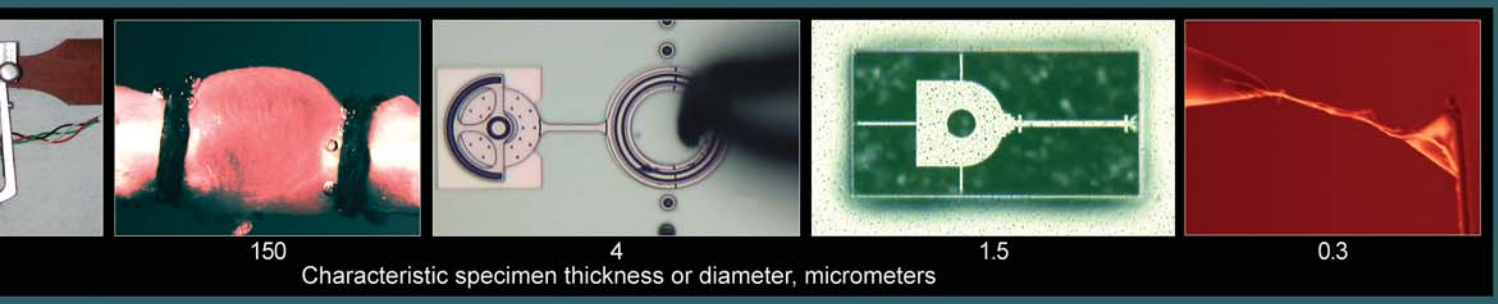
Materials Reliability Division



Materials Science and Engineering Laboratory

FY 2002 Programs and Accomplishments

NIST
National Institute of Standards and Technology
Technology Administration
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NISTIR 6905
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On the Cover:

Reliability of Modern Materials

Materials Reliability Division research focuses on reliability issues in structures on many scales. Advanced measurement and modeling methods are developed and applied to evaluation of failure modes and mechanisms along with research on the basic physics and materials science of failure. The images on the cover show the size range of tensile test specimens measured this year. They are the following (the numbers are dimensions in micrometers): 0.3 – carbon nanotube; 1.5 – CMOS aluminum film; 4 – MEMS polysilicon; 150 – rat pulmonary artery; 8000 – structural steel.

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Materials Science and Engineering Laboratory

FY 2002 Programs and Accomplishments

Materials Reliability Division

Fred R. Fickett, Chief

Thomas A. Siewert, Deputy

NISTIR 6905

September 2002

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Executive Summary

The Materials Reliability Division mission is to develop and disseminate measurement methods and standards enhancing the quality and reliability of materials for industry. Our focus in FY02 continues development of measurements for materials evaluation in micro- and optoelectronics. These nanoscale measurement issues have also led into areas not specific to the two electronics technologies. The use of advanced atomic force microscopy with probes based on nanotubes has opened up the broad field of nanotechnology. Division expertise in property measurement development is also being applied in biomaterials metrology in collaboration with local universities and research hospitals. We see this area as one with significant potential for growth. In spite of the small-scale focus and the associated new materials, our work continues to cover the full range of traditional materials with dimensions from the very small to tall buildings, gas pipelines and bridges. The addition of Homeland Security to the national agenda has brought new life to some of these activities. Foremost among these is the commitment of NIST to a major role in the investigation of the World Trade Center disaster. The Division expertise in measurement of properties of metals under both normal and unusual conditions has resulted in a significant activity in this area. Our Standard Reference Materials program continues to provide support of the many instruments worldwide that insure the accurate determination of impact resistance of structural steels. In FY02, the three Groups in the Division pursued the following research directions.

Microscale Measurements

Constrained volume materials systems are studied in terms of their mechanical, thermal, and electrical behaviors. This often necessitates the development of new precision measurement techniques. In the past year, we have broadened our traditional interconnect and electronics packaging industry focus to include work that ties to optoelectronics and biomaterials. This means branching into classes of materials beyond the metals, polymers and ceramics that we have traditionally studied. We now also develop very fine scale measurements for semiconductors and biological tissue. Structure–property relationships are investigated using high-spatial resolution imaging techniques such as electron microscopy and scanned probe microscopy, coupled with precision property measurement methods such as microtensile testing, thin film biaxial testing, scanning thermal microscopy, and wafer-level electrical testing. Key features of our research include concentration on fundamental changes in behavior due to dimensional scaling and bi-material interfaces, and mapping of microstructure and microproperty distributions.

Microstructure Sensing

FY02 activities involved a variety of nondestructive techniques to evaluate the microstructural properties of materials. Many projects involved acoustics and ultrasonics, for instance: development of Brillouin light scattering facilities to probe phonon-magnon interactions in magnetic switching applications; resonant ultrasound experiments to examine intrinsic loss mechanisms in langasite materials under consideration for next-generation microelectronics; and optical measurements of surface acoustic waves to determine elastic properties of thin films such as low-k dielectrics and superhard nanocomposites. Other projects involved new approaches to scanned probe microscopy for nanoscale materials characterization, such as carbon nanotube microscopy for electrical properties and atomic force acoustic microscopy for mechanical properties. Theoretical work included development of Green's function and multiscale modeling methods for diverse applications such as nanoindentation and quantum dots.

Process Sensing and Modeling

The projects in this group are designed to develop measurement technology for determining a material's characteristics or to characterize a measurement system. In FY02, we reduced our activities in welding, and expanded our activities in lead-free solders and biomaterials. Specifically, we continued to update our material property database for lead-free solders, while taking a larger role in a NEMI round robin on solder reliability and alloys. In the Charpy SRM program, we published a collection of our studies for the past decade, and finished a video on machine verification. In biomaterials, we installed several new systems to measure material properties, and we have developed tests to verify these systems. For other agencies, we studied repairs proposed for Folsom Dam, developed repair procedures for the U.S. Capitol dome, and will be measuring properties of steels used in the World Trade Center.

Division Chief's Commentary

The Materials Reliability Division has expanded into new directions in FY02. This research is concentrated in the NIST Strategic Focus Areas of Nanotechnology and Health Care as well as Homeland Security. The growth has been based mainly on our existing expertise. In areas where components were lacking, we have arranged successful collaborations with outside individuals and research groups. We continue to be able to acquire equipment that allows maintenance of our ability to be a leader in development of test techniques for material behavior on finer and finer scales. We also maintain a capability in infrastructure support work, for which the Division was well known for many years. As discussed above, this capability proved to be especially valuable this year. The Division management has changed with the assignment of two new Group Leaders. New staff has been added, both in the research and technical support areas, and staff reassignments to new Groups and research areas were completed. We continue to rely on a large cadre of guest scientists, postdocs, and students. Morale and productivity continue at a high level.

Fred Fickett
Chief, Materials Handling Division

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. The Technical Highlights include:

- Carbon Nanotubes: Characterization and Application for Nanometer Scale Devices
- Strain in Compound Semiconductor Photonic Systems
- Biomaterials Metrology Project

Carbon Nanotubes: Characterization and Application for Nanometer Scale Devices

The Materials Reliability Division believes that research on carbon nanotubes and their applications can significantly support economic opportunities in nanotechnology. Carbon nanotubes have a unique set of characteristics that offer promise for a variety of nanometer scale applications. The carbon–carbon bond is among the strongest known atomic bonds, and produces amazing mechanical strength. Because of their outstanding thermal conductivity, nanotubes are also being considered for thermal transport applications. In addition, recent reports envision nanotubes performing multiple functions in future computers: both as switching elements and as interconnections. We are, therefore, developing nanometer scale measurement, manipulation, and bonding techniques to enhance future nanometer scale devices.

Technical Description

The Materials Reliability Division has begun research on carbon nanotubes in two application areas: as probes in advanced imaging and as materials for mechanical and electrical structures. Starting this research required us to develop an understanding of the different types of nanotubes available, their sources, and techniques for handling them. Because nanotubes are such new materials, and new to this Division, a brief introduction is given.

Carbon nanotubes are essentially graphite sheet structures that are rolled into tubes. They can be single sheet tubes, referred to as single-walled, or they can be multiple concentric tubes, referred to as multi-walled. Several types of nanotubes are available commercially in quantities of the order of a gram. One manufacturer's catalog lists five types of multi-walled nanotubes and one type of single-walled. The type that we used for probe tips for atomic force microscopy (AFM) differs slightly. These are a number of multi-walled nanotubes twisted together and referred to as nanotube ropes. These ropes are large enough to view in an optical microscope. Figure 1 shows various diameter multi-walled nanotubes.

Accomplishments

This year, FY02, we have developed methods to handle the nanotubes and used them in several experiments. We have attached nanotubes to an AFM probe, to map surface conductance on a nanometer scale, and to a mechanical strain-testing device mounted in a scanning electron microscope (SEM) that allows imaging of the nanotube as it is strained. We have also joined with the Electronics and Electrical Engineering Laboratory to explore the high-frequency response of nanotube AFM probes. Within the Division, we are also working on multiscale modeling, so that designers can model, for example, the effect of imposed deflection on the attachment of a nanotube to its mount within some device.

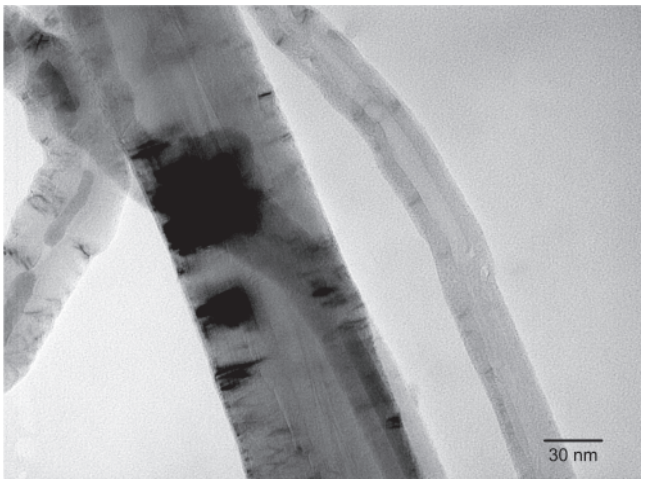


Figure 1: *Transmission electron microscope (TEM) image showing multi-walled nanotubes of different diameters. These are large-diameter tubes, from a set obtained for testing as AFM probes. Single-walled tubes are much smaller, from 1 to 5 nm in diameter.*

Developing the manipulation and attachment processes has required the largest effort so far. To attach the tubes, the first handling step is to remove impurities from the bulk nanotubes with solvents. Next a drop of solvent with nanotubes is dispensed on a substrate such as a glass slide and allowed to dry, leaving the tubes lying flat. Adhesive tape is then used to pick up and hold several nanotubes for access and manipulation under the optical or scanning electron microscope.

The next operation is to prepare the target to which the nanotube will be attached. The target is mounted on a micromanipulator. We have used a micromanipulator controlled by manual micrometers under an optical microscope and one driven by piezo-stepper motors in the SEM. To practically view thick-walled nanotubes optically, an optical microscope, with a very long working distance and a very bright oblique illuminator, was obtained.

To mount a nanotube to an AFM tip under the optical microscope, the AFM tip is mounted on the micromanipulator and then touched to a carbon impregnated conductive adhesive. A small submicrometer-sized bead of adhesive is subsequently attached to the tip as it is pulled away. The AFM tip is

then positioned to an appropriate nanotube, and the nanotube is applied to the tip. A nanotube rope attached to an AFM tip is shown in Figure 2. The bead of adhesive can be seen at the end of the pyramidal AFM tip. From this image it is clear that the sensing portion of the nanotube is much smaller than the end of the AFM tip. Claims of an order of magnitude increase in image resolution have been reported in the literature.

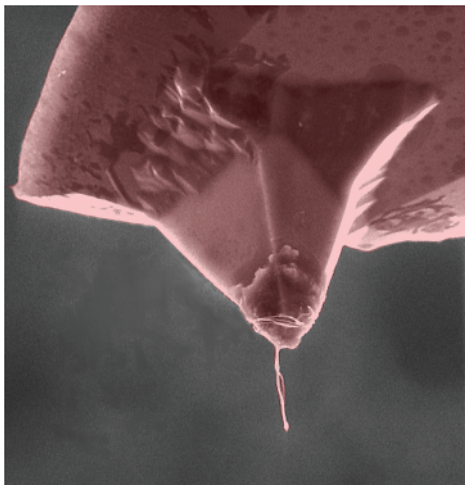


Figure 2: *Scanning electron microscope (SEM) image of nanotube attached to an AFM tip with applied adhesive. The nanotube is actually a rope of several tubes about 100 nm in diameter.*

Attaching nanotubes in the SEM begins with a sharp tungsten probe mounted in a piezo-driven micromanipulator. The tungsten probe is brought in contact with the nanotube, and the electron beam is focused and left stationary where the two come together, producing a carbonaceous deposit. This technique, called electron beam deposition (EBD), produces a strong bond on the nanometer scale. Next, the nanotube

Figure 3: *SEM image of tungsten probe (bottom of image) attaching a 20 mm long nanotube rope to the end of an AFM tip (upper left) using EBD. After this nanotube rope was attached, it was pulled until the tube separated from the tungsten probe.*

is moved to the AFM tip or other device for attachment, again using EBD to bond the two together. We have made preliminary tensile measurements using this procedure by viewing the deflection of the AFM cantilever as the tungsten probe is pulled away from the AFM cantilever. We have found the EBD bond can survive micronewton forces (Figure 3).

Microelectrical mechanical systems (MEMS) are developing rapidly. Nanotubes can be used with MEMS as mechanical and electrical components. Mechanical and electrical characterization of the bonding techniques is critical to developing the full potential of nanotubes on MEMS. We can easily apply the micromanipulation procedures we have developed to test various bonds (Figure 4).

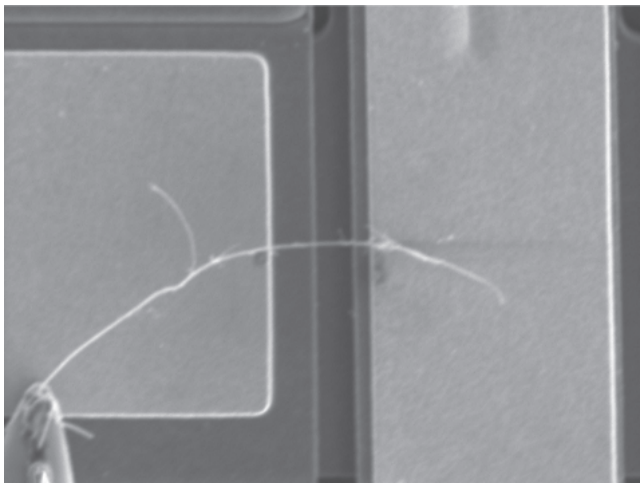


Figure 4: *SEM image of attaching a nanotube to a MEMS device, in progress. Development of carbon deposits can be seen near the edges of the MEMS under the nanotube. The nanotube rope is about 100 nm in diameter. The tip of the tungsten probe that maneuvers the nanotube can be seen in the lower left corner of the image.*

Future Research

More details on our research on the use of nanotubes as probes in advanced imaging appears later in this report. Future research on nanotubes as mechanical materials includes developing systematic procedures for mechanical measurements on nanotubes of different types. In addition, we plan on applying these attachment techniques to mechanical measurements of other submicrometer tube or fiber-like materials, such as collagen fibrils, and developing a practical modeling capability for nanotube-device attachments.

For More Information on this Topic

P. Rice, D. Read

Strain in Compound Semiconductor Photonic Systems

The photonics technology industry develops products that often contain strain. Sometimes the strain is intentionally introduced, as in the case of highly strained quantum wells, while at other times it is undesirable, as in the case of oxide-confined vertical cavity surface-emitting lasers (VCSEL)s. Industry seeks to better understand and control strain development in such systems. We approach this problem in the Materials Reliability Division with two efforts — one concentrating on high spatial resolution measurement of strain using electron microscopy methods; and the other on theoretical modeling of development of strain energy during heterostructure growth using Green's Functions and related boundary element methods.

Background

Strain is a major factor in the compound semiconductor photonics industry. It can be intentionally used for the self-assembly of quantum dots (QDs) or for tuning the energy band structure of heteroepitaxial layers. Deleterious effects of strain can lead to delamination of buried oxide apertures in vertical cavity surface-emitting lasers (VCSELs) or shifting of operating wavelengths for InGaAsP/InP-based devices.

We have begun an effort, which is partially ATP-funded, to understand and control strain development in III-V semiconductor materials systems. One aspect of the work involves developing experimental methods for measuring elastic strains with spatial resolution in the range of tens of nanometers. This involves the use of electron diffraction in the scanning electron microscope (SEM) and also in the transmission electron microscope (TEM). At present, this is being applied to strains induced by phase transition arising from the wet oxidation of AlGaAs layers confined between GaAs layers. Such oxides are used as optical apertures for VCSELs and, upon formation, result in volumetric compressive strains in the semiconducting layers in excess of 6%.

The second aspect of our contribution to solving this problem is to develop a multi-scale theoretical method for modeling the self-assembly of InAs QDs on GaAs substrates. This involves the combined use of Green's functions (GF) and boundary element methods (BEMs) incorporating anisotropic elasticity. Strain, in this case, occurs from the mismatch in natural lattice parameters between the two materials. We determine both numerically and analytically the elastic strain energy distribution in these systems. A nice aspect of the work is that we are able to provide experimental data for direct incorporation into the theoretical model using electron microscopy, due to the well-defined conditions often present during epitaxial growth of crystals.

Strain Measurement with Electron Diffraction

We describe the SEM method of electron backscatter diffraction (EBSD) here due to some recent advances in our lab. The advantage of using a SEM method over a TEM method for strain determination lies primarily with the fact that less specimen preparation is required. This reduces the likelihood of significantly changing the initial strain state and, therefore, removes the need to model the preparation-induced relaxation. The main disadvantage of the SEM method is that strain sensitivity is not as good (detectability ~ 0.1% at best) as that possible by use of TEM. We estimate the EBSD spatial resolution for strain measurement in these systems to be ~ 50 nm.

Figure 1 shows a cross-sectional SEM image of an AlGaAs/GaAs multilayer structure, with an aluminum oxide layer partially grown in. The diffuseness of the EBSD patterns is an indication of the amount of strain present around the oxide growth front. More specifically, pattern diffuseness is a direct measure of the magnitude of the elastic strain gradient present within the sampling volume of the electron beam. The strain is greater near the growth front. With automated beam scanning and pattern collection, we are able to determine the spatial extent of the distortion field caused by the oxidation. Preliminary measurements suggest that the distortion field associated with a single oxide layer extends more than 1 μm beyond the position of the front. The diffraction measurements are made only in crystalline portions of the specimen and, hence, do not sample the oxide strain directly, but rather the resulting effect on AlGaAs and GaAs layers in the vicinity.

More quantitative determinations of elastic strain are made by measuring the widths of the bright bands visible in the EBSD patterns, as shown in Figure 2. One bandwidth is directly proportional to the Bragg angle for the diffracting planes.

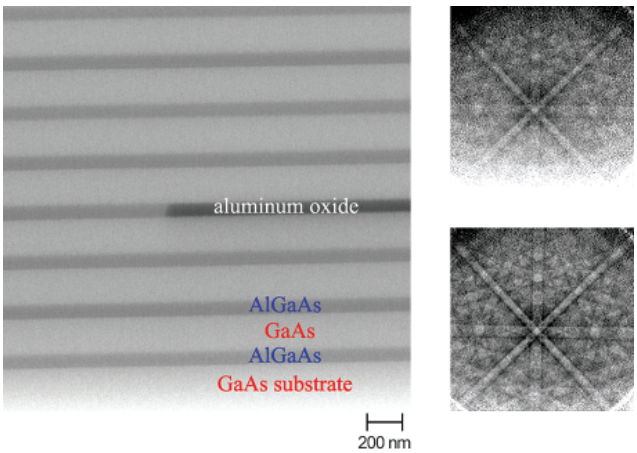


Figure 1: *SEM image of multilayer structure, with oxide growth front visible. EBSD patterns obtained from positions indicated by line segments. Image to far right is an indexed pattern. Scale bar = 200 nm.*

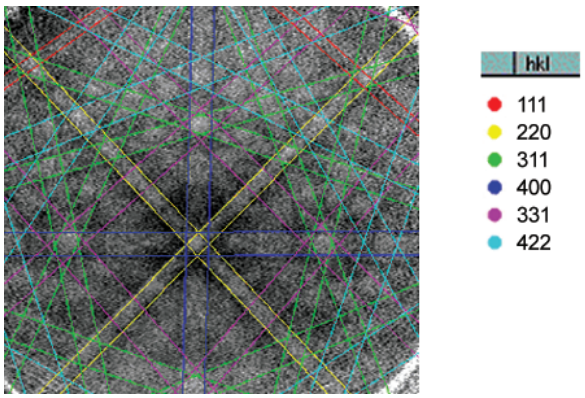


Figure 2: *Indexed EBSD pattern used for elastic strain determination.*

Energetics of Self-Assembly of Quantum Dots

We are developing advanced theoretical techniques using Green's functions (GF) and boundary element methods (BEMs) to model the self-assembly of arrays of quantum dots in anisotropic semiconductors. We consider the energetics of growth of a small QD in the strain field of an existing "grown" QD. We introduce a new parameter Γ, the elastic energy release rate (EERR), that determines the growth of a QD. The EERR, as in fracture mechanics, is defined as the elastic relaxation energy per unit volume of growth and is given by dW/dV_{QD}, where W is the elastic strain energy and V_{QD} is the volume of the growing QD. Assuming uniform misfit strain in QDs,

$$W = \tfrac{1}{2}\int_{\partial D} T_i u_i dS - \tfrac{1}{2}\sum_{n=1}^{N}\int_{\partial\Omega_n} (C_{ijkl}\varepsilon^0_{ij} n_l) u_k dS + \tfrac{1}{2}\int_D C_{ijkl}\varepsilon^0_{ij}\varepsilon^0_{kl} dV, \quad (1)$$

where the first term is the work done by external traction, the second term is the work done by the intrinsic traction at the boundary of QDs, and the last term represents the energy of the reference state.

We use eq. (1) to evaluate W by using GF and a special BEM that requires numerical discretization only along the boundaries of the uncapped QDs and the interfaces between the substrate and QDs. Our BEM is computationally more efficient than the conventional BEM and the domain-based numerical techniques such as the finite-element method.

Equation (1) takes into account the strain created by the growing QD. This effect has been neglected in the earlier papers that consider only the strain field of the "grown" QDs. Equation (1) also accounts for the fact that the material parameters of QDs are different from the host solid and include the continuity conditions at the interface between QDs and the host solid. These contributions have been neglected in earlier calculations using Green's functions.

We have applied our theory to InAs QDs in GaAs. We have calculated EERR for a QD growing on the free (001) surface for a buried seed dot as well as a seed dot on the surface (Figure 3). We find that the grown QD reduces the EERR of a new QD. For the grown QD at the surface, the EERR for the new dot is lower in the <100> and <010> directions. However, the change in EERR is small when the grown QD is at the surface. In contrast, the effect of a buried QD on EERR of the new QD is very pronounced.

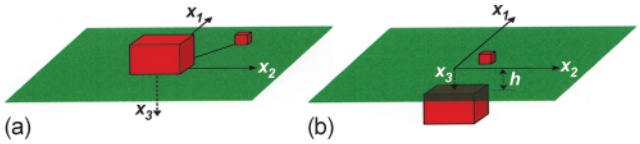


Figure 3: *Formation of a new QD under the influence of a grown surface QD (a) or buried QD (b).*

We find that the maximum of EERR occurs vertically above the grown QD only for some values of the depth of the QD (Figure 4). This shows that, for QDs covered by a thick spacer medium, a vertical array of self-assembled QDs is energetically favorable. In the case of a thin covering, an oblique stacking of QDs could occur. We also find that there is an optimum depth of the buried QD for the formation of a new QD vertically above it.

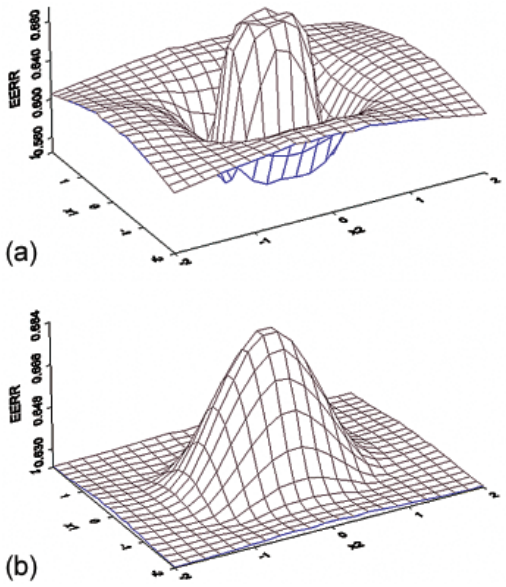


Figure 4: *Variation of the EERR for formation of a new QD with locations at different depths of the buried QD (Figure 3b): (a) h = 0.1a; (b) h = 0.6a.*

For More Information on this Topic

Electron diffraction strain measurement: R.R. Keller; *GF/BEM modeling:* V.K. Tewary

Biomaterials Metrology Project

The knowledge base of physicians and biologists is complemented by that of materials scientists and mechanical engineers when attacking the complex problems of cardiovascular, neurological, and oncological diseases. Mechanical testing, failure analysis, mechanical design, and methods to introduce, measure, and evaluate physical stimuli fall within the realm of materials scientists and mechanical engineers. Members of the Materials Reliability Division have initiated this project with the goal of applying our knowledge of material mechanics, nanotechnology, and sensor development toward improving reliability of medical treatments, and understanding and manipulating cellular function.

Background

Materials scientists have much to contribute in the realm of biosciences. Our perspective is uniquely different from that of biologists and physicians. The physical properties that are the manifestation of biological functions can be measured and evaluated using the same principles that the materials scientist uses on more traditional materials. Our approach to measurements of physical properties is grounded on identifying and testing material properties. The development of test methodologies to obtain these material properties is our specialty. Additionally, we have extensive experience in metrologies at the micro and nano scales.

This fledgling project was first conceived at the start of the fiscal year, at a convergence when division scientists were looking for new directions and the results of the NIST 2010 exercise were first made public. Health care was designated as one of the four key areas to develop at NIST and we had several staff members who had already demonstrated an interest in this area. We hired a contractor who, over a course of several months, evaluated our capabilities and offered directions in the health sciences. Dr. Finch started by interviewing individuals, then holding topical meetings to gauge response to different areas that fit our Division's unique expertise. A summary meeting was convened to determine commitment to several broad areas. He reported to us that our measurement capabilities and our interests indicated that we should direct our research toward cardiovascular disease, tissue engineering, Bio-MEMS, and/or thermal properties for treatments or optimizing recovery from surgery.

During that time, Dr. Finch also acted as liaison between the (local) medical research community and ourselves. Those relationships are proving fruitful: in this atmosphere of interdisciplinary approaches to research, our expertise in measuring mechanical properties of materials and nanotechnology are valued, and collaborations have been initiated between the Materials Reliability Division and researchers at the University of Colorado, the University of Colorado Health Sciences Center, Children's Hospital of Denver, members of the Biotechnology Division of CSTL, and within the Polymers Division of MSEL. Work has begun on three different subprojects within Biomaterials Metrology. They are Metrologies for Tissue Engineering Scaffolds, Pediatric Pulmonary Hypertension, and Cellular Engineering Micro Systems.

Approach

Metrologies for Tissue Engineering Scaffolds is an ATP-sponsored subproject that was awarded in FY02 in collaboration with the Polymers Division of MSEL and headed by Dr. Newell Washburn (Polymers Division). The premise of the proposal is that the regulatory approval process for tissue scaffolds is complicated and requires rigorous testing hurdles before they can gain commercial acceptance. By developing tests on scaffolds with systematic variations of parameters, it will be possible to determine the influence and interrelationships of material and biological variables and give producers a roadmap that allows predictions for performance of tissue engineered medical products. Our role in this proposal is the characterization of mechanical properties of the scaffolds and the scaffold–tissue constructs. The initial test phase toward this goal has been to measure the compressive strength and the compressibility of the scaffold material alone. An on-going project within the Division necessitated the acquisition of a load frame and cells that lend themselves ideally to the size scales required for the scaffold measurements.

Pediatric Pulmonary Hypertension is a subproject in collaboration with Dr. Robin Shandas, who has a joint appointment with the University of Colorado Health Sciences Center and the Mechanical Engineering Department. The objective of this subproject is to determine the factors (mechanical, genetic, proteonomic) that contribute to the development of pulmonary hypertension in children. The prevalence of this disease is much greater at high altitudes than at sea level, and only limited studies have been conducted to date. Previously, Dr. Shandas studied pediatric pulmonary hypertension from a fluid-dynamics viewpoint. With our ability to measure mechanical properties, a fuller picture of how the arteries remodel due to the effects of the disease is possible. A biaxial pressure test is being conducted to compare the stress–strain behavior of diseased versus healthy arteries. The data from the tests are analyzed for the constants to a constitutive model that fully describes the viscoelastic non-linear characteristics of arterial tissue.

Figure 1 shows images of a rat pulmonary artery at the start of the test and at two pressure levels. A biaxial test fixture is necessary to account for the directional differences in materials properties, as is evinced by the protruded tissue having an elliptical shape. Through-thickness anisotropy will be evaluated by conducting biaxial tests with each artery yielding two samples: one to be tested with the inner surface of the artery up and the other with the outer surface up. The cells comprising the arterial wall will be separated and studied microscopically to determine morphological and histological changes due to the disease.

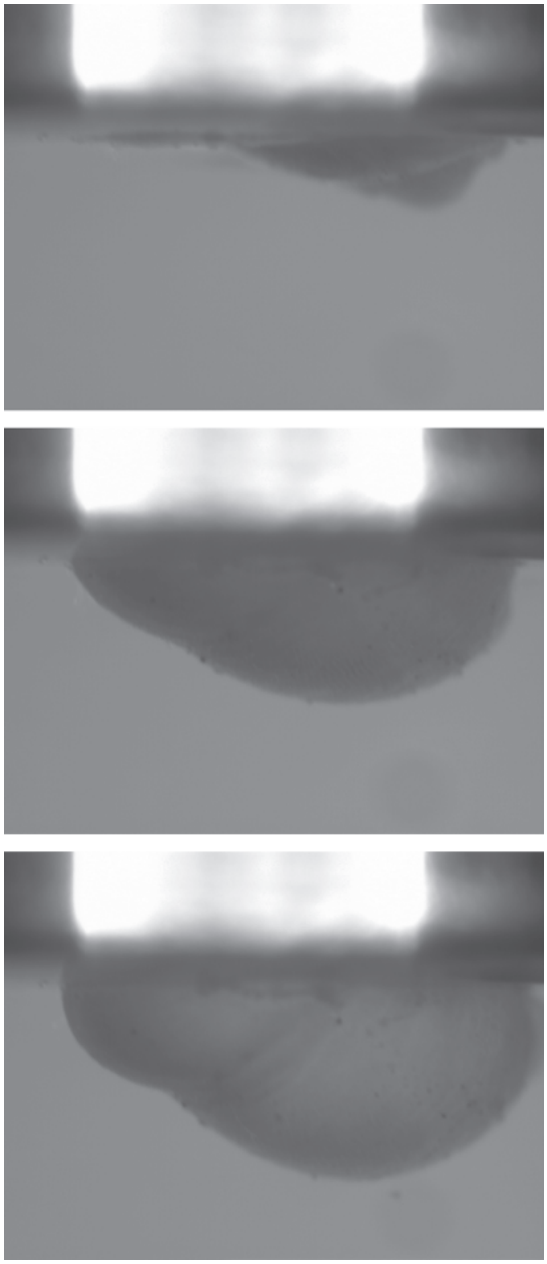


Figure 1: *Images of a rat pulmonary artery at the start of the biaxial test, at ~2 psi, and at ~4 psi. Field width = 4 mm.*

Cellular Engineering Micro Systems is a broad, ambitious program involving a team that includes members from NIST, multiple departments in Engineering and Arts and Sciences at the University of Colorado at Boulder, and the University of Colorado Health Sciences Center. Specialists in the fields of MEMS, microfluidics, neurons, smooth muscle cells, cell functionalization, microgravity cell growth, neural networks, AFM, thermal properties, surface interaction, and various analytical techniques have pooled their collective intellect to design tests and test fixtures appropriate for an individual living cell.

The first phase of this subproject will pursue two directions simultaneously. The first is the measurement of mechanical forces exerted by vascular smooth muscle cells, as well as their cellular response to external mechanical stimuli. Designed to offer insight into the relationship between stimulus and response in cardiovascular development, individual or multiple cells will be mechanically stimulated while being monitored for changes in gene expression. A prototype design has been submitted to a MEMS fabrication company for manufacturing.

The second direction is the electrical stimulation of neurons to observe how neural pathways develop when established pathways have been severed. Carbon nanotubes will be used to detect neuronal signals resulting from electrical stimulus. Carbon nanotubes offer a 10–100x improvement in spatial resolution over the currently used patch-clamp system. A clear understanding of the complex signal integration performed by neurons is impossible without the spatial resolution made possible with carbon nanotubes.

Summary

The Materials Reliability Division in FY02 has embarked on a new research direction, Biomaterials Metrology, with considerable forethought and deliberation. We have chosen to build upon our expertise in the areas of developing measurement techniques for physical properties and in the nanoscale regime. The subprojects, Metrologies for Tissue Engineering Scaffolds, Pediatric Pulmonary Hypertension, and Cellular Engineering Micro Systems, fulfill that objective.

For More Information on this Topic

E. Drexler, T. Quinn, D. Finch (NIST)

Materials For Micro- And Optoelectronics

U.S. microelectronics and related industries are in fierce international competition to design and produce smaller, lighter, faster, more functional, and more reliable electronics products more quickly and economically than ever before. At the same time, there has been a revolution in recent years in new materials used in all aspects of microelectronics fabrication.

Since 1994, the NIST Materials Science and Engineering Laboratory (MSEL) has worked closely with the U.S. semiconductor, component, packaging, and assembly industries. These 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. The vision that accompanies this program — to be a key resource within the Federal Government for materials metrology development for commercial microelectronics manufacturing — is targeted 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 models of small, complex structures to substitute for, or provide guidance for, experimental measurement techniques; and
- Develop fundamental understanding of materials needed in future micro- and opto-electronics and magnetic data storage.

With these objectives in mind, the program presently consists of projects led by the Metallurgy, Polymers, Materials Reliability, and Ceramics Divisions that examine and inform industry on key materials-related issues. 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) at NIST. Materials metrology needs are also identified through industry groups and roadmaps including the International Technology Roadmap for Semiconductors (ITRS), International SEMATECH, the IPC-Embedded Passive Devices Taskgroup, the IPC Lead-free Solder Roadmap, the National Electronics Manufacturing Initiative (NEMI) Roadmap, the Optoelectronics Industry Development Association (OIDA) roadmaps, and the National [Magnetic Data] Storage Industry Consortium (NSIC).

Although there is increasing integration within various branches of microelectronics and optoelectronics, the field can be considered to consist of three main areas. The first, microelectronics, includes needs ranging from integrated circuit fabrication to component packaging to final assembly. MSEL programs address materials metrology needs in each of these areas, including, for example, lithographic polymers and electrodeposition of interconnects, electrical, mechanical, and physical property measurement of dielectrics (interlevel, packaging, and wireless applications), and packaging and assembly processes (lead-free solders, solder interconnect design, thermal stress analysis, and co-fired ceramics).

The second major area is optoelectronics, which includes work that often crosses over into electronic and wireless applications. Projects currently address residual stress measurement in optoelectronic films, and wide bandgap semiconductors. Cross-laboratory collaborations with EEEL figure prominently in this work.

The third area is magnetic data storage, where the market potential is already large and growing and the technical challenges extreme. NSIC plans to demonstrate a recording density of 40 times today's level by 2006. To reach these goals, new materials are needed that have smaller grain structures, can be produced as thin films, and can be deposited uniformly and economically. New lubricants are needed to prevent wear as the spacing between the disk and head becomes smaller than the mean free path of air molecules. MSEL is working with the magnetic recording industry to develop measurement tools, modeling software, and magnetic standards to help achieve these goals. MSEL works in close collaboration with the Electronics and Electrical Engineering Laboratory, the Physics Laboratory, the Information Technology Laboratory, and the Manufacturing Engineering Laboratory as partners in this effort.

Contact: Robert Keller

Micrometer-Scale Reliability: Mechanical Behavior of Thin Films

Techniques for characterizing the mechanical behavior of thin films are being developed and applied. This is necessary since thin-film microstructures and properties are usually quite different from those of the same materials in bulk form. While the general principles of conventional mechanical testing apply to thin films, special test equipment and techniques are required. The ultimate goal of this effort is to test specimens produced by semiconductor fabrication equipment and similar in size to features on integrated circuit chips.

David T. Read

Technical Description

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

- To develop experimental techniques to measure basic tensile properties, fatigue behavior, and fracture resistance in thin-film 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 µm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths of 0.1 to 1 µm).

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.

Accomplishments

Last year we reported the application of our recently developed force-probe technique to two new types of materials: 1) aluminum interconnect layers made in a commercial CMOS (complementary metal oxide semiconductor) fabrication facility, obtained through the MOSIS service, and 2) polycrystalline silicon made at Sandia National Laboratories. This year, we have demonstrated that the same technique is applicable to polymer materials used in electronic devices. We tested a commercial photodefinable polymer, a polyimide, between room temperature and 200°C.

The tensile section was 0.6 x 10 x 190 µm. Our success with these specimens from the three main families of materials, metals, ceramic-like, and polymers, and positive reports from other laboratories using similar techniques, has made us optimistic that standardized testing of thin films may be near.

The cured polyimide was very well behaved, with results as expected and consistent with the manufacturer's product bulletin; the elongation to failure was over twice the minimum value given by the manufacturer. This establishes that in this material, no "thin film" effects alter the tensile properties, down to a thickness of 0.6 µm. Changes in polymer mechanical properties in films thin enough to constrain the arrangement of the chain-like polymer molecules have been anticipated and may still occur for thinner specimens.

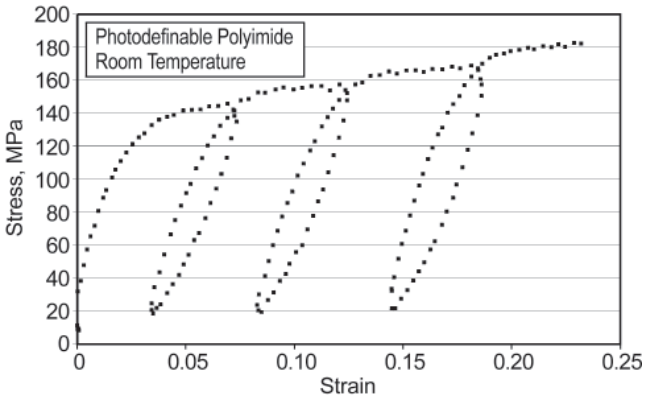


Figure 1: *Stress-strain curve of photodefinable polyimide at room temperature showing hysteresis loops.*

Three other efforts were pursued in this project: continuation of our testing of CMOS aluminum; fabrication and testing of microtensile specimens representative of copper interconnect lines in microelectronics devices; and extension of our microtensile test technique to exotic materials such as carbon nanotubes. While CMOS aluminum specimens down to 2 µm wide were successfully fabricated, this set of specimens behaved in a very brittle manner, more brittle than the previously tested set. We are applying advanced analytical tools to find the cause of this behavior. Our preliminary results on copper are similar to those for other metals we have tested, but the details are currently covered by a nondisclosure agreement with the supplier of the specimens. Our initial experiments with carbon nanotubes are described elsewhere.

Contributors and Collaborators

Y.-W. Cheng, J.D. McColskey (NIST); R. Emery, T. Scherban (Intel); B. Yeung, (Motorola); C. Litteken (Stanford University)

Micrometer-Scale Reliability: Stress Voiding and Electromigration

The microelectronics industry continues to strive for better device performance through a combination of dimensional scaling and incorporation of new materials for chip-level interconnects. Reliability of the resulting new systems is poorly known at present. We conduct studies to understand the mechanisms of thermal strain — and electric current — induced degradation phenomena, with emphases on the roles of localized stress and localized variations in microstructure on interconnect lifetimes.

Robert R. Keller and Roy H. Geiss

Technical Description

The two primary reliability-limiting phenomena that chip-level interconnects undergo are stress voiding (SV) and electromigration (EM), occurring during thermal- and electric current-induced stressing, respectively. Both involve the development and subsequent relaxation of stress and take the form of surface topography development and void formation. Consequent device failure occurs in the form of passivation cracking, short circuiting, and open circuiting. Previous studies have demonstrated that even small variations in localized microstructure can have large impacts on these types of failure.

We began an effort about a year and a half ago that shows that both mechanical and diffusion-based degradation processes can occur due to alternating current (AC) stressing at low frequencies and high current densities. Interconnect damage under such conditions appears to occur due to thermomechanical fatigue resulting from Joule heating of metal interconnects well-adherent to oxidized silicon substrates. Thermal expansion mismatch strains lead to large stresses in the metal and cause cyclic deformation. This work has suggested both a new testing technique as well as a potentially new reliability threat to narrow interconnects. AC stressing can be used to perform fatigue testing of very small structures in this manner. The reliability threat appears as we consider such testing in metals encapsulated by soft dielectrics, which may not suppress the surface damage. Here, we report new observations of microstructural changes accompanying AC stressing of Al-alloy interconnect lines.

Accomplishments

We conducted a quasi *in situ* test whereby a line was characterized for surface structure and crystallographic orientation distribution prior to testing, as well as during periodic (every 15–60 s) test interruptions. Figure 1 shows a SEM image and the corresponding orientation map obtained by automated electron backscatter diffraction (EBSD) after less than one minute of stressing. The topography in the large grain towards the left of the field of view can be explained by the schematic to the far left. Given the observed orientation, threading dislocations could glide on the indicated slip system leaving slip offsets at the surface. During cycling, the severity of the offsets increases considerably.

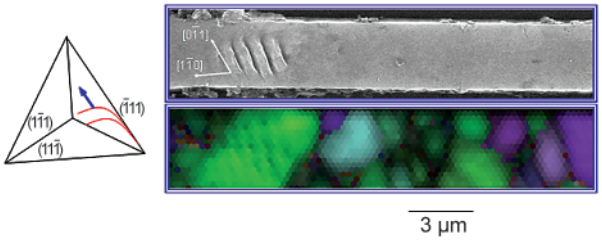


Figure 1: *Dislocation mechanism for topography formation induced by AC stressing in Al-1Si.*

An unexpected observation is shown in Figure 2, where the orientation maps show that significant grain growth can occur even within the first minute of stressing, prior to the formation of slip offsets. This demonstrates that the effect induced by low-frequency cycling of high-current density is not simply mechanical fatigue, but that diffusion must also play a significant role. Interestingly, we have seen this effect only in the aluminum alloy. Similar ongoing studies on copper done by our colleagues at the Max-Planck-Institut have shown no evidence of grain growth in the absence of topography development. Rather, that work shows grain growth that takes place concurrently with or after the formation of slip offsets.

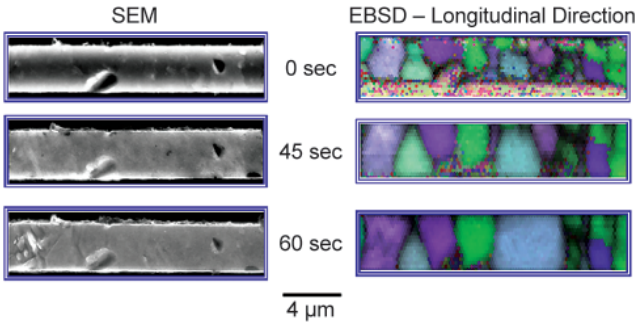


Figure 2: *Grain growth induced within first minute of AC stressing, prior to occurrence of topography.*

Contributors and Collaborators

R. Mönig, C. Volkert (Max-Planck-Institut für Metallforschung)

Micrometer-Scale Reliability: Strain in Photonic Semiconductors

The compound semiconductor photonics industry seeks to both measure and control strain that develops during the course of device fabrication. In the Materials Reliability Division, we are developing electron diffraction methods for measuring and mapping elastic strain distributions. The methods are applied to phase transition-induced strains for the case of oxide formation in vertical cavity surface-emitting lasers, and to lattice parameter mismatch strains for self-assembled quantum dots.

Robert R. Keller and Roy H. Geiss

Technical Description

Vertical cavity surface-emitting lasers (VCSELs) have, as an integral component, oxide layers to act as apertures resulting in record low threshold currents and high efficiencies. These apertures are grown by selective wet oxidation of AlGaAs layers (compositions typically contain 98–100% AlAs) situated between GaAs layers. Upon transformation from AlGaAs, the aluminum oxide undergoes a large volume contraction in excess of 6%. The exact magnitude of the contraction is not well-characterized as the theoretical volume change exceeds 20% while the few experimental measurements suggesting 6–7% are based on TEM images of layer thickness changes. The contraction can be manifested as elastic strain in adjacent GaAs layers, dislocations in GaAs and in the interfaces, and also as delamination.

We are using TEM and SEM electron diffraction methods to measure elastic strain states via determination of lattice spacings and angles. While the TEM method provides superior spatial and strain resolutions, the SEM method is not subject to the difficulties associated with thin foil preparation, namely site-specificity and stress relaxation induced during thinning. Here, we report progress on SEM measurements by electron backscatter diffraction (EBSD).

Accomplishments

The EBSD method has seen little successful application to the measurement of elastic strains due primarily to the low signal-to-noise inherent in the detection system. We have made exciting progress along two avenues for strain measurement in oxidized AlGaAs/GaAs multilayer structures. We started with the most direct measure of strain available in the diffraction patterns, namely the spacing of the Kikuchi-like bands of high intensity. These bandwidths are proportional to Bragg angles and therefore provide a direct link to interplanar spacings in the crystal. Figure 2 of the Technical Highlight on this subject shows an example of an indexed pattern with a computer-simulated pattern overlaying an experimental result. Rather than measure bandwidths directly from the data, we have begun to apply simple image mathematics to accentuate the differences between patterns showing strain and those showing no strain.

We have, to date, been able to detect very small differences in strain from locations within the crystal that are only tens of nanometers apart. Quantification of these strains is underway.

The other aspect of EBSD-based strain measurement makes use of automated collection of diffraction patterns, followed by analysis of pattern sharpness. If the elastic strain varies considerably within the volume of material sampled by the electron beam (of approximate diameter 50 nm), then there will be a continuously changing lattice parameter that causes a continuously changing Bragg angle. In the pattern, this effect broadens the edges of the Kikuchi-like bands and makes the pattern more diffuse. A larger strain gradient leads to more diffuseness. The extent of diffuseness can be quantified and this effect mapped as shown in Figure 1. Here, each pixel represents a different beam position, and greater diffuseness is represented by a darker intensity. In this figure, we concentrate on the colored pixels within the GaAs layers only.

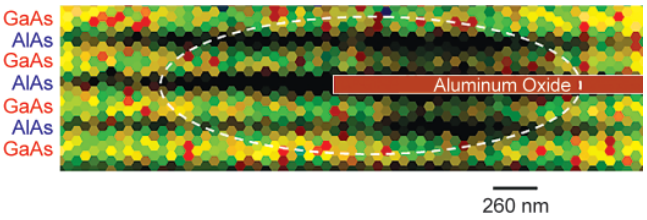


Figure 1: *Crystallographic orientation map with darker pixels indicating greater pattern diffuseness or strain.*

The strain field around the aluminum oxide growth front (indicated schematically in brown) can be qualitatively seen to extend over a few GaAs/AlAs bilayers in directions normal to the layers. It extends even further in directions parallel to the layers as indicated schematically by the dashed white oval.

We are in the process of evaluating means for quantifying the relationship between EBSD pattern diffuseness and magnitude of elastic strain gradients.

Contributors and Collaborators

A. Roshko, S. Lehman, K. Bertness (Optoelectronics Division, EEEL)

Electronic Packaging and Components: Packaging Reliability

We are developing methods to examine materials and interfaces in electronic packaging applications and elucidate the damage mechanisms. Our current focus is on advanced packaging structures and embedded passive materials using electron-beam moiré to measure mechanical strain, and thermal microscopy to measure heat flow and thermal properties on increasingly smaller size scales.

Andrew Slifka and Elizabeth Drexler

Technical Description

The microelectronics industry is moving rapidly toward higher-density components of smaller size using less expensive materials. One move in this direction is the advent of embedded and integrated passive components in printed circuit boards (PCBs) and another is the use of PCBs in various types of grid array packages. These organic-based PCBs can have a large coefficient of thermal expansion (CTE) in comparison with many materials found in their proximity. This CTE mismatch can reduce the reliability of electronic packaging systems by causing localized stress. The CTE mismatch in flip-chip packages is also a source of failure and fatigue.

We are investigating the damage induced from CTE mismatches between organic materials, organics and metals, organics and ceramics, and organics, metals and ceramics to determine the initiation of damage and the ultimate failure mechanisms in these systems. Electron-beam moiré is employed to measure displacements and calculate mechanical strain due to thermomechanical loading. Thermal microscopy is used to measure changes in interfacial thermal resistance in order to detect the onset of thermomechanical damage at that interface before any surface manifestation is visible.

We are developing new measurement methods, both thermal and mechanical, using scanned-probe microscopy (SPM) in order to characterize packages at increasingly smaller size scales.

Accomplishments

Building on the preliminary measurements made in FY01, we developed the thermal SPM into a quantitative technique. We used an industrial embedded resistor (Figure 1) to make comparative measurements between the SPM thermal system and the IR microscope. Similar temperature differences can be achieved with both the SPM and IR systems, although two different heating techniques are used due to geometric constraints. Improved spatial resolution is motivating the move to the SPM. With increased miniaturizing in electronic packaging, the IR microscope no longer has the needed resolution to detect materials and interfaces in future, and even some current, packages.

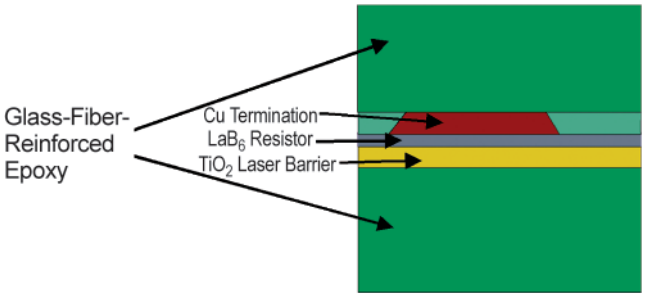


Figure 1: *Schematic of the materials contained in the embedded resistor specimen and their relative positions.*

Mapping of the change in interfacial thermal resistance in an embedded resistor specimen, shown in Figure 2, among the various material combinations shows the increase in degradation with respect to thermal cycling (–55 to 125°C). The interface between the TiO_2-filled epoxy (laser barrier) and the ceramic embedded resistor shows signs of damage within 5 thermal cycles. Corresponding electron-beam moiré data show that maximum strains are found in the laser barrier and in the resin-rich regions of the printed circuit board, particularly between the resistors, where unfilled resin collects during lamination.

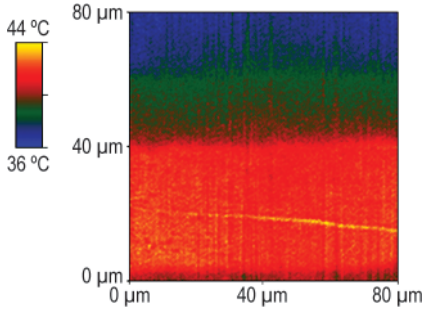


Figure 2: *Thermal SPM image showing the laser barrier, resistor, and copper (from top to bottom), 8°C, full scale.*

In collaboration with the Jet Propulsion Laboratory, we are looking at techniques to mitigate the destructive consequence of CTE mismatches in flip-chip packages. Strains in specimens were measured before, after, and without the microwave process.

Contributors and Collaborators

J. Felten (DuPont Technologies, Research Triangle Park, NC); V. Shah (MicroFab Technologies, Plano, TX); R. Snogren (SAS Circuits, Inc., Littleton, CO); N. Budraa (Jet Propulsion Laboratory, Pasadena, CA)

Electronic Packaging and Components: Acoustic Loss in Piezo Crystals

Langasite, langatate, and langanite are piezoelectric materials that show promise of providing characteristics superior to quartz for a variety of electronic oscillator and filter applications. The acoustic loss of these crystals is being studied in our laboratory to identify and quantify the dominant physical mechanisms that degrade performance, including the intrinsic phonon–phonon mechanism. Through this work, the project seeks to provide guidance to other researchers in minimizing the acoustic loss and determining the most promising material to pursue among this group of compounds.

Ward Johnson and Sudook Kim

Technical Description

Langasite ($La_3Ga_5SiO_{14}$), langatate ($La_3Ga_{5.5}Ta_{0.5}O_{14}$), and langanite ($La_3Ga_{5.5}Nb_{0.5}O_{14}$) have attracted significant attention in recent years as candidate materials for improved electronic oscillators and filters. The potential advantages of these crystals over quartz include higher piezoelectric coupling (which enables devices to be made smaller), lower acceleration sensitivity, and higher Q (which reduces phase noise and enables higher-frequency operation). These materials also have no phase transition below the melting point, which allows devices to be operated at high temperatures and provides the potential of producing large-diameter wafers (for surface-acoustic-wave devices) more easily and cheaply than with quartz.

Values of the acoustic loss Q^{-1} reported in the literature vary widely and may not represent the intrinsic limits of these materials. Therefore, current data provide little guidance in establishing a focus of further research. This project seeks to remedy this situation by identifying the dominant contributions to Q^{-1} in langasite, langatate, and langanite. The known physical mechanisms that can significantly contribute to Q^{-1} include dissipation through external mechanical contact, defect anelasticity, nonlinear coupling to other modes, and intrinsic coupling to thermal phonons (phonon–phonon loss through the Akheiser mechanism).

Accomplishments

In FY02, an experimental system was constructed for measuring Q^{-1} and frequencies of resonant shear modes of Y-cut plano-convex disks in vacuum at temperatures from 150 K to 1000 K. The plano–convex geometry of the sample has the effect of localizing the vibrations of certain modes near the center, so damping from mechanical support at the edges is essentially eliminated. Acoustic resonance measurements are performed using tone-burst direct piezoelectric coupling with planar electrodes above and below the sample. Q^{-1} and resonant frequencies are determined through analysis of free-decay waveforms in the time domain. The configuration of the sample, supporting structure, and copper electrodes are shown in Figure 1.

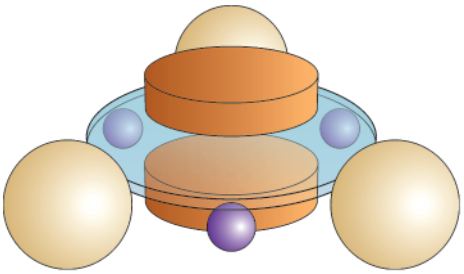


Figure 1: *Plano-convex single-crystal disk supported on sapphire spheres and centered laterally by larger alumina spheres. Driving electrodes are above and below sample.*

Measurements of Q^{-1} in langatate have been performed as a function of frequency, temperature, external mechanical contact, and vibrational amplitude. As shown in Figure 2, Q^{-1} varies significantly between samples which are taken from the same crystal boule. The dominant contributions to Q^{-1} in these measurements have not yet been identified. However, the contribution from external damping has been determined to be insignificant, except for the lowest mode near 2 MHz. At higher amplitudes, a nonlinear contribution is observed.

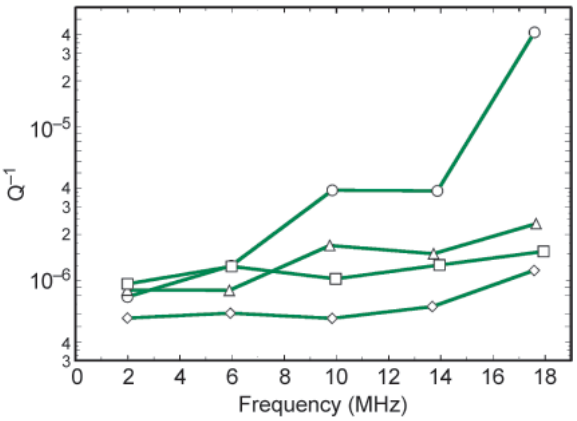


Figure 2: *Q^{-1} versus resonant frequency in a Y-cut plano–convex disk of langatate at ambient temperature.*

Contributors and Collaborators

R. Smythe (Piezotechnology, Inc., Orlando, FL)

This project is partially supported by a grant from the U.S. Army Research Laboratory.

Solder Reliability: Lead-Free Solder

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. 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 develop and disseminate such data on lead-free solders.

Tom Siewert

Technical Description

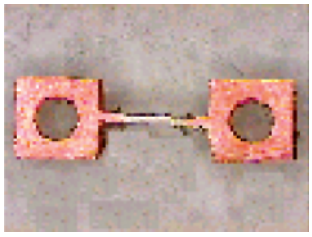
The worldwide "green" movement in the electronics industry to replace lead–tin eutectic solders with lead-free solders creates a need for critical data on the industry's new lead-free solder compositions for these design and reliability models. In fact, the NEMI website describes their view of the situation 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; however, our interests fit with only two: the Alloy Group (whose mission is to select a lead-free alloy, pursue an industry standard, and gather data) and the Reliability Group (who is analyzing data from a round robin that compared the performance of lead and lead-free alloys).

Development of our database started in April 2000 after we became members of the NEMI Lead-free Alloy Group, and we 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 addresses all of these issues.

The database focuses 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 comparative 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 the existing physical and mechanical property data that have been developed by researchers around the world. The most recent version of the database (4.0) is posted on the Materials Reliability Division's website at: *http://www.boulder.nist.gov/div853/*. We have reported on the availability of the database in posters at national technical meetings, and the interest in the data is shown by this website being one of the top 5 sites for our Division (based on number of hits in the past year). Feedback from users of the database is guiding the addition of new data. We are also publishing a Standard Practice Guide that describes standardized procedures to facilitate the comparison of data between laboratories and to permit the combination of data from different sources into a single, comprehensive database.



We have developed the capability of tensile-testing solder samples that have test dimensions on the order of the sizes of solder balls used in flip-chip packages. The sample shown at right has a gauge length of 500 µm and a rectangular cross section that is 250 µm by 200 µm. A hydraulic tensile machine was outfitted with a control system that can reliably apply strain rates of 10^{-5} s^{-1} to the specimen. The strain is measured by capturing video images of the gauge section at magnifications of up to 750x. The images are then processed using image correlation techniques to measure the strain across the whole gauge section. Studies can now be conducted on the effects of phase sizes vs. structure size, lead free solder properties, and/or strain rate effects.

Contributors and Collaborators

T. Quinn, Y.-W. Cheng, C. McCowan, C. Handwerker (Metallurgy Division and Chair of NEMI Lead-free Alloy Group); Members of the NEMI Alloy and Lead-free Alloy Task Groups; J.C. Madeni, S. Liu (Colorado School of Mines)

Materials Property Measurements

This program responds both to MSEL customer requests and to the DOC 2005 Strategic Goal of "providing the information and framework to enable the economy to operate efficiently and equitably." For example, manufacturers and their suppliers need to agree on how material properties should be measured. Equally important, engineering design depends on accurate property data for the materials that are used.

The MSEL Materials Property Measurement Program works toward solutions to measurement problems, on scales ranging from the macro to the nano, in four of the Laboratory's Divisions (Ceramics, Materials Reliability, Metallurgy, and Polymers). The scope of its activities goes from the development and innovative use of state-of-the-art measurement systems, to leadership in the development of standardized test procedures and traceability protocols, to the development and certification of Standard Reference Materials (SRMs). A wide range of materials is being studied, including polymers, ceramics, metals, and thin films (whose physical and mechanical properties differ widely from the handbook values for their bulk properties).

Projects are directed to innovative new measurement techniques. These include:

- Measurement of the elastic, electric, magnetic, and thermal properties of thin films and nanostructures (Materials Reliability Division);
- Alternative strength test methods for ceramics, including cylindrical flexure strength and diametral compression (Ceramics Division); and
- Coupling micromechanical test methods with failure behavior of full-scale polymer composites through the use of microstructure-based object-oriented finite element analysis (Polymer Division in collaboration with the Automotive Composites Consortium).

The MSEL Materials Property Measurement Program is also contributing to the development of test method standards through committee leadership roles in standards development organizations such as ASTM and ISO. In many cases, industry also depends on measurements that can be traced to NIST Standard Reference Materials (SRMs). This program generates the following SRMs for several quite different types of measurements.

- Charpy impact machine verification (Materials Reliability Division);
- Hardness standardization of metallic materials (Metallurgy Division);
- Hardness standardization and fracture toughness of ceramic materials (Ceramics Division); and
- Supporting the Materials Property Measurements Program is a modeling and simulation effort to connect microstructure with properties. The Object-Oriented Finite-Element (OOF) software developed at NIST is being used widely in diverse communities for material microstructural design and property analysis at the microstructural level.

In addition to the activities above, the Materials Reliability, Metallurgy, Ceramics, and Polymers Divisions provide assistance to various government agencies on homeland security and infrastructural issues. Projects include assessing the performance of structural steels as part of the NIST World Trade Center Investigation, advising the Bureau of Reclamation (BOR) on metallurgical issues involving a refurbishment of Folsom Dam, advising the Department of the Interior on the structural integrity of the U.S.S. Arizona Memorial, advising the U.S. Customs Service on materials specifications for ceramics, and advising the Architect of the Capitol on repair procedures for cracks in the outer skin of the Capitol Dome.

Contact: Donna Hurley, Thomas Siewert

Metrology for Nanoscale Properties: Conductive AFM using Nanotubes

With submicrometer features defining the latest optical and electrical integrated circuitry, very small defects in processing can have major influence on product yield. We are developing a technique using carbon nanotubes mounted to atomic force microscope tips to measure coating defects with sub-nanometer resolution, for example holes in nanometer thick, dielectric coatings. The development of these tips also leads to methods for probing high-frequency electronic circuitry and measuring the electrical properties of biological specimens.

Paul Rice

Technical Description

We are mounting carbon nanotubes on the end of conductive atomic force microscope (AFM) tips and scanning them across a sample. This provides an electrical conduction map of the surface of the sample with nanometer resolution.

Very thin films are prevalent in today's new optical and electrical circuitry. Films of only a few nanometers are routinely deposited on structures for devices such as narrow bandwidth filters used in the telecommunications industry for separating the individual data signals from the main carrier signal. Since nanometer-thick films are only a few atomic layers, the completeness of the film is crucial in the operation of the device. Visualizing thin film coverage has always been an issue. Two widely-used techniques are optical- and scanned-probe based. Optical techniques, in general, average over several square micrometers and thus have difficulty resolving defects that are nanometers in dimension. Scanned probe techniques can resolve the defects if there is enough vertical contrast around the edge of the defect. However, for very thin coatings, it is difficult to distinguish the film from the substrate. To explore this thin film coverage, a nanometer-thick dielectric film is deposited on a conductive surface. By scanning a nanotube tip across the surface, flaws can be distinguished in the dielectric film as changes in the electrical conductivity.

Having an electrical probe with a few square nanometer contact area lends itself to several other small feature measurements. These include low-invasive high frequency probing of electronic circuits and exploration and injection of electrical signals in living tissues.

New electronic devices, besides being smaller, are expected to operate at much faster speeds. Where a few years ago megahertz signals were commonplace, gigahertz signals are now expected. Measuring these devices in operation requires a probe which does not affect the device. The nanotube tips provide such a small contact area that current loss to the probe is very small.

Living tissue is extremely sensitive to measurements. A carbon nanotube is so small that it has minimal effect on even single cells. With this nanometer-sized probe, the electrical potential within a living neuron can be measured on a nanometer scale.

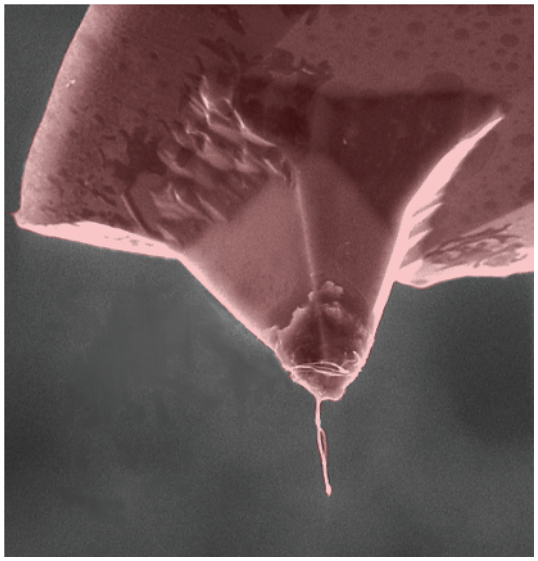


Figure 1: *SEM micrograph of carbon nanotubes attached to the end of an AFM tip. The long slender tip is 100 nm in diameter and is made of several multiwalled nanotubes wound together similar to a rope.*

Accomplishments

In the first few months of this project, a micromanipulator capable of positioning the micrometer-length nanotubes was constructed, and the appropriate sized nanotubes were obtained from a university source. Conductive AFM tips were fabricated, and techniques were developed for attaching the nanotubes to the tips. Electronics has been developed for the AFM to measure the microamp currents that these tips will detect.

Contributors and Collaborators

NIST contributors: D. Read, J.D. McColskey, D. Finch (MSEL); P. Kabos (EEEL); A. Plant (CSTL); *Academic collaborators:* E. Grulke (University of Kentucky); M. Stowell (University of Colorado)

Metrology for Nanoscale Properties: Nanoscale Mechanical Properties

We are developing in situ *measurement techniques to investigate mechanical properties on the nanoscale. Our approach, called atomic force acoustic microscopy, involves dynamic excitation of an atomic force microscope cantilever at acoustic frequencies. The technique enables quantitative point measurements of elastic moduli as well as images of relative stiffness. The mechanical-property information obtained will prove valuable for a variety of applications involving thin films or small-scale structures.*

Donna Hurley

Technical Description

Ever-decreasing length scales in many industries present a serious challenge for materials characterization. Tools must be developed to accommodate submicrometer dimensions. Specifically, the need to determine nanoscale mechanical properties exists in applications from microelectronics to biotechnology. Knowledge of mechanical properties such as elastic modulus and interfacial quality (defects, adhesion, strain) is critical to the successful development of new film materials and device assemblies. Likewise, such information could help assess the integrity or reliability of biocompatible coatings, tissue scaffolding, and so forth.

To meet these needs, we are developing nondestructive tools that exploit the spatial resolution of atomic force microscopy (AFM). Our approach is called atomic force acoustic microscopy (AFAM) and involves vibrating the cantilever at ultrasonic frequencies to excite mechanical resonances. The resonant frequencies shift as the tip comes 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. The small tip diameter (~10–100 nm) means that we can obtain *in situ* elastic-property information with nanoscale spatial resolution. Furthermore, AFM's scanning ability enables 2D imaging of mechanical properties.

Accomplishments

In FY02, we worked to understand how quantitative AFAM measurements on thin films should be made. Experiments were performed on different thin-film samples using cantilevers with different geometries. The indentation modulus M [$M = E/(1-\nu^2)$, where E is Young's modulus and ν is Poisson's ratio] is determined using a calibration sample with known elastic properties. Two data analysis methods were used. The first was an analytical model for beam dynamics that assumed a uniform cantilever cross section. The second method involved a new finite element model (FEM) specifically developed to accommodate variations in thickness and width along the length of the cantilever.

With AFAM we measured M in an Al film about 1 µm thick and a Nb film 280 nm thick. We found reasonably good agreement between AFAM results and those from surface acoustic wave methods (SAWs) and instrumented indentation techniques (IIT). Better agreement resulted from combining results from two calibration samples with different modulus. For a cantilever with nearly uniform cross section, the analytical model gave values for M agreeing with other techniques within 5%. FEM analysis results were consistently lower by 2–4 GPa (about the same as the measurement repeatability). For a cantilever with a nonuniform cross-section, values of M for the Nb film were in good agreement with other values regardless of analysis approach. However, M in the Al film was 15–40% lower than other results. We are investigating whether this discrepancy could arise from tip-sample damping effects. Our experiments indicate that the analytical model is valid for the types of cantilevers currently used. However, we continue to refine the FEM approach, since it allows greater flexibility to include effects such as adhesion, damping, and lateral forces.

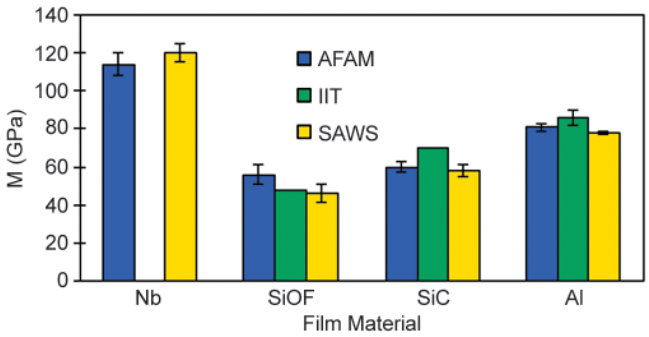


Figure 1: *Comparison of values for the indentation modulus M obtained on the same four samples by three techniques.*

We also used AFAM to measure M in a SiOF film 3 µm thick and a SiC film 0.82 µm thick and compared the results with values obtained by IIT and SAWs. The agreement in values ranged from good to excellent. We plan to evaluate the reasons for the discrepancies in order to obtain a better understanding of each method. However, our results show the validity of our basic approach for quantitative AFAM measurements of elastic properties on the nanoscale.

Contributors and Collaborators

Division participants: D.C. Hurley, P. Rice; *NIST collaborators:* P.M. McGuiggan (MSEL Polymers Division); D.T. Smith (MSEL Ceramics Division); *External collaborators:* J.A. Turner (U. Nebraska–Lincoln); M. Manghnani (U. Hawaii); A. Rar (Oak Ridge National Laboratory)

Metrology for Nanoscale Properties: Brillouin Light Scattering

A Brillouin light scattering facility has been constructed for measuring phonon and magnon properties at gigahertz frequencies in micron-scale volumes of thin-film materials. The current focus of research using this system is on characterizing interactions between magnetic modes and thermal phonons which play a central role in determining the switching times of magnetic-storage devices, spin-valve sensors, and other thin-film magnetic devices.

Ward Johnson and Sudook Kim

Technical Description

Brillouin light scattering is the inelastic scattering of incident photons with elastic waves (phonons) or spin waves (magnons) in a material. This scattering can involve either the generation of waves (Stokes process) or annihilation of waves (anti-Stokes process) in the material. Fabry–Perot interferometric techniques for measuring the shifts in photon frequencies arising from Brillouin scattering have evolved rapidly over the past couple of decades, such that they now provide powerful and practical methods of characterizing the dynamic properties of bulk and thin-film materials. In our division, these techniques are being pursued because of their capability to characterize both elastic and spin waves at gigahertz frequencies in thin-film material with surface areas typically on the order of 50 µm in diameter.

The research is currently focused on the interactions of magnons and phonons in ferromagnetic thin films. This subject is of great importance with respect to maximizing the speed of magnetic-storage devices, spin-valve sensors, and other thin-film magnetic devices. The coupling of directly excited spin waves to other waves in the material determines the time to achieve equilibration of the magnetization during a switching event. Brillouin light scattering may provide a particularly powerful tool for probing these interactions because the detection generally can be switched between magnons and phonons simply by rotating a polarizing filter in the path of the scattered light. The plan of research includes measurements of changes in the populations of magnons and phonons induced by ferromagnetic resonant excitation of permalloy ($Ni_{81}Fe_{19}$) thin films having varying amounts of rare-earth doping, which affects the damping constants of magnetic switching. Also, measurements will seek to determine whether rare-earth doping affects the peak line widths, which are proportional to the damping constants of the individual acoustic and spin waves.

Accomplishments

Over the past year, we have assembled a Brillouin-scattering system and placed it in operation. Initial measurements of thermal magnons and phonons in thin films of pure permalloy ($Ni_{81}Fe_{19}$) and Tb-doped permalloy have been performed. Figure 1 shows typical magnon and phonon spectra from $Ni_{81}Fe_{19}$ (1.2% Tb). The central peak that extends off scale in both plots arises from a direct reference beam. Apparent peaks at frequencies near −2.7 GHz and +3.5 GHz in Figure 1(b) are experimental artifacts arising from elastic scattering off the sample. Peaks to the left and right of the central peak arise, respectively, from Stokes and anti-Stokes interactions with magnons and phonons having various symmetries.

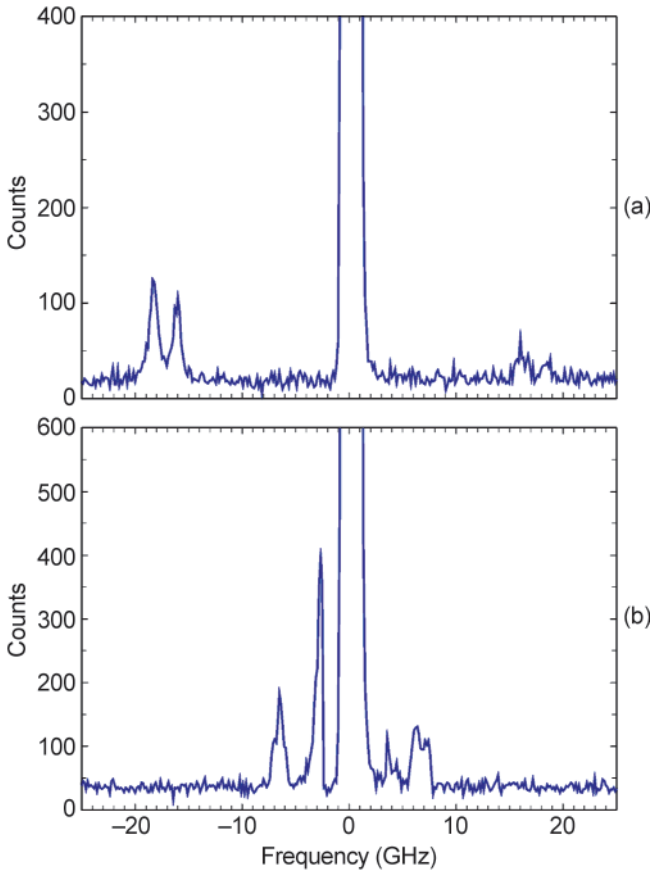


Figure 1: *Brillouin spectra from a $Ni_{81}Fe_{19}$ thin film doped with 1.2% Tb. (a) Magnon spectrum. (b) Phonon spectrum.*

Contributors and Collaborators

P. Kabos (NIST Radio-Frequency Technology Division); S. Russek (NIST Magnetic Technology Division); C. Patton (Colorado State University)

This project was partially funded by the National Nanotechnology Initiative.

Physical Properties of Thin Films and Nanostructures: Green's Function Methods

Computational methods based upon the use of elastodynamic, elastostatic, and lattice Green's functions are developed for calculating surface acoustic wave velocities in layered solids, stress and strain due to a surface load in a semi-infinite solid, quantum dots and formation energy of self-assembled array of quantum dots, change in thermodynamic functions due to quantum dots, and multiscale modeling of point and extended defects in materials. These calculations will result in better methods of characterization of thin films and quantum dots, and bridge the length scales in materials' measurements.

Vinod K. Tewary

Technical Description

Mathematical modeling of the elastic response of anisotropic materials is required for design and interpretation of experiments leading to industrial applications of advanced materials. We have developed computationally-efficient techniques for calculation of elastic (elastodynamic and elastostatic) and lattice static Green's functions that are powerful tools for modeling. The elastodynamic Green's functions are used for modeling the propagation of elastic waves, and elastostatic Green's functions are used for calculations of stress and strain. We calculate the elastic Green's functions by using a delta-function representation in slowness space that we had developed earlier.

The efficiency of a nanoscale solid-state device depends upon interaction between point defects and extended defects in the solid. Whereas extended defects can be modeled using continuum theory, the point defects must be treated atomistically. We are developing a hybrid Green's-function method for multiscale modeling of solids that treats the point defects such as vacancies and interstitials at the atomistic scale and extended defects such as free surfaces and interfaces at the continuum scale in the same formalism. We define a lattice static Green's function in terms of interatomic potentials, which reduces to the continuum Green's function in the asymptotic limit. In the hybrid Green's function, the defect space is treated atomistically, whereas the long-range Green's function is expressed in the continuum model.

Accomplishments

The work on interpretation of SAW velocities in anisotropic films on anisotropic substrates has been extended to multilayered solids (Figure 1).

We have calculated stress and displacement fields due to a surface load in semi-infinite anisotropic solids. These results should be useful for interpretation of nanoindentation measurements. We have also calculated the stress and the displacement fields due to a quantum dot near a free surface in an anisotropic solid. By using the boundary-element method along with the Green's function, we have calculated the formation energy of a quantum dot in the strain field of existing quantum dots. These results should be useful for interpretation and design of self-assembled array of quantum dots and related devices. Using the Debye Green's function, we have calculated the change in thermodynamic functions of a solid due to a quantum dot. This should be useful for characterizing the material properties of quantum dots.

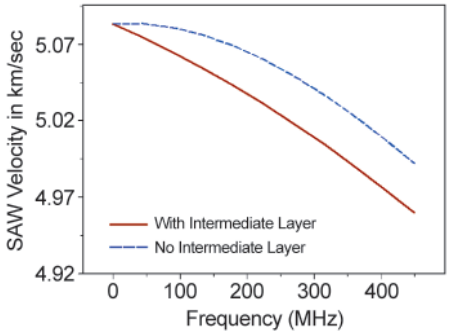


Figure 1: *SAW velocities in TiN film (206 nm) on Si with and without Ti intermediate layer (100 nm).*

As a first step towards multiscale modeling of nanoscale electronic materials, we have calculated the elastic interaction between a vacancy and a free surface in Cu and Al using simple model potentials. This would help in bridging the gap between length scales in materials' measurements.

We are collaborating with Kent State University and MIT in setting up a digital library of Green's functions on the Internet. This project, which was initially supported by NIST CTCMS, is sponsored by the National Science Foundation. We organized a Green's functions experts' meeting at Boulder to discuss the digital library in which 21 people from universities, industry, and Government labs participated.

Six research papers on various applications of the Green's functions have been submitted for publication. Computer programs have been written for calculation of various Green's functions, SAW velocities, and inversion algorithms for determination of material parameters of films from observed SAW velocities in films on anisotropic materials containing an intermediate layer. A preliminary version of the digital library is accessible through the CTCMS website.

Contributors and Collaborators

B. Yang (NIST Guest Researcher); L. Bartolo (Kent State University); A. Powell (MIT)

Physical Properties of Thin Films and Nanostructures: Mechanical Properties

We are developing noncontact, nondestructive tools to measure thin-film mechanical properties such as elastic modulus and residual stress. Our approach utilizes laser-ultrasonic methods to determine the frequency-dependent velocity of surface acoustic waves. The 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

Successful industrial implementation of thin films frequently requires knowledge of film mechanical properties. This knowledge is needed to estimate surface residual stresses, to predict component reliability or performance, and to assess film-substrate adhesion. Yet current methods are generally limited to destructive tests or even "try it and see." Moreover, film properties usually cannot be extrapolated from those of bulk samples (if they even exist). We are developing nondestructive methods to quantify the elastic properties of thin films. Our objective is to relate physical properties such as elastic moduli, residual stress, or adhesion to quality or performance factors like component lifetime.

Our approach makes use of surface acoustic waves (SAWs). SAWs are well suited for our purposes, since their energy is concentrated near the surface but the energy decay away from the surface depends on wavelength. Hence the SAW velocity depends on the ratio of wavelength to film thickness. We optically generate and detect SAWs to obtain the SAW phase velocity over a broad frequency range (dispersion relation). Results are then compared to the predictions of an analytical model to determine quantitative elastic moduli.

Accomplishments

In FY02, a series of superhard nanocomposite $Ti_{1-x}Si_xN_y$ films developed for enhanced wear resistance were examined. Values greater than 40 GPa for the microhardness H were measured by instrumented indentation techniques (IIT). Our SAW values for Young's modulus E ranged from 325 to 417 GPa and were in good agreement with destructive IIT results. Both H and E were observed to depend on the magnetron power or atomic fraction of Si in the films. From the results, the ratio H/E, an indicator of wear resistance, was found to be relatively high. Work is underway to explore relationships between modulus, microstructure, and growth parameters. We also compared SAW results with those obtained by IIT for Al, Nb, and (three) TiN films. Results differed by $<15\%$, and no systematic errors were observed. Discrepancies could be attributed to effects like film thickness or residual stress.

We also started SAW evaluation of films developed for microelectronic applications. Highly porous films like silica aerogels hold great promise as insulators due to low values of the dielectric constant k. Although E is only $\sim$1 GPa in such films, they must withstand strenuous chemical–mechanical processing. We began modifying the experimental apparatus to accommodate these materials. SAW experiments on several porous silica and polymer films showed clear relations between E, porosity or density, and k. Accuracy, in determination of porosity (from density), was about 1%.

Another activity in FY02 examined residual stress. Residual stress changes a film's effective density and elastic constants and therefore alters SAW propagation. We did preliminary calculations to examine stress effects on SAW dispersion in TiN films. We also calculated the effect of equibiaxial residual stress on SAW dispersion using an elastodynamic Green's function method. For a Ge film with 1 GPa compressive stress on a Si substrate, the change in wavespeed due to stress reaches a minimum of -0.5% at a finite frequency. Furthermore, the effect of residual stress on the distortion and evolution of finite-amplitude (nonlinear) SAWs was investigated theoretically. The thickness of a single-crystal Ge film on a Si substrate was varied so that the system possessed weak, moderate, and strong dispersion relative to its nonlinearity. Stress had the largest effect on waveform components in thicker films, of higher harmonics, and at longer propagation distances. For example, changes in magnitudes of the first five harmonic components were 3% to 15% in the case of moderate dispersion.

In FY02, the delta-function representation of the elastodynamic Green's function developed previously was extended to systems with multiple film layers. We developed a new algorithm to find the material parameters (density, elastic moduli, thickness) of a film/interlayer/substrate system for general anisotropic materials. In this algorithm, we insert the observed values for the velocities directly into the expression for the poles and solve for the material parameters of the film. This approach is more efficient than conventional methods of inversion that require many forward calculations and least-squares fitting between the observed and the calculated velocities. We are now using this formulation to examine anisotropic TiN films (thickness 300–3000 nm) on Si substrates with a 100 nm intermediate layer of metallic Ti.

Contributors and Collaborators

Division participants: V.K. Tewary, R.E. Kumon, C.M. Flannery, D.C. Hurley; *NIST collaborators:* D.T. Smith (MSEL Ceramics Division); *External collaborators:* J.A. Berger (Colorado School of Mines); D. Fei (Caterpillar); A.F. Jankowski (LLNL); A.J. Richards (CSIRO–Australia)

Physical Properties of Thin Films and Nanostructures: Thermal Barrier Coatings

Steady-state measurement techniques and appropriate reference materials for thermal conductivity of ceramics and ceramic coatings used in advanced engines have been developed. This provides industry with calibration and transfer between transient and steady-state data, allowing understanding of the relationship between thermal performance and microstructure.

Andrew Slifka

Technical Description

Thermal-barrier coatings have applications primarily in gas turbines and diesel engines to allow higher operating temperatures and longer lifetimes to increase efficiency. New materials with designed microstructures for lowering thermal conductivity are being developed, as well as nanoscale superlattices for thermal barriers. Industry uses a measurement of thermal diffusivity, which is fast and generally reliable, but a bridge is needed to provide designers with thermal conductivity and to calibrate diffusivity measurement apparatus. To this end, we provide steady-state thermal conductivity measurement methods and data and develop appropriate reference materials.

Accomplishments

We continue to measure ceramic coatings made by electron-beam directed vapor deposition, a high-deposition-rate variant of electron-beam physical vapor deposition (EB-PVD), which are designed to lower thermal conductivity by controlling the coating microstructure. In collaboration with the University of Virginia, we found that coatings with porosity controlled on three spatial size levels can reduce thermal conductivity by 50% compared to current-technology coatings from industry. In addition, the deposition process used at the University of Virginia is two or three orders of magnitude faster than the standard EB-PVD process. Figure 1 shows thermal conductivity data, which include results showing that this advanced process can produce coatings comparable in morphology and thermal performance with industrial EB-PVD coatings. Controlling porosity results in the controlled reduction of thermal conductivity.

Also in collaboration with the University of Virginia, we are measuring thermal conductivity of new zirconate materials. Evidence in the literature shows that lanthanum and samarium zirconates have lower thermal conductivity than currently-used yttria-stabilized zirconias with similar microstructures. Preliminary measurements on a lanthanum zirconate ($La_2Zr_2O_7$) coating produced using EB-PVD shows a low thermal conductivity.

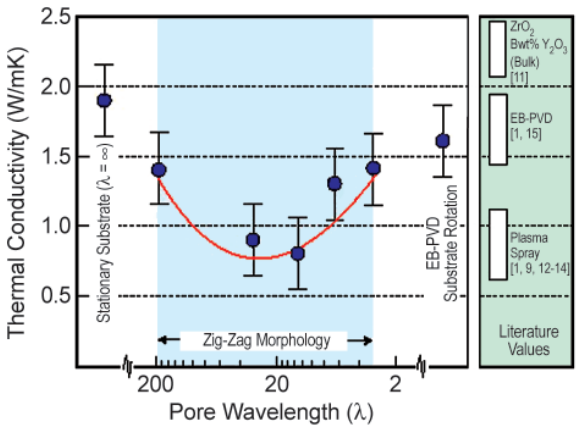


Figure 1: *Thermal conductivity of EB-PVD thermal barrier coatings, including an industrial EB-PVD sample and corresponding literature values.*

Measurements were made on an aluminum nitride material from CoorsTek. Even though our infrared microscopy system was not designed to measure high-conductivity materials such as this, we made measurements to determine whether the measurement method was feasible in that range. CoorsTek was interested in transferring the measurement technology. However, our infrared microscopy method proved to be unable to make measurements in that high conductivity range. In an answer to this need and others, we are developing a measurement system using scanned-probe microscopy that works similarly to the infrared system, but is designed to operate over a wide range of thermal conductivity. Future applications will include materials ranging from thermal barrier coatings to heat removal films used in electronics.

We have received thermal barrier coating specimens that use 4 elements with graded composition across the face of the specimen. This specimen is very large when compared to our typical specimens. Therefore, we have built a system to manipulate the specimen, and we will have a new IR microscope by the end of the year which will enable the automation of thermal measurements in a combinatorial fashion.

Contributors and Collaborators

B. James Filla (NIST, Information Technology Laboratory); D. Hass (University of Virginia); H.N.G. Wadley (University of Virginia); N. Nichelson (CoorsTek, Golden, Colorado)

Physical Properties of Thin Films and Nanostructures: X-ray Methods

We strive to correlate and explain macroscopic properties of technologically interesting materials by their underlying microstructure. We focus especially on x-ray diffraction studies of ferroelectric, optoelectronic, 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 refinement and determination of short-range and long-range crystal structure in materials.

Tom Siewert and Davor Balzar

Technical Description

Many macroscopic materials properties depend on the crystalline structure, internal stress, texture, and defect concentration. We develop methods to obtain this information from diffraction measurements, mostly for microelectronics and wireless communications applications. Diffraction measurements are compared to measurements by other experimental techniques, such as atomic-force microscopy (AFM) and scanning electron microscopy (SEM). The properties are then correlated with other physical properties, typically dielectric, ferroelectric, magnetic, and electric.

Accomplishments

In this period, we focused on the development of methods for the characterization of crystallite size and size distribution and influence of residual stresses and defects on ferroelectric and dielectric properties. We analyzed seven sets of measurements conducted on a ceria sample that was prepared for a size-strain round robin, which was carried out last year with 18 participating laboratories. The sample was prepared at University of Rennes from a precursor hydrated ceria by heating in a silica crucible at 650°C for 45 hours. Another ceria sample was prepared at NIST to correct for the effects of instrumental broadening by annealing commercially obtained ceria at 1300°C for 3 h and slowly cooling it in the furnace. The diffraction measurements were carried out on two laboratory (University of Birmingham and University of Maine) and two synchrotron (NSLS and ESRF) x-ray sources, two constant-wavelength neutron (NIST and ILL) and a TOF neutron (ISIS) source. SEM micrographs have shown predominantly spherical grain shape with a lognormal size distribution. Diffraction measurements were analyzed by three methods: a model assuming a lognormal size distribution of spherical crystallites, Warren-Averbach analysis, and Rietveld refinement. The last two methods have detected a relatively small strain in the sample, as opposed to the first method. Assuming a strain-free sample, the results from all three methods agree well. The average real crystallite size, on the assumption of a spherical crystallite shape, is about 180 Å. The scatter of results given by different instruments is relatively small, although significantly larger than estimated standard uncertainties. The complete results were published in the open literature.

The influence of stresses on the ferroelectric properties (Curie–Weiss temperature) was studied on pristine, W and Mn 1% doped $Ba_{0.6}Sr_{0.4}TiO_3$ epitaxial thin films that were deposited by pulsed laser deposition (PLD) on the $LaAlO_3$ substrate. X-ray diffraction was used to determine both residual elastic strains and defect-related inhomogeneous strains by analyzing diffraction line shifts and line broadening, respectively. We found that both elastic and inhomogeneous strains are affected by doping. This strain correlates with the change in Curie–Weiss temperature. To quantitatively explain the experimental findings, we model the ferroelectric properties in the framework of Landau–Ginsburg–Devonshire thermodynamic theory. Both compressive in-plane elastic strains (stresses) and especially inhomogeneous strains influence the observed increase in Curie–Weiss temperature. For a good agreement with the thermodynamic theory, it is necessary to consider the contribution of inhomogeneous strains, as shown in Figure 1, where a consideration of only elastic strain gives a poor fit to the thermodynamic theory.

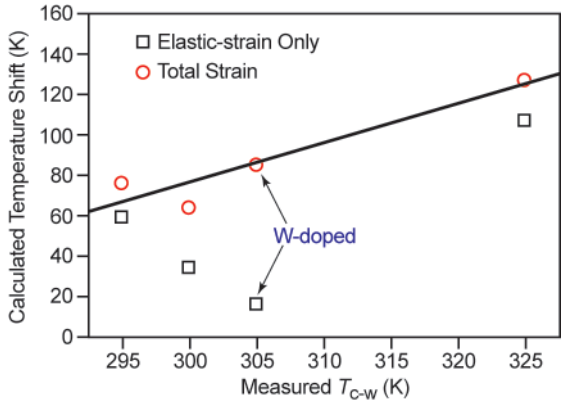


Figure 1: *Temperature shift of the Curie–Weiss temperature, calculated from the thermodynamic theory, for both elastic strain and total (elastic and inhomogeneous) strain contribution to the Gibbs free energy as a function of Curie–Weiss temperature determined from the dielectric measurements.*

Contributors and Collaborators

S. Mani, C.N. McCowan, N.C. Popa, P. Spagnol, B. Toby (NIST); S. Lewis, M. Moench (University of Denver); A.M. Hermann, P.A. Ramakrishnan (University of Colorado); N. Audebrand, D. Louër (University of Rennes); M.R. Daymond (ISIS, Rutherford–Appleton Laboratories); A. Fitch, O. Masson (ESRF, Grenoble); A. Hewat (ILL, Grenoble); J.I. Langford (University of Birmingham); A. Le Bail (University of Maine, Le Mans); P.W. Stephens (SUNY at Stony Brook)

Infrastructure Reliability: Charpy Impact Machine Verification

We assist owners of Charpy impact machines in achieving conformance with the requirements of ASTM Standard E 23. We interact with the ASTM Committee responsible for the Charpy impact standard to improve the service and to maintain a high-quality verification program. We also participate in the activity in ISO Committee TC 164, so our specimens and procedures remain compatible with the associated international and regional standards.

Daniel P. Vigliotti

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 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 verification 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 (3 J to 400 J capacities) that are used for research purposes.

Accomplishments

We had about 1000 customers for this service in FY02, 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, many users took advantage of our support services, as shown by over 2000 emails, 650 faxes, and 4000 phone calls. We immediately contact the machine owner when it fails to meet the verification criteria. In this contact (by phone, mail, email, or fax), we suggest corrective measures. Also, in our laboratory, we tested the 2635 specimens necessary to confirm that 17 new lots of reference specimens were suitable to enter the SRM inventory.

We are in the middle of a three-year test program that is collecting 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 E 23) 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). The next symposium on this topic is being planned for 2004.

Chris McCowan serves as the Chairman of ISO TC164 SC4 P on pendulum impact and also as the Chairman of ASTM Subcommittee E28.07 on impact testing. Dan Vigliotti continues as the Chairman of the Task Group that oversees Standard E 23, 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 these data.

Currently, we are working on some enhancements to the program, including a training video that explains the operation of the verification program.

Contributors and Collaborators

NIST participants: D. Vigliotti (Charpy Program Coordinator); C. McCowan, T. Siewert, J. Alcorn, A. Rodriguez, D. Cyr, N. Neumeyer, L. Leininger, C. Farrell; *Industrial collaborators:* Members of ASTM Subcommittee E 28.07

Analysis of Structural Steel in the World Trade Center Investigation

In 2002, NIST became the lead agency in a planned investigation of the World Trade Center collapse. The investigation addresses many aspects of the catastrophe, from occupant egress to factors affecting how long the Twin Towers stood after being hit by the airplanes, with the goal of gaining valuable information for the future. A critical aspect of the investigation is the metallurgical and mechanical analysis of structural steel from the Twin Towers and WTC 7. The analysis includes characterization of properties, failure modes, and temperature excursions seen by the steel.

David McColskey

The collapse of the twin World Trade Center towers on September 11, 2001 was the worst building disaster in human history. Engineers, emergency responders, and the nation did not anticipate, and were largely unprepared for, such a catastrophe. Among other national needs, these events highlight the following technical priorities:

- To establish the probable technical causes of the collapses and derive the lessons to be learned;
- To develop and disseminate immediate guidance and tools to assess and reduce future vulnerabilities; and
- To produce the technical basis upon which cost-effective changes to national practices and standards can be developed.

NIST has prepared a technical investigation plan to address these issues (see <http://wtc.nist.gov/>). A primary objective of the investigation is to determine why and how the World Trade Center Buildings collapsed after the initial impact of the aircraft. To aid in this investigation, more than 100 WTC steel structural elements were brought to NIST to be studied. The NIST Metallurgy and Materials Reliability Divisions' project to study the steel is outlined here.

Task 1 — Identification of steel. Steels in the exterior and core columns, welds, spandrels, trusses, truss seats, and fasteners of the WTC Towers 1 and 2, and WTC 7 will be identified based on composition, microstructure, and mechanical properties, and compared to specifications from the period.

Task 2 — Failure mechanisms based on visual evidence. Steel pieces at NIST, and evidence available from other sources, will be examined and documented as to failure mechanisms.

Task 3 — Property data to support structure performance and airplane impact modeling studies. In addition to the mechanical properties provided in Task 1, the steels in the exterior and core columns, welds, spandrels, and fasteners of WTC 1 and 2 will be tested for high strain rate properties, including tensile and yield strength, ductility, and impact properties.

Task 4 — Steel property data to support models of steel frame performance in fire. Creep and high temperature tensile properties will be measured for columns, bolts on columns, trusses, hangers, and bolts or welds associated with truss seats.

Task 5 — Steel property data to support models of steel performance during collapse. High-strain-rate, room-temperature tensile properties will be measured for truss seats and associated bolts and welds.

Task 6 — Models of elevated temperature deformation behavior. Models of elevated temperature deformation as a function of load and time at temperature will be provided for relevant steels, giving deformation information for steels with any given history of loading and temperature.

Task 7 — Determination of maximum temperatures exposure. The steel will also be analyzed to estimate maximum temperatures reached, although it is recognized that high temperature exposure before the collapse may be difficult or impossible to distinguish from exposure during post-collapse fires. Studies of paint condition on the steel, microstructural changes in the steel, and stress relief in high strength bolts, washers, and weld zones will be done.

(Skilling Ward Magnusson Barkshire Inc.)

Figure 1: *Exterior columns and floor trusses during WTC tower construction. These critical elements of the steel structure will be characterized in the NIST investigation.*

Contributors and Collaborators

T. Siewert, C. McCowan, R. Santoyo (Materials Reliability Division, MSEL); F.W. Gayle, R.J. Fields, S.W. Banovic, T. Foecke, M. Williams, D. Kelley, S. Claggett, W. Luecke, J. Cahn (Metallurgy Division, NIST Materials Science and Engineering Laboratory); P. Brand (NIST Center for Neutron Research, MSEL); J. Gross (NIST Building and Fire Research Laboratory)

Infrastructure Reliability: U.S. Capitol Dome

Corrosion is leading to cracking of the outer castings of this well-known landmark. We are leading a team of experts from the welding industry in developing innovative repair procedures, which will restore the dome's castings to their original integrity.

Tom Siewert

Technical Description

The dome of the United States Capitol is undergoing a rehabilitation program. The work has been undertaken by the Architect of the Capitol to ensure the structural integrity of the 130-year-old dome as well as its protection and preservation into the indefinite future. Built between 1856 and 1866, the dome is made of 8,909,200 pounds of cast iron. Corrosion between poorly protected plates is leading to cracking of the outer castings (the skin) of this well-known landmark. We are working with the Office of the Architect to develop repair procedures that will restore the outer casting to their initial integrity.

We have assembled a team with broad expertise in welding consumables and weld repair of castings. (The seven team members come from major manufacturers that produce welding consumables or repair cast irons, and they have a total of over 200 years of experience.) This team is developing procedures that can be used to repair the existing cracks and to reattach fractured pieces in the outer cast iron shell of the Capitol dome. The major challenges are: 1) the cast iron has very poor tensile properties (due to its age and composition), and 2) the shell cannot be disassembled for repair. We are evaluating various possible procedures that meet the criteria for the onsite repair, and preparing to demonstrate the optimal procedure on typical cracked joints in the dome.

The team has reviewed the wide range of repair alloy compositions that have been developed over the years. This was particularly important because the dome cast iron has phosphorus levels as high as 0.8 mass %, typical of castings produced over 100 years ago. The combination of high levels of phosphorus with low strength means that traditional repair compositions and procedures would transfer too much stress to the castings and lead to further cracking. Therefore, we concentrated on evaluating the repair materials with the lowest strengths.

Laboratory trials have identified two weld materials (a low-fuming bronze and an aluminum bronze) that best meet the criteria. We are now planning to test these procedures on parts of the dome.

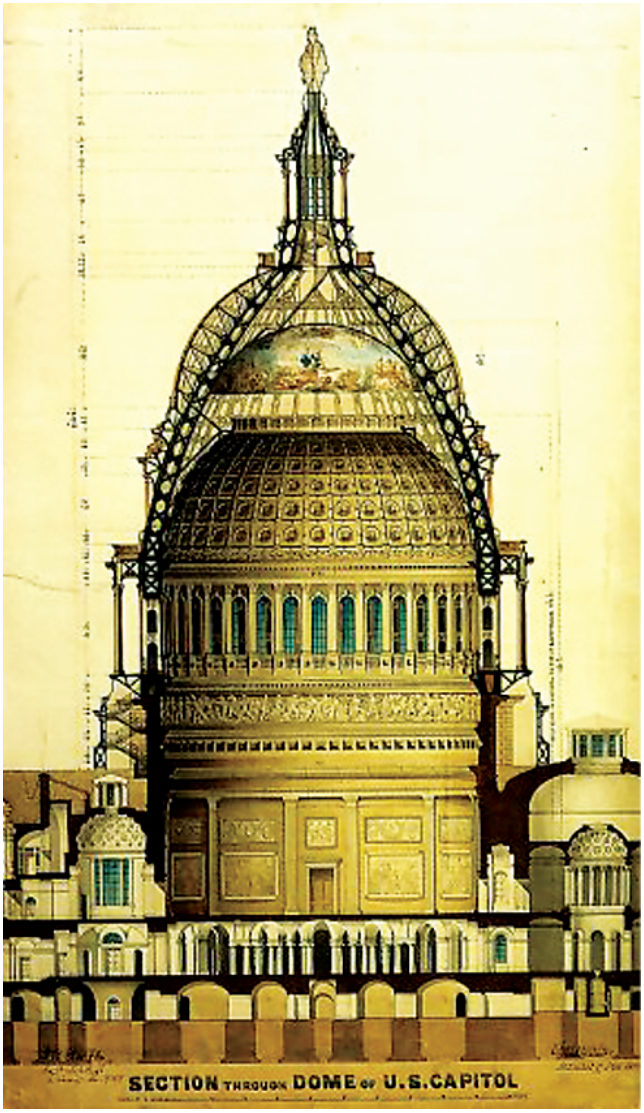


Figure 1: *Section through the Capitol dome, drawn in 1859. The castings that have experienced corrosion are only on the very outer skin of the dome.*

Accomplishments

The laboratory tests were successful, and we are now ready to try the procedures on the dome.

Contributors and Collaborators

C. McCowan, R. Bushey (ESAB Corp.); S. Kiser, E. Hinshaw (Special Metals Corp.); D. Kotecki (Lincoln Electric); B. Myers (recently retired from Dresser Rand Corp.); D. Olson (Colorado School of Mines)

Infrastructure Reliability: Service to Bureau of Reclamation

We 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. Currently, we are assisting on the renovation of Folsom Dam.

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, relies on their Technical Service Center. When particularly complex problems call for additional expertise, the center recruits experts from other government agencies and from the private sector. Our services are frequently 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.

Over the past several years, we have studied the failures of pre-stressing wires on pipes in the BOR's Central Arizona Project (CAP). Our examination of failed wires from the reinforced pipe indicated that stress corrosion cracking initiated at pre-existing flaws in the wire, leading to the fracture of individual wires. Our study of these failures contributed to a change in the type of pipes considered as options for these applications. The original siphons have been replaced by steel-lined concrete pipes, and the BOR has placed a moratorium on the use of the pre-stressed pipe.

Other examples of the NIST–BOR interaction for this past year include the evaluation of brass tubes that failed on an air cooler at the Colorado Big–Thompson Project and fractured connection bolts from Hoover Dam.

We have just started to review proposed revisions to the Folsom Dam sluice gates. The plan is to increase the volume of water that can flow through these gates

Figure 1: *View of Folsom Dam.*

by about a factor of three, by increasing their internal dimensions and adding two more gates. Three of us have visited the dam to gain first-hand knowledge of the general metallurgical, corrosion, and welding issues. Our task is to issue a report that comments on the suitability of the plans in the areas of materials, welds, inspection procedures, corrosion, and the effect of dissimilar metals.

We had a chance to look closely at all the various details of the proposed configuration and the condition of the present gates. We learned that the current gates (nearing 50 years old) are performing well, and there do not seem to be problems with the current materials. However, current manufacturing capabilities do not give BOR an option to build the new gates with the same materials. We were able to offer some preliminary advice and will submit our final report in the next few months.

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.

Contributors and Collaborators

C. McCowan, T. Siewert, R. Ricker (NIST); J. LaBoon, D. Read, T. Johnson (Bureau of Reclamation)

Infrastructure Reliability: Waveform-Based Acoustic Emission

The major project objective is to develop the scientific underpinnings necessary to enhance acoustic emission (AE) technology through increased, high-sensitivity bandwidth. Current secondary objectives include: (1) developing for many users the missing element of modeling AE signals for multiple sources in specimens with and without edge reflections; and (2) developing rational analysis approaches to AE waveforms to solve the real-world problems of reliable identification and location of sources of AE signals.

Tom Siewert and Marvin Hamstad

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result of micro-sized transient energy releases in a material. Monitoring these waves can provide fundamental information about the location and mechanism(s) of the transient-energy release as well as the time/stress history of such releases. 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 relevant to a wideband application of AE technology. These include development of wideband high-sensitivity sensor/preamplifiers; high-speed digital recording data-gathering systems of wide dynamic range; finite-element modeling to predict near- and far-field displacement waves from AE sources; wideband experimental AE displacement waveforms from 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 FY2002 covered three related phases: 1) additional finite-element modeling of displacement signals (for radiation angles from 0 to 90 degrees) resulting from buried point sources in large specimens; 2) computation of wavelet-transform (WT) results for a variety of AE source types, depths (relative to a plate's top surface) and radiation angles; and 3) analysis of the application of WT results to location of AE sources.

Accomplishments

1. By use of the NIST developed finite-element code, an extensive database of AE signals exists for 11 source types of single- and multi-dipole sources in an aluminum plate 4.7 mm thick at six or seven source depths. The plate has transverse dimensions sufficient that plate edge reflections do not superimpose on direct signal arrivals. For each source type and depth, AE signals at seven different radiation directions have been calculated. From the signal database, a WT database can be computed from the out-of-plane displacement signals at three far-field distances from the AE sources.
2. WT results demonstrate how the energetic portions of the AE signals change as a function of source depth and radiation angle for a microcrack initiation source (See Figure 1). Note amplitude of WT is shown by color (red highest), and also shown are the superimposed fundamental Lamb modes.

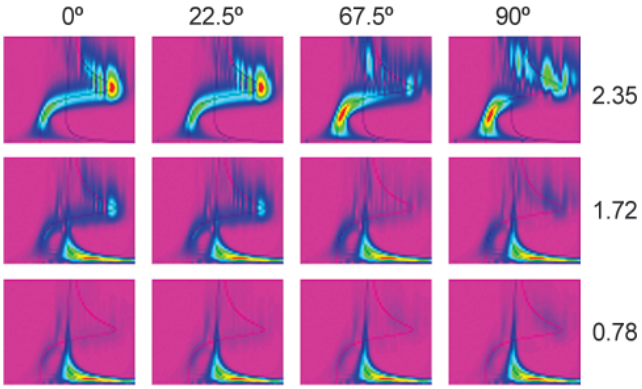


Figure 1: *WTs at 180 mm propagation distance showing frequency (0 to 1 MHz) versus time (0 to 120 ms), crack initiation source at three depths (in mm), and four radiation angles.*

3. Additional results show that an accurate arrival time of a specific energetic frequency and mode (with associated group velocity) can be obtained from a frequency-based WT maximum, even in the presence of a low signal-to-electronic-noise ratio. (See Table 1).

Table 1. *Arrival times (ms) from the peak WT magnitude at 522 kHz for the original AE signal dominated by a higher-frequency region of the fundamental symmetric mode.*

S/N ratio	Low freq. noise	Distributed freq. noise
No noise	97.9	97.9
5:1	97.9	97.9
2:1	97.9	97.9
1:1	97.9	97.8
1:2	97.9	87.8

Contributors and Collaborators

NIST participants: D. McColskey, D. Hodgson (MRD); *Cross OU collaborators:* A. O'Gallagher, J. Gary (NIST-ITL); W. Prosser (NASA Langley)

Infrastructure Reliability: Elastic-Stiffness and Related 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.

Hassel Ledbetter and Sudook Kim

Technical Description

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

During the 2002 fiscal year, we completed the following eleven studies:

1) Elastic stiffnesses, Debye temperatures, and T_c in cuprates (review);
2) The internal-friction "tensor" (review);
3) Monocrystal elastic coefficients of natural quartz;
4) Monocrystal elastic and piezoelectric coefficients of lithium niobate;
5) Monocrystal elastic and anelastic coefficients of lithium niobate;
6) Internal-friction "tensor" in langasite;
7) Elastic coefficients of layers in laminates;
8) Acoustic studies of composite-material interfaces (review);
9) martensite crystallography and elastic coefficients;
10) extended Landau theory of phase transitions; and
11) low-temperature monocrystal elastic coefficients of the charge-density-wave material lutetium-iridium-silicon.

Against most existing theories, study one shows that phonons must participate in the high-T_c mechanism. Study two shows how to use acoustic spectroscopy to determine not only the elastic-stiffness tensor C_{ijkl}, but also its complete imaginary parts, the internal-friction tensor Q^{-1}_{ijkl}. The first application of acoustic spectroscopy to trigonal-symmetry spheres appears in study three. The fourth study represents the first-ever simultaneous measurement of the complete elastic-stiffness tensor C_{ijkl} and the piezoelectric tensor e_{ijk}. It opens the way for a completely new, improved approach to the e_{ijk}. A companion study, number five, gives the anelastic tensor Q^{-1}_{ijkl} and shows how to deduce from the C_{ijkl} that lithium niobate possesses both covalent and ionic bonding, contrary to the literature. Another piezoelectric, langasite, provided the basis for the sixth study, which concluded that the internal friction must arise from phonons scattered by vibrating dislocations. The seventh study focused on laminated composites with different compositions of tungsten-particle copper-matrix. New theory permitted determining layer properties from macroscopic composite properties. The eighth study (invited for an international conference) provided a chance to review six of our studies related to composite-material interfaces and to argue for careful acoustic measurements combined with solid-mechanics modeling. In the ninth study, we answered the old question, "How do the elastic-stiffness coefficients affect the martensite crystallography?" Landau's theory of near-second-order phase transitions provided the starting point for the tenth study, where we included terms through third order in the order parameter Q and showed how elastic-stiffness coefficients behave above, at, and below the phase transition. Remarkable in the eleventh study is that monocrystals C_{ijkl} were measured through the phase transition, representing one of the very few such cases. Oddly, in a Landau-theory sense, some C_{ijkl} behave as expected for a second-order transition, while others behave as for a first-order case.

Late in the year, Dr. Ledbetter accepted a senior-research position at Los Alamos National Laboratory where he will continue these and related researches.

Contributors and Collaborators

H. Ogi (Osaka University); M. Dunn (University of Colorado); P. Heyliger (Colorado State University); A. Migliori (Los Alamos National Laboratory)

Interface of Materials with Biology

New materials and devices are radically changing the medical treatment of injury and disease, yet because of the rapid growth of this segment of the materials industry, an adequate measurement infrastructure does not yet exist. The program on the Interface of Materials with Biology develops measurement methods, standards, and fundamental scientific understanding at the interface between materials science and biological science. Within the health care industry, we focus on dental and medical sectors that apply synthetic materials for replacement, restoration, and regeneration of damaged or diseased tissue.

Five major activities constitute this program:

- The dental industry is primarily composed of small manufacturers with very little R&D capability. The dental materials projects, carried out in collaboration with the American Dental Association, located in the Polymers Division, fill that gap by developing improved materials and techniques, patenting and licensing these inventions, and, most importantly, providing technical assistance to the licensees for producing and improving their products. This has provided U.S. companies with products that successfully compete in a worldwide market. Our research focuses on improved understanding of the synergistic interaction of the phases of polymer-based composites and the mechanisms of adhesion to dentin and enamel. This approach will ultimately lead to materials with improved durability, toughness and adhesion to contiguous tooth structure.
- In this era of interdisciplinary approach to research, the Materials Reliability Division is providing an added dimension to studying diseases and cellular function. By taking a physical/mechanical approach to how cells function, respond, and remodel, we are able to provide insight into the progression of diseases using knowledge and skill sets typically absent in the biomedical community. We concentrate on mechanical property metrology for several biological systems, including natural as well as engineered tissue and spanning a considerable size range from individual neurons and muscle cells to complete pulmonary arteries. This necessitates the development of unique mechanical testing platforms ranging from electrical and mechanical probes of individual living cells to biaxial stressing systems. We interpret our measurements by focusing on the roles of structural elements such as cells, composition, and tissue anisotropy. This classical materials science approach to understanding properties is proving invaluable to the biomedical community.
- The tissue engineering industry shows the potential for explosive growth in the coming years as biomedical research is moving from academic science to industrial application at an increasing rate. Work in the Polymers Division seeks to bridge the gap between knowledge generation by cell biologists and product development in industry. In collaboration with the Chemical Science and Technology Laboratory, we are developing measurement methodologies and reference materials to use in assessing interactions in complex systems of living cells with synthetic materials. The expected outcomes of this work include methods to use reference substrates that induce specific cellular responses and engineered DNA vectors that act as fluorescent reporters of cellular responses.
- Regenerating form and function to bone defects in an elderly, osteoporotic population of Americans is a daunting challenge. To meet this challenge, the Polymers Division is collaborating with the American Dental Association to develop metrology methods to characterize the biocompatibility of synthetic bone grafts. Quantitative methods being developed include assays for adhesion, viability, proliferation, and differentiation of bone cells, as well as optical coherence tomography and confocal microscopy for measuring tissue ingrowth. The combinatorial approach is used to rapidly identify compositions and surface features that provide desirable properties such as biocompatibility and mechanical durability.
- The NIST Center for Neutron Research and the NIST Biotechnology Division are engaged in the study of biomimetic films that serve as models of cell membranes and which are of fundamental importance in understanding such key biological processes as phospholipid self-assembly, molecular recognition, and cell-protein interactions. Recent improvements in neutron reflectometry at the NCNR, coupled with advances in biomimetic film fabrication at metallic interfaces pioneered in the Biotechnology Division, afford enhanced sensitivity for probing membranes and membrane-protein complexes. New phase sensitive measurement techniques and model-independent data analysis methods have demonstrated the feasibility of obtaining reliable depth profiles of membrane structures in contact with biologically relevant aqueous environments, achieving subnanometer spatial resolutions.

Fundamental to much of the work in this program is the recognition that surfaces and interfaces play a critical role in biological systems and, in particular, in the interactions of synthetic or designed materials with biological systems and function. By providing the unique expertise in the NIST Materials Science and Engineering Laboratory to characterization of surfaces and interactions at interfaces in biomaterials, we will accelerate the introduction of improved materials and help provide the means to assure quality control that is critical to this industry.

Contact: Elizabeth Drexler, Robert Keller

Biomaterials Metrology: Pediatric Pulmonary Hypertension

Pulmonary hypertension is a potentially fatal complication of congenital heart defects in children who live at high altitudes. Discovering the cause and expression of the disease may open the way to finding new, more effective treatments. Our contribution to this goal is to provide data on the mechanical properties of the pulmonary artery and its constituents in healthy and diseased tissue for use in a constitutive model that fully describes the viscoelastic behavior of arterial tissue.

Elizabeth Drexler

Background

We in the Materials Reliability Division have a unique opportunity to help the children in our area. Children born with heart defects (approximately 10%) at the high altitudes found in the Rocky Mountain region have an increased risk of developing pulmonary hypertension. The mechanisms for developing this potentially fatal complication are not clearly understood, nor are the physical modifications that result in clinical expression. It is known that with the hardening of the pulmonary artery, the heart becomes enlarged trying to maintain a constant flow volume. This, in turn, exacerbates the condition of the artery and eventually leads to congestive heart failure. By measuring how, where, and to what extent the pulmonary artery remodels, we hope to contribute to a comprehensive study on how to prevent or, at least, mitigate the effects of pediatric pulmonary hypertension.

Technical Strategy

With a team of seven scientists from the Materials Reliability Division, we are collaborating with Robin Shandas, a faculty member at University of Colorado with a joint appointment in the Mechanical Engineering Department and at the Health Sciences Center, and his team of graduate students.

It is well known that arteries have non-linear viscoelastic behavior. Additionally, the properties are not isotropic, but directional, similar to those of a reinforced composite. Our approach has been to study the literature and develop a constitutive model in order to ascertain which measurements are needed to adequately describe the tissue behavior. In order to fully characterize the artery material, we have developed test methods that address the anisotropy of the tissue and support this model.

In FY02, we are providing data on the mechanical properties of healthy and diseased rat pulmonary arteries. The strategy is to start with rats, as they are easily bred to be genetically identical: thus we can ensure that property differences are due to the disease, not to genetic variability. However, the hypertension is induced chemically in rats. The next phase will be to measure mechanical properties of calf pulmonary arteries in which the disease is effected in a hypobaric chamber.

Accomplishments

In FY02, we have concentrated our efforts on the biaxial pressure/bubble test. With this test, we dissect the artery into two ~ 4 × 4 mm sections. Each section is placed on an O-ring, constrained by the top annulus, and tested, one with the adventitia side up and one with the intima side up, in the fixture shown in Figure 1. The entire test fixture is submerged in an ionically balanced fluid in order to keep the tissue from dehydrating, and it is tested within 24 h of harvesting to minimize the effect on properties. The arterial wall is loaded with saline solution under pressure in a step ramp and images from the three axes are collected at each step. We have the option to collect multiple off-axis images if the displacements are not biaxial. The images are analyzed for displacements in order to generate stress–strain curves. We are focusing on the elastic region of the curve, as plastic deformations such as ruptures and aneurysms are outside the scope of this study.

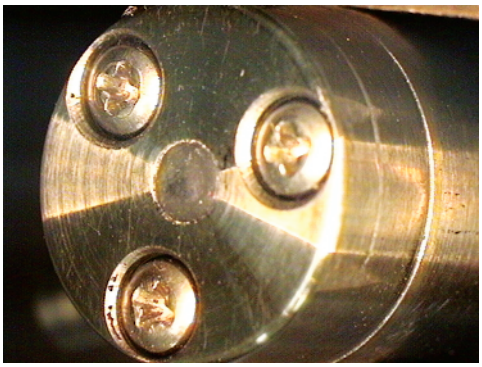


Figure 1: *Biaxial test fixture developed at NIST. The tissue is tested by pressurizing the backside of the 3 mm diameter gauge section located at the center.*

An additional aspect of the test program is to chemically separate the various types of cells (e.g., collagen, elastin, smooth muscle) from the tested material. The purpose is to determine the effect of the disease on the quantity, microstructure and physical properties of the cells, particularly in the adventitia, as the remodeling is thought to be concentrated there.

Contributors and Collaborators

D.S. Finch, J.D. McColskey, C.N. McCowan, T.P. Quinn, A.J. Slifka, J.E. Wright (NIST); R. Shandas, M. Salehi (University of Colorado)

Biomaterials Metrology: Tissue Engineering Scaffolds

Tissue engineering offers the hope that diseased or injured structures within the body can be replaced with materials that are grown outside the body. To be effective, test methods for the mechanical properties of the polymeric scaffold with and without tissue ingrowth must be developed and the properties themselves measured.

Timothy P. Quinn

Background

A proposed method for treating disease and injury in the human body is to replace the diseased tissue with tissue that is grown outside the body. Typically, a seed of healthy tissue is cultured to grow into a polymeric scaffold. The scaffold supports the tissue and coaxes it to grow into the proper geometrical shape. The tissue and scaffold are then implanted in the patient to replace the injured or diseased tissue.

It is essential to know the mechanical response of the scaffold before, during, and after tissue is grown in it. For example, bone replacement materials have particularly severe mechanical requirements: the bulk modulus of cancellous bone ranges from 50 to 100 MPa and the longitudinal fracture toughness of cortical bone between 1.5 and 2.5 MPa·√m.

Technical Strategy

Together with the Polymers Division, a set of materials with different structures will be selected. The structures of the selected materials will be representative of the different kinds of microstructures (porous, woven, etc.) and compositions (plastics, hydrogels, etc.) that are now being considered for tissue engineering applications. Each class of material with variations in structure will be tested in compression and/or tension. The resulting stress–strain curve will be fitted by use of phenomenological models that correlate mechanical properties with microstructure and thereby provide a quality control assay to discriminate whether a processed scaffold meets manufacturing tolerances.

Because of the relatively small size of the samples, ASTM test methods will be modified to test specimens in tension and compression. The structure of the specimens will first be analyzed statistically for important characteristics (such as pore size) and crosslink percentage. A hydraulic tensile machine will be used to apply loads to the specimen. Strain will be measured using video-microscope images of the specimen as load is applied. Strain rate dependencies will be carefully analyzed to fully characterize the phenomenological models.

Accomplishments

In FY02, we have developed the test methods and collected preliminary data on scaffolds manufactured by the Polymers Division. For compression tests, sample geometry of 6 x 6 x 3 mm was selected. A dog-bone sample with a gauge length of 6 mm was selected for tension tests. The samples are first patterned by staining with osmium or sputtering with platinum to allow image correlation techniques to measure the strain. The response of the scaffold pictured in Figure 1 to compression is shown in Figure 2.

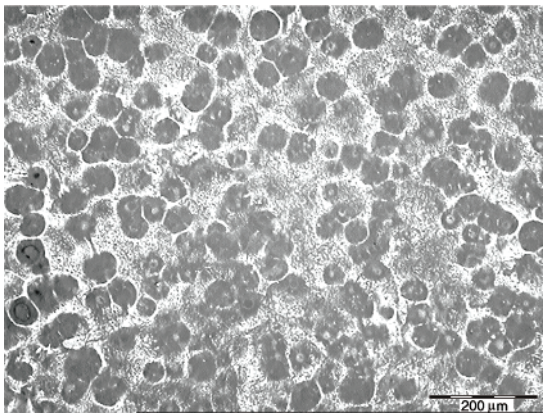


Figure 1: *Microstructure of a typical polymeric scaffold.*

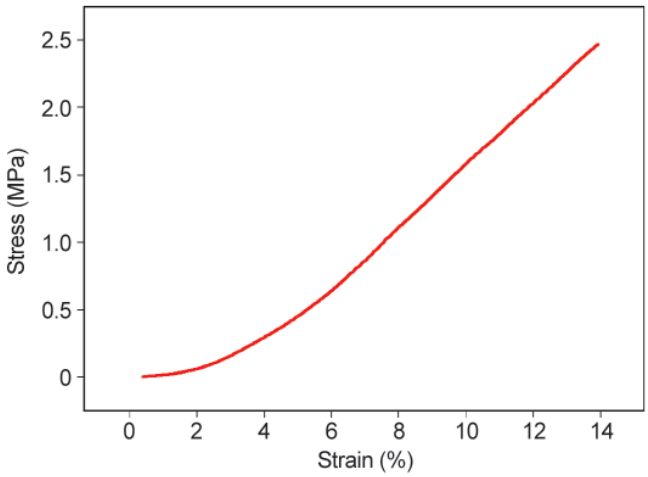


Figure 2: *The response of the scaffold in compression.*

Contributors and Collaborators

J.D. McColskey, C.N. McCowan (NIST); N.R. Washburn, F.W. Wang, C.G. Simon, L.A. Swanegan (Polymers Division)

Biomaterials Metrology: Cellular Engineering Micro Systems (CEMS)

The biomedical community seeks a research platform for the study of fundamental chemical and physical processes at the single-cell scale, as an integral component for understanding disease processes. We are developing CEMS to enable stimulation of single cells and cellular arrays incorporating in situ *monitoring of responses at the cellular, intracellular and intercellular levels.*

Dudley Finch

Background

Cells by nature are heterogeneous, and, therefore, information at the single-cell scale is required to understand many complex cellular processes. The objective of CEMS is to enable stimulation (chemical, electrical, optical, mechanical, magnetic, electromagnetic, acoustic, etc.) of single cells and cellular arrays incorporating *in situ* monitoring of their response. This requires that the CEMS device be capable of maintaining cell viability which, among other considerations, will require an aqueous environment with close control of pH, nutrients, oxygen levels, etc.

One application is the measurement of the mechanical forces exhibited by vascular smooth muscle cells and their response to mechanical forces. Many clinical investigations have established that elevation in blood pressure is a powerful risk factor for the development of coronary artery disease. However, the mechanisms whereby hypertension leads to atherosclerosis are not well understood. Arterial hypertension produces structural changes in the arterial wall, which accelerates the atherogenic process. These responses include growth of smooth muscle cells (hyperplasia or hypertrophy) and vascular remodeling. By studying the response of vascular smooth muscle cell to a range of stimuli, it is envisaged that a more detailed insight into cell function can be gained.

Accomplishments

Since the beginning of this project in March 2002, three prototype designs have been submitted for manufacture using the MUMPS process. An example of one such design to measure the magnitude of the contractile forces exerted by a smooth muscle is shown in Figure 1.

In this figure, a series of circular areas can be seen varying in diameter from 40 to 100 µm. Each of these areas is divided into four or eight pads as wedges,

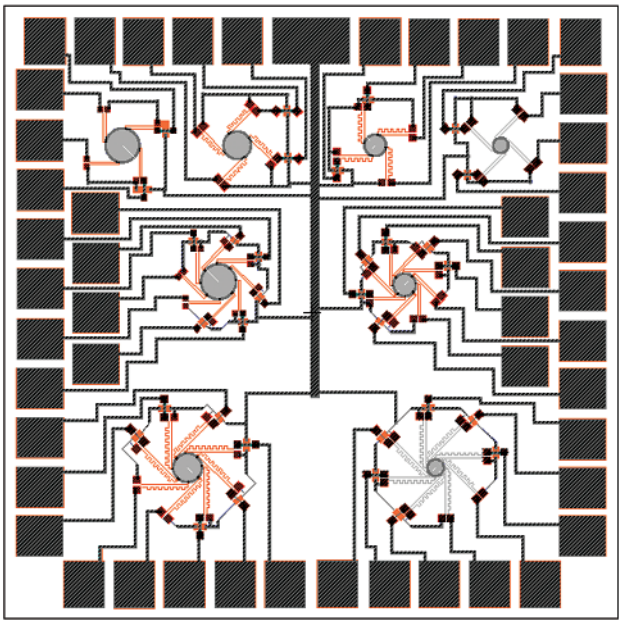


Figure 1: *CEMS design for measuring cellular forces exhibited by vascular smooth muscle cells.*

each of which is attached to a silicon cantilever designed to allow four-point resistance measurements. The separation between each wedge is approximately 2 µm. It is known that cells will not extend through such gaps and will therefore bridge between the wedges, allowing the magnitude and direction of forces exerted by the cells to be measured. Modeling shows that a force of 20 nN, typical of the forces expected, will give rise to a 0.5% change in resistivity.

The cell's function is controlled by its interaction with the substrate material. In order to ensure that cells adhere only to the pads, these areas will be coated with nanometer-thick layers of collagen. Cells will generally not adhere to gold surfaces, and these have been incorporated into the design where cells are not to be deposited. In addition, single cell manipulation is currently being investigated.

Packaging of the final design will be undertaken to enable functioning of the CEMS device in an aqueous environment.

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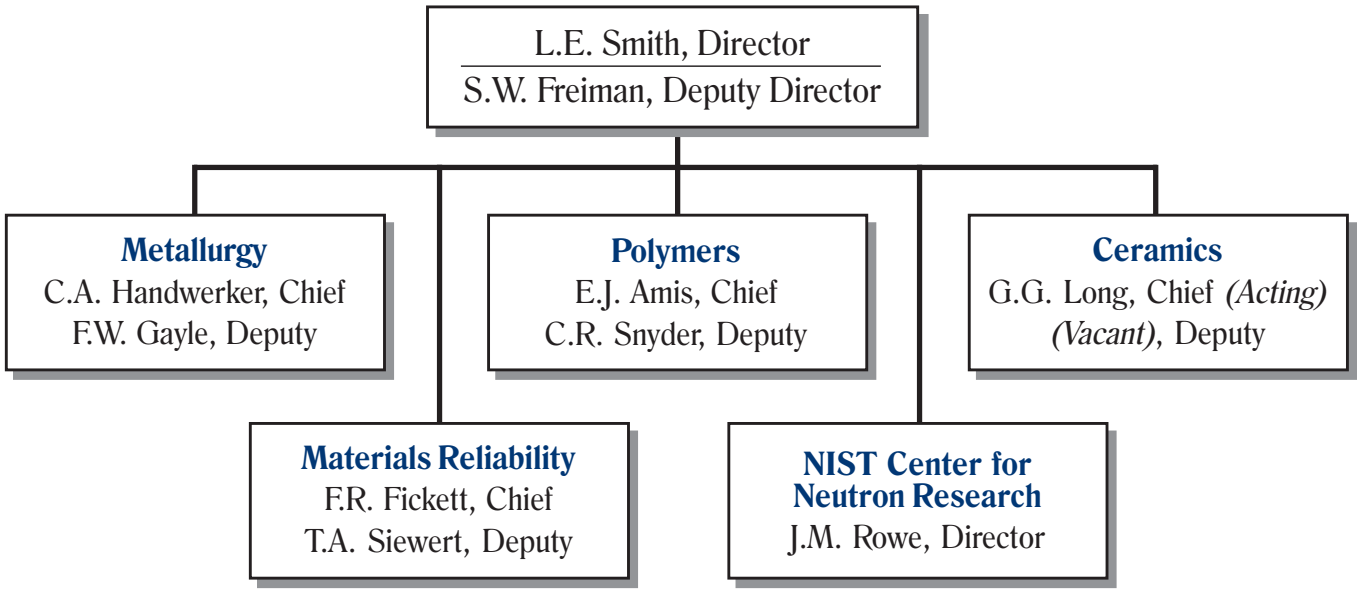

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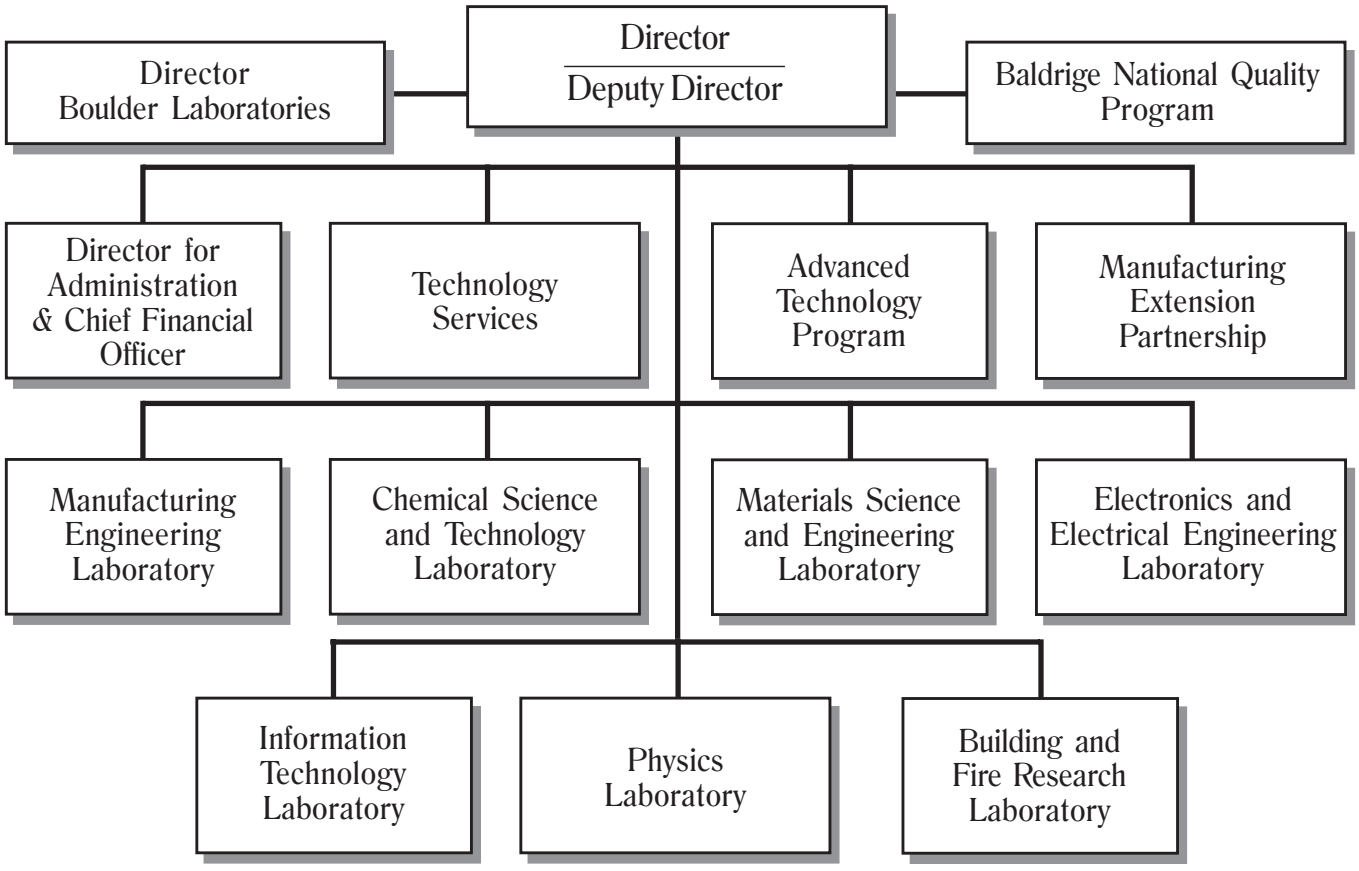

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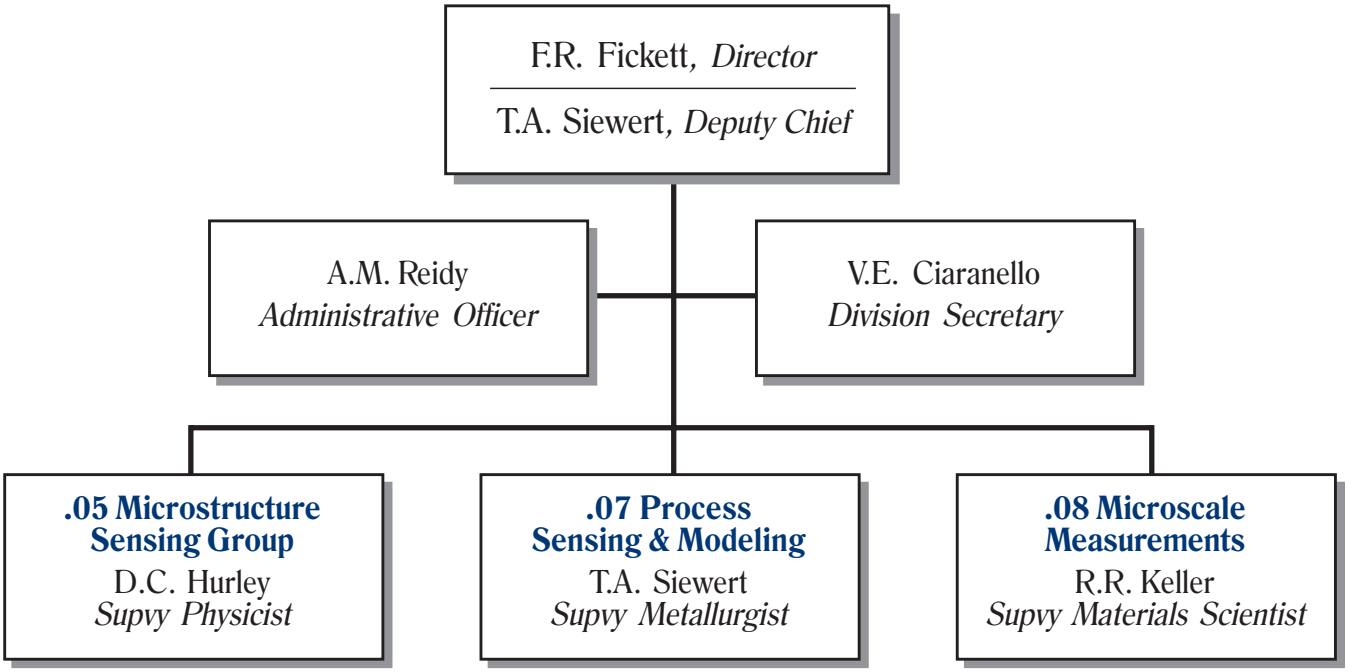

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