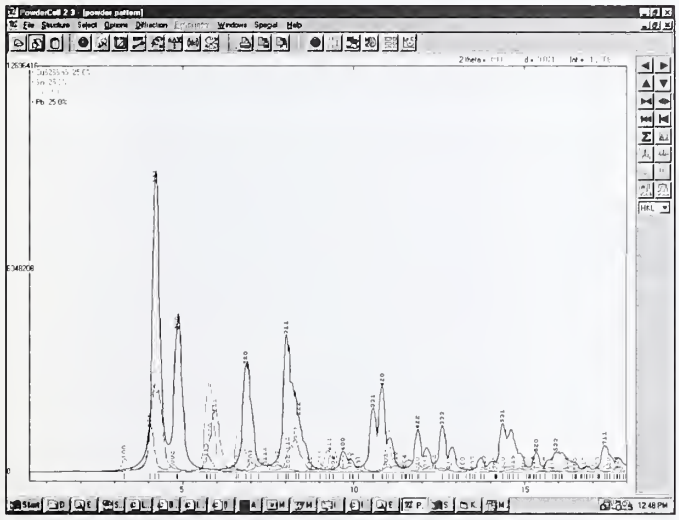

Figure 2. Total calculated diffraction pattern at 59 keV, as given by four crystallographic phases: Cu, Sn, Pb, and intermetallic $Cu_{6.26}Sn_5$.

To test how much a high degree of diffraction-line overlap prevents a positive identification of the intermetallic layer, we prepared two samples: one was normally prepared by soldering and in the other solder was mechanically pressed and glued to the back of the Cu board, and so does not contain any intermetallic phase. Figure 3 shows comparison diffraction scans, where the soldered sample appears to show an additional diffraction line. However, this example illustrates that, although there is a strong indication that the additional peak is given by an intermetallic phase, the final proof is likely to require significantly improved instrumental resolution. We just acquired a new high-brightness x-ray source and a very precise positioning stage that overcome the limitations of our existing equipment. New experiments with such improved equipment are under way, which will hopefully prove a possibility in the near future for nondestructive quantification of intermetallic buried layers.

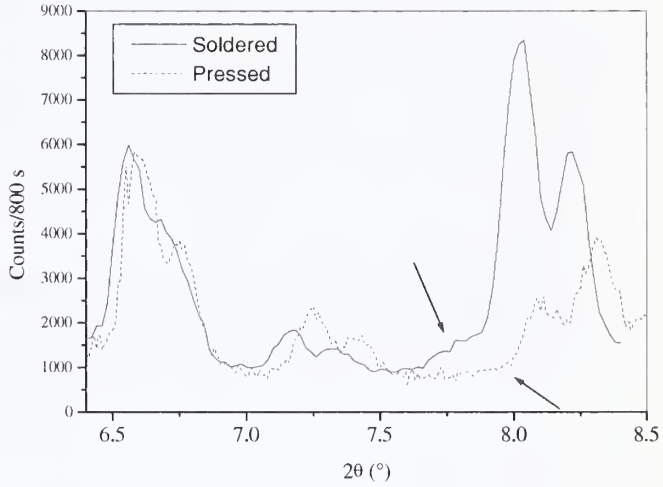

Figure 3. Diffraction patterns of two specimens: soldered Cu board (solid line), where an additional reflection (indicated by an arrow) is possibly due to an intermetallic phase, and another specimen where solder was mechanically pressed and glued to the back of Cu board (dashed line).

For More Information On This Topic

Tom Siewert, Davor Balzar, Chris McCowan

Database on Lead-free Solders

The worldwide movement in the electronics industry to replace lead-tin eutectic solders with lead-free solders creates a need for critical data on the industry's new lead-free solder compositions for these design and reliability models. Our team is working with the NEMI Lead-free Alloy Task Group to develop a comprehensive database.

Technical Description

With product cycle time being slashed to keep up with consumer demand and competitive pressure, new electronic products are going directly from computer-aided design to full-scale production. The worldwide movement in the electronics industry to replace lead-tin eutectic solders with lead-free solders creates a need for critical data on the industry's new lead-free solder compositions for these design and reliability models. In fact, the NEMI web site describes their view of the situation as "The NEMI Lead-free Assembly Project was launched in 1999 to help North American companies develop the capability to produce lead-free products by 2001, with an eye toward total lead elimination by 2004. The goals and focus of the project were determined by the findings of NEMI's 1998 roadmap and an industry task force formed by NEMI to investigate process and material considerations of lead-free electronics assemblies." Their program plan is being implemented by the Lead-free Assembly Project. Project work is organized into five groups, including an Alloy Group whose mission is "to select a lead-free alloy, pursue an industry standard, and gather data."

Development of our database started in April 2000 after we became members of the NEMI Lead-free Alloy Group, and learned that modelers and production engineers need more data before they can switch their production lines to lead-free solders. The long history in the use of current lead-based solders means that these data sets are quite complete and widely available. The modelers and production engineers need equally complete sets of data on the various lead-free alternatives, so they can make informed decisions for their production applications. Researchers are rapidly developing corresponding data on lead-free alloys, but the data are widely distributed among the various technical journals and proceedings. In addition, it is beginning to appear as though differences in test procedures (e.g. loading rates and dwell times) may make some of the data inconsistent from laboratory to laboratory. Finally, individual researchers may be repeating some of the work of others, while other critical data needs are being overlooked. Making the existing data more widely available will address all of these issues.

We have been requested to focus on the three lead-free compositions that seem to have the widest interest: near the tin-silver-copper eutectic (Sn-4Ag-0.8Cu), near the tin-silver eutectic (Sn-4Ag), and near the tin-copper eutectic (Sn-0.8Cu). We have added data for eutectic tin-lead composition (Sn-37Pb) for comparison purposes, and have added data for other lead-free alloys as we find them. We have not added data for electronic materials other than solder.

Accomplishments

Our team continues to work with this NEMI Lead-free Alloy Group to gather existing physical and mechanical property data that have been developed by researchers around the world, into a single database. The most recent version of the database is posted on the Materials Reliability Website at: *http://www.boulder.nist.gov/div853/* In addition, our team is working with NEMI to develop a list of missing high-priority data, with the list serving as a roadmap for research in lead-free solders. NIST and NEMI are planning to host a joint national workshop on these issues, perhaps in Gaithersburg by February 2001.

We plan to add additional data and to critically evaluate more of the existing data each month.

Database for Solder Properties with Emphasis on New Lead-free Solders

Lead free SOLDERS

National Institute of Standards & Technology and
Colorado School of Mines

Lead-Free Solders Research Programs at Universities

The following are stablished research centers with Lead-Free Solders programs:

- Alabama Microelectronics Science and Technology Center - Auburn University
- Packing Research Center - Georgia Institute of Technology
- Reliable Microelectronics Packing Program - University of California at Berkeley
- Center for Welding, Joining and Coatings Research - Colorado School of Mines
- Integrated Electronics Engineering Center - University of Binghamton
- Ames Laboratory and Iowa State University

The following is a list of universities also involved in this research. The contact person would be a faculty member:

- Purdue University
- University of Wisconsin
- Michigan State University
- Northwestern University
- University of Toronto

OBJECTIVE

The purpose of this web site is to provide an on-line database for solder properties emphasizing new lead-free solders. Lead-free solder data is being developed rapidly, but is still difficult to find. (See the Alloy Database section in the August 29, 2000 press release on the NEMI web site - www.nemi.org). Therefore, we hope this web site will allow us to collect this information in one place, and update it frequently. If you have additional data to contribute, please send it to the contact at the bottom of this page. The data reported in this site has been collected from reliable sources, critically reviewed and ordered. There is no restriction to access the datafile. The user is able to read the data on HTML format and download in WORD format, which then can be formatted in EXCEL for easier manipulation.

DATAFILES

The datafiles are ordered by the date in which they were placed on-line, to see them click on one of the links below. If you would like to download the file, click on WORD FORMAT, then go to file and save the document.

HTML FORMAT	WORD FORMAT
• Properties of Lead-free Solders. RELEASE 2.0 2000 October 10 4:30:50 pm	• Properties of Lead-free Solders. RELEASE 2.0 2000 October 10 4:30:50 pm

"NEMI" DATA REQUEST

The NATIONAL ELECTRONICS MANUFACTURING INITIATIVE - NEMI is very interested in contacting university centers and professors that are involved in the characterization of lead free solders. If you are interested on this information click here: "NEMI Data Request".

For More Information On This Topic

http://www.boulder.nist.gov/div853/

Tom Siewert, Carol Handwerker

Predicting and Evaluating Failure Modes of Electronic Packages

Failure in electronic devices usually occurs because of physical damage to the device or thermomechanical degradation of the complex structures caused by the different thermal expansion properties of the wide variety of materials used in the package. Evaluation of the exact failure modes, critical to their resolution, is a difficult problem that becomes increasingly more so as the size of the components and their connections decreases. In this program, a variety of thermal, mechanical, and electron-optical techniques are used to make detailed analyses of the failure of actual packages and the stress buildup that precedes the failure.

Background

Electronic packages are extremely complex structures and rapidly becoming more so as the technology of devices advances at a breakneck pace. The physical size of nearly all electronic devices is decreasing rapidly and, at the same time, their capabilities are increasing dramatically. A classic example of this is the cell phone, which now supports capabilities in email, internet access, and soon even operation of vending machines and GPS location and direction finding. Each new capability requires more electronics that must be packed into a smaller space.

Computer systems that now operate at speeds into the GHz range require CPU chips and associated memory that operate closely packed elements at very high current densities. The chip structure may have many layers; currently seven is not unusual, and the problem of distributing power and signals among these layers creates materials problems. The large amount of heat generated creates a difficult environment for the elements of the package and removal of the heat becomes a primary design consideration. Furthermore, as the device is operated, the heating and cooling cycles create large cyclic stresses on the components because of large differences in thermal expansion of the materials.

The list of materials found in modern electronic packages is staggering, ranging from the silicon of the chips to the metals of the interconnects and on to the dielectrics, both polymeric and ceramic, that make up the electronic elements. Additionally, the materials that hold the package together and the circuit board structures, which may contain embedded passive devices and certainly contain integral passives, contribute to the complexity. When wireless capabilities are required, additional problems arise because of the high frequencies involved. This requirement leads to new materials for dielectric applications at both the high- and low-k end.

Materials measurement problems in this field fall into several categories. Some of the dielectric materials do not exist in bulk form, so data on mechanical and electrical behavior must be acquired on thin-film samples. The metals are also used in very thin films, and most often in small dimensions normal to the thickness as well. The films are prepared by a Number of techniques and each results in quite different mechanical properties for the metal. The bulk "handbook" properties are almost never seen in these structures. Also, the measured properties depend critically on the method of film preparation. In addition, the size, and even the shape, of the element may lead to variations in properties. Measurement techniques for these materials are quite different from those used to measure traditional mechanical and thermal properties.

When these materials are combined in actual devices, such as the relatively simple structure shown in Figure 1, the additional issues of interactions among them further complicate the issue. Both mechanical and thermal measurements must be designed that allow for analysis of the actual behavior of the package. The problem is further complicated, especially in the case of chip packages, by the fact that traditional measurement techniques are not scalable to the sub-micrometer sizes now seen in many applications. Worse yet, the very real possibility of nanometer-scale structures in the near future leaves even the more advanced measurement techniques wanting.

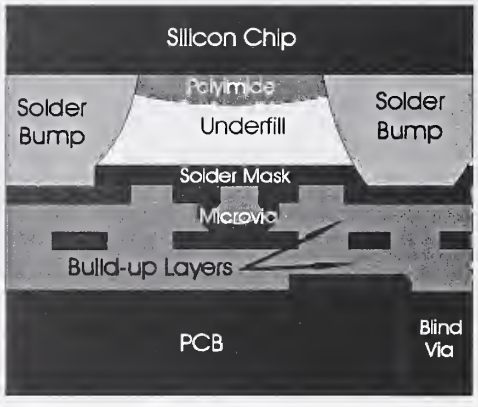

Figure 1. Structure of a flip chip package showing the solder bump connections and the multilayer wiring structure.

Division projects in this area have made significant advances that are reviewed in the following discussion. The separate project reports provide more specifics. The goal here is to indicate how the various project accomplishments fit into a major activity in support of the electronic packaging effort.

Approach

Evaluating thermomechanical behavior of both the elements of the package and the package structure itself requires an array of measurement and analysis techniques. Each of them must operate on the microscale, yet be able to relate back to the large body of data developed using larger scale measurements. Fortunately, the Division staff has a long history of excellence in large-scale measurement and, although the move to the microscale has not been without problems, the understanding of the large-scale issues has helped a great deal in developing the techniques and apparatus for these new adventures. The main areas of investigation are the following: mechanical properties of component materials in thin-film form; strain behavior of packages under thermal or mechanical cycling; thermal properties and heat transport at interfaces in prototypes and actual packages. The work in FY00 has concentrated on measurements at the microscale on actual materials and structures, usually obtained from industry, but also in the form of specialized test specimens created in our laboratories. The development of techniques allowing expansion of our capabilities to nanoscale structures is also underway, with several successful demonstrations of new systems.

The thin-film mechanical properties are measured using traditional force-displacement measurements, but with microscale test systems of our own design. Samples for testing are supplied by industry as well as being made in our own laboratory. The first such apparatus was the subject of a NIST best-practice guide publication and has been replicated by several organizations. The most recent device allows testing in the scanning electron microscope.

The main tool for determination of package strain and failure is the electron beam moiré system allowing measurement of displacement on the scale of 50 to100 nanometers. This system and the associated analysis techniques were described in another best practice document. The technique has been applied to numerous industrial problems. Demonstrations and presentations of the method have been made to industrial groups. Techniques under development use the scanned-probe microscope, in conjunction with gratings produced by bacteria or self-assembling polymers, to increase the displacement detection sensitivity to as low as 5 to 20 nanometers.

The newest capability in the package evaluation program is the measurement of thermal transport through structures and across interfaces. Most measurements rely on our infrared microscope, which has a detection limit on the order of 5 to 10 micrometers and a field size of hundreds of micrometers. Our test system allows heating of a prepared sample with either a laser pulse or by energizing the package electrically. By just using optical observation of the image, flaws in packages can be easily detected, as can electrical problems that lead to excessive heating. With proper calibration for variations in emissivity, thermal conductivity can be measured. To address future problems involving smaller regions, our scanned-probe microscope has been modified to allow thermal imaging measurements. This capability, which is still under development, should allow thermal measurements on the nanometer scale.

Accomplishments

The force-probe tensile test technique and associated apparatus for thin-film mechanical properties testing was completed and its operation in the SEM demonstrated. The imaging system was upgraded to allow higher speed data acquisition and application of advanced image correlation techniques for strain determination.

Application of moiré measurements to a commercial high-density interconnect (HDI) package showed that the microvias resisted deformation despite the highly expansive material surrounding them. New software for fringe analysis was developed and demonstrated. Thermal cycling of bacteria layers on Si and Cu without debonding was achieved. Use of the layers as a mask for deposition of metal grating was demonstrated.

Heat flow across interfaces between conductive adhesives, printed wiring board, and stainless steel was measured by infrared microscopy to determine changes during thermal cycling. The interface regions were also measured with electron-beam moiré, indicating where damage was most likely to initiate.

Involvement with industry is a critical issue in understanding the real problems with failure. Dr. David Read spent much of the year as a NIST Industrial Fellow at the Motorola Advanced Technology Center. In this capacity, he pursued research on user-induced physical damage to portable electronic devices, while gaining insight into the industrial approach to reliability assessment.

Summary

The success of the program and the resulting interest from industry has been excellent this year. Our capability to measure the next several size generations of devices in all research areas means that the work will continue to be of value for some time to come. Staying up with current needs of industry and ahead of projected needs continues to be our goal.

For More Information On This Topic

Slifka, A. J. and Drexler, E. S., "Characterization of Interfaces Involving Electrically Conductive Adhesives using Electron-Beam Moiré and Infrared Microscopy," Proceedings of the 50th Electronic Components and Technology Conference, Las Vegas, NV, 403 (2000).

Andrew Slifka, Elizabeth Drexler, David Read

Combinatorial Methods for Materials Characterization

In order to develop new materials rapidly and efficiently, industrial laboratories are turning toward combinatorial methods to focus attention quickly on compositions or microstructures that optimize the desired properties. The objective of this effort is to apply the skills and expertise in materials characterization techniques that are already available within the Division to the problem of rapid mapping of combinatorial libraries.

Background

The combinatorial method of screening many materials to find the particular composition or microstructure that optimizes a particular property has proven very valuable for the development of new materials. In the past, the Materials Reliability Division has developed several techniques for making a detailed characterization of bulk materials on a specimen-by-specimen basis. This particular program was initiated in mid year to investigate the feasibility of applying at least some of these techniques to the multi-element libraries that are the basis for the combinatorial approach. To be considered practical, a method has to be applicable to a thin film on a substrate and show promise for making the required measurements rapidly and automatically . In addition, the sensor used should be able to interrogate an area no larger than a few millimeters on a side and be mobile so that maps of properties could be constructed..

Approach

Following a survey of the available experimental methods, five were chosen for additional development. These were:

1. X-ray diffraction using a beam focused to a diameter less than a millimeter.
2. Acoustic microscopy using frequencies in the range of 40 to 100 MHz and focusing transducers attached to a raster scanning mechanism.
3. Atomic force microscopy modified by adding acoustic vibration sensors to measure the local elastic properties of a surface.
4. Infrared microscopy using specimen geometries designed to yield local values of thermal conductivity.
5. Magnetostrictive Ultrasonic Transducers (MUTs) whose sensitivity is determined by the magnetostriction coefficients of the material under the sensor.

Theoretical Analysis

To support and organize the program, theoretical tools are being assembled to take the output of each experimental procedure and deduce the physical properties of the local area within the library. This requires modeling each technique in a way that allows the influence of the surrounding material as well as the substrate to be taken into account. To date, a preliminary mathematical solution based on scalar waves has been obtained and is being extended to a tensor wave equation.

Experimental Techniques

The progress achieved before the end of FY 00 is summarized below.

Library Procurement. In order to test the techniques being developed, three sample libraries commonly used in combinatorial studies have been obtained. These are:

(a) A magnetic film library was fabricated by EEEL in Boulder by sputtering Tb, $Ni_{0.8}Fe_{0.2}$ and Co on a silicon substrate.

(b) A ferroelectric library composed of a ternary mixture of $PbTiO_3$, $PbZrO_3$ and $Pb(Ni_{0.33}Nb_{0.67})O_3$ on a $LaAlO_3$ substrate was obtained from LBNL in California.

(c) A transparent conducting oxide film library formed from Cd-Sn-0 on a silieon substrate was obtained from NREL in Golden, Colorado.

X-ray Diffraction. The Tb-Ni0.8Fe0.2-Co magnetic library was diced to enable lattice structure information to be obtained with existing x-ray diffraction equipment. The results are being analyzed and will be reported in the open literature shortly. These benchmark measurements will allow for a subsequent comparison with the measurements made on undieed libraries which is planned for FY01 using recently acquired x-ray microdiffraction beam controls and precise positioning equipment.

Acoustic Microscopy. The Division's acoustic microscope has been upgraded by the installation of a large F-number focusing transducer that can deliver ultrasonic energy at frequencies up to 100 MHz into a water bath. The large F-number is necessary to insure that Rayleigh waves can be excited on the surface of the sample in a small area. Software to automatically scan the distance between the transducer and the sample surface (the z coordinate of the microscope) has been written to permit collection of reflected signal amplitude data versus z. These V(z) curves can be used to deduce quite

accurate values for the Rayleigh wave velocity inside the area of the focal spot at the frequency of operation. By repeating the V(z) measurement at several different frequencies and applying the Green's Function techniques developed in another project, values for the local elastic moduli of the thin film can be deduced.

Atomic Force Acoustic Microscopy. In a conventional Atomic Force Microscope (AFM), the topography of a surface is mapped on a nanometer scale by recording the position of a small stylus on the end of a flexible cantilever beam as the cantilever is scanned over the surface. Our AFM was modified by adding the capability to vibrate the surface under the stylus at an ultrasonic frequency and for detecting these vibrations in the cantilever beam. By measuring the change in the response of the cantilever when it is moved close to the surface, the elastic compliance of the surface under the stylus can be deduced. Since the cantilever mechanism can be scanned over an area, a map of the elastic modulus as a function of position can be displayed. Thus, the elastic modulus of each element in a combinatorial library can be determined.

Infrared Microscopy. An infrared microscope measures the temperature and emissivity of a surface at the focal point of the instrument. In our microscope, this focal spot is less than a millimeter and, thus, it can map the distribution of temperature over a combinatorial library whose thermal boundary conditions are well defined. Two types of boundary conditions will be imposed on library samples mounted on the microscope stage. One library sample will be constructed of rows of similar materials insulated from one another by gaps and having gradations of thermal conductivity along their length dimensions. When the two ends of the rows are held at a fixed temperature difference, the temperature distribution along the rows and across the gaps will map the local thermal conductivity of each element of the library. The second library sample type will be formed by a thin film on a substrate maintained at a uniform temperature. In this case, the temperature distribution on the top of the film can be interpreted in terms of the local thermal conductivity of the film.

Magnetostriction. A Magnetostrictive Ultrasonic Transducer (MUT) can excite and detect ultrasonic waves in the surface of a ferromagnetic material by a magnetic interaction across a small air gap under a coil held near the surface. The amplitude of these waves is determined by the magnetostriction coefficients of the material under the sensor coil. For measuring the magnetostriction of a combinatorial library, apparatus is being assembled to launch a fixed amplitude ultrasonic wave into the substrate supporting a thin film of magnetic material whose magnetostrictive coefficients are expected to vary from point-to-point on the surface. A MUT receiver coil located at some point over the film will measure the amplitude of the ultrasonic wave at that point and thus determine the magnetostrictive coefficient of the film under the coil. By scanning the coil over the substrate, a map of the magnetostrictive coefficients of the film can be displayed.

Magnetic Properties. A library formed by co-deposition of Tb, $Ni_{0.8}Fe_{0.2}$ and Co was fabricated by Division 814 to study the magnetostrictive properties of this ternary alloy system. On-wafer measurements were performed by a Magneto-optical Kerr Effect (MOKE) system but the library had to be diced into individual sites to use the alternating gradient magnetometer instrumentation currently available in our laboratory. The results yielded hysteresis loops at selected locations on the library and exposed very dramatic variations in magnetic properties as a function of composition. A paper describing these measurements will be presented at the Intermag 2001 Conference in January, 2001.

For More Information On This Topic

V. Tewary (Modeling), D. Balzar (X-ray Diffraction), W. Johnson (Acoustic Microscopy), D. Hurley (Atomic Force Acoustic Microscopy), A. Slifka (Thermal Conductivity), G. Alers (Magnetostriction), S. Russek (Magnetization).

Ultrasonic Techniques for Measuring the Mechanical Properties of Thin Films

An ever increasing number of products derive their utility from exotic materials deposited in the form of a thin film on a sturdy, inexpensive substrate. To help insure the quality and reliability of these products, we are developing ultrasonic techniques to measure the mechanical properties of the films. Measurements of the elastic moduli are proving useful for inferring the quality of the microstructure of the film as well as for estimating the level of residual stresses produced by thermal treatments

Technical Description

Modern industry depends heavily on materials with unique properties. In fact, the composition and microstructure of new materials are often engineered to optimize a particular property. In most cases, these properties cannot be achieved when the material is in its bulk form, but can be obtained when it is in the form of a coating layer or a thin film on some kind of easily obtained substrate. Some examples are the thermal-barrier coatings on gas turbine blades for high- temperature applications, chromium or zinc plating on steel for decoration or corrosion resistance, dielectric or magnetic films on ceramics for microelectronic devices and polymer coatings for biomedical implants. The thicknesses of these practical films range from the submicron level in microelectronic devices to hundreds of microns in thermal-barrier coatings. Substrates are often single crystals whose crystallographic orientation has been chosen to ensure that the film will be a single crystal or will have a high degree of preferred orientation in its microstructure. Thus, very special properties can be obtained with small amounts of expensive materials, and a wide variety of deposition techniques can be utilized for the manufacture of valuable products.

The quality of these final products is usually assured by simply measuring the desired property at the end of the manufacturing process. However, making improvements or assuring reliability after extended service often require making other measurements on the film in order to characterize its physical state or to detect defects in its microstructure. For bulk materials, ultrasonic testing has proven to be a very important tool for accomplishing this purpose, and there are many models that relate the ultrasonic quantities that can be measured to the microstructural features that control the desired properties or to the flaws that limit the service life.

Approach

During FY2000, we initiated a program to develop ultrasonic testing techniques that could be applied to materials in the form of thin films deposited on substrates with anisotropic elastic properties. The basic challenges were twofold. One was to develop models that would describe the interaction of ultrasonic waves with films having thicknesses that are much less than the probing ultrasonic wavelength. (At an ultrasonic frequency of 1 GHz, the wavelength of a longitudinal wave is about six microns, which is more than ten times the thickness of films commonly used in most microelectronic devices.) The second challenge was to develop experimental techniques for measuring the ultrasonic wave propagation coefficients at frequencies approaching 1 GHz with accuracy sufficient to separate film and substrate effects. Since direct scaling of conventional ultrasonic pulse-echo techniques to propagation through a film with submicron dimensions demands operation at frequencies much greater than 100 GHz, it was decided to focus attention on ultrasonic wave modes that propagate in the plane of the film. This led to models for surface-acoustic-wave propagation on substrates supporting a thin surface film and to experimental techniques that would measure the wave velocity of these surface or Rayleigh waves with high accuracy.

Accomplishments – Models

Mathematical models for surface-wave propagation on layered media have attracted considerable attention in recent years to guide the design of surface-acoustic-wave (SAW) devices used as resonators and filters in the commercial electronics industry. We have extended these models by applying a dynamic Green's function formalism to develop a more rapid and computationally efficient way to calculate the wave velocity of a surface wave on a single crystal substrate covered with a very thin film of an anisotropic material. It uses the properties of the substrate as well as the elastic constants, density and thickness of the film as input parameters. Because of the computational efficiency of the model, it has been possible to develop a rapid inversion procedure whereby some of the elastic moduli of the film can be deduced from measurements of the dispersion of the SAW velocity. In addition, the effects of inaccurate knowledge of the density and thickness of the film can be taken into account. The elastic constants that have been deduced in this way are being used to infer the quality of the film and the degree of preferred

Orientation present in its grain structure. In the future, it is anticipated that the Green's function approach will allow the effects of the film-to-substrate adhesion to be studied by introducing different boundary conditions into the equations of motion.

Accomplishments – Experimental

Three different experimental approaches to measuring the frequency dependence (i.e. the dispersion) of the SAW velocity are being developed for application to films on different scales of thickness. At low (1-10 MHz) frequencies, EMAT techniques have demonstrated sufficient accuracy to measure the dispersion produced by electroplated films 10 to 20 micrometers thick on aluminum. The same technique is also being applied to the measurement of sub-surface stress gradients on rolled or forged products by replacing the film in the Green's function model with a layer whose elastic properties vary continuously with depth below the surface. For the intermediate-frequency range extending from 10 to 100 MHz, an acoustic microscope is being modified to rapidly produce V(z) curves. Here, the voltage amplitude, V, of the signal reflected from the surface of a part immersed in a liquid coupling bath is measured as a function of the distance, z, between the transducer and the part. If the transducer is a focusing type, this V(z) curve can be used to deduce the velocity of the Rayleigh wave on the surface of the part with an accuracy sufficient to deduce an elastic modulus of a film with a thickness dimension in the range of 1 to 10 micrometers. For a film whose thickness is less than 1 micrometer, we have assembled an ultrasonic system that uses a pulsed laser beam focused to a line on the substrate to produce a plane SAW wave front that is detected by a Michelson interferometer attached to an accurate translation stage. Fourier analysis of the signal from the interferometer at various propagation distances allows the dispersion of the SAW velocity to be measured over a range of frequencies extending from 20 to 400 MHz. This system was used to measure some of the elastic constants of films of TiN 0.1 to 0.5 micrometers thick and deposited on a silicon substrate. Because the range of frequencies was large and the Green's function model was available, it was possible to demonstrate that the films had the strong elastic anisotropy expected for a highly oriented crystalline film.

A fourth experimental technique for measuring the mechanical properties of thin films is also applicable to bulk materials. It is the Atomic Force Acoustic Microscope (AFAM) technique and is performed in an atomic force microscope by observing the resonant frequency of the cantilever as its tip is brought into contact with the sample surface. The shift in resonant frequency between the free-space value and the surface-coupled value can be related to the Young's modulus of the surface directly under the tip. Since the tip's diameter is less than 0.1 micrometer, the modulus value deduced by this technique is characteristic of a region of space that is usually less than the thickness of the films encountered in practice. Thus, the modulus value can usually be considered to be that of the film independent of the substrate. Because the AFM tip can be scanned over an area, it is possible to create a 2D map of Young's modulus and show geometrical features on the surface with nanoscale resolution. During FY 00, the Division's AFM was modified to permit measurements of the cantilever's resonant frequency as a function of the distance between the tip and the surface of a sample. Validation of the model used to deduce Young's modulus is being carried out by fabricating the sample out of a material such as silicon with well known elastic properties.

For More Information On This Topic

See the Programs championed by G. Alers, D. Hurley, W. Johnson, or V. Tewary.

Harmonization of Charpy Impact Reference Materials

NIST has developed an agreement for an international comparison of Charpy impact verification specimens. The partners are the two other National Measurement Institutes, IRMM (Belgium) and NRLM (Japan), that sell verification specimens. We are attempting to make our programs more transparent to industrial users and national standards-writing bodies. Since some of the most important differences in the verification procedures used around the world are related to the verification specimens themselves, we think that interactions addressing these differences can have a strong influence on the direction and implementation of future harmonization efforts.

Problem

A meaningful harmonization (equivalency) of Charpy V-notch (CVN) standards is unlikely until the reference materials used for the verification of impact machines in Europe, Japan, and the United States (EN-10045-2, JIS B 7722, and ASTM E23) share a more common method of certification.

Background

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, or encoder, and/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. The NIST 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.

The National Measurement Institutes (NMIs), which supply the standardized materials used to verify the performance of Charpy impact machines around the world, have different procedures for assigning certified values to the verification specimens. The principal difference is how the laboratories calibrate (and/or control) their measurement systems. This difference stems from the lack of a sufficiently robust method for the direct calibration of Charpy impact machines. As a result, several distinct approaches to the certification of impact verification specimens have developed. For example, the procedure used by IRMM to produce Community Bureau of Reference (BCR)-certified reference materials for impact testing relies on "Master Specimens" as a control for the system. The certified values for BCR specimens are adjusted by a calibration factor with reference to "Master Specimens" that are tested in parallel on the same machine (and same day). The United States and Japan both use "Master Reference Machines" as the control in their systems. The values produced by these "Master Machines" are the correct values by definition. The United States, for example, has 3 impact machines (located at NIST in Boulder, Colorado) that are defined as the reference machines for ASTM verification testing. Japan has two reference machines that are used to determine certified values for JIS impact verification specimens. Although recent comparisons of the results from our respective systems showed good agreement, currently there is no process in place that can continually ensure the equivalency of our systems. Full harmonization of our calibration procedures would facilitate international trade, and help guarantee the quality and safety of products.

Objective

Collect and analyze data during a three-year test program on "International Master Batches" of Charpy impact verification specimens. The results of this testing will be used to evaluate the use of Master Specimens as a common control in the certification procedure for CVN verification specimens between the three NMIs, and to evaluated machine variables, offsets, uncertainty, and other factors relevant to the harmonization of our respective systems.

Test Matrix Details

Batches of AISI type-4340 steel CVN specimens have been produced at energy levels near 15 J, 30 J, and 110 J for use as "International Master Specimens". Participants use their own certification procedures to assess the quality of the batch they produced for the test matrix, and report the data and procedure used for the evaluations. The test matrix includes testing 10 specimens at each energy level on each machine every six months for three years.

All machines used in our respective verification programs are included: two for IRMM, two for NRLM, and three for NIST. This allows us to compare each of the complete verification systems used by our laboratories to one another, and also to compare all individual machines used in the systems. To compare the actual procedures, NIST uses an 8 mm striker radius, and IRMM and NRLM use a 2 mm striker radius for the testing. The small difference in the results due to the striker radius (for type-4340 steels) is being treated as a machine effect in the study.

The test results are being evaluated using the current BCR procedures to determine an initial value for the International Master Specimens. IRMM is analyzing the data according to the BCR procedures. Following each set of tests, the data

will be sent to NIST. NIST will compile a raw data set and distribute it to the laboratories for correction. IRMM will analyze the corrected data set and distribute the results. NIST will perform other types of data analysis to determine other values of interest to the study, but not included in the BCR procedures. In addition, any laboratory is welcome to submit its analysis of the data for our consideration. The results for each test on each machine will be compared to the grand average for the machines (and the initial value determined for the international batch) to evaluate machine performance and stability. The characteristic behavior of machines (relative to the others) will be evaluated in an attempt to better understand specimen/machine interactions and machine design variables that may contribute to the apparent discrepancies between machines. The grand averages from each test (and the data accumulated in the three-year study) will be used to track the stability of the specimens and machines and to evaluate the uncertainty for the specimens (and systems). The data will also be used to evaluate and discuss a harmonized measurement of "uncertainty" for CVN verification specimens.

Reporting

The results from each test (every six months) will be made available to the participants in the study as soon as possible. It is agreed, however, that these data are not to be published until the three-year study is completed (unless there is a unanimous decision to proceed otherwise). Informal presentations to update internal groups and national standards writing bodies on our progress are agreed to be acceptable uses for the data (during any stage of the study). The final report will be published and widely circulated, after approval by all participants. NIST has taken responsibility for the overall coordination and writing of reports for the study.

Benefits to the NMI's

These three years of testing will provide each NMI with information relevant to their internal programs. The testing will provide data for uses such as:

- A second level of control in monitoring the stability of our respective impact verification programs (which can be used to evaluate existing control systems);
- A robust evaluation of specimen stability (since seven machines will test the "same" specimens twice a year, even subtle changes due to the specimens should be distinguishable);
- A comprehensive evaluation of the specimen/machine interactions for the three different types of CVN verification specimens.

This three-year study is also an opportunity to develop a strong working relationship with the other NMIs that provide CVN verification specimens, and the results will provide a common platform for meaningful discussions of harmonizing our programs. The testing is expected to help address some basic questions pertinent to harmonization efforts, which include:

How consistently do the seven machines perform in relationship to one another;

How characteristically do the machines perform at the various energy levels evaluated;

What are the advantages and disadvantages of using "Master Specimens" versus "Master Machines" as the primary control for the certification of CVN verification specimens;

Would the use of both "Master Specimens" and "Master Machines" as a dual control in our programs significantly improve the stability of our existing programs; and

Are there technically correct actions the NMI's can take that would remove unnecessary barriers to the harmonization of CVN verification testing.

Benefits to Users (National and International)

The energy scales used to measure absorbed energy by the United States, Europe, and Japan could be calibrated for equivalency.

If an "International Master Specimen" approach is clearly validated, and practical enough for adoption by the laboratories, it may be possible to remove the barriers in impact verification test standards (EN, ISO, CEN, ASTM and JIS) to attain full harmonization of the relevant standards.

If impact verification standards are fully harmonized, the users of verification specimens will have multiple suppliers for their samples, and the samples will meet the requirements of various national and international impact standards.

Even if "International Master Specimens" are not adopted by our laboratories as a common control method, it is virtually certain that this testing will help to improve both our respective control procedures and our understanding of how a certified value from one laboratory relates to the other laboratories. This will result in improved impact standards and specimens for our customers.

For More Information On This Topic

Chris McCowan

MATERIALS FOR MICROELECTRONICS

Today's U.S. microelectronics and supporting infrastructure industries are in fierce international competition to design and produce new smaller, lighter, faster, more functional, and more reliable 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, component and packaging, and assembly 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 advanced materials and material processes within new product technologies, as outlined within leading industry roadmaps.[1] The vision that accompanies this program – to be the key resource within the Federal Government for materials metrology development for commercial microelectronics manufacturing – may be realized through the following objectives:

- develop and deliver standard measurements and data;
- develop and apply in situ measurements on materials and material assemblies having micrometer- and submicrometer-scale dimensions;
- quantify and document the divergence of material properties from their bulk values as dimensions are reduced and interfaces contribute strongly to properties;
- develop fundamental understanding of materials needed in future microelectronics.

With these objectives in mind, the program presently consists of twenty separate projects that examine and inform industry on key materials-related issues, such as: electrical, thermal, microstructural, and mechanical characteristics of polymer, ceramic, and metal thin films; solders, solderability and solder joint design;[2] interfaces, adhesion and structural behavior; electrodeposition, electromigration and stress voiding; and the characterization of next generation interlevel and gate dielectrics. 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] at NIST. 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 Materials for Microelectronics can be found at: (http://www.msel.nist.gov/research.html

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[1] International Technology Roadmap for Semiconductors, 1999, and National Technology Roadmap for Semiconductors, 1994 and 1997, Semiconductor Industry Association, San Jose, CA; National Technology Roadmap for Electronic Interconnections, IPC, Lincolnwood, IL, 1995, 1997; National Electronics Manufacturing Technology Roadmap, National Electronics Manufacturing Initiative, Inc., Herndon, VA, 1996, 1998, 2000.

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

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

Contact Information: **David Read**

Mechanical Behavior of Thin Films

David T. Read

Techniques for characterizing 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, special test equipment and techniques are required. The ultimate goal of this project is to test specimens produced by semiconductor fabrication equipment and similar in size to features on integrated circuit chips.

Technical description

Thin films are an essential component of all advanced electronic devices. Understanding of failure modes in these devices, especially interfacial delamination, requires knowledge of the mechanical behavior of the films. Techniques for characterizing 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.

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;
- to relate thin film mechanical behavior to microstructure;
- to extend test techniques from their present level (1 μm thick, 10 m wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths of 0.1 to 1 μm).

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. Previous annual reports cover the silicon-frame tensile specimen and the piezo-actuated tensile tester, which operate successfully for specimens 100 μm wide and larger. We have continued to support one such apparatus at NIST and one at Motorola in Tempe.

Accomplishments

Because problems were encountered with narrower specimens, a new technique, called the force-probe tensile-test technique has been developed. The apparatus includes a loading system operable within the SEM for testing specimens as shown in figure 1. This year, the imaging system was upgraded to capture SEM images approximately every 15 s during the tensile test.

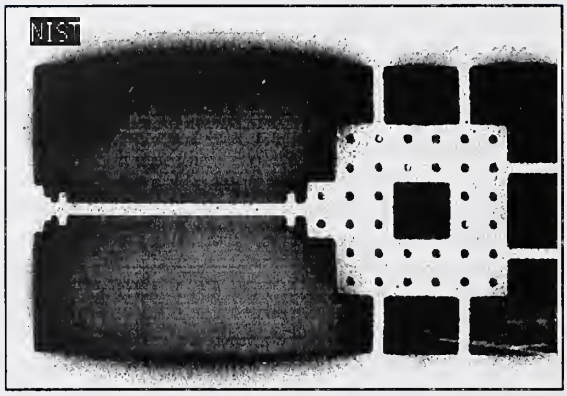

Figure 1. Electron-beam-deposited aluminum specimen with 6 tethers intact. The gauge section, which is the long strip at the left, has a width of 10 μm. The large square hole at the center of the tab is for loading with a needle-like tungsten probe. It is 50 μm across. For comparison, the diameter of a human hair is often quoted as 75 μm.

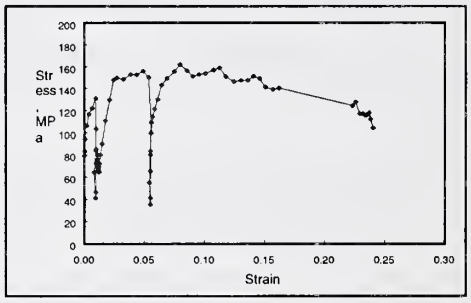

Figure 2. Stress-strain curve with 2 unloading-reloading experiments for measurements of Young's modulus.

These images were analyzed by digital image correlation, to obtain strain, as plotted on the x-axis of figure 2. Observations of ductility of 25 to 30 % in e-beam-evaporated aluminum were confirmed in tests in the SEM.

Publications

Microstructural and Mechanical Characterization of Electrodeposited Gold Films, Long, G. S., Read, D. T., McColskey, J. D., and Crago, K, *ASTM STP 1413, Mechanical Properties of Structural Films*

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Electromigration and Stress Voiding in Interconnects

Robert R. Keller

Ever-improving performance of microelectronic devices is accomplished by scaling down the dimensions of metal interconnections. Generic, empirical lifetime models are insufficient for lifetime prediction since the roles of internal stresses and microstructure in such small structures are not thoroughly considered. This project addresses the mechanisms that control reliability in interconnections. Specific goals include development of methods for locally measuring mechanical strain during electrical stressing and assessing quantitatively its role in electromigration failure.

Background

Stress voiding (SV) and electromigration (EM) are failure phenomena that limit the reliability of narrow interconnects. They occur during thermally and electric current-induced stressing, respectively. The end result is the formation and growth of hillocks and voids in the metal due to the development of severe mechanical stresses and stress gradients; hillocks can lead to short-circuit failures, and voids 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 non-uniform local diffusion during electrical current stressing. Unless a more complete mechanistic understanding is developed, the impact of SV and EM is projected to worsen as the dimensions of interconnect structures continue to scale downward, and as new materials are introduced into interconnect architectures.

Interconnects become less homogeneous as dimensions scale downward, since the structures then comprise individual grains through the film thickness and across the line width. Behavior also becomes less homogeneous, and even small variations in microstructure can detrimentally affect stress development and reliability. Understanding and solving the problems of stress relaxation through void formation and growth at the micro- and nanostructural levels are essential to the continued development of metallizations on a submicron scale. We use electron microscopy to quantitatively characterize on a local scale the microstructures of films and narrow metallizations for interconnects. Electron backscatter diffraction, crystallographic 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 reliability behavior. Results are interpreted in terms of both the energetics and kinetics of void formation and growth, and correlated to interconnect lifetimes.

Accomplishments

This year, we began refinement of the convergent-beam electron diffraction (CBED) method for evaluating strains at high temperatures, in preparation for accelerated electromigration testing in the transmission electron microscope (TEM).

Through collaboration with Max-Planck-Institut, we found that CBED is well capable of providing strain information at temperatures ranging from –180 °C to 225 °C, and the hydrostatic thermal strains track nicely with published values for thermal expansion. At the highest temperatures investigated, bare copper does not undergo oxidation within the TEM, which is encouraging for the electromigration tests; the strongly dynamical scattering present in Cu was earlier a concern, but it seems now that we are able to simulate those effects with little trouble. Figure 1 shows a CBED pattern obtained at –180 °C.

We have begun development of software to evaluate lattice constants from CBED patterns. This software can take an experimental pattern as input, detect the experimental high-order diffraction line positions with sub-pixel accuracy, and overlay simulated patterns onto the experimental pattern. At this point, it allows us to evaluate lattice parameters semi-automatically using optimization algorithms. The user interface is now in a state such that what used to require many hours for manual pattern fitting now requires only minutes. Further efforts will automate the process more fully, leading to the evaluation of three-dimensional strain and stress states in interconnects, with spatial resolution of better than 100 nm.

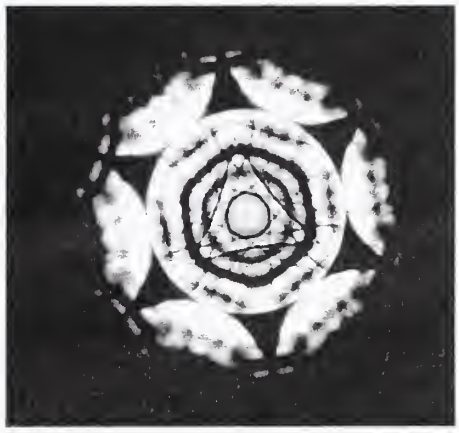


Figure 1. Convergent-beam electron diffraction pattern from copper, showing (111) crystal orientation and high-order diffraction lines used for measuring lattice parameters.

Contributors and Collaborators

NIST Contributors: Fred Fickett, Tim Quinn, Michael Varney

Mihal Gross, Lucent Technologies; Stephan Krämer, Julie Nucci, Cynthia Volkert, Max-Planck-Institut für Metallforschung

X-ray Studies of Intermetallics in Solder Joints

Tom Siewert, Davor Balzar

Objective: Develop high-energy x-ray diffraction, as a nondestructive technique, to identify and quantify potentially harmful intermetallic layers in solder joints. The long-term integrity of the solder-ball — copper-pad joints is crucial to the reliability of printed circuit boards. Yet, brittle fracture is often observed at the interface of the solder and a buffer layer, which is used as a protective surface finish for the contact pads. The fracture is attributed to the formation of brittle intermetallics.

Background

The quality of the interface between solder and the copper pad (on the circuit board) is a key component in the reliability of printed circuit boards. The reliability of these joints is becoming even more important with the advent of higher pin counts and finer pitch products, such as ball grid array (BGA) technology and chip-scale packages (CSP). Brittle failure of these joints (and so of the entire board or system) is observed in service under conditions of extended thermal cycling, vibration, and thermal or mechanical shock. The brittle fracture often initiates in and propagates through the intermetallics that grow at the interface between the solder and a protective buffer layer on the contact pads. Currently, the industry is switching to electroless Ni/Au, which is increasingly used in high-density BGA packages and flip-chip applications, where the problem of intermetallic growth is even more severe. The current interest in lead-free alloys, especially those high in Sn, increases the need for better measurement techniques for these degradation mechanisms.

Currently, the growth of intermetallics is diagnosed destructively; shear testing is used to measure the joint integrity, then the fracture surfaces are examined by light and scanning electron microscopy (SEM). Unfortunately, the SEM's energy-dispersive x-ray capability has some limitations. The technique has a limited penetration depth (a few micrometers) and then is able to identify only chemical elements and approximate stoichiometry of intermetallic compounds, not the crystallographic phases present at the interface. Perhaps the greatest disadvantage of optical or SEM examination of the intermetallics is that the short penetration depth requires the solder joint to be cross-sectioned.

The x-ray diffraction technique permits direct and unequivocal identification of the crystallographic phases in a solder joint. Furthermore, an x-ray beam of high energy (up to 320 keV) can penetrate through a circuit board and its attached devices to the buried intermetallic layer and therefore *nondestructively* identify compounds detrimental to the package reliability. Because the diffraction intensity is proportional to the concentration, the method can also estimate the relative concentration of the intermetallics.

Accomplishments

We prepared bulk intermetallic samples from Cu and Sn powders by solid-state reaction at elevated temperature. The reference diffraction patterns of the Cu-Sn intermetallic phases were then recorded by 8 keV x-rays (Cu-K_α characteristic radiation), and found to be close to the $Cu_{6.26}Sn_5$ structure. Furthermore, we made samples with an intermetallic layer by prolonged aging of soldered copper boards at elevated temperature. We used lead-free solders with the composition Sn-3.7 Ag. The existence of the intermetallic layer was confirmed by both light microscopy and SEM. Figure 1 shows a micrograph with intermetallic layer clearly visible. Its average thickness is estimated at 5 μm and the approximate composition, as determined from the line EDS scan, is somewhat richer in Sn than the $Cu_{6.26}Sn_5$ structure is.

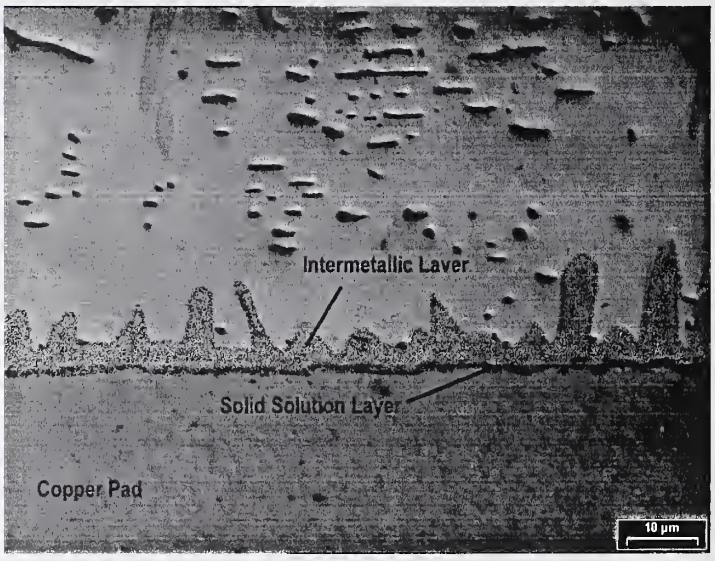


Figure 1. Optical micrograph of the copper-solder interface. Adjacent to the copper (at the bottom) is a 1 μm thin darker region, most likely Cu-rich-Sn solid solution, followed by the irregularly shaped Cu-Sn intermetallic layer, which ranges between 5 and 10 μm thick.

Contributors and Collaborators

Chris McCowan

Lead-free Solder Database

Members of the Materials Reliability Division are working with members of the Metallurgy Division, the Colorado School of Mines, and the National Electronics Manufacturing Initiative (NEMI) to expand a database on the properties of lead-free solders.

Tom Siewert

Technical Description

With product cycle time being slashed to keep up with consumer demand and competitive pressure, new electronic products are going directly from computer-aided design to full-scale production. The worldwide "green" movement in the electronics industry to replace lead-tin eutectic solders with lead-free solders creates a need for critical data on the industry's new lead-free solder compositions for these design and reliability models. In fact, the NEMI web site describes their view of the situation as "The NEMI Lead-free Assembly Project was launched in 1999 to help North American companies develop the capability to produce lead-free products by 2001, with an eye toward total lead elimination by 2004. The goals and focus of the project were determined by the findings of NEMI's 1998 roadmap and an industry task force formed by NEMI to investigate process and material considerations of lead-free electronics assemblies." Their program plan is being implemented by the Lead-free Assembly Project. Project work is organized into five groups, including an Alloy Group whose mission is to select a lead-free alloy, pursue an industry standard, and gather data.

Development of our database started in April 2000 after we became members of the NEMI Lead-free Alloy Group, and learned that modelers and production engineers need more data before they can switch their production lines to lead-free solders. The long history in the use of current lead-based solders means that these data sets are quite complete and widely available. The modelers and production engineers need equally complete sets of data on the various lead-free alternatives, so they can make informed decisions for their production applications. Researchers are rapidly developing corresponding data on lead-free alloys, but the data are widely distributed among the various technical journals and proceedings. In addition, it is beginning to appear as though differences in test procedures (e.g. loading rates and dwell times) may make some of the data inconsistent from laboratory to laboratory. Finally, individual researchers may be repeating some of the work of others, while other critical data needs are being overlooked. Making the existing data more widely available will address all of these issues.

We have been requested to focus on the three lead-free compositions that seem to have the widest interest: near the tin-silver-copper eutectic (Sn-4Ag-0.8Cu), near the tin-silver eutectic (Sn-4Ag), and near the tin-copper eutectic (Sn-0.8Cu). We have added data for eutectic tin-lead composition (Sn-37Pb) for comparison purposes, and have added data for other lead-free alloys as we find them. We have not added data for electronic materials other than solder.

Accomplishments

Our team continues to work with this NEMI Lead-free Alloy Group to gather into a single database existing physical and mechanical property data that have been developed by researchers around the world. The most recent version of the database is posted on the Materials Reliability Division's Website at: *http://www.boulder.nist.gov/div853/*. In addition, our team is working with NEMI to develop a list of missing data of high priority, with the list serving as a roadmap for research in lead-free solders. NIST and NEMI are planning to host a joint national workshop on these issues, perhaps in Gaithersburg by February 2001.

We plan to add additional data and to critically evaluate more of the existing data each month.

Contributors and Collaborators

David Smith
Carol Handwerker, Metallurgy Div. and Chair of NEMI Lead-free Alloy Group
Members of NEMI Lead-free Alloy Task Group
Juan Carlos Madeni and Steven Liu, Colorado School of Mines

MATERIALS FOR WIRELESS COMMUNICATION

The current revolution in wireless communications would not have been possible without the discovery and development of oxide ceramics exhibiting the coincidence of high, temperature-independent permittivity with low dielectric loss. Advanced ceramics are used primarily as building blocks for filters and oscillators in components that are critical to the performance of the end application. One of the first ceramics widely used for cellular base stations was discovered, and its processing phase diagram determined, in a collaborative effort between Bell Labs and NBS in early 1970. Today, wireless technologies constitute one of the most important growth areas in the world electronics industry. Paramount in the cost-cutting and miniaturization process is the need for research that will facilitate the rational design of advanced materials to provide temperature stability, frequency, and size-reduction requirements for the next generation of devices for cellular personal communications system (PCS) and many other niches of the wireless communications industry.

Technically, important ceramic materials fall into two major dielectric categories: bulk ceramics for base station resonators/filter; and those needed for low-power, miniaturized hand-held devices. Activities there include research on both types of dielectric materials. The primary goal of the program is to determine methods that can be used to predict high-frequency dielectric behavior to enable rational design of next-generation ceramics.

A collaboration between MSEL and the Electrical and Electronics Engineering Laboratory at NIST on ceramic materials for base-station applications includes experimental determination of selected complex-oxide phase diagrams integrated with in-depth structural (X-rays, electrons, neutrons), crystal-chemical, spectroscopic, and dielectric property characterization. The objective of this multidisciplinary project is to determine the fundamental relationships between phase chemistry, crystal structure, and dielectric performance at wireless frequencies.

Another project uses first-principles methods to elucidate the roles of cation order-disorder and ferroelastic phenomena in determining the phase relations and physical properties of complex ceramic oxide systems. First-principles calculations are used to predict cation ordering phenomena, physical properties, and how they vary with chemical composition. Critical experiments are performed to test the predictions.

Microstructural modeling and experimental studies are also underway to determine the dimensional changes in low-temperature-cofired ceramics used for portable communication devices. Models developed in this study were identified by a large segment of industrial producers as being crucial to reducing the time for the design and production of components.

Related work includes development of noncontact acoustic metrology to characterize wireless materials in both thin-film and bulk form. The mechanical properties of thin films are investigated using laser-ultrasonic methods to generate and detect surface acoustic waves (SAW). The elastic-property information obtained will result in improved predictive modeling of film performance. Also, resonant-ultrasonic techniques are applied to new piezoelectric materials for SAW devices, used extensively as oscillators in hand-held devices.

Electromagnetic properties of polymer composite films are also being investigated for applications in wireless communications. These materials can be used to construct RLC cells and de-coupling power planes integrated within chip substrates and printed circuit sub-assemblies. Current technologies utilizing discrete components cannot provide adequate solutions at frequencies above 1 GHz which are critical for the high-speed electronics needed by wireless technologies. In partnership with the National Center for Manufacturing Science and industry, a research consortium was organized to address this problem.

Contact Information: **Fred Fickett**

Thermomechanical Studies of Electronic Packaging Materials

Andrew Slifka, Elizabeth Drexler

Objective: Develop methods to examine interfaces in electronic packaging materials and elucidate the damage mechanisms. Our current focus is on integral passive materials using moiré interferometry to measure mechanical strain and thermal microscopy to measure heat flow and thermal properties on increasingly smaller size scales.

Background

The trend in electronics is toward components of higher density and smaller size using less expensive materials. One move in this direction is the advent of integral passive components and another is the increasingly prevalent use of organic (polymeric) conductors and fillers. These organic materials have a large coefficient of thermal expansion, which can reduce the reliability of electronic packaging systems. We are investigating interfaces between organic materials and between organics and metals to determine the initiation of damage and failure mechanisms in these material systems. We have previously developed the electron-beam moiré technique for measuring mechanical strain and are applying it in conjunction with thermal microscopy to obtain a more complete picture of thermomechanical failure in electronic packaging material systems.

Figure 1. IR micrograph of an integral resistor embedded between printed wiring board and thermally conductive adhesive.

Accomplishments

We developed infrared (IR) microscopy for measurement of heat flow and examined interfaces in a sample containing two conductive adhesives, printed wiring board, and stainless steel. The sample was measured at various stages of thermal cycling from –55 °C and 125 °C. The regions examined by IR microscopy were also measured with electron-beam moiré. Moiré was able to show the regions where damage was most likely to initiate, while IR microscopy tracked changes in interfacial thermal resistance at the interfaces. IR microscopy was able to detect damage before it was manifested at the surface and viewable by common techniques such as optical or electron microscopies. Electron-beam moiré showed accumulated strain in the materials and evidence of residual accumulated stress at interfaces as thermal cycling progressed. This work was presented at the 50th Electronic Components and Technology Conference (ECTC).

Work has begun on IR microscopy of organic electronic elements using Joule heating. This approach will localize heat as it would occur in-service and allow us to assess the validity of thermal cycling as the accepted method of accelerated failure in electronic packaging materials. Figure 1 shows an IR microscope image of an integral resistor sandwiched between printed wiring board and a thermally conductive adhesive.

We have begun development of scanned-probe microscopy (SPM) techniques for both moiré and thermal microscopy. Figure 2 shows height and thermal SPM images of the sandwiched integral resistor specimen.

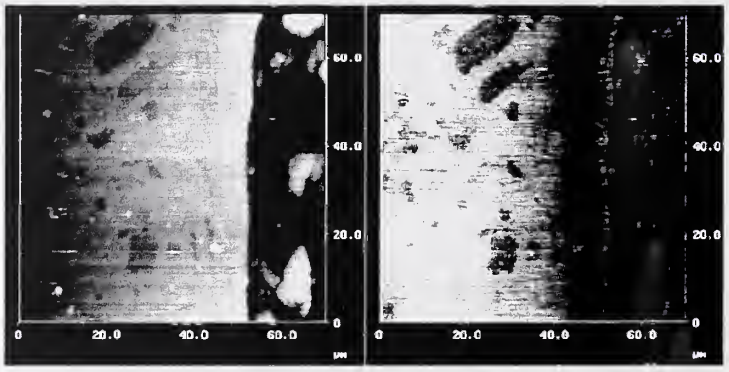


Figure 2. SPM images of the sandwiched integral resistor specimen with height on the left and thermal contrast on the right.

Contributors and Collaborators

Paul Rice
Richard Adams

Experimental Micromechanics by Electron-beam Moiré

Elizabeth S. Drexler

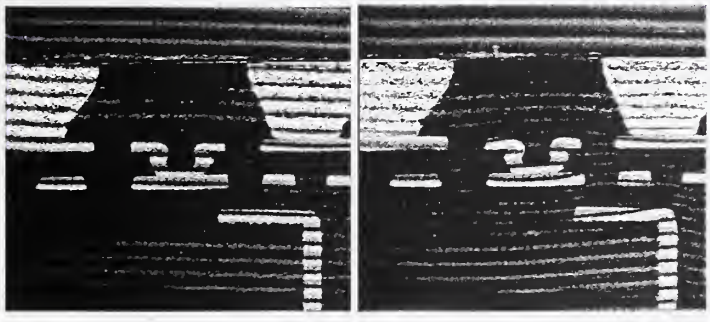
The objectives of the program are to develop and apply the electron-beam moiré technique to measurement of strain and observation of deformation at high magnification. The observations are used to characterize failure modes and to verify mathematical models and simulations of microscale mechanical behavior. The technique is modified to remain current with industrial requirements for resolution and conditions. We reach out to industry to make them aware of the technique and its potential to solve their reliability issues.

Technical Description

The electron-beam moiré program seeks to offer support and verification of models conducted or sponsored by the microelectronics industry. Electron-beam moiré is unique,as it can measure small displacements (minimum displacements range from approximately 25 nm to 100 nm, depending on the pitch of the grating lines) at temperatures between –55 and 150 °C, the maximum range used by industry for qualification of packages. The technique allows the researcher to observed the substrate throughout the thermal cycling so that one can identify the locations and materials in which deformations are occurring.

An experiment was conducted for Motorola in Austin, TX to look at the microvias contained in their high-density interconnect (HDI) substrates. The figure shows the microvia located near the center of the Si die in the HDI package at room temperature and at 125 °C. The image on the right shows that the build-up layers containing the microvia are highly expansive, raising concerns regarding potential deformation of the microvias. Over the course of 34 thermal cycles (–55 to 125 °C), the microvias remained virtually free of deformation, but cracks developed elsewhere in the package at interfaces between materials of differing coefficients of thermal expansion.

The resolution of the electron-beam moiré technique is appropriate for measuring displacements in package features such as solder bumps, (micro) vias, board interconnections, wire bonds. However, for on-chip features, or chip-on-glass or other low-expansion packages, increased resolution is necessary to measure displacements. In FY00 endeavors to improve resolution were conducted on two fronts. For the first, a new Field Emitting Scanning Electron Microscope (FE-SEM) was purchased and delivered just at the end of the fiscal year. It is expected that the lithographic activities will be moved to that microscope, which has a smaller, tighter electron beam than the one used earlier. The smaller beam spot should yield a smaller grid pitch, and, therefore improved resolution.



The other area of development is seeking to conduct moiré experiments on the Scanning Probe Microscope (SPM). The tip of the SPM rasters in much the same way that the electron beam rasters in an SEM. We have verified that the scanning tip will interfere with a topographical grating to generate moiré fringes. Improving the resolution will also require a smaller pitch on the specimen grating. No lithographic techniques available can give grating pitches on the order of 20 to 25 nm. For that, the natural hexagonal grating found in the outer protein layer of the *Sulfolobus acidocaldarius* bacteria is being used. We have successfully attached the protein layers to two different substrates and heated the substrates to 125 °C or more. The protein layers remained attached throughout the thermal loading.

Accomplishments

--A test on the Motorola HDI specimen was completed, and it was determined that the microvias resisted deformation despite the highly expansive material surrounding them. The results were presented and well received at ECTC in May 2000. The accompanying paper was published in the Proceedings of ECTC.
--We commissioned an outside contractor to develop software to aid in fringe identification and analysis. The software was received and it greatly facilitates the reduction and analysis of the moiré fringe data.
--A new FE-SEM was purchased. It is hoped that we will be able to transfer our lithographic capabilities to that microscope.
--A heating stage for the Dimension 3000 SPM was acquired. This will enable us to thermally load microelectronic packages during Scanning Probe Moiré testing.
--The bacterial protein layers were deposited on Si and Cu substrates, then subjected to a thermal cycle up to 125 and 150 °C, respectively. The protein layers adhered to the substrate throughout the thermal loading.

Contributors and Collaborators

Robert Munroe—Motorola, Austin, TX
T. Andrew Winningham—University of Colorado
Joe Kuczynski—IBM, Rochester, MN

Anelastic Damping in Piezoelectric Materials for Crystal Resonators

Ward Johnson

This project seeks to identify defects that degrade the acoustic properties of langasite, langatate, and their isomorphs, which are candidates for replacing quartz in electronic oscillators and filters. Ultrasonic measurements as a function of temperature and frequency are used to provide information on anelastic mechanisms that reduce the Q of the material. These data will assist researchers in the selection of materials on which to focus within this group of compounds and provide information useful for the development of crysta-growth techniques.

Technical Description

During the past decade, a number of research groups have sought to grow and characterize high-quality single crystals of langasite ($Li_3Ga_5SiO_{14}$), langatate ($Li_3Ga_{5.5}Ta_{0.5}O_{14}$), and their isomorphs for electronic-oscillator and filter applications. The potential advantages of these materials over quartz include higher piezoelectric coupling, which enables devices to be made smaller, and higher Q, which provides lower phase noise and operation of higher-frequencies. These new materials also have no phase transition below the melting point, which allows devices to be operated at high temperatures and provides the capability of producing large crystals more easily and cheaply than with quartz. This last feature is important because the increasing use of large wafer processing techniques for surface acoustic wave (SAW) devices is pushing the limits of quartz crystal-growth technology.

The current situation in this area of research is similar to that in the 1950s and 1960s when techniques for growing synthetic quartz were being refined. However, one difference is that systematic studies of the temperature dependence and frequency dependence of the anelastic loss are almost nonexistent for the langasite family of compounds, whereas numerous studies of this type were performed on quartz to provide information on the identity of defects that degrade the Q. Because of the lack of systematic research on contributions to the loss, current conclusions regarding the superiority, with respect to the Q, of one compound over another in the langasite family must be considered tentative; observed differences may not represent the intrinsic limits of the materials.

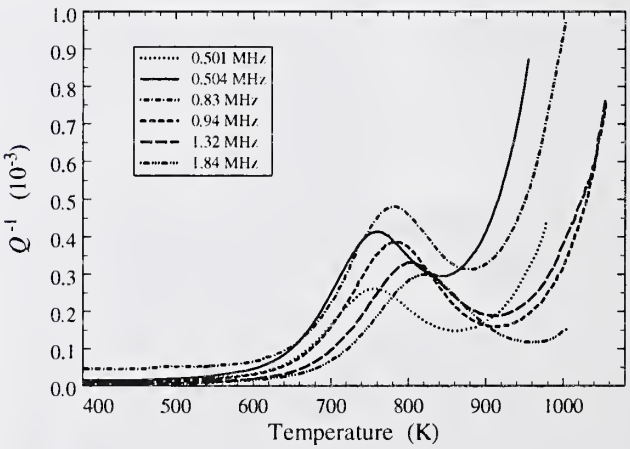
Our laboratory has developed unique capabilities for measuring anelastic properties of piezoelectric and metallic materials as a function of temperature and is applying these to the characterization of this family of compounds. Two experimental systems provide a combined capability of measuring ultrasonic-resonance from 77 K to 1100 K. A novel transduction technique has been developed that employs inductive piezoelectric coupling to cylindrical single-crystal specimens and minimizes the effects of mechanical contact.

Accomplishments

After developing experimental techniques during the past year, an exploratory study of acoustic damping (Q^{-1}) as a function of temperature and frequency was performed on single-crystal langatate. Measurements in the range from 80 to 300 K suggest that dislocations produced during grinding of the crystal may dominate Q^{-1} in this temperature range. At elevated temperatures, point-defect relaxations produce large peaks in Q^{-1} (see, for example, the figure below), some of which are eliminated by brief annealing at 1080 K. These peaks rest on a background that increases rapidly at the highest temperatures and is believed to arise from diffusing interstitial impurities.

The large defect-related contributions to the damping at elevated temperatures have significant implications for the development of high-temperature devices. Even modest performance and reliability at these temperatures can be achieved only if the defects responsible for the anelastic effects observed in this study are identified and eliminated.

The complete results of this study were presented at the 2000 IEEE/EIA International Frequency Control Symposium (proceedings to be published).



Q^{-1} of six resonant modes of langatate during cooling from 1055 K.

Contributors and Collaborators

NIST: Ward Johnson and Sudook Kim
Industrial collaborator: Robert Smythe, Piezo Technology, Inc., Orlando, FL

Elastic Properties of Thin Films Using Surface Acoustic Waves

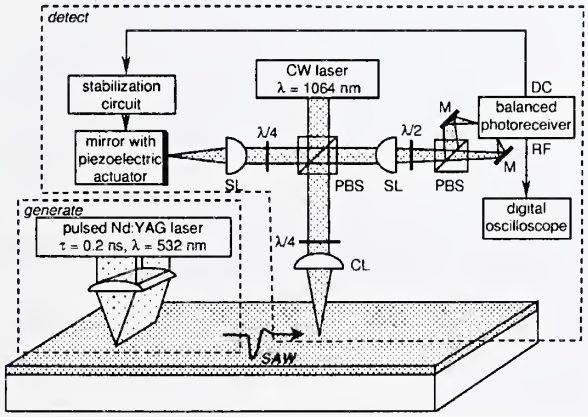
We are developing noncontact, nondestructive tools to measure the mechanical properties of thin films. Our approach utilizes laser-ultrasonic methods to determine the frequency-dependent velocity of surface acoustic waves. The elastic-modulus information provided by our methods will aid in developing processes for new materials. Furthermore, such mechanical-property information is needed to predict the reliability and performance of new thin-film components.

Donna Hurley

Technical Description

Industrial uses of thin films range from providing specialized electrical or magnetic properties for device applications to giving ordinary materials extraordinary resistance to wear or corrosion. Whatever the application, successful development of a film requires a thorough understanding of its mechanical properties. Such knowledge is needed to estimate surface residual thermal stresses, to predict component reliability or performance, and to determine if there is good adhesion to the substrate. However, current methods are generally limited to destructive tests or even to "try it and see." We are developing nondestructive methods to quantify thin-film mechanical properties. Our objective is to relate measurable physical properties such as elastic moduli, residual stress, or adhesion to quality factors such as component lifetime.

The experimental approach involves surface acoustic waves (SAWs). SAWs are well suited for our purposes, since their energy is concentrated near the surface but the decay of energy away from the surface depends on wavelength. The SAW velocity is thus determined by the SAW wavelength relative to the film's thickness. To obtain elastic-property information, we measure the SAW phase velocity over a broad frequency range (dispersion relation). Comparison of measurements to the predictions of an analytical model for wave propagation lead to quantitative values for the film's elastic moduli.



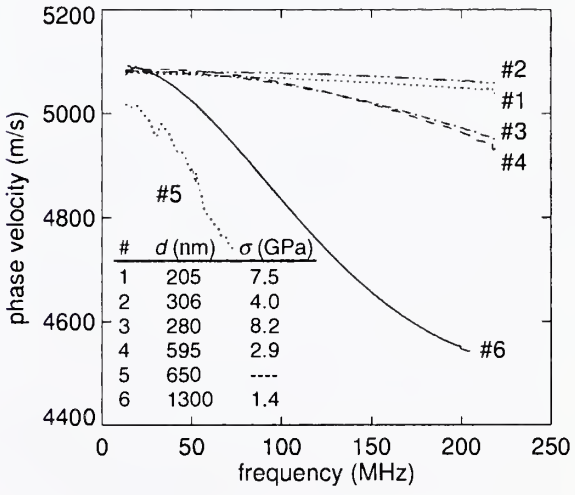
Schematic of laser-ultrasonic SAW apparatus.

Accomplishments

In this first year of the project, we created a laser-ultrasonic apparatus for broadband SAWs. The system incorporates a newly acquired 0.2-ns pulsed laser for SAW generation. We also significantly increased the bandwidth of our existing Michelson interferometer detector to over 800 MHz. We used this apparatus to measure dispersion relations in several samples, including TiN films on Si (thicknesses 200 to 1300 nm) and TiN films on stainless steel (thicknesses 250 to1000 nm). The maximum detected frequency with broadband methdods was over 200 MHz. To increase the frequency, we used different optical methods to generate narrowband SAWs up to 275 MHz. We are currently applying models developed in another project to extract elastic moduli from the experimental dispersion relations. The experimental results are also being compared with those obtained on the same samples using nanoindentation methods.

Output

The first results from this project were presented at the Review of Progress in Quantitative NDE conference (July 2000). A comprehensive journal article is in preparation.



SAW dispersion relations for Si samples with TiN films. The thickness d *and residual stress* σ *for each film are shown.*

Contributors and Collaborators

NIST participants: D. C. Hurley and V. K. Tewary
D. T. Smith, MSEL Ceramics Division

External collaborators: A. J. Richards, CSIRO Telecommunications & Industrial Physics (Australia)

X-ray Studies of Wireless Materials

Tom Siewert, Davor Balzar

Objective: We strive to correlate and explain macroscopic properties of technologically interesting materials by their underlying microstructure. We especially focus on studies of x-ray diffraction (XRD), ferroelectric, photovoltaic, semiconducting, and other materials relevant to the microelectronics and wireless communications industries. In particular, studies of microstructural properties, such as strain or stress, crystalline defects, and texture, complement the information obtained by the determination of short-range and long-range order in materials.

Background

Many macroscopic materials properties depend on internal stress, texture, and defect concentration. We develop methods to obtain this information from diffraction measurements and apply it to different materials of interest, in particular for microelectronics and wireless communications uses. Diffraction measurements are complemented with measurements by other experimental techniques, such as atomic-force microscopy (AFM) and scanning electron microscopy (SEM). Properties determined are then correlated with other physical properties measured, such as dielectric, ferroelectric, magnetic, and electric.

Accomplishments

In this period, we concentrated on three classes of materials: thin films of ferroelectric $Ba_{0.6}Sr_{0.4}TiO_3$ (BST) for tunable microwave devices, Si-B-C-N high-temperature dopable electronic materials, and $Cu(In,Ga)Se_2$ (CIGS) semiconductors for use as absorbers in photovoltaic devices. BST thin-film ferroelectric materials have received considerable attention because of their growing use in integrated, nonvolatile and dynamic random-access memories (DRAM), pyroelectric detectors, and acoustic transducers. Because these materials also exhibit nonlinear dielectric properties under external electric fields, they are exploited in tunable microwave devices, such as microstrip line phase-shifters and tunable filters. For these applications, it is imperative that the films exhibit high dielectric constant and tunability, and low dielectric loss. Losses can be decreased by doping small fractions of some metal ions. However, they depend greatly on residual stresses and defects. We studied the correlation between dielectric and ferroelectric (Curie-Weiss temperature) properties and residual elastic strain (stress) and defect concentration in pristine and W thin films of BST doped with 1% Mn and grown on substrate of $LaAlO_3$. Elastic strains and defect concentration increase for the doped specimens, as compared to the pristine BST thin film. This correlates with the change in both relative permittivity and Curie-Weiss temperature, and can explain the observed increased dielectric loss. These changes are explained in terms of Landau-Ginsburg-Devonshire thermodynamic theory. Agreement between theory and experiment is very good for our samples.

Amorphous, non-stoichiometric boron-doped silicon carbonitride (SiBCN) ceramics show excellent high-temperature structural properties, such as oxidation, creep resistance, and insensitivity to high-radiation environments. They are considered for similar applications as for SiC: Device types include RF and microwave power devices, cellular-phone base stations, phased-array radar systems, and small, lightweight RF and microwave transmitters. Other applications include low-intensity blue LEDs, aircraft and automotive engine sensors, jet-engine ignition systems, transmitters for deep-well drilling, and a number of measurement and control systems for industrial processes. The short-range structural properties in the amorphous phase are very important. We studied it by the radial-distribution-function (RDF) formalism. The preliminary results show it to resemble that of SiCN. The local structure comprises Si tetrahedra with B, C, and N at the corners. Measurements using x-rays of significantly higher energy (59 keV), with improved real-space resolution, are under way.

Thin film polycrystalline CIGS semiconductors have been investigated extensively for use as absorbers in photovoltaic devices because of their high optical absorption (exceeding 10^5 cm^{-1} at photon energies above band gap), optimum band gap (1.1 - 1.2 eV), which has an excellent match with the solar spectrum, and high quantum efficiency. Significant progress has been made in recent years and small laboratory-scale cells of CdS/CIGS have been demonstrated to have solar-to-electric conversion efficiency exceeding 18 %. However, difficulty arises in retaining the high efficiency when scaled up to square-foot modules. The CIGS films in this study were grown by sequential sputtering and physical-vapor-deposition processes. A maximum efficiency in excess of 10 % was achieved. Detailed characterization of the films revealed the fundamental parameters controlling the efficiency of the solar cells made from these films. The effect of annealing conditions (temperature and duration) on the CIGS film's microstructure and corresponding device performance has been investigated. Structure-property correlations were made using diffraction studies and Rietveld analysis. Auger spectroscopy and Rietveld refinement of x-ray diffraction patterns indicated that films deficient in copper resulted in higher efficiencies, which can be attributed to the p-type conductivity. Furthermore, presence of impurity phases such as $Cu_{1.8}Se$, which shows x-ray diffraction patterns very similar to those of the parent CIGS film, is detrimental to the solar cell's performance.

Contributors and Collaborators

Fred Fickett

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C.H. Marshall (Lockheed Martin Astronautics), C. Haluschka and R. Riedel (Technical University, Darmstadt, Germany)

COMBINATORIAL METHODS

The Combinatorial Methods Program develops new measurement techniques and experimental strategies needed for rapid acquisition and analysis of physical and chemical data of materials by industrial and research communities. A multi-disciplinary, multi-OU team from the Measurements and Standards Laboratories of NIST participates to address key mission-driven objectives in this new field, including needed measurement infrastructure, expanded capability, standards, and evaluated data.

Measurement tools and techniques are developed to prepare and characterize materials over a controlled range of physical and chemical properties on a miniaturized scale with a high degree of automation and parallelization. Combinatorial approaches are used to validate measurement methods and predictive models when applied to small sample sizes. All aspects of the combinatorial process from sample "library" design and library preparation to high-throughput assay and analysis are integrated through the combinatorial informatics cycle for iterative refinement of measurements. The applicability of combinatorial methods to new materials and research problems is demonstrated to provide scientific credibility for this new R&D paradigm. One anticipated measure of the success of the program would be more efficient output of traditional NIST products of standard reference materials and evaluated data.

Through a set of cross-NIST collaborations in current research areas, we are working to establish the infrastructure that would serve as a basis for a broader effort in combinatorial research. Within MSEL, novel and elegant methods for combinatorial library preparation of polymer coatings have been designed to encompass variations of diverse physical and chemical properties, such as composition, coating thickness, processing temperature, surface texture and patterning. Vast amounts of data are generated in a few hours that help understand how these variables affect material properties, such as a coating's wettability or phase miscibility. Additional focus areas for both organic and inorganic materials include multiphase materials, electronic materials, biomaterials assay, and characterization of materials structure and properties. State-of-the-art on-line data analytical tools, process-control methodology, and data-archival methods are being developed as part of the program.

In order to promote communication with and technology transfer to a wide range of industrial partners, an industry-National laboratories-university combinatorial consortium is being organized by MSEL. The consortium will facilitate direct interactions on combinatorial measurement problems of broad industrial interest and on efficient transfer of the methods developed to U.S. industry.

Contact Information: **Donna Hurley**

Modeling and Characterization of Combinatorial Libraries

Vinod K. Tewary

A variety of measurement techniques are being developed and adapted for screening and characterization of combinatorial libraries of functional materials. Methods include x-ray diffraction, scanning acoustic microscopy, dynamic atomic force microscopy, point-probe ultrasonics, thermal screening, and magneto-acoustic techniques. A theoretical model based upon the elastodynamic Green's functions is being developed that would give the local acoustic response of the library.

Technical Description

This year we have started a new program on modeling and characterization of combinatorial libraries of functional materials. A combinatorial library of materials is in the form of a thin film containing several (10^2-10^4) sites of new materials on a common substrate. A fundamental understanding of the physical properties of new materials requires knowledge of their elastic characteristics such as stress, elastic constants, and texture. Stress is a key factor that can influence mechanical, dielectric, thermodynamic, and optical properties of thin films. It limits the maximum thickness of epitaxial films, and alters ferroelectric domain structure, the Curie temperature, and tunability of microwave devices such as resonators, filters, and phase shifters.

Our goal is to develop a mathematical model and measurement techniques for evaluating the structural, mechanical, thermal, and magnetic properties of libraries of new electronic, magnetic, and polymeric materials. The technical challenge lies in developing quantitative measurement techniques with micron-scale spatial resolution, capable of rapidly scanning areas of a few square centimeters. New theories are required for modeling the elastic response of the library and developing techniques for inversion of the measured values to determine the parameters characterizing the library.

Accomplishments

A variety of measurement techniques are being developed and adapted for library screening and characterization. A theoretical model based upon the elastodynamic Green's functions is being developed that would give the local acoustic response of the library. A preliminary model that gives the local acoustic velocities in individual sites of the library for scalar waves has already been constructed. Measurement techniques include x-ray diffraction, scanning acoustic microscopy, dynamic atomic-force microscopy, point-probe ultrasonics, thermal screening, and magneto-acoustic techniques. Libraries of giant magnetostrictive alloys have been fabricated by our collaborators and are one of the combinatorial systems currently under investigation.

The Division's existing atomic-force microscope was adapted for measurements on libraries. An automated lock-in technique was implemented to acquire frequency spectra of the cantilever's response to dynamic excitation. Work is in progress to analyze the measured resonance shifts so that quantitative information (e.g., Young's modulus) can be obtained.

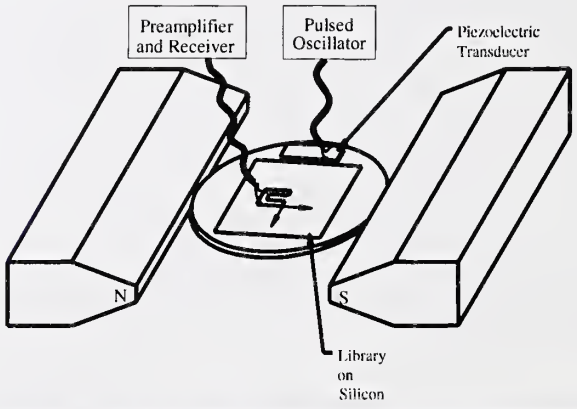
Acoustic microscopy is being explored as a technique for characterizing libraries, because acoustic microscopy can rapidly measure surface-wave velocities with sub-millimeter resolution. This year, a system for performing measurements in the 40-100 MHz range was implemented and tested on solid materials and homogeneous thin films.

X-ray measurements were made on a ternary (NiFe-Co-Tb) library on a 3 inch wafer. This library was fragmented to enable magnetic and x-ray measurements with existing equipment. The results are being analyzed and will be reported in the open literature shortly. These benchmark measurements will allow for a subsequent comparison with the measurements made on an identical library, but without fragmented sites, using just-acquired microdiffraction and precise positioning equipment.

A new method of measuring magnetostriction will be applied to libraries of magnetic films on a silicon substrate. The technique uses an ultrasonic wave in the substrate to generate dynamic magnetic fields above the film through its magnetostrictive coefficients. These fields are detected by a meander line coil carefully tuned to both the frequency and wavelength of the ultrasonic wave. A diagram is shown below.

Output

A preliminary mathematical model for scalar waves in combinatorial libraries.



Contributors and Collaborators

V.K. Tewary, G.A. Alers, D. Balzar, D.C. Hurley, W.L. Johnson, D.T. Read, A.J. Slifka, B.K. Alpert (ITL), S.E. Russek (EEEL)

ULTRASONIC CHARACTERIZATION OF MATERIALS

The program on Ultrasonic Characterization of Materials is directed toward development of model-based methods for measuring those ultrasonic properties of a material that will permit characterization of microstructural features with size scales ranging 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 as well as to provide physical-property values to support the design of improved microelectronic structures.

A primary focus of this program is the characterization of microelectronic structures such as interconnects and dielectric or magnetic layers as well as the microstructural features of structural alloys, composites, and engineered surfaces. The idea is to establish models that relate microstructure to measurable physical properties so that from measurements of appropriate ultrasonic properties, the salient microstructural features can be ascertained. For example, measurements of the ultrasonic wave velocity and attenuation can be related to the elastic properties of microstructural elements and mechanical relaxations that reflect the dynamics of atomic diffusion and redistributions of magnetic moments. These model-based measurements will enable industry to replace destructive microscopic methods with nondestructive techniques 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 material property modeling. We have worked with industry to commercialize noncontact ultrasonic transducers, wave-form-based acoustic-emission testing, magnetostrictive transduction, and nonlinear ultrasonics. Modeling advances include Green's function methods for rapid solution of problems involving wave propagation in layered, anisotropic materials, data-analysis procedures for deducing elements of the elastic modulus tensor of anisotropic polycrystals, and techniques to separate internal-friction values from ultrasonic attenuation measurements that are dominated by losses from scattering and beam diffraction. Current emphasis is directed toward measuring the elastic properties of the thin films used in microelectronic devices.

Contact Information: **George Alers**

Elastic-Stiffness Coefficients and Related Physical Properties

Our main goal is to understand, through measurements and modeling–theory, the elastic and related properties of solids that possess high scientific or technological interest. As required, we develop new measurement and modeling–theory methods.

H. Ledbetter, S. Kim, H. Ogi

Technical Description

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

Beside the elastic coefficients, we study sound velocities, internal friction, thermal expansivity, the Debye characteristic temperature, atomic volume, anharmonic properties (such as the Grüneisen parameter), creep, stress-strain behavior.

From the theoretical side, we can consider any physical property representable as a tensor. For example thermal conductivity, piezoelectricity, dielectric behavior, and so on.

Accomplishments

For the 2000 fiscal year, our accomplishments were enormous. This report's publications list gives titles of thirty-five of our manuscripts either published in the archival literature, submitted for publication, or to be submitted for publication soon.

These studies focused on the following materials:

- Cementite (Fe_3C)
- Ceramics
- Composites
  - Cu/steel
  - Diamond/Cu
  - NbTi/Cu/epoxy
  - NiTi/Al
  - SiC_f /Al
  - SiC_p/glass
  - SiC_p/Ti
- Covalent compounds
- Diamond crystals
- 8-N Compounds
- Langatate ($La_3Ga_{5.5}Ta_{0.5}O_{14}$) crystal
- Oxides
- Oxide superconductors
- Steel, low-carbon
- Steel, Cu-precipitated

Our 2000-fiscal-year publication list includes numerous review papers.

Perhaps our most notable achievement this year was contributing eleven chapters (about 250 manuscript pages) to the in-press Handbook of Elastic Properties of Solids, Liquids, and Gases, which will provide the standard elastic-property reference for many years. Also, to the forthcoming Encyclopedia of Materials, we contributed an invited review on solid-state lattice-vibrational properties.

Output

This report's publications list shows thirty-five of our manuscripts that emerged during the 2000 fiscal year.

Contributors and Collaborators

University collaborators (principal):

H. Ogi, Visiting scientist from Osaka University

M. Dunn, University of Colorado

P. Heyliger, Colorado State University

Dislocation Damping in Structural Alloys

This project seeks (1) to develop ultrasonic techniques for measuring load-induced changes in internal friction in structural alloys and (2) to develop theoretical models that relate ultrasonic measurements to changes in dislocation network structure and/or pinning. The accomplishment of these objectives will enable real-time evaluation of dislocation dynamics and provide a basis for nondestructive ultrasonic evaluation of the integrity of structural material during production or service.

Ward Johnson

Technical Description

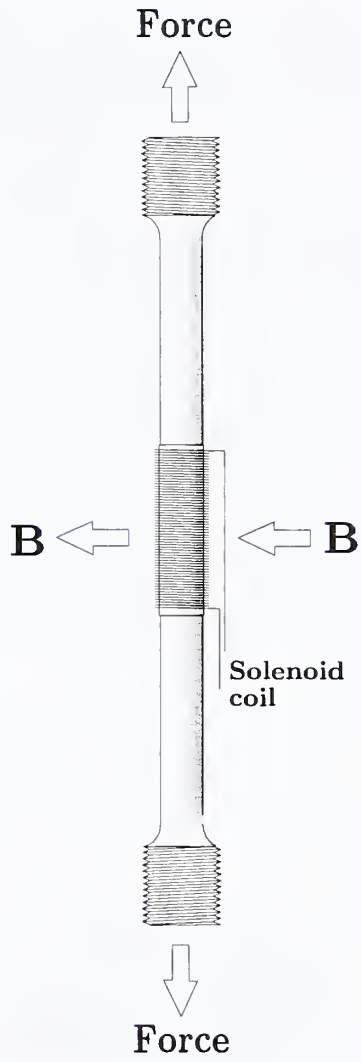
The dynamics of dislocations play a central role in determining the mechanical properties of structural materials. Measurements of acoustic damping and velocity are highly sensitive to these dynamics because of the anelastic response of dislocations. Although dislocation damping has been studied extensively in high-purity metals, it has been studied little in technologically important alloys and is poorly understood in these materials.

The experimental approach used in this research employs a unique laboratory system to measure anelastic ultrasonic effects in alloys during loading in a tensile testing machine. Specimens are designed to have resonant modes trapped in a central section with a slightly larger diameter, so that insignificant acoustic energy is lost through the machine grips (see figure). Loss of energy through transducer coupling also is eliminated by employing noncontacting electromagnetic-acoustic transducers. With this system, 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.

Accomplishments

Research during the past year has focused on a model Al (0.2% Zn) alloy. Measurements of resonant frequencies and damping were performed as a function of temperature following loading below the plastic regime. The observed changes in acoustic properties are found to be inconsistent with a single relaxation mechanism. (A similar conclusion was reached for previous measurements on interstitial-free steel.) The value of the activation energy for repinning of dislocations after loading, determined from the dependence of the recovery rate on temperature, indicates that vacancies are the pinning agents.

The results of this study were presented at the Dislocations 2000 conference in Gaithersburg, MD (proceedings to be published).



Specimen and ultrasonic transducer. A solenoid and a static B-field are used to excite and detect resonant vibrations with Lorentz-force coupling. Tensile forces are applied with a mechanical testing machine.

Contributors and Collaborators

Ward Johnson

Measurement of Residual Stress and Plastic Flow in Engineered Structures

The main objective of this program is to develop ultrasonic techniques and procedures that would allow measurement of residual stress distributions in large structures, surface layers and thin films. The frequency (wave length) of the waves used is expected to set the dimensional scale for the application

George Alers

Technical Description

Nondestructive measurement of the level of residual stress in large structures, coated parts and thin films is a long-term goal of the NDE community for predicting service-life and reliability. Since stress modifies the velocity of ultrasonic waves, it has often been suggested that an accurate measurement of the velocity of sound in a component could be used to infer or even measure the residual stress. Unfortunately, preferred orientation or texture in the grain structure of most materials introduces effects on the velocity of sound that mimic and often exceed the effects of the residual stress.

The objective of this project is to develop a procedure for measuring several ultrasonic wave velocities so that the effects of stress and texture can be measured separately. A secondary objective is to use those techniques that could ultimately be implemented on structures in the field or on components at a manufacturing facility. To date, our program has focused on the use of electromagnetic acoustic transducers (EMATs) for launching and detecting the ultrasonic waves because these transducers can operate in harsh environments, can interrogate the material with a wide variety of wave modes, and allow the velocity of sound to be measured very accurately.

During FY 2000, a mathematical model was developed to describe the effects of both stress and texture on ultrasonic wave propagation. This model described the material in terms of the two Lame' elastic constants of a random collection of cubic crystal grains, the three Orientation Distribution Coefficients (ODCs) that describe a polycrystal made up of cubic crystal grains with preferred orientations and the three third-order elastic coefficients (TOECs) that describe the response of an ultrasonic wave to a stress. Application of the model to samples with simple geometries subjected to uniform applied stresses and supporting the wave propagation modes available to EMATs have allowed the definition of calibration tests that can be used to determine all eight of these material constants.

Accomplishments

1. EMATs and jigs for making very accurate measurements of the phase velocity of Rayleigh waves, Love waves and surface-skimming longitudinal waves have been assembled so that the two Lame' constants, the three ODCs and the three TOECs of structural materials can all be measured from one surface of a plate.

2. Application of the model to thick plates and forgings of commercial alloys indicates that an EMAT probe can be developed for use on a factory floor to measure the residual stress gradients that distort parts when they are being machined from these materials.

3. Devices that focus x-rays and neutrons onto a sub-millimeter volume below the surface of plates containing sub-surface residual stress gradients have been assembled to verify the ultrasonic measurements described above with the x-ray and neutron-diffraction techniques commonly used to measure residual stress.

4. A rotating EMAT probe was used to measure the effects of elastic and plastic deformation on the velocity of ultrasonic waves propagating in the thickness dimension of a plate. The results are being used to assess the permanent damage to buried gas pipelines caused by dents or by earth movement.

Output

A chapter for a book was prepared on EMAT techniques for precision measurement of elastic constants.

A report on the ultrasonic measurement of residual stress and plastic deformation in the walls of a section of natural gas transmission pipeline permanently deformed in three-point bending was prepared for the Gas Research Institute.

Contributors and Collaborators

G.A. Alers, P.D. Panetta, J.D. McColskey, M. Dunn (Univ. of Colorado), H. Ogi (Osaka University), H. Haynes, The Gas Research Institute, D. Kerr, Pacific Gas and Electric.

Waveform-Based Acoustic Emission

G. A. Alers, M. A. Hamstad

The major project objective is to develop a firm scientific base that will provide the underpinnings upon which the potential enhancements to AE technology from an increased, high-sensitivity bandwidth can be obtained. Current secondary objectives include: (1) developing for many users the missing element of modeling AE signals for all relevant geometries; (2) developing rational application of artificial intelligence technology to the real-world problems of reliable source identification and source location of AE signals.

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result micro-sized transient energy releases in a material. Monitoring these waves can provide fundamental information about the location and mechanism of the transient-energy release as well as the history of such releases. Often the energy release is due to local micro-damage processes as a response to applied stress. The technical approach, which is beyond that currently commercially offered for either resonant or waveform-based AE technology, is to successively examine different aspects of 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 sensors or preamplifiers; high-speed wide-dynamic-range digital recording data-gathering systems; finite-element modeling to predict near- and far-field displacement waves from relevant AE sources; wideband experimental AE displacement waveforms from typical sources in materials of interest; signal-processing techniques to accurately identify source types and their locations; and experimental studies of simulated AE wave propagation. The scope in FY2000 covered three phases: (1) finite-element modeling of displacement signals (in the near and far fields) resulting from buried dipole point sources in laboratory-size samples as compared to large field-size samples; (2) examination of the parameters (hardware and software) required to move AE finite-element calculations from dedicated multiprocessor workstations to multiprocessor commodity PC systems; and (3) study of the application of artificial intelligence approaches applied to modeled AE signals for the purpose of identification and location of sources.

Accomplishments

1. With modeled AE wideband and narrowband signals it was demonstrated in samples with plate geometry that the nearby sample edges significantly distort the AE signals as compared to field-type samples without nearby edges. Specifically, the signal amplitude is reinforced by up to 14 dB in the small samples studied. This result significantly enhances signal detectability in the laboratory as compared to that in field applications. Also, the frequency spectrum experiences significant complications in the laboratory sample due to early arrival of edge reflections and mode conversions not present in the field sample. Thus source identification is more difficult in the laboratory samples as compared to field samples.

2. It was determined that commodity-type PC processors Currently have sufficient computational speed to carry out meaningful finite-element computations at total computation times similar to those for workstation type processors. But, current PC based systems lack the required large shared RAM memory. This result requires a Beowulf cluster approach with a distributed RAM memory. Key elements identified to enhance the computations with PC based systems include: size of L1 cache, size of L2/L3 cache, bandwidth between processor and memory and a switch-based hub (in the case of a cluster).

3. Initial studies of the application of artificial intelligence Techniques (e.g., neural nets) have strongly shown that initial signal processing of the wideband AE waveforms by a wavelet transform is one essential way to preserve the frequency-versus-time "fingerprint" of the AE source type (See Figure 1).

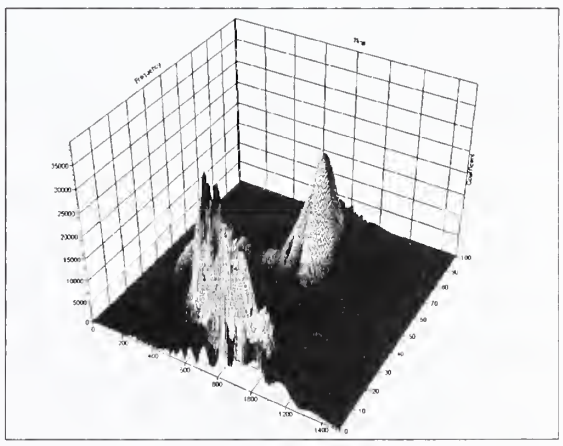


Figure 1. Wavelet transform showing fundamental extensional and flexural modes from an in-plane dipole source in a plate.

Contributors and Collaborators

NIST participants: John Gary, Abbie O'Gallagher, David McColskey, Jen Newton

NASA Langley participant: William Prosser

Green's Functions for Modeling Elastic Response of Electronic Materials

Vinod K. Tewary

A computationally efficient representation of the Green's function has been developed to model the elastostatic and elastodynamic characteristics of an anisotropic thin film on an anisotropic substrate. The model can account for surface texture, and imperfect interfacial bonding. The elastodynamic model is used to calculate the dispersion of surface acoustic waves in the film and also provides an efficient algorithm for the inverse problem of characterizing the film from measured values of the dispersion. The elastostatic model is used to develop a boundary-element computer program for stress analysis and is available on our interactive web site on Green's functions.

Technical Description

Green's functions give the response of a solid to a probe and are also called response functions. Green's function provides a powerful tool for modeling the elastic response of anisotropic solids that is useful in interpreting measurements of their elastic characteristics. The elastostatic Green's functions are used for time-independent problems, and the elastodynamic Green's functions are used for modeling the propagation of elastic waves.

We calculate the elastodynamic Green's functions by using a delta-function representation in slowness space that we had developed earlier. This representation has been found to be computationally very efficient for elastostatic as well as elastodynamic problems. In the delta-function representation, the Green's function at position vector **x** at time t is written as

$$\mathbf{G}(\mathbf{x},t) = \int \mathbf{g}(\mathbf{q})\, \delta\,[\mathbf{q}.\mathbf{x}-t]\, d\mathbf{q}, \qquad (1)$$

Where

$$\mathbf{g}(\mathbf{q}) = \mathrm{Lim}_{\varepsilon\to+0}\, \mathrm{Im}\,[\mathbf{\Lambda}(\mathbf{q}) - (1-\imath\varepsilon)\rho\mathbf{I}]^{-1},$$

$$\Lambda_{ij}(\mathbf{q}) = c_{ikjl}\, q_k\, q_l$$

is the Christoffel matrix in slowness space, ρ is the density of the solid, **I** is the identity matrix, **q** is a vector in the slowness space, and the integration is performed over the entire vector space of **q**.

We have applied this representation to model the elastic-wave propagation in thin films of electronic materials and combinatorial libraries of materials. The model accounts for the texture in the film by integrating over the orientation distribution function of the grains and also for defective interfacial bonding. Because of its computational efficiency, this model is also useful for the inverse problem of determination of g(q) from the measured values of the displacement field and wave velocities.

Equation (1) reduces to the elastostatic Green's function at t=0. Using this representation of the elastostatic Green's function, a computer program for boundary-element analysis is being developed by our collaborators at MIT.

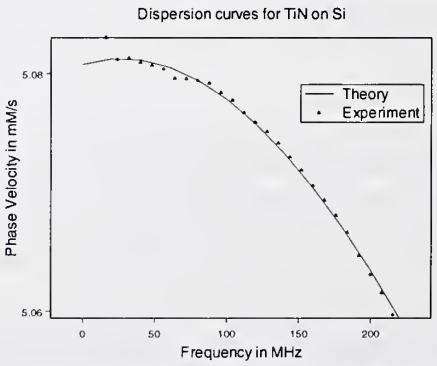
Accomplishments

We have applied the Green's function to calculate the velocities of surface acoustic waves (SAWs) in thin films of electronic materials. An efficient algorithm has been formulated for estimating the elastic constants from the measured SAW dispersion. The dispersion relations are used directly in the equation that determines the poles of the Green's function, which is then solved for the elastic constants. We have applied this method to estimate the elastic constants of thin polycrystalline films of TiN on Si. As shown in the figure given below, excellent agreement is obtained between the theoretical and measured values of the SAW velocities for polycrystalline TiN films on Si. The experimental values used in this work were obtained in a related project.

An interactive web site, sponsored by the Center for Theoretical and Computational Materials Science of NIST, has been set up that provides a platform for interaction amongst the researchers on Green's functions and serves as a repository of software and technical literature.

Output

1. A paper on elastostatic Green's functions is ready to be sent for publication.
2. A web site for Green's functions is ready: http://nistgf.kent.edu.
3. A paper on the elastodynamic Green's functions for SAW in thin anisotropic films of electronic materials is in preparation.
4. A computer program for estimating elastic constants from measured dispersion curves is ready and is available on request.



Contributors and Collaborators

NIST: V.K. Tewary and D.C. Hurley

External: L. Bartolo (Kent State University), Adam Powell (MIT), and J.R. Berger (Colorado School of Mines).

Atomic Force Acoustic Microscopy

We are developing in-situ *measurement techniques to probe mechanical properties on the nanoscale. Our methods involve dynamic excitation of an atomic force microscope cantilever at ultrasonic frequencies. The mechanical-property information obtained with this method will prove valuable for a wide range of applications involving thin films or small-scale structures. For example, we are developing AFAM tools to monitor the cryopreservation process in order to improve viability rates.*

Donna Hurley

Technical Description

Ever-decreasing length scales in many fields of technology presents a serious challenge for materials characterization. New nondestructive measurement tools must be developed to accommodate submicrometer dimensions. Specifically, the ability to determine mechanical properties on the nanoscale is needed in many applications, particularly in microelectronics. Knowledge of mechanical properties including elastic moduli and interfacial quality (defects, strain, adhesion, etc.) is critical to the successful development of new film materials and device assemblies. Likewise, nanoscale mechanical information could provide an assay tool for combinatorial methods of materials discovery.

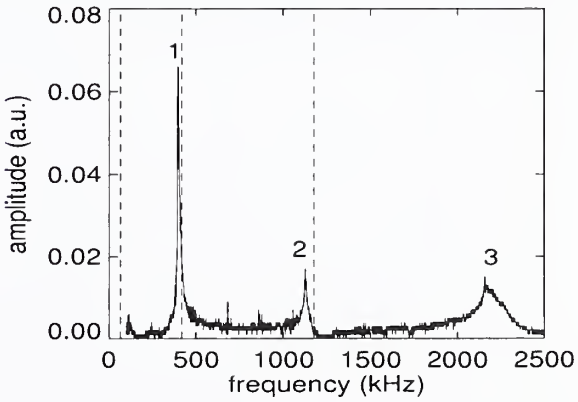
Applications in biotechnology could also benefit from nanoscale mechanical-property information. For instance, such information could be used to assess the integrity or reliability of biocompatible coatings, tissue scaffolding, and the like. Another example involves tissue cryopreservation. Although the ability to cryopreserve tissue underpins much medical biotechnology, success is limited by our understanding of the cryopreservation process. By sensing mechanical-property variations, nanoscale elastic techniques might differentiate between crystalline and non-crystalline ice formation and cell survival. In this way, such tools would help industry to better understand and optimize the cryopreservation process.

To meet these needs, we are developing measurement tools that exploit the spatial resolution of atomic force microscopy (AFM). Although standard AFM measures topography, other new techniques sense elastic properties. One promising approach, called atomic force acoustic microscopy (AFAM), involves vibrating the cantilever at ultrasonic frequencies (~0.1-3 MHz) to excite mechanical resonances. The resonant frequencies shift as the tip is brought in contact with a sample. By measuring the resonant frequencies under both free-space and surface-coupled conditions, quantitative information about the sample's elastic properties can be extracted. A major advantage of AFAM is that the small tip diameter (~10-100 nm) enables determination of *in situ* elastic-property information with nanoscale resolution. Also, the AFM's scanning ability offers the attractive possibility of obtaining 2D images of mechanical-property information.

Accomplishments

Since the start of this project in midyear FY 2000 we have adapted the Division's existing atomic force microscope to enable AFAM measurements. We have also implemented a lock-in technique to obtain frequency spectra of the cantilever's response to dynamic excitation. The data acquisition process has been automated since each spectrum would take several hours to acquire manually. The software sweeps the excitation frequency and records the resulting signal at that frequency as detected by the lock-in amplifier. A typical resonance spectrum acquired in this way is shown below. The figure indicates that the cantilever's resonant frequencies shift when the tip is in contact with a material.

We have acquired qualitative images of the relative elasticity by exciting the sample at a near-resonant frequency. However, we also wish to determine the elastic properties quantitatively. We are working to analyze the measured resonance shifts to obtain quantitative elastic property information (*e.g.*, Young's modulus). Trials on model materials with well-known properties, such as silicon, are in progress to validate our experimental methods. In addition, we have begun a collaboration to model the complex tip-sample interaction for a deeper understanding of the physical behavior. Although quantitative measurements are now made at a single point only, elastic imaging will follow naturally.



First three flexural resonances for a rectangular Si cantilever in contact with <100> Si. Dashed lines indicate the first three free-space resonances.

Contributors and Collaborators

NIST participants: D. C. Hurley, P. Rice, M. Varney, R. Adams
External collaborators: J. A. Turner, University of Nebraska-Lincoln

METALS CHARACTERIZATION

Engineering design depends on specification of the properties of the materials that are used, and manufacturers and their suppliers need to agree on how these properties should be measured. In many cases, they depend on measurements that can be traced to Standard Reference Materials (SRMs). This program generates SRMs for several quite different types of measurement. NIST is now providing certified Rockwell hardness test blocks for the C scale and is developing standards for additional scales. NIST is also providing leadership at national and international levels in the development of standardized test procedures and traceability protocols. Standards are also produced for microhardness.

The need for reliable magnetic measurements is becoming increasingly acute because of new technologies involving magnetic phenomena in data storage and microelectronics. Such measurements require calibration of magnetometers using certified magnetic standards in several different shapes and magnetic strengths, and with a wide range in magnetic character. These standards are now being produced under this program.

Coating thickness standards are produced by electrodeposition and are widely used for calibration of coating thickness measuring instruments. Coupons are produced with a wide range of thickness and are bar coded to allow analysis of degradation and life expectancy when the standards are returned for verification.

The Charpy impact machine verification project provides rapid, accurate assessment of test data generated by our customers using NIST SRMs, and, where merited, certifies the conformance of Charpy impact test machines to ASTM Standard E 23. NIST staff participates in ISO Committee TC 164, to ensure that our specimens and procedures remain compatible with associated international and regional standards

NIST provides SRMs for ferrite in stainless steel welds and maintains 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.

In the broader sense, a vast range of characterization technologies is needed by U. S. industry to understand the behavior of metals during processing and use. The Metals Characterization Program includes measurements of thermophysical properties of high-temperature materials, advanced magnetic measurement technologies, and measurement and analysis of the deformation properties of nanolayered materials.

The NIST effort in metals characterization has a strong emphasis on electron microscopy, which is capable of revealing microstructures within modern nano-scale materials and atomic-resolution imaging and compositional mapping of complex crystal phases with novel electronic properties. Our NIST microscopy facility is now being enhanced by the addition of a high-resolution field-emission scanning electron microscope capable of resolving features down to 1.5 nm. This FE-SEM maintains high resolution even on uncoated samples, and is equipped with a new detector to provide topographic and compositional images on the nanometer scale.

Contact Information: **Tom Siewert**

Charpy Impact Machine Verification

Tom Siewert

1. Provide rapid, accurate assessment of test data generated by our customers, and, where merited, certify the conformance of Charpy impact test machines to ASTM Standard E 23.
2. 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.
3. Participate in the activity in ISO Committee TC 164, so our specimens and procedures remain compatible with the associated international standards and other regional standards.

Technical Description

The Charpy impact test uses a swinging hammer to assess the resistance of a material to brittle fracture. The absorbed energy is measured from a calibrated scale, encoder, and/or an instrumented striker. The low cost and simple configuration of the test have made it a common requirement in codes for metals used in 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 letter 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 certified values for the NIST reference materials sold through the Standard Reference Materials Program Office. In addition, we have several more machines capacities of 3 to 400 J that are used for research purposes.

Accomplishments

We had about 1000 customers for this service in FY2000, 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 E 23. As usual, we found that many users took advantage of our support services, as shown by over 650 faxes and 2000 phone calls. This year, we implemented a new procedure to contact every customer whose machine fails to meet the verification criteria. In this contact (by phone, mail, email, or fax), we suggest corrective measures. 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.

We have started a new, three-year test program that will collect data on "International Master Batches" of Charpy impact verification specimens. A meaningful harmonization (equivalency) of Charpy V-notch standards around the world is unlikely until the reference materials used for the verification of impact machines in Europe, Japan, and the United States (EN-10045-2, JIS B 7722, and ASTM E23) share a more common method of certification. The results of this test program will be used to evaluate the use of Master Specimens as a common control in the certification procedure for CVN verification specimens between the three National Measurement Institutes. It will also evaluate machine variables, offsets, uncertainty, and other factors relevant to the harmonization of our respective systems. A major outcome will be the multi-year comparison of the equivalency of the energy scales used to measure absorbed energy by the United States, Europe, and Japan.

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 E 28 in Seattle, Washington. The 26 papers presented at the symposium have now been published in an ASTM Special Technical Publication (STP 1380).

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 (percentages of machines that meet the requirements and the distribution of data around the mean). The technical committee members have been quite pleased with our openness in sharing this data.

Output

NIST authors contributed to four of the papers in ASTM Special Technical Publication (STP 1380).

Contributors and Collaborators

NIST participants: Dan Vigliotti (Charpy Program Coordinator), Chris McCowan, Tom Siewert, Samantha Dimmick, Jesse Sycura, and James Alcorn. Industrial and academic collaborators: Members of ASTM Subcommittee E 28.07

Ferrite in Stainless Steel Welds

The major project objective is to maintain a supply of these weld ferrite SRMs for national and international users. Secondary objectives include: (1) improving the accuracy and traceability of the system used to assign ferrite numbers (FNs) to stainless weld-metal specimens, and (2) developing a fundamental calibration system based on primary units, such as magnetic field strength.

Tom Siewert, Chris McCowan

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. 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 distributed over the range of 30 to 100 FN.

In FY2000, we finished assigning certified values to all in-process material, and sent them to SRMP. At present, they have at least a five-year inventory of both ranges.

Our measurements identified several previously overlooked variables in the procedures and the instruments used to make the FN measurements. In addition, our measurements showed several ways in which the procedures in Standard AWS 4.2 might be improved. We continue to work with the Welding Research Council Subcommittee on Welding of Stainless Steel and Commission II of the International Institute of Welding (IIW) to incorporate these improvements in the next versions of the standard.

Long-Term Strategy

We plan 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 experts in IIW Commission II, 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.

Accomplishments

1. Assigned certified values and prepared the certificates and reports of error so that 56 additional sets of secondary FN standards could enter the SRM inventory.
2. 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.
3. Hosted the meetings of the ASTM Task Group on Revision of Standards A799 and A800, and the Steel Founders Society, May 3 to 4, 2000, where new markets for these SRMs were discussed.

Output

1. Prepared an overview of the measurement procedures (including the recently discovered improvements) and published this as NIST SP 260-141.
2. Reported on the inventory and technical issues at the Annual meeting of IIW Commission II, July 11, 2000.

Contributors and Collaborators

NIST participants: Chris McCowan, Tom Siewert, Dan Vigliotti, Samantha Dimmick, and Jesse Sycura.

Industrial collaborators: Malcolm Blair (Steel Foundry Society), Ron Bird (Steel Foundry and Engineering), Damian Kotecki (Lincoln Electric Company)

Service to Bureau of Reclamation

Project Objective: Provide assistance to the Bureau of Reclamation (BOR) on metallurgical issues that arise during the maintenance and inspection of dams and water conveyance infrastructure projects in the western United States. Typically, our advice regarding a particular issue is used by the BOR engineers as an independent check of other opinions they gather.

Chris McCowan

The physical infrastructure of the United States contains diverse elements, and the material issues continue to become even more complex as these structures age. To support their federally maintained infrastructures, the Bureau of Reclamation (BOR), within the Department of the Interior, has a Technical Service Center. When particularly complex problems call for additional expertise, they recruit experts from other government agencies and from the private sector. Our services are typically requested by staff in the BOR Materials Engineering and Research Laboratory, located at the Federal Center in Denver, Colorado. The close proximity makes it easy to distribute specimens or to meet in the microscope laboratory for comparison and discussion of observations on various specimens. Here we describe the interaction between BOR and NIST-Boulder on several recent materials issues.

One example of work we have done for the BOR is our evaluation of pre-stressing wire that failed on the Central Arizona Project (CAP). Examination of failed wires from the reinforced pipe, Figure 1, indicated that stress corrosion cracking initiated at pre-existing flaws in the wire, leading to the fracture of individual wires. Eventually, multiple wire failures severely degraded the structural integrity of the siphons, which convey water under roads and rivers in the CAP system. Our study of these failures has contributed to a change in the design of the pipes. The original siphons were replaced by steel-lined concrete pipes and the BOR has placed a moratorium on the pre-stressed pipe design that failed in the CAP. Repair costs have exceeded $35 million.

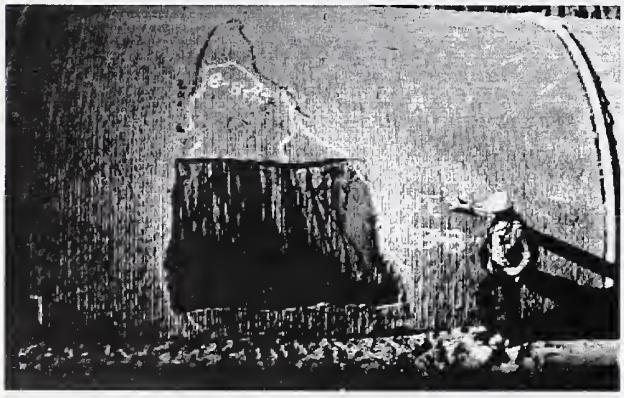


Figure 1: A 6.4 m diameter siphon from the CAP. This section was excavated for inspection in about 1990 to help determine the extent of distress in the CAP system.

Another example of the NIST-BOR interaction is the evaluation of a failed rod from a post-tensioned tendon in the Morrow Point Dam in central Colorado. In this case the engineers involved were fairly certain that corrosion was not an issue, since the rods are packed in grease and corrosion products were not apparent. However, the BOR Technical Service expert advised that the rod should be more closely examined and arranged for us to evaluate it. Cursory examinations at NIST revealed several sub-critical transverse cracks in the wire sample, likely due to stress corrosion cracking (Figure 2), and inhomogeneities in the microstrueture of the rod (not shown). The results indicated that a general cracking problem might be present in other tendons in the Dam. The project engineers are now considering replacement of all the tendons in the Dam.

This interaction between the BOR and NIST has continued for over 8 years, and has led to a sharing of expertise and increased public safety.

Figure 2: Example of a sub-critical transverse crack found in the post-tension rod from the Morrow Point Dam. The photo was digitally enhanced to show the elongated grain morphology in the rod.

Contributors and Collaborators

Chris McCowan

Mike Peabody and Tom Johnson, Bureau of Reclamation

METALS PROCESSING

The Metals Processing Program directs its attention to problems identified as important by industries ranging as widely as automotive, aerospace, coating, and microelectronics. The general nature of the problem is to understand the processing steps that will lead to products having the desired form and properties, at an acceptable cost. The types of processes considered range from melting and solidification of castings or single crystals to powder production and consolidation, and coating production by thermal spray and electrodeposition. The work thus uses NIST expertise in a wide range of disciplines, including thermodynamics, electrochemistry, fluid mechanics, diffusion, x-ray, and thermal analysis, and many others.

The use of metals and their alloys is based on physical properties that are developed through careful design of their processing cycle. This cycle can include many steps including a formation process such as solidification or electrodeposition, a heat-treatment process, and a deformation process such as rolling or stamping. In each of these processes, the distribution of crystal phases, the grain structure, the alloy's compositional segregation, and the defect structure are altered, with resulting changes in properties such as strength, ductility, corrosion resistance, and conductivity, which properties form the basic rationale for the use of metals in industrial products. 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.

This program applies advanced sensor and measurement systems to monitoring, diagnostics, and control of processes such as thermal spray, welding, and electrodeposition. All of these areas are important to the automotive industry, where low cost, high reliability, and rapid development of new products are critical. Sensors, usable in either a laboratory or industrial environment, are developed and applied to better understanding of processing phenomena or to feedback and control systems. Modeling plays a central role in several projects, allowing prediction of the correlation between processing conditions and the material response. Current modeling effort includes prediction of solidification, grain growth, and electrodeposition geometries by the phase field method, and prediction of solute diffusion in multicomponent materials.

Metals Processing projects with an especially strong focus on areas that are of special interest to MSEL have evolved to become part of other program areas such as Materials for Microelectronics and Forming of Lightweight Materials. Because processing plays such a basic role in determining the properties and performance of metals, we expect this program to continue providing a foundation for advanced metals technologies.

Contact Information: **Tom Siewert**

Weld Process Sensing, Modeling, and Control

Tim Quinn

Develop a better understanding of the underlying physics governing arc welding process through advanced instrumentation and data analysis techniques; develop simple, nonintrusive, and robust sensors that provide meaningful information about the status of the welding process; develop physics-based theoretical models of the process; and assist in the development of industrial standards for information exchange between intelligent components in a robotic arc welding cell

Accomplishments

A welding cell has been set up with its own web server to demonstrate the capabilities of the Internet to remotely monitor welding operations. The system demonstrated that a remote welding engineer could watch the weld being made (Fig. 1); receive and display sensor information; and communicate with the operator over the network. The sensor information is stored in real time in a networked database. The welding engineer can review the data from a particular weld, but can also manipulate the data from a web page to spot trends. The remote-sensing technology was demonstrated at a large industrial trade show, and the technology has successfully been transferred to industry and has resulted in a commercial product.

NIST personnel actively lead the development of a network standard for communication between components in the welding cell. The American Welding Society's A9 committee (chaired by NIST) is writing a standard (A9.4) to create a "plug and play" capability to integrate components in the welding cell. Currently, communication with a component in the welding cell is specific to that particular component, causing expensive, proprietary solutions to be generated for every weld cell's configuration. The A9.4 standard will allow any component in the weld cell to communicate with any other component using standard protocols and cabling.

Researchers at Ben Gurion University and NIST have developed a coupled model of the arc, electrode and weld pool in gas metal arc welding (GMAW) by use of a commercial finite-element code. In the GMAW process, a metal electrode carries a large current (i.e. 300 A). An arc is created between the end of the electrode and the work piece in the inert shielding gas. As the electrode melts, droplets detach and travel into the molten welding pool. Experiments were conducted this year to verify the results of the finite-element model. Cross-sections of the solidified weld pool were made for various welding conditions. The weld pool shapes can now be compared to the model's predictions. In collaboration with researchers at Ohio State University, the temperature of the arc at specific locations has been made and will be compared to the model as well.

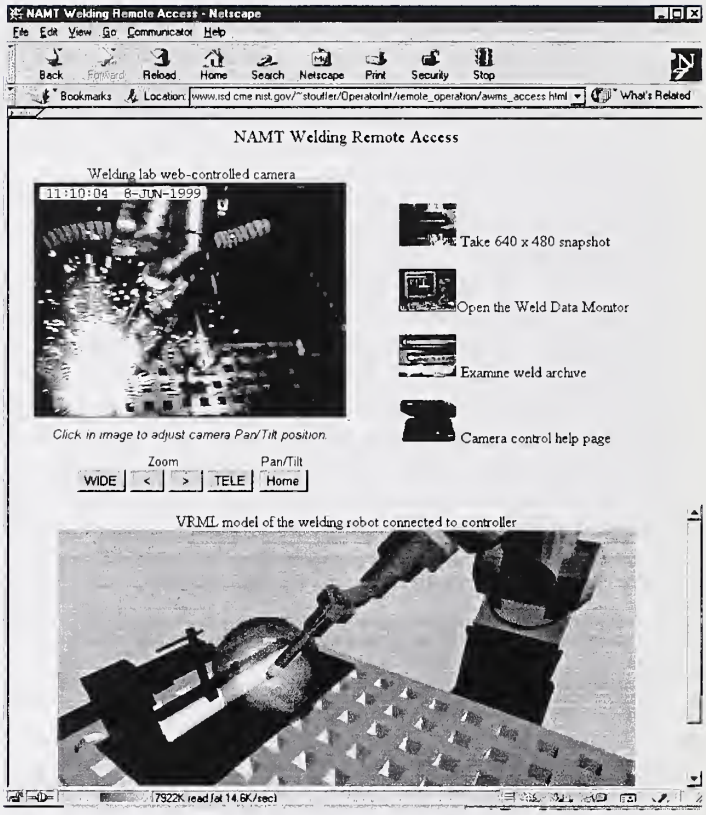


Figure 1: The video image of the part as it is being welded and a solid model image of the robot are displayed on a web page.

In conjunction with researchers at the Colorado School of Mines, NIST has developed a model of the wire in GMAW as it is being pulled through the conduit. The model can predict the friction between the wire and the liner. Experiments were conducted using a linear actuator to smoothly pull the wire through a bend in the conduit at a specific angle. Results show agreement with the model within a few percent.

Contributors and Collaborators

Tom Siewert
Bill Rippey, MEL
Toby Padilla and David Munoz, Colorado School of Mines
Moti Szanto, Ben Gurion University, Israel
Dave Farson, The Ohio State University

High-energy X-ray Diffraction Studies

Tom Siewert

Once high-value products are manufactured, it is often impossible to remove sections for destructive analysis without damaging the component. Yet, destructive mechanical tests are often specified as the reference method in evaluating whether an overheated or mechanically damaged structure is still fit for service. This project investigates the use of high-energy x-ray diffraction as an alternate, nondestructive option to the conventional destructive methods for measuring physical properties.

Background

The x-ray diffraction technique permits direct and unequivocal identification of the crystallographic phases in a component. Furthermore, the diffraction intensity is proportional to the concentration, so the method can also estimate the relative concentration of the phases. Yet, x-ray diffraction is often overlooked as a nondestructive analysis tool because of the limited penetration depth (a few micrometers) of conventional diffraction systems (often using 8 keV x-ray energies).

We use high-energy x-ray tubes with potentials up to 320 kV. In previous studies, we have demonstrated the ability of these x-rays to penetrate more than 10 mm of nickel alloy, as we monitored the solidification of a turbine blade while it was in a vacuum furnace. We have now moved this equipment to a general-purpose shielded room, where we can perform a wider range of experiments. We have a high-quality x-ray imager with hardware and software for acquisition and processing of the 2-D diffractograms and a solid-state detector for energy-dispersive measurements. New specimen-manipulation equipment was installed and calibrated in FY1999 and 2000. Toward the end of FY2000, new high-brightness tubes arrived and have been integrated with new collimators and the precise sample-positioning system. While many laboratories have SEMs and low-energy x-ray diffraction systems (under 20 keV), we are among the few laboratory facilities (other than synchrotron sources) in the U.S. that conduct x-ray diffraction studies at high energy. Thus, our goal has been to use our system as a testbed for demonstrating its capability through industrial interactions.

Accomplishment

This year we collaborated with Digiray Corporation on the examination of several specimens of 7150 aluminum specimens. These specimens had been subjected to various heat treatments that affected the mechanical properties. The specimens were evaluated to determine whether x-ray diffraction could serve as a nondestructive sensor to distinguish the microstructural changes associated with the heat treatment. More important, however, for industrial users is the ability to nondestructively assess possible microstructural changes on parts in service, which could be buried or otherwise not directly accessible. This would require the use of high-energy penetrating radiation in most cases.

While our equipment was being upgraded, we examined the specimens with Cu characteristic radiation (8 keV). The comparison between an "optimally annealed" and "overheated" specimens has shown differences in the respective diffraction patterns (see Figure 1). Although the diffraction lines stemming from major crystallographic phases present in the specimens were unchanged, there was significant broadening of diffraction lines belonging to a minor phase in the "overheated" specimen. Although this change is easily registered in our specimens, further work at higher energies will have to show how feasible the measurements would be in the field, where the beam has to penetrate through different obstacles, such as paints, coatings, and layers of other materials.

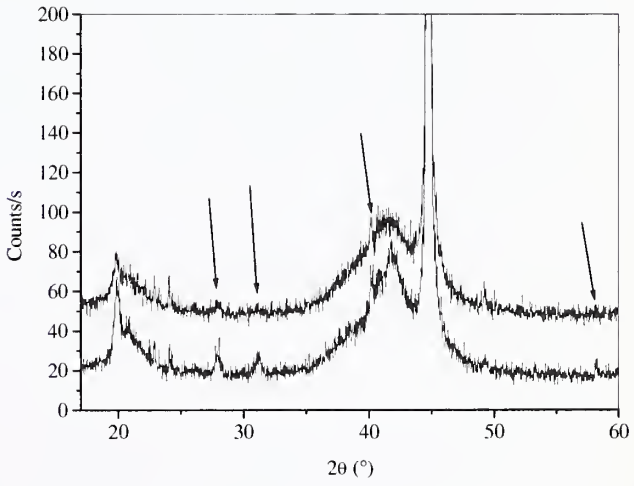


Figure 1. Diffraction patterns of optimally annealed (lower) and overheated (upper) samples. Arrows mark positions of affected diffraction lines.

Contributors and Collaborators

Tom Siewert, Davor Balzar

Dick Albert, Digiray Corporation

FORMING OF LIGHTWEIGHT MATERIALS

Automobile manufacturing is a materials-intensive industry that involves about 10 % of the U.S. workforce. In spite of the use of the most advanced, cost effective technologies, this competitive industry still has productivity issues related to measurement science and data. Chief among these is the difficulty encountered in die manufacture for forming of sheet metal. In a recent Advanced Technology Program sponsored workshop (The Road Ahead, June 20-22, 2000, at USCAR Headquarters), the main obstacle to reducing the time between accepting a new design and actual production of parts was identified as producing working die sets. This problem exists even for traditional alloys with which the industry is familiar. To benefit from the weight saving advantages of high-strength steel and aluminum alloys, a whole new level of formability measurement methods and data is needed, together with a better understanding of the physics behind metal deformation.

To meet these industrial needs, the Metallurgy Division has developed a program that encompasses standard formability test methods, multiscale, physically-based constitutive laws, and consolidation of aluminum-matrix composites. In the past year, we have established a sheet-metal formability laboratory. A state-of-the-art formability testing machine equipped with an advanced surface displacement analysis system permits us to investigate industrially important measurement problems in formability and pursue standard test methods for formability. The facility provides test samples of biaxially deformed metal for other aspects of this program. For example, deformation-induced surface roughening of sheet metal is a poorly understood phenomenon that is highly relevant to industry. We are currently performing controlled experiments on biaxially strained sheets to develop a surface roughening database and a generic model that industry has identified as a need of high priority. On a more fundamental level, we are using MSEL's advanced characterization capabilities (TEM, Synchrotron Radiation, NCNR) to understand the basic dislocation patterning responsible for the observed behavior of metals. A predictive model based on percolation theory has been developed from the measurements and observations. All aspects of the research at NIST will impact our customers by improving the commercially available, finite-element computer codes that are heavily used by this industry. A key element in the design of this program is that an insight or advancement gained in one area can be immediately used in a piecewise fashion in the design process, i.e., total success of the program is not required to have an impact. Other means of transferring this technology, such as through standardizing organizations and by direct interaction with industrial counterparts, are being pursued. While targeting the auto industry, our research will have extended applications to all other industries that employ metal forming in their production lines.

Contact Information: **Yi-Wen Cheng**

Aluminum Formability

1. *To develop test specimens from which data compatible with finite-element formability analyses can be generated.*
2. *To generate data for input to finite-element analyses.*
3. *To characterize the full-field plastic-flow behavior of aluminum sheets during biaxial deformation.*

Yi-Wen Cheng

Technical Description

Using aluminum sheets for automotive components has been restricted by 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 method 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, 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.

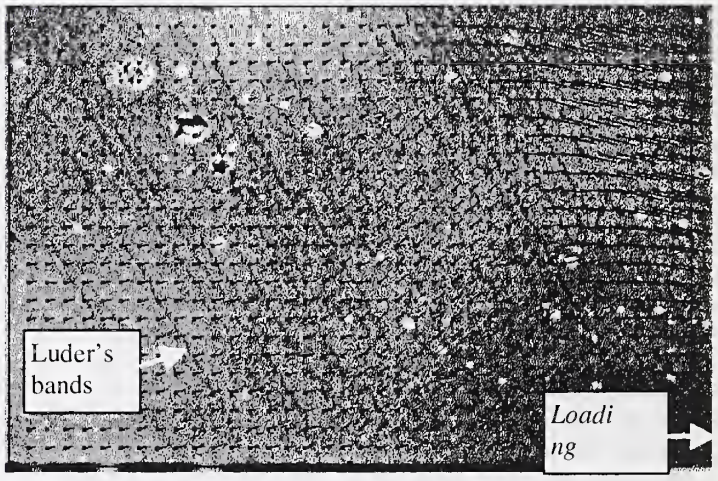
Accomplishments

An apparatus for loading tensile panels with the capability of constant image and load acquisitions has been set up. The apparatus and a software, which performs digital-image-correlation analysis, are being used to measure the tensile properties and to characterize the strain pattern of tensile panels of the 2024-T3 aluminum alloy.

The results of testing 88.9 mm wide tensile panels showed that the specimens were under predominately plane-strain condition. Mechanical properties of Young's modulus, 0.2 percent yield stress, and ultimate tensile strength determined from the tests were consistent with values cited in the literature. The results also showed that the Luder's bands began to develop at a nominal axial strain of 7.15 percent. Results were sent to Metallurgy Division for incorporation into the finite-element analysis.

Output

Full-Field Strain Measurement of Aluminum-alloy Panels, to be published.



The above image shows plastic-flow pattern and Luder's bands in a test of aluminum tensile panel. Lines emanating from dots are displacement vectors displaying the magnitude and direction of displacement at a given point. The panel was stretched at a nominal axial strain of 10.6 percent.

Contributors and Collaborators

J. David McColskey
Richard J. Fields and Tim J. Foecke, Metallurgy Division
Brian Armstrong, University of Colorado

CERAMIC COATINGS

The Ceramic Coatings Program addresses plasma spray deposited and physical vapor deposited ceramic thermal barrier coatings (TBC) used in aircraft, land-based turbines, and diesel engines as well as wear-resistant coatings used in many applications. These materials are a significant portion of the nearly one-billion-dollar North American market for ceramic coatings. A primary goal of this program is to improve the reliability of ceramic coatings. Collaborations have been established with 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, to enable research on relevant materials and to transfer results to users. Collaborations are also underway with Bundesanstalt für Materialfürschung 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. 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.

Participants in the NIST program are located in MSEL, i.e., Ceramics Division, Materials Reliability Division, Metallurgy Division, and the NIST Center for Neutron Research, as well as in the Chemical Science and Technology Laboratory.

The program has the following elements:

- Development of predictive models for the long-term reliability of ceramic coatings under operating conditions.
- Relating microstructural characteristics such as fine voids and phase stability to thermal and mechanical properties.
- Developing and validating microstructure based models that predict coating performance.
- Development of measurement methods such as online instrumentation for improved control of thermal spray processes and thermal properties.

Contact Information: **A.J. Slifka**

Thermal Conductivity of Ceramics and Ceramic Coatings

Andrew Slifka

Objective: To develop steady-state measurement techniques and appropriate reference materials for thermal conductivity of ceramics and ceramic coatings. This provides the thermal-barrier-coatings industry with calibration and transfer between transient and steady-state data, allowing understanding of the relationship between thermal performance and microstructure.

Background

Thermal-barrier coatings have many applications, but the primary use is in engines, to allow higher operating temperatures and longer lifetimes to increase efficiency. New materials with designed microstructures for lowering thermal conductivity are being developed by industry. Industry uses a measurement of thermal diffusivity, that is fast and generally reliable, but a bridge is needed to provide designers with thermal conductivity and to calibrate diffusivity measurement apparatus. We have provided steady-state thermal conductivity measurement methods and data, and development of appropriate reference materials.

Accomplishments

We have measured two compositions of yttria-stabilized zirconia materials for potential use as high-temperature reference materials for thermal conductivity. We are currently measuring a third composition because it may prove to have greater phase stability than either of the others. We are measuring the phase composition and phase stability as a function of thermal aging of the two measured compositions. Figure 1 shows thermal conductivity data for the two measured compositions. One material has a predominantly tetragonal phase composition and the other is predominantly cubic. The cubic material has low enough and constant enough thermal conductivity to have a wide range of application as a standard reference material for thermal conductivity.

We are measuring ceramic coatings made by physical vapor deposition (PVD). These coatings are designed to lower thermal conductivity by controlling the coating microstructure. By adjusting process parameters, not only can the porosity be controlled, but the microstructure of the porosity as well. Figure 2 shows a PVD coating with low thermal conductivity due to a 3-level porous microstructure. The results of this study have been submitted for publication.

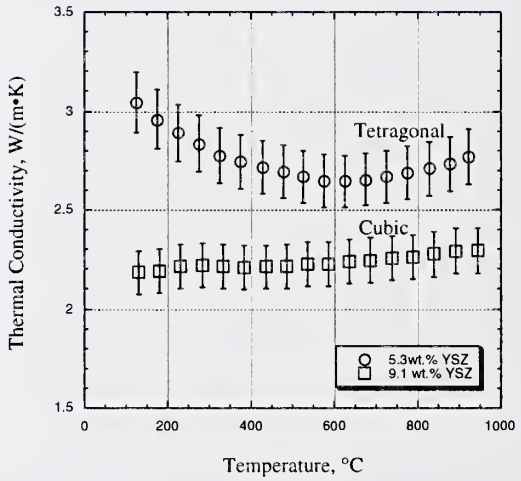


Figure 1. Thermal conductivity results for 2 candidate standard reference materials for thermal conductivity.

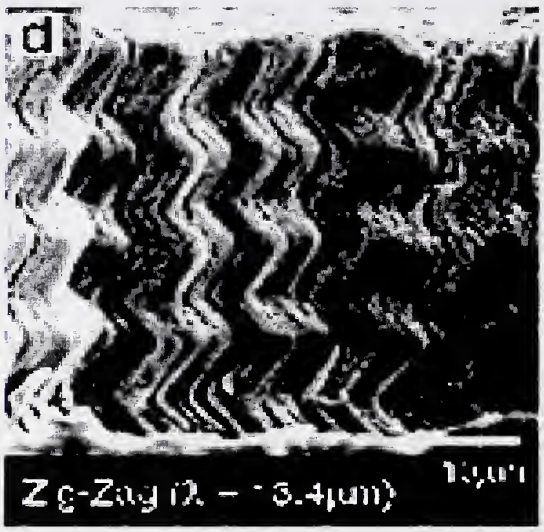


Figure 2. Ceramic coating made by PVD that exhibits low thermal conductivity due to a controlled porous microstructure.

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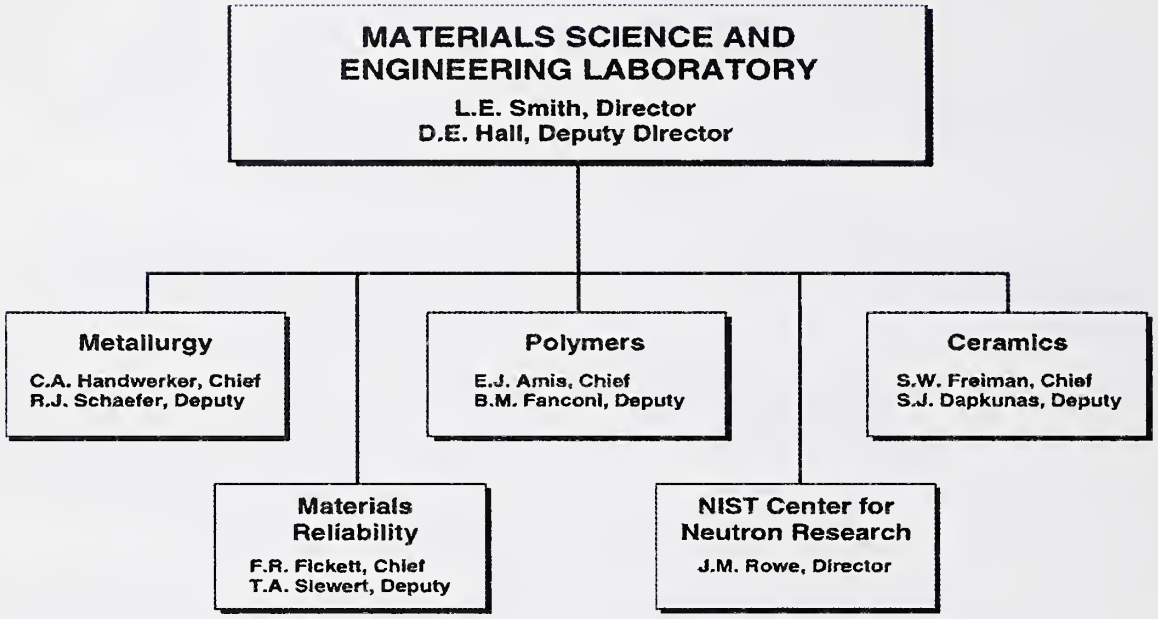
Organizational Charts



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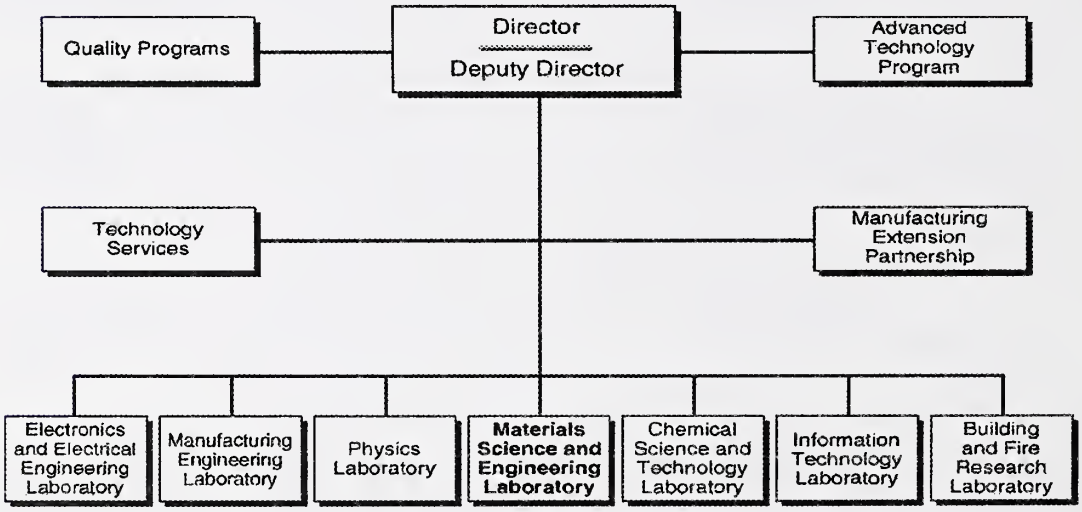
Organizational Chart



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