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. We have changed our portfolio of research projects slightly this past year, as we have found new, critical technology gaps that limit the reliable use of materials. Yet, we have maintained our competence in our core areas. Much of our research is concentrated in the NIST Strategic Focus Areas of Nano-technology, Health Care, and Homeland Security. Our work spans a wide range of materials, and with a dimension span that extends from nanometer-scale devices — carbon nanotubes and single cells — to tall buildings, gas pipelines and bridges. For FY05, we organized our research into the following focus areas:

Materials for Micro- and Optoelectronics: The U.S. microelectronics and related industries continue to face fierce international competition. The International Technology Roadmap for Semiconductors (ITRS) has long recognized the importance of metrology to the advancement of the industry by devoting an entire chapter to the subject. The 2003 ITRS calls specifically for solutions to the Interconnect Difficulty Challenge (through 2009) of achieving necessary reliability due to the fact that "new materials, structures, and processes create new chip reliability (electrical, thermal, and mechanical) exposure. Detecting, testing, modeling and control of failure mechanisms will be key." We continue to support the industry through the following projects aimed at improved measurements, modeling, and advanced materials science: measurement of mechanical properties, strains, and micrometer-scale reliability of thin films; improved under-standing of chip-level electrical and thermomechanical reliability in metallic interconnects; improved measurements and understanding of thermal flow and damage related to packaging and thin film reliability; linking models for structure and behavior of nanostructures from interatomic, to nano-, to continuum length scales; and molecular dynamics simulations of experimentally observed structure and behavior of thin films.

Nanocharacterization: Metrology, the science of measurement, plays a key role in the development and commercialization of nanotechnology. Manufacturing commercially viable nano-scale products, so widely envisioned in the press, demands vast improvements in our ability to measure material dimensions, characteristics, and structures at the nano-level. The overarching goal of our nanocharacterization effort is to develop reliable and accurate measurement techniques for a broad range of materials and material properties at the nano-scale. We are applying a variety of techniques to meet this challenge, such as atomic force acoustic microscopy (AFAM), Brillouin light scattering, surface acoustic wave spectroscopy (SAWS), x-ray diffraction, and multiscale modeling. We continue to explore synergies between these techniques as well as application of these measurement tools to a wide range of materials: ceramics, polymers, thin films, low-k dielectrics, self-assembled structures and others.

Biomaterials Metrology: The medical research community has recognized a burgeoning need to understand the role that the mechanical behavior of biological materials plays in normal and diseased tissues. This focus area addresses these needs on multiple length scales: cellular, cell + matrix, and tissue levels. On the cellular level, we are using MEMS (microelectromechanical systems) devices to apply and measure loads and displacements on single living cells. Techniques to calibrate the MEMS devices are being developed by use of an optical trap and an atomic force microscope. A novel bioreactor was developed to apply biaxial stress to tissue-engineered vascular grafts. Functionality is substandard if the grafts are cultured in a stress-free environment. Empirical models were identified that predict the compressive response of a porous scaffold used for tissue engineering. On the tissue level, work is ongoing using a rat tissue model to predict the mechanical response of arteries to the onset of pulmonary hypertension. Two models for inducing hypertension were compared. Complementary work was conducted using quantitative ultrasonic techniques. These physical acoustic techniques have also been applied to other tissue systems. Strong and diverse collaborations are growing with the University of Colorado, National Jewish Medical Research Center, Children's Hospital of Denver, and the Colorado School of Mines.

Infrastructure: The projects in this group are designed to develop measurement technology for determining a material's characteristics or for characterizing a measurement system. In FY05 we finished measuring properties of steels used in the World Trade Center as part of the NIST-led study (final reports now available on the NIST WTC web site), but continue to look at the issues that impede the use of fire-resistant steel. In addition, our mechanical testing capabilities are being applied to pipeline safety issues (in a joint project with the Metallurgy Division),with funding and technical assistance of DOT. The Charpy SRM program had over 850 customers in FY05, and we have shipped more than 1200 copies of our new training video in the past three years. A three-year international intercomparison of Charpy impact verification specimens was completed, confirming the equivalence of impact energy scales among the international producers of reference specimens.

Tom Siewert, Acting Chief

Quantitative Nanomechanical Imaging

We are developing SPM metrology for rapid, nondestructive assessment of elastic properties with nanoscale spatial resolution. Atomic force acoustic microscopy methods enable modulus measurements at either a single point or as a map of local property variations. The information obtained furthers our understanding of the nanomechanical properties of surfaces, thin films, and nanoscale structures.

Donna C. Hurley

As critical dimensions shrink well below 1 µm, new tools are required to investigate materials properties on commensurate scales. In particular, information about nanoscale mechanical properties is needed. Knowledge of properties such as elastic modulus and interfacial quality (defects, strain, adhesion, etc.) is critical to successful development of new films and nanoscale assemblies. Such information could assess integrity or reliability in applications from microelectronics to biotechnology. It is also increasingly important to assess the spatial distribution in properties rather than just the "average" properties. Existing methods for mechanical-property measurements have drawbacks: they may be destructive or limited to specialized test specimens, or cannot provide the needed spatial resolution.

To meet these needs, we are developing scanned-probe microscopy (SPM) methods to measure and image elastic properties. Atomic force acoustic microscopy (AFAM) involves the vibrational modes of the atomic force microscope (AFM) cantilever when its tip is in contact with a sample. With AFAM, the indentation modulus M of the sample can be determined. [For an isotropic material $M=E/(1-\nu^2)$, where E is Young's modulus and ν is Poisson's ratio.] The small radius of the AFM tip (~5-50 nm) means that we can obtain *in situ* elastic stiffness images with nanoscale spatial resolution.

In FY05, we combined single-point AFAM methods with SPM scanning to achieve *modulus mapping*—that is, quantitative images of elastic properties. We developed a frequency-tracking circuit to pinpoint the contact-resonance frequencies at each image pixel. A digital signal processor architecture enables rapid data acquisition (~20 min. per 256 x 256 image). Figure 1 shows an example of a modulus map calculated from resonant-frequency images. The calculations use a Hertzian model for the tip-sample contact. The values of M agree with both single-point AFAM measurements on the same sample and literature values for the constituent materials in bulk form. Work to improve the speed and performance of our methods is underway. This mapping capability greatly expands our ability to evaluate the properties of multicomponent nanostructures.

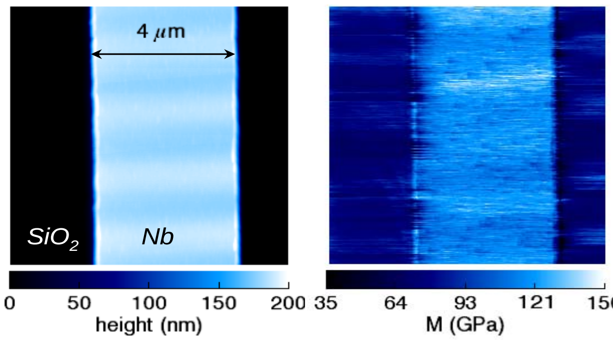

Fig. 1: *AFM topography image (L) and AFAM modulus map (R) of a thin-film sample containing a niobium (Nb) stripe ~200 nm thick on top of a silica (SiO_2) blanket film ~350 nm thick.*

Progress towards improved measurement accuracy was made in FY05. Currently, our data analysis uses an idealized (Hertzian) model, in which the tip is a perfect hemisphere. Experiments were performed in which 10 different AFM cantilevers were subjected to a series of AFAM test runs and imaged in the scanning electron microscope (SEM) between runs. In this way, the actual tip dimensions could be compared to the values inferred from the Hertzian model. The graph in Fig. 2 shows that even for cantilever beams that are virtually identical, the tips behave quite differently when in contact. From such results, we concluded that standard contact-mechanics models do not yield tip dimensions consistent with the actual (SEM) values. This result is not surprising if one realizes that the tip-sample interaction region is only about 1-5 nm in depth and 5-50 nm in diameter. Even

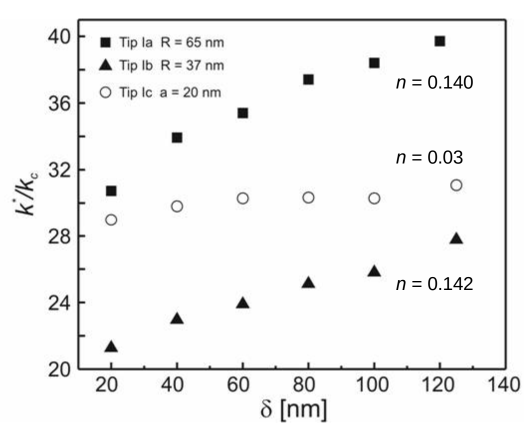

Fig. 2: *Contact stiffness k^*/k_c versus deflection δ for three different cantilevers. Shown are values for the hemispherical radius R or flat-punch radius a of each tip obtained from SEM images, as well as the exponent n used in the power-law approach (see text).*

high-resolution SEM techniques cannot sufficiently characterize the tip shape on this scale. Better agreement was obtained using a power-law approach that allowed for tip shapes intermediate between a flat punch (exponent $n = 0$) and a hemisphere ($n = 1/3$). Current AFAM techniques circumvent this issue through frequent measurements on reference samples with known elastic properties. This work represents the first step towards the development of a better contact-mechanics model for improved measurement accuracy.

Measurement accuracy is also affected by the ability to measure and control the static force applied to the tip. The force is usually determined by the stiffness of the cantilever, but this quantity may not be well known. We began work on this issue in FY05 using NIST's electronic force balance (EFB). The EFB provides SI-traceable force calibration at the nano- to micronewton scale. With the EFB, we determined the voltage-versus-force relationship for two piezoresistive cantilevers. The cantilevers will serve as force transfer standards to calibrate cantilevers of unknown stiffness used in our experiments. Experiments performed to develop a calibration methodology demonstrated the basic proof of concept and identified several issues to resolve. For instance, measurement precision is limited by drift of the output voltage over times of a few minutes. Reasons include instability in the AFM scanner and tip-sample creep or slip. This and other issues will be resolved in further experiments that are in progress.

In addition, the effects of surface energy and relative humidity (RH) on nanomechanical properties were studied using self-assembled monolayers (SAMs) of n-octyldimethylchlorosilane, whose surface energies ranged from hydrophobic to hydrophilic. Qualitative AFAM images of a micropatterned, surface-energy-gradient SAM sample acquired at different RH values (Fig. 3) showed that image contrast depended on both RH and surface energy. In quantitative AFAM point measurements (Fig. 4), the contact stiffness remained roughly constant for the hydrophobic SAM but increased monotonically for the hydrophilic SAM. To correct for this physically unrealistic behavior, a viscoelastic damping term representing capillary forces between the tip and the SAM was added to the data analysis model. The contact stiffness calculated with this revised model remained constant with RH, while the damping term increased strongly with RH for the hydrophilic SAM. Such results enhance our measurement capabilities for a range of materials and environmental conditions.

FY05 project results were described in 6 contributed and 2 invited journal articles, 5 conference presentations (3 proceedings), and 2 invited workshop presentations.

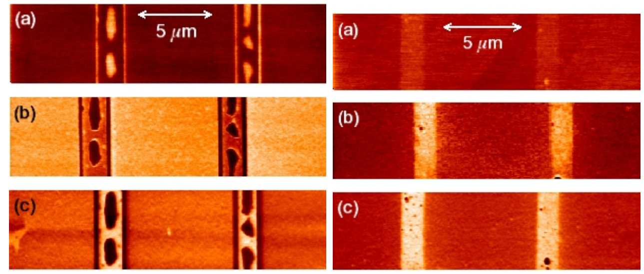

Fig. 3: *Hydrophilic (L) and hydrophobic (R) regions of a micropatterned SAM on Si sample. For each sample region, image (a) is of the sample topography (z scale 3 nm), while (b) and (c) are AFAM images acquired at 5 % and 42 % RH, respectively.*

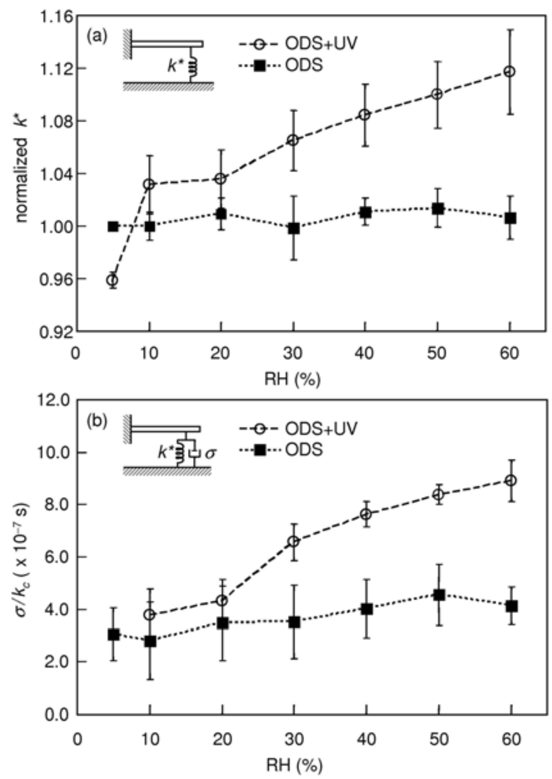

Fig. 4: *AFAM results for ODS (hydrophobic) and ODS+UV (hydrophilic) SAMs. The insets indicate the model used. (a) Apparent contact stiffness k* assuming a purely elastic interaction. (b) Damping term σ normalized by the cantilever stiffness k_c assuming an elastic-viscoelastic interaction and that k* is constant.*

Contributors and Collaborators

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Brillouin light scattering: Dynamic elastic and magnetic properties of nanostructures

Brillouin light scattering can be used to provide information on acoustic waves and spin waves at gigahertz frequencies in nanometric thin-film materials and devices. During FY05, we focused on developing the metrology and models for characterizing waves in several material systems, including nanoimprinted polymers and ferromagnetic thin films with nanoscale edge effects.

Ward Johnson, Sudook Kim, and Colm Flannery

Brillouin light scattering (BLS) is an experimental technique that measures the intensity of spectral components of light that is inelastically scattered by vibrational waves (acoustic phonons) or spin waves (magnons) in a material. Fabry-Perot interferometric techniques are used to acquire accumulated spectra through repeated mechanical sweeping of the etalon spacing.

In the Materials Reliability Division, BLS is being pursued as a technique for characterizing dynamic elastic and magnetic properties of a variety of materials and devices with nanoscale dimensions. During FY05, research on elastic waves focused on nanoimprinted polymeric lines (highlighted below), carbon nanotubes, and polymeric membranes. Research on spin waves focused on magnon-magnon interactions in $Ni_{81}Fe_{19}$, modes localized near edges of ferromagnetic thin-film structures, and modes excited in spin-momentum-transfer devices.

Research on nanoimprinted polymers was pursued in collaboration with the NIST Polymers Division, Colorado State University, and the University of Akron. The goal of this work is to develop experimental and analytical methods for characterizing elastic properties, which are expected to deviate from bulk properties when one or more dimensions of a nanoline are less than a few tens of nanometers. Figure 1 shows a typical BLS spectrum from an array of imprinted polymethyl-methacrylate (PMMA) nanolines and plots of dispersion curves from a series of spectra obtained at various scattering angles. The general character of the observed modes, except for the lowest, is determined through Fresnel-Adler calculations (black lines) for a uniform film with a thickness equal to the height of the nanolines plus the thickness of a residual PMMA layer beneath the lines. The lowest-frequency set of points arises from transverse flexural modes of the lines. The identification of these modes is based partly on the correspondence of the data with calculations of the lowest-order flexural modes (antisymmetric Lamb waves) of a plate with thickness equal to the width of the nanolines (blue curve in Fig. 1). Finite-element calculations* have been employed to provide detailed information on the vibrational displacements, such as those of the flexural mode shown in Fig. 2.

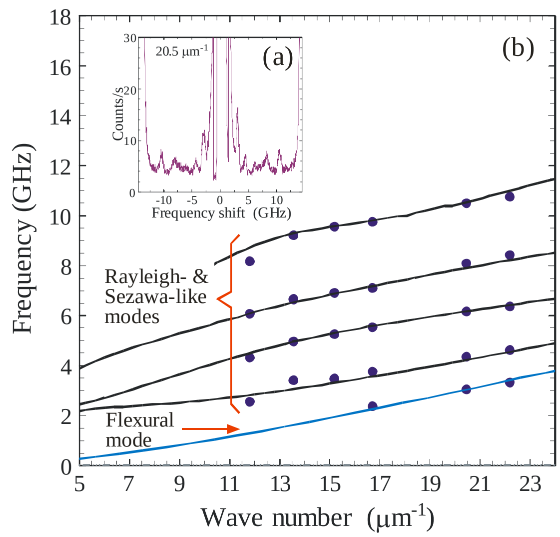

***Fig. 1 (a):** BLS spectrum for PMMA nanolines. **(b)** Measured dispersion curves (blue circles), calculations for a uniform film (black curves), and Lamb-wave calculations (blue curve).*

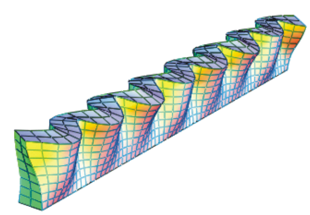

Fig. 2: Finite-element calculation of displacements of a flexural mode of a PMMA nanoline.

Contributors and Collaborators

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Mechanical Behavior of Biological Materials

There are 28 different types of tissue that perform a myriad of different functions in the human body. Virtually every type of tissue has the ability to adapt to its mechanical environment. In some cases, this adaptation is beneficial (a broken bone mends itself). In other cases, such as arterial stiffening with hypertension, such adaptation is detrimental. In still other situations, the degeneration of mechanical properties is an indication of disease, such as the loss of tone of the bladder, resulting in incontinence. Mechanical properties are even important in the spreading of viral infections, as it takes a mechanical force to unwrap the DNA of a virus for it to replicate and spread.

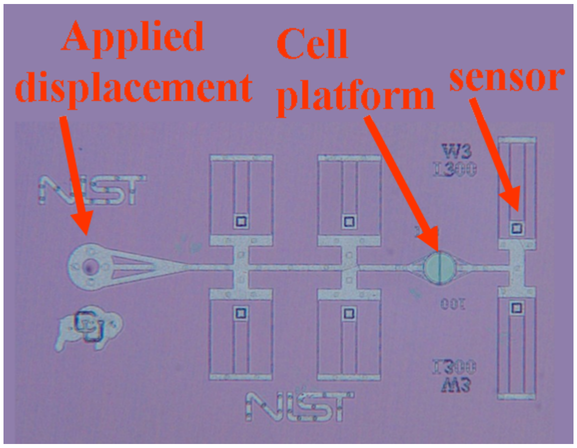
Mechanical response of cells and tissues is at the heart of many disease mechanisms, and can be exploited for diagnosis and treatments of disease. Mechanical properties also limit the efficacy of engineered tissues. Biological materials offer unique challenges to mechanical measurements, as they are functionally graded, anisotropic, nonlinear, viscoelastic, and continually adapting to their environment.

The Materials Reliability Division (MRD) is using its core competency in force and strain measurement to bring new and unique tools and procedures for biological materials. Whether the issue is measuring the mechanical properties to assess the presence of disease or the efficacy of treatments, or applying mechanical forces to facilitate understanding mechanisms of remodeling, we have the expertise to develop test platforms appropriate to the needs of medical researchers.

We have taken the established technology of the optical tweezers and have combined elements to make a versatile and load-sensitive tool. Through the use of a piezo-actuated mirror, our instrument is capable of sharing the laser to perform biaxial tensile tests with picoNewton force resolution.

Using the MEMS fabrication facility on the NIST Boulder campus, we have designed and built a series of MEMS devices specifically designed to test living cells. Our fabrication facility offers us the flexibility to use processes that are not commonly available in other facilities. Prototype bio-MEMS devices have been built for testing the adhesion and mechanical response of single cells on various protein substrates (see Figure) and for stimulating cells at different strains and frequencies to observe how changes affect cell behavior.

A bubble test system for testing membrane-like tissues has been adapted for different thicknesses of tissue, and the set-up has been refined so that test operation, image collection, and the extraction of data has been made efficient and routine. We are also working with a modeler from the University of South Carolina who has developed an analytical solution that preserves the anisotropic nature of the tissue mechanical properties.



A MEMS based load-displacment frame developed at NIST.

Mechanical stimulation can be used to improve the mechanical properties of engineered tissues, and to enhance drug delivery. We have designed bioreactors that can be used to study how best to stimulate scaffold/tissue constructs. These reactors can conduct mechanical tests without removing the sample from the reactor. Together with researchers at NIH, we are conducting experiments to identify the mechanisms that allow pulsed and focused high-intensity ultrasound to enhance drug delivery.

Contact: Timothy Quinn

Response of Tissues and Tissue Engineered Constructs to Mechanical Stimulation

Mechanical stimulation of tissue as it is being grown in a bioreactor has been shown to make tissue engineered constructs more like healthy, natural tissue. However, little effort has gone into studying exactly how the stimulation should be done[1]. Mechanical stimulation can also be used for enhanced drug delivery. Researchers at the National Institutes of Health have been developing an ultrasonic system to enhance drug delivery in tumors that are not easily treated with chemotherapy. We are developing instrumentation and models to optimize these methods.

Timothy P. Quinn, Tammy L. Oreskovic, and Brian E. O'Neill

Mechanical Stimulation of Tissue Engineered Constructs

A bioreactor that can provide both mechanical stimulation to a tissue engineered (TE) construct and mechanical testing while the tissue is being grown has been developed. The reactor was designed to facilitate studies to determine the optimal variables for growing TE constructs for vascular grafts. In this bioreactor, we can stimulate a planar sheet of tissue/scaffold construct with an arbitrary waveform. The reactor can be configured to apply a given force or a given displacement. It is equipped with actuators, load cells, and viewports to conduct online biaxial stress-strain tests without removing the sample from the reactor.

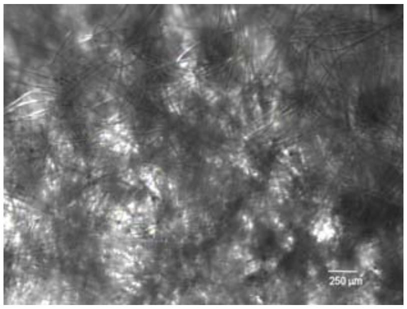


***Fig. 1**: The biodegradable scaffold with tissue ingrowth taken during a stress-strain test. Image correlation is used to measure strain.*

Human coronary smooth muscle cells (passages 7–9) were obtained from a commercial source and used in the experiments. The coronary smooth muscle cells propagated on the scaffold and were seen without difficulty by day 7 (Figure 1). No evidence of contamination was noted in the reactor at any time in the verification testing. Stress-strain testing was also accomplished during the trials. An example of the utility of online monitoring was evidence of damage to the scaffold in the form of fiber de-adhesion at a strain of 10%.

Mechanisms for the Increased Bioavailability of Materials Using High Intensity Focused Ultrasound

At low powers, the mechanical stimulation of high intensity focused ultrasound (HIFU) can be used to increase the permeability of tissues and therefore could be used to increase the effectiveness of drugs delivered to treated tissues. At these power levels, the tissue is not permanently damaged but can heal and return to its original pre-HIFU state.

In order to optimize the HIFU treatment, we need to know the mechanism by which the tissue is made more permeable. To understand this, a simple model of the sound propagation is being developed together with a parameterized model of the openings ("cracks") between cells and between cells and the extracellular matrix. Concepts from the field of damage mechanics (usually reserved for materials such as metals) are being used to develop this model.

Supporting experiments using bovine cardiac tissue are being conducted to identify the unknown parameters of the models and to identify the mechanical mechanism that leads to the increased permeability. This newly implemented system relies on confocal therapeutic and imaging ultrasonic elements, packaged together commercially. A gated function generator and power amplifier is used to drive the outer, therapeutic HIFU element in pulsed mode at 1 MHz. An ultrasonic pulser/receiver simultaneously drives the inner 10 MHz imaging element and collects data for processing. The system has a number of unique capabilities for biological application. The imaging element is used to monitor the radiation force, which has been proposed as the main mechanism behind the effect. Following designs used in the literature, we have constructed a device to measure the permeability of the tissue samples.

[1] Martin *et al*., Ann Biomed Eng, (27), p. 656, 1999

Contributors and Collaborators

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Mechanical Behavior of Tissue

Measurements of the structure and mechanical properties of biological materials elucidate mechanisms of disease and permit quality assessment of tissue-engineered constructs. Certain diseases may be identified by changes in the mechanical properties of the affected tissue before loss of function is detected, thus enabling earlier diagnoses and intervention. Our objective is to provide support to clinicians by developing techniques to measure and by measuring the properties of tissue.

Elizabeth Drexler

Research on the mechanical properties at the tissue level has focused on the properties of pulmonary arteries with the onset of pulmonary hypertension (PHT). The onset of PHT is known to cause remodeling of the walls of the pulmonary arterial system, and, if left untreated, can lead to right side heart failure. We have tested the left and right main arteries, and the trunk (the arteries most accessible for clinical diagnostics) in the direction of blood flow and in the circumferential orientations. The goal is to determine the onset of pulmonary hypertension by correlating our measured mechanical properties to the output of tissue Doppler and ultrasound, techniques currently being developed as diagnostic tools and to develop structure-property relations.

Using a rat model as a first approximation to the behavior of human arteries, we have measured the stress-strain properties of pulmonary arteries from four different populations: control, hypoxia-treated, monocrotaline-treated, and hypoxia-treated genetically modified to disable a receptor responsible for activating vasodilators. Figure 1 shows the stress-strain behavior of the treated populations compared with the controls. The difference between the two hypoxic populations and the controls is readily obvious. However, the monocrotaline-treated data indicate that the arteries have not remodeled similarly to those of the hypoxic rats, and an entirely different mechanism, other that arterial wall remodeling, may be operating.

Complementary work is underway on the histology and quantitative ultrasonic properties of the pulmonary arteries. The reduction in elastic fiber, which accompanies an increase in thickness in the hypoxic samples, is consistent with the change in mechanical properties typically associated with hypertension, where the arteries become less compliant. The increased thickness of the medial layer is due mostly to thicker muscular layers between elastic lamellae. At constant pressures, increases in thickness and reductions in elastin content contribute to a stiffer response. We have used quantitative ultrasonic characterization to correlate mechanical properties due to remodeling to the ultrasonic properties of the unstressed remodeled tissue. Ultrasonics is also used to predict fracture risk in osteoporitic patients. Dispersion (or how the speed of sound changes with frequency) appears to be sensitive to changes in bone mineral density. We organized a topical meeting on ultrasonic characterization of trabecular and cortical bone that was sponsored by the Acoustical Society of America.

The results comparing the arteries from the hypoxia-treated rats to those of the controls, along with this histology were presented at the ASME Summer Bioengineering Conference in Vail, CO in June. The comparison between the monocrotaline and hypoxic to the controls were presented at the Biomedical Engineering Society Annual Fall Meeting in Baltimore, MD in September. A manuscript on the technique and the control results was submitted to the Journal of Biomechanics. Three papers have been published on the topic of improving quantitative ultrasonics for tissue characterization, and a NIST IR is now available summarizing the Inaugural Workshop on Computational Tools for Modeling Acoustic Propagation in Real-World Materials. Additional presentations have been made at the IEEE International Ultrasonics Symposium in 2004 and the 148th meeting of the Acoustical Society of America.

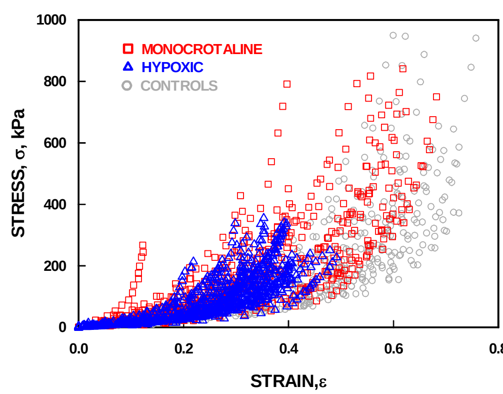

Fig. 1: *Stress-strain behavior of the control, hypoxic, and monocrotaline-treated rat pulmonary arteries.*

Contributors and Collaborators

C. McCowan, K. Waters, A. Slifka, T. Quinn (Materials Reliability Division, NIST); R. Shandas, D. Ivy, C. Cool, and K. Colvin (University of Colorado Health Sciences Center).

Cellular Level Measurements

Techniques and tools that facilitate the exposure of single cells (and arrays) to controlled mechanical environments and quantification of mechanical forces, and at the same time allow for the characterization of other biological phenomena, are needed for the study of tissues and cells. The development and evaluation of one of these tools, a bio-MEMS cell puller, is the focus of this year's effort.

Andrew J. Slifka

Research on the mechanical response of biological materials at the cellular and sub-cellular levels is being done with the development of a number of tools. Optical trapping for cellular mechanics and small-force materials science is one. We use an optical trap with a scanned laser to trap multiple dielectric spheres. These spheres can be attached to a cell. We plan to use four balls to perform a biaxial mechanical measurement of a single cell. We are developing data analysis routines to allow measurement of transient mechanical response. The optical trap is also being used to study attractive forces by measuring those forces required to pull apart two-dimensional islands of self-assembling nanoparticles.

Another tool uses bio-MEMS for cell pulling and adhesion. Both adhesion and the mechanical response of a cell to varying mechanical environments are fundamental to understanding cell motility and numerous disease mechanisms. A device that has a transparent platform on to which a single cell can adhere has been designed and built. The focal adhesions of the cell can be viewed by use of reflection interference microscopy or appropriate staining. The platform is split so that the cell can be strained from one side and forces measured on the other by way of thin cantilever beams (see Fig. 1). Image analysis can yield strain as well as the cross-sectional size of the cell so that stress can be determined from the force information. Therefore, force- or stress-strain response of a single cell can be determined in addition to force as a function of adhesion area. The device and instrumentation can also be operated in a cyclic mode, which can be used to determine the change in mechanical response of the cell as a function of cyclic fatigue. The design of this device and results on vascular smooth muscle cells were presented at the Biomedical Engineering Society Annual Fall Meeting in Baltimore, MD in September.

The last tool is development of a bio-MEMS device for quartz-crystal microbalance (QCM)-type measurements. The QCM is used extensively in the medical research field for characterizing antibody systems. It is a macroscale device with high sensitivity due to its oscillating nature. We believe that a bio-MEMS device that works similarly could yield a thousand-fold increase in sensitivity and have as wide an application as the QCM. We have made measurements on a model antibody system as a baseline and have designed and built a bio-MEMS device that mimics the QCM. We have developed a model of the device response in order to optimize future designs for particular applications. A manuscript on the device design and model has been written for the *Journal of Applied Physics.* We are pursuing simple methods for electrical measurement of the change in oscillator response, which is the heart of the QCM measurement technique.

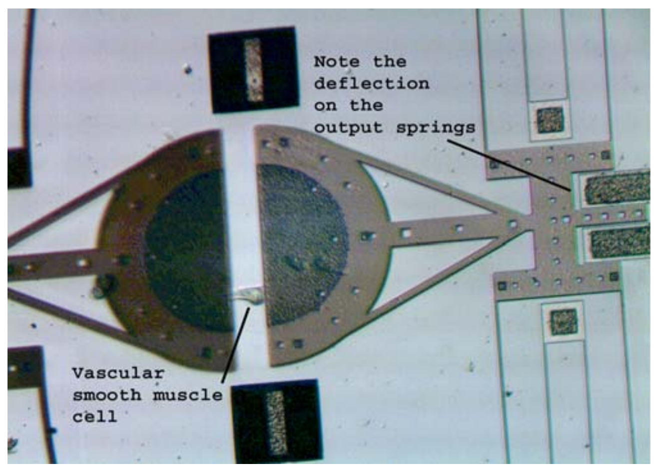

Fig. 1: *Detail of the cell platform of the bio-MEMS device used for mechanical and adhesion measurements, showing a cell being stretched.*

Contributors and Collaborators

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Materials for Electronics

The U.S. electronics industry faces strong international competition in the manufacture of smaller, faster, more functional, and more reliable products. The continual advances in this industry are due in part to the continuing development and application of new reliable materials that are capable of providing ever-increasing performance. A persistent, critical challenge to further progress includes improving capabilities for the measurement and prediction of the performance, stability, and reliability of extremely fine-scale structures used in electronics.

The Materials Reliability Division (MRD) of the NIST Materials Science and Engineering Laboratory (MSEL) works closely with U.S. industry, with a focus on the semiconductor manufacturing sector. We also have efforts addressing complementary emerging areas such as optoelectronics.

MSEL has a multidivisional approach, committed to addressing the most critical materials measurement and standards issues for electronic materials. MRD addresses several of the Lab's key objectives:

- Develop and deliver standard measurements and data;
- Develop advanced measurement methods needed by industry to address new problems that arise with the development of new materials;
- Develop and apply *in situ* as well as real-time, factory floor measurements, for materials and devices having micrometer- to nanometer-scale dimensions;
- Provide the fundamental understanding of the divergence of thin film and nanoscale material properties from their bulk values;
- Provide the fundamental understanding of materials needed for future nanoelectronic devices, including first principles modeling of such materials.

The MRD program consists of projects that are conducted in collaboration with partners from individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs in the government such as the National Semiconductor Metrology Program (NSMP). Metrology needs are also identified through the International Technology Roadmap for Semiconductors (ITRS).

MRD researchers have made substantial contributions to meeting the most pressing technical challenges facing industry, from development of novel measurement methods, to assessment of mechanical response of the dimensionally-constrained materials ubiquitous to this industry, to insight into deformation phenomena that limit reliability. Following are examples of MRD contributions over the past year.

Electrical Methods for Mechanical Testing of Thin Films and Interconnects. The continual decrease in feature size has been the driving force for advances in the semiconductor industry. Current structures have 90 nm dimensions with planned nodes at 65 nm and 45 nm structures in 2007 and 2010. Advanced measurement methods suitable for evaluating mechanical properties and reliability under conditions involving severe electrical and/or thermal stressing are under development. Such methods are applicable even to extremely fine structures, which may be buried, as long as electrical access is possible. The accuracy of this novel technique is insured by: (a) comparison to established techniques, particularly microtensile testing and instrumented indentation; and (b) use of powerful analysis techniques like TEM (transmission electron microscopy) to verify the physical failure mechanisms.

Multiscale Modeling of Quantum Dots in Semiconductors. A classical mechanical modeling approach that relates physical processes at the interatomic level to measurable lattice distortions at the nanometer level and to mechanical stresses and strains at the macroscopic level is under development. Molecular dynamics, lattice statics Green's functions, and continuum Green's functions are seamlessly linked to predict the mechanical response of nanometer-scale structures that may form the basis for future nanoelectronic devices.

Contact: Robert R. Keller

Electrical Methods for Mechanical Testing of Thin Films and Interconnects

The International Technology Roadmap for Semiconductors calls for solutions to the near-term challenge (through 2009) of achieving necessary reliability because "new materials, structures, and processes create new chip reliability (electrical, thermal, and mechanical) exposure. Detecting, testing, modeling and control of failure mechanisms will be key." To this end, we develop test and detection methodologies for mechanical reliability of dimensionally constrained materials, and further materials science understanding of the observed mechanical behavior.

Robert R. Keller, Nicholas Barbosa III, Roy H. Geiss, David T. Read, Andrew J. Slifka

During 2005 we have consolidated three distinct projects addressing different aspects of the reliability of materials for microelectronics into one division program. The present focus is on electrical methods for measuring thermal fatigue lifetime and mechanical strength of patterned metal films. The methods are based on the principle of applying joule heating to specimens in a controlled manner, by use of low frequency, high current density a.c. electrical signals. Thermal expansion mismatch between film and substrate then leads to thermal strains, which can be used as the basis for mechanical testing.

A key aspect of developing time-varying electrical methods for mechanical metrology is knowledge of the temperature of the specimen with sufficient spatial and temporal resolutions. This year a key advance was made in the first known demonstration of time- and spatially resolved measurements of joule heating in metal interconnects during application of alternating currents. The measurement is made with an atomic force microscope (AFM) that uses a Pt-Rh alloy probe tip as a resistive element in a Wheatstone bridge circuit. Figure 1(a) shows a thermal AFM image depicting temperature differences measured at the surface of a non-passivated Al interconnect carrying an rms current density of 7 MA/cm^2. A rapid decrease in temperature with distance away from the interconnect is apparent from the thermal color scale. Figure 1(b) shows the temperature variation at one position of the AFM probe, over 0.2 s, for a 10 Hz current; the oscillation is approximately 15 °C. These measurements are used to estimate temperature on a local scale, for evaluating very small structures, and for characterizing the development of local hot spots. They are also being correlated to electrical resistance measurements of global interconnect temperature.

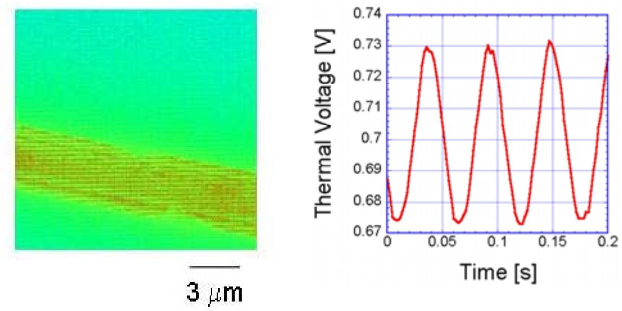

Fig. 1: *Scanning thermal microscope results from Al interconnect undergoing a.c. stressing at 10 Hz, 7 MA/cm^2: (a) thermal map showing decrease of temperature with distance away from line; (b) time-resolved measurement showing 15 °C temperature oscillation with stationary probe tip.*

We have made additional progress in documenting the damage processes that can lead to failure during thermal fatigue of patterned interconnects, using Al-1Si as a model material. Figure 2 shows an inverse pole figure (IPF) indicating surface normal grain orientations before and after 1.4 x 10^5 temperature cycles induced by a.c. stressing. Blue dots represent orientations prior to stressing, and red dots represent orientations of severely deformed regions after stressing. Black dots along IPF edges indicate 10° increments. Reorientation is consistent with a recent analysis of slip asymmetry during cyclic deformation of bulk face-centered cubic crystals. The analysis showed that for the case of fully reversed loading, a single crystal reorients such that during the tensile half-cycle, the loading axis rotates toward the primary slip direction, [011], and during the compressive half-cycle, it rotates toward the normal to the primary slip plane, $(1\bar{1}1)$. The net result of this ratcheting action is an ultimate crystal orientation near (113).

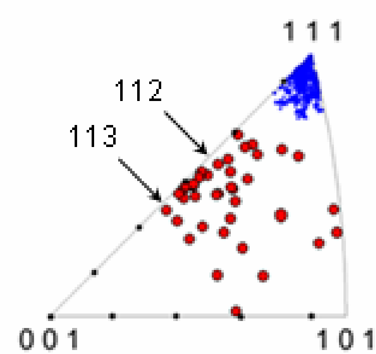

Fig. 2: *Inverse pole figure showing surface normal orientations before and after a.c. stressing of Al-1Si at 12.2 MA/cm^2 for 1.4 x 10^5 temperature cycles.*

Development of methods to electrically measure strengths of thin films is now underway this year. Measurement of yield strength is based on detection of the onset of plasticity by use of changes in residual resistance after accumulated cyclic damage. While this particular measurement method is still in its infancy, we offer here a qualitative description of the principles of the approach.

Plasticity detection involves periodic, intermittent monitoring of d.c. resistance at low current density, during a slowly increasing ramp of a.c. current density. After an initial d.c. resistance measurement prior to any stressing, alternating current is then applied for a predetermined time, during which behavior is nominally elastic. The a.c. current is then interrupted, the sample is allowed to cool, and another low current d.c. measurement is taken. Stressing resumes with a higher level of a.c. current density, followed by another cycle of cooling and d.c. measurement, and so on. Eventually, an a.c. current density is reached where there is a measurable change in the residual d.c. resistance, as compared to the initial state. We hypothesize that such a change is induced by permanent damage to the test specimen, caused by a.c. stressing at or near the yield point. Such damage could take the form of either the introduction of a significant density of lattice defects such as dislocations, or the onset of a local cross-sectional area change due to deformation. Additional studies involving microtensile testing and transmission electron microscopy are planned, in order to clarify the issue.

Results from our work appeared this year in two archival journal articles, and we target three more journal submissions by January 2006. One paper written with German colleagues (R. Mönig, R. R. Keller, C. A. Volkert, "Thermal Fatigue Testing of Thin Metal Films," *Rev. Sci. Instrum.* **75**, 4997-5004 (2004)) represents the first archival journal publication of this work, and describes in detail the test methodology.

We also completed the final report of our August 2004 workshop on nanomaterials (R. R. Keller, D. T. Read, and R. Mahajan, "Report of the Workshop on Reliability Issues in Nanomaterials," *NIST-SP 1043*; available from the Materials Reliability Division website: http://www.boulder.nist.gov/div853/RIN/report.htm), which was reviewed by nine non-NIST plenary speakers. This workshop established us as being among the recognized leaders in the field of reliability of nanomaterials.

Eight presentations of this project were given at six conferences (MRS, ASME, Characterization and Metrology for ULSI Technology, GOMACTech, Mechanics and Materials, Advanced Metallization Conference).

Contributors and Collaborators

Y. Cheng (NIST/Protiro, Inc.); R. Mönig (MIT); C. Volkert (Forschungszentrum Karlsruhe); B. Sun (Intel).

Multiscale Modeling of Quantum Dots in Semiconductors

A computationally efficient multiscale model is developed for a quantum dot in a semiconductor. The model links the subnano, nano, and macro length scales by integrating the powerful techniques of molecular dynamics, lattice-statics, and continuum Green's functions. The model is applied to Ge quantum dots of realistic sizes up to 7 nm in Si. The topography of a free surface of Si containing a buried quantum dot is calculated and can be measured and used to characterize the QD. The model can also predict the formation of arrays of quantum dots and can be a useful tool for strain engineering of quantum dots.

Vinod Tewary and David Read

Technical description

Currently there is a strong interest in modeling the mechanical characteristics of quantum dots (QDs) in semiconductors because of their potential application in powerful new devices such as huge memory systems, ultralow-threshold lasers, and quantum computers. A QD has to be modeled at the following scales: (i) the core region (subnanometer), where the nonlinear effects may be significant, (ii) the region of the host solid around the QD (nanometer), and (iii) free surfaces and interfaces in the host solid (macro). Modeling is needed for interpreting measurements and design of new devices. A multiscale model is especially useful for strain engineering of QDs and their arrays.

A QD causes lattice distortion in the host solid, which manifests itself as strain and displacement fields throughout the solid. Strain and displacement fields at a free surface can be measured and used to characterize the QD. Strain field determines the elastic energy of the system and is mainly responsible for the formation of arrays of QDs. The strain and displacement field are essentially continuum-model parameters, whereas the lattice distortions are discrete variables that must be calculated by use of a discrete lattice theory. Hence one needs a multiscale model that relates the discrete lattice distortions at the microscopic scale to a macroscopic parameter such as strain.

We have developed a computationally efficient multiscale model that links the length scales from sub-nano to macro and can be used on an ordinary desktop computer. The model integrates classical molecular dynamics (MD) with Green's functions. We use molecular dynamics at the core of the QD to account for the nonlinear effects and the lattice-statics Green's function **G** near the quantum dots, which reduces asymptotically to the continuum Green's function near a free surface. The displacement field in this model containing N atoms is given by

$$\mathbf{u}(\mathbf{l}) = (1/N)\,\Sigma_k \mathbf{G}(\mathbf{k})\,\mathbf{F}(\mathbf{k})\,\exp(i\mathbf{k}.\mathbf{l}),$$

where **l** is a lattice site, **k** is a reciprocal space vector, and **F**(**k**) is the Kanzaki force, which is calculated by using MD. For large l, the above equation reduces asymptotically to description of a macroscopic continuum, while the discrete lattice effects are retained in **F**(**k**). Thus, our model is truly multiscale, as it (a) seamlessly links the discrete atomistic effects in **F**(**k**) to macroscopic scales through the GF, and (b) directly relates microscopic lattice distortion at the nanoscale to measurable macroscale parameters.

Accomplishments

We are now able to model QDs of realistic sizes, up to about 7 nanometers, on a desktop computer. To model such a large QD, it is necessary to include at least a million atoms in the host lattice. An attempt to model such a large system using only MD would need huge computational effort and will involve somewhat arbitrary assumptions for relating discrete lattice distortion to continuum parameters. We have calculated surface strains and surface topography (Figure 1), which can be measured and used to characterize the QDs. Note the local minimum at (0,0). A similar minimum occurs in the strain energy for certain QDs. The position of the minimum is a possible favorable location for the nucleation of a new QD.

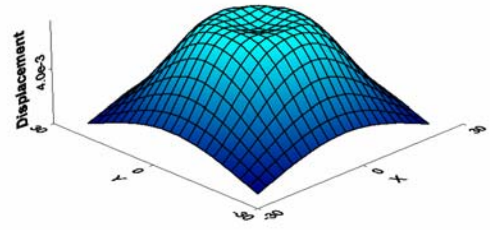

Fig. 1: *Ttopography of a free surface in Si due to a buried 1.1 nm Ge QD in Si*

Contributors and Collaborators

R.R. Keller (Materials Reliability Division, NIST); Bo Yang (Florida Tech); R. Pandey (Michigan Tech Univ).

Safety and Reliability

We take for granted that the physical infrastructure around us will perform day in and day out with consistent reliability. Yet, failures occur when these structures degrade to where they no longer sustain their design loads, or when they experience loads outside their original design considerations. In addition, we have become increasingly aware of our vulnerability to intentional attacks. The Safety and Reliability Program within MRD was created to develop measurement technology to clarify the behavior of materials under extreme and unexpected loadings, to assess integrity and remaining life, and to disseminate guidance and tools to assess and reduce future vulnerabilities.

Project selection is guided by identification and assessment of the particular vulnerabilities within our materials-based infrastructure, and focusing on those issues that would benefit strongly by improved measurements, standards, and materials data. This year we have contributed to the MSEL work with the Department of Homeland Security and the Office of Science and Technology Policy in developing the National Critical Infrastructure R & D Plan, which will provide guidance across much of the national infrastructure. Ultimately, our goal is to moderate the effects of acts of terrorism, natural disasters, or other emergencies, all through improved use of materials.

Our vision is to be the key resource within the Federal Government for materials metrology development as realized through the following objectives:

- Develop advanced measurement methods needed by industry to address reliability problems that arise with the development of new materials;
- Develop and deliver standard measurements and data;
- Identify and address vulnerabilities and needed improvements in U.S. infrastructure; and
- Support other agency needs for materials expertise.

This program responds both to customer requests (primarily other government agencies) and to the Department of Commerce 2005 Strategic Goal of “providing the information and framework to enable the economy to operate efficiently and equitably.” For example, engineering design can produce safe and reliable structures only when the property data for the materials are available and accurate. Equally importantly, manufacturers and their suppliers need to agree on how material properties should be measured.

The Safety and Reliability Program works toward solutions to measurement problems on a variety of scales, most of them large. The scope of activities includes the development and innovative use of state-of-the-art measurement systems; leadership in the development of standardized test procedures and traceability protocols; development of an understanding of materials in novel conditions; and development and certification of Standard Reference Materials (SRMs). Many of the tests involve extreme conditions, such as high rates of loading or high temperatures. These extreme conditions often produce physical and mechanical properties that differ significantly from handbook values for their bulk properties under traditional conditions. These objectives will be realized through innovative materials property measurement and modeling.

The MRD Safety and Reliability Program is also contributing to the development of test method standards through committee leadership roles in standards development organizations such as the American Society for Testing of Materials (ASTM) and the International Standards Organization (ISO). In many cases, industry also depends on measurements that can be traced to NIST Standard Reference Materials (SRMs).

In addition to the activities above, MRD provides 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, working with standards organizations to allow the use of fire-resistive steel, collaborating with both the Department of Transportation and the Department of Energy on pipeline safety and bridge integrity issues, and advising the Bureau of Reclamation on metallurgical issues involving pipelines and dams.

Contact: Tom Siewert

Analysis of Structural Steel from the World Trade Center

In 2005 NIST completed the three-year Federal Building and Fire Safety Investigation of the World Trade Center Disaster. The investigation addressed many aspects of the catastrophe, from occupant egress to factors affecting how long the WTC towers stood after being hit by the airplanes, with a goal of gaining valuable information for the future. A critical aspect of the investigation was the metallurgical analysis of the recovered structural steel. The analysis included characterization of mechanical properties, failure modes, and structural response determined from photographic evidence.

David McColskey, Chris McCowan, Tom Siewert, and Raymond Santoyo

The collapse of the World Trade Center (WTC) towers was the worst building disaster in human history. Engineers, emergency responders, and the nation were largely unprepared for such a catastrophe. The task of the NIST investigation was to determine the details of why and how the towers collapsed. As part of this investigation, the Metallurgy and Materials Reliability Divisions characterized the recovered structural steel.

The project comprised five tasks:

1. Collect and catalog physical evidence.
2. Categorize failure mechanisms from visual evidence.
3. Determine steel properties to support modeling.
4. Correlate determined and specified steel properties.
5. Analyze steel to estimate temperature extremes.

Analysis of fracture surfaces of recovered perimeter columns struck by the aircraft showed that the steel remained ductile even at high deformation rates. Data such as this were important in assuring the accuracy of the steel properties supplied to the modeling efforts.

Photographic analysis demonstrated that perimeter columns of WTC 2 began to pull into the building almost 30 minutes before collapse. Our staff developed pull-in maps based on pre-collapse photographs, Figure 1, which were instrumental in constraining the finite element models of the building deformation.

Tensile tests of the dozens of steel types and grades recovered showed that their yield and tensile strengths are consistent with the expected values. In a few cases, the strengths of the NIST-tested specimens were slightly less than called for, but the number of under-strength samples is consistent with the natural variability in steel strength and the damaged state of the recovered steel. Figure 2 shows the ratio of measured to specified yield strength for recovered perimeter column steels.

Photographic evidence indicated that 16 of the recovered perimeter column panels from WTC 1 were exposed to fire before the collapse. Our staff developed a forensic test based on paint cracking due to thermal expansion of the steel. This test placed limits on the time and temperature exposure of the recovered columns. Results indicate that only three locations on these 16 recovered columns reached temperatures above 250 °C.

The final investigation report is available on the NIST WTC web site at wtc.nist.gov

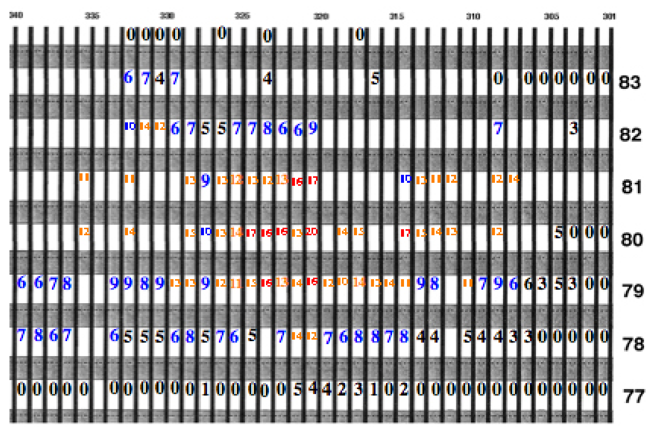

Fig. 1. *Pull-in map of perimeter columns (in inches) on the east face of WTC 2, 9:03 am.*

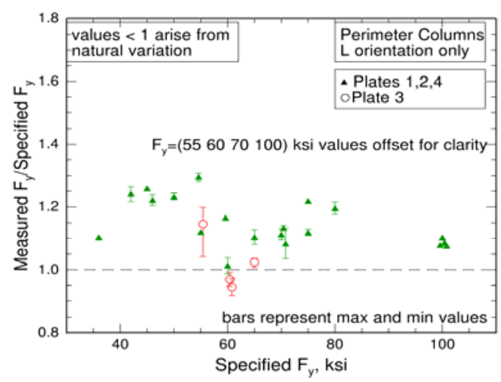

Fig. 2. *Measured yield strength of perimeter column specimens.*

Contributors and Collaborators

W.E. Luecke, S.W. Banovic, T. Foecke, R.J. Fields, F.W. Gayle, and M. Iadicola (Metallurgy)

Standard Test Methods for Fire-Resistive Steel

The fires and subsequent collapse of the World Trade Center focused attention on the vulnerability of structural steel to fire. Recently steels designated as "fire-resistive" have become available. This project is developing a standard test method for quantitatively evaluating and comparing the resistance to high-temperature deformation of structural steels.

David McColskey and Bill Luecke

All steels lose strength with increasing temperature. By 600 °C, most structural steels have lost more than half their strength. At intermediate temperatures the strength is independent of time, but above 500 °C, creep, or time-dependent deformation, further reduces the load-carrying capability. To combat this loss of load-carrying capability, structural steel in buildings is insulated to keep it cool in fire.

Fire resistive (FR) steels are intended to be drop-in replacements for existing grades of structural steel. They can meet the same specifications, have similar weldability, cost only marginally more, but retain superior elevated temperature strength. Their superior high-temperature properties have the potential to provide extra time for building occupants to escape a fire.

In Japan and Europe, FR steels are qualified based on high-temperature retained yield strength. Although this definition employs a simple, familiar test, because it is a short-term test, it ignores the time dependence of the deformation resistance. Domestic standards for structural components use a critical temperature criterion for failure of steel, so effectively all steels are identical, regardless of high-temperature deformation resistance.

We are studying three possible test methods for standardization. The first is the conventional high-temperature retained yield strength. The second is a slow-rate (several hours to failure) tension test, which should capture the time-dependent deformation effects. The third method is a hybrid of a creep and a conventional tension test in which the test specimen is held under constant load as the test temperature ramps upward linearly. Over a narrow temperature range, which can be approximated as a critical temperature, the deformation rate increases drastically and the specimen fails. This critical temperature can be used as a measure of the fire resistance.

One goal of this project is to build on recent worldwide research on similar tests, but to take the proof of concept to a draft standard. Each potential method has advantages, but there has been no research to compare the results of each method to the others. Our research focuses on understanding the limitations, repeatability, and reproducibility of the methods by characterizing several different classes of construction steels. In the near term, after selecting a single method, we will organize an interlaboratory study (ILS) to probe the limitations and establish precision and bias.

Figure 1 compares the behavior of two FR steels evaluated using high-temperature, tensile yield strength, F_y, measured in a high-temperature tensile test and the critical temperature evaluated using the temperature ramp test. Although the normalized yield strengths diverge significantly above 600 °C, the critical temperatures measured in the ramp tests are very similar.

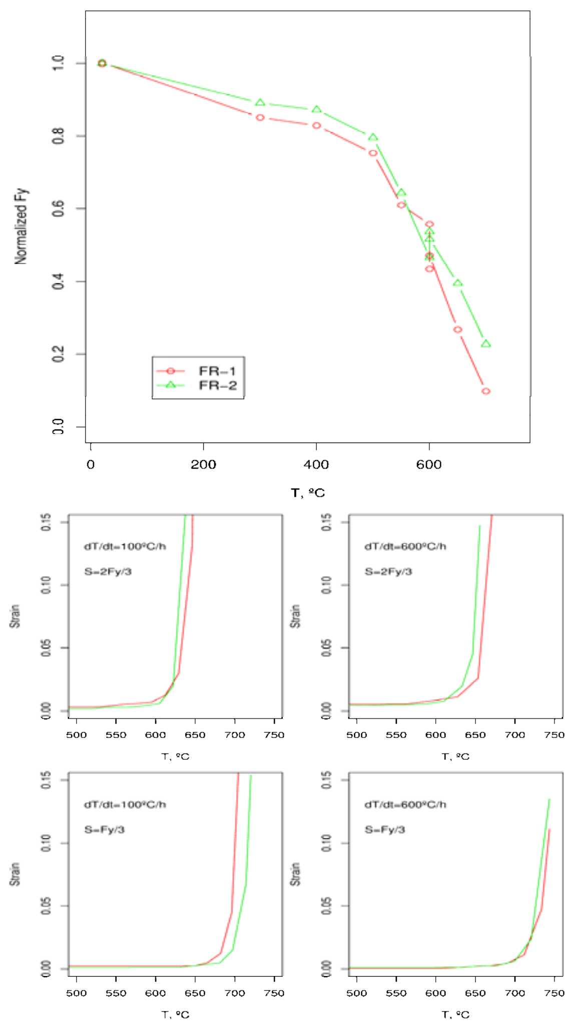

***Fig. 1**. Comparison of tensile and ramp tests.*

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.

Raymond Santoyo

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 (of the 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 three master Charpy impact machines (all 300 J to 400 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

Ray Santoyo continues in his role as Charpy Coordinator. We have served about 850 customers in the past year, a slight increase over the year before. The great majority of these machines were within tolerances required by ASTM Standard E 23. As usual, many customers took advantage of our support services, as shown by over 760 emails, 750 faxes, and 400 phone calls in the first 9 months of FY05. 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.

NIST's support of ISO Standard 17025 means that we have been reformatting our quality manual for the Charpy program to match the new NIST styles, and to fit with the overall NIST quality manual (QM-I). Thus, Chris McCowan has refined our Division quality manual (QM-II) and the Charpy program manual (QM-III) to match those of the other Laboratories and Divisions. We performed an independent audit in Fall 2005, and then formally adopted these new quality manuals.

We helped to organize another international symposium, the Second Symposium on Pendulum Impact Machines: Procedures and Specimens, held in conjunction with the November 2004 meeting of ASTM Committee E 28 in Washington, D. C. We also helped to lead the symposium, and contributed to three of the papers. Previous symposia have provided valuable insight into improvements in our program.

This year, we produced the first batch of Izod impact verification specimens, which has been tested and is undergoing statistical evaluation. It is expected that it will soon enter inventory as SRM 2115.

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.

Contributors and Collaborators

NIST participants: Ray Santoyo (Charpy Program Coordinator), John Clark, Chris McCowan, Ross Rentz, Tom Siewert, and Zack Vasicek.

External collaborators: IRMM (Europe), NRLM (Japan), Members of ASTM Subcommittee E 28.07.

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 approaches to analyze AE waveforms to solve the real-world problems of reliable identification and location of sources of AE signals.

Tom Siewert, 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 develop 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 (FEM) to predict near- and far-field displacement waves from AE sources in large and small specimens; wideband experimental AE displacement waveforms from sources in materials of interest; signal-processing techniques to accurately identify source types and their locations; and experimental characterizations of simulated AE wave propagation. The scope in FY2005 covered studies on the effects of electronic background noise on the accurate determination of AE signal arrival times through the use of modal arrivals obtained from time-frequency analysis.

Accomplishments

Previous work demonstrated, with FEM-generated signals, a technique to determine very accurate AE signal arrival times using modal arrivals. The modal arrivals correspond to Lamb-wave modes with known group velocities. The finite-element code modeled AE signals for dipole-type sources in an aluminum plate 4.7 mm thick. The plate had transverse dimensions sufficient that plate edge reflections did not superimpose on direct signal arrivals. The FEM signals were essentially noise free.

To examine how the presence of electronic noise (from the sensor/preamplifier) alters the ability to correctly determine the AE signal arrival times, detailed analysis was carried out by superimposing experimental wideband noise on the FEM-based AE signals. A wavelet transform (WT) was used to carry out the time-frequency analysis. The WTs of typical noise signals demonstrated that the WT magnitudes of the noise at each frequency vary with time in a random fashion. Hence, it was necessary to do a statistical study of the effects of noise. Fifty noise segments were prepared. The noise segments were added to the noise-free FEM signals. At each signal-to-noise (S/N) ratio, fifty FEM-plus-noise signals were used for three different source types at different source depths. The S/N ratio was calculated based on the peak signal amplitude in the assumed direction of applied stress for each signal case. Using the same successful technique for noise-free signals, the FEM-plus-noise signals were processed and the arrival times of the energetic frequencies in the signal were compared to those from the noise-free signals. To quantify the results in a practical way, the source location errors were calculated using the errors in arrival times. Figure 1 shows even at very low signal-to-noise ratios the location errors were very small for the majority of the cases.

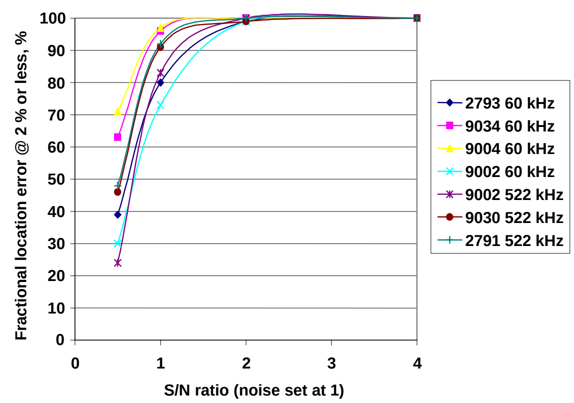

Fig. 1: Fraction (%) of the 1225 calculated locations for each case with an error of 2 % or less as a function of the S/N ratio for all cases (source type and depth). Note the numbers in the key before the frequency denote the different source and depth cases.

Contributors and Collaborators

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

Pipeline Safety: Corrosion, Fracture and Fatigue

A critical element of the nation's infrastructure is the more than 3 million km of pipelines for natural gas and hazardous liquid that provide almost 2/3 of the nation's energy. Following the passage of the Pipeline Safety Improvement Act in 2002, NIST began working with the pipeline industry, DoE, DoI's Minerals and Management Service, and DoT's Office of Pipeline Safety to provide the measurement methods, standards, and data needed to understand corrosion, fracture and fatigue failure mechanisms in this critical element of the nation's infrastructure.

Tom Siewert, David McColskey, Chris McCowan, and Ray Santoyo

After Congress passed the Pipeline Safety Improvement Act in 2002, NIST, DoE, and DoT developed a memorandum of understanding detailing a coordinated program of research, development, demonstration, and standardization. The goals of the program are to continue improvements in the safety and operation of pipelines and related facilities. NIST has a long history of contributions to pipeline safety and in this project is responsible for materials research addressing concerns with corrosion, fatigue, and fracture, especially as pipelines are pushed to higher performance by use of new materials and higher pressures.

At the urging of the Pipeline Research Council International (PRCI) and DoT's Office of Pipeline Safety (OPS), NIST reexamined data from the original NBS underground corrosion studies conducted between 1920 and 1957, involving over 36,000 samples buried at 128 different sites across North America. The original study was found to be thorough and ahead of its time with respect to statistical analysis. Fig. 1 compares the results of the power law exponent fit determined in the original study to that determined by modern computer curve fitting routines. This figure shows (i) strong evidence of nonlinear kinetics and decreasing corrosion rates and (ii) that only minor differences were obtained with modern analysis. In both original and new analyses, the scatter in the data limits the conclusions. The origin of this scatter needs to be understood before less conservative nonlinear kinetics can be used for rate models.

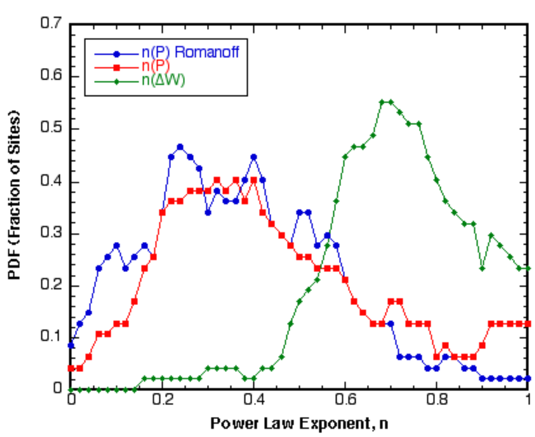

***Fig. 1**: Corrosion analysis (historical data), power law exponents.*

Fracture and fatigue studies are underway on both traditional and modern steels. By acquiring and publishing data on these properties, NIST hopes to enable better modeling of performance. The focus of this study is to assess the ability of different pipeline steels to arrest crack propagation. Crack tip opening angle (CTOA) was measured on samples cut from the pipeline samples. Fig. 2 shows the CTOA measurements on two baseline steels and the region of stable crack growth.

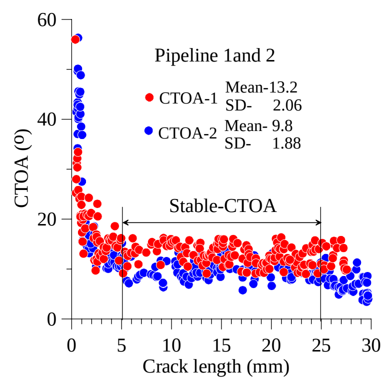

***Fig. 2:** Crack tip opening angle behavior – conventional steel.*

Working with OPS, MMS (DoI), DoE, PRCI, American Gas Association, the Gas Technology Institute, ASTM Intl., NACE Intl., CANMET, and the National Energy Board (Canada), NIST organized a workshop on Advanced Coatings for Pipelines and Related Facilities held at NIST June 9-10. Sixty representatives of the pipeline industry, suppliers, and government agencies from the US, Canada, and the UK attended this meeting. NIST also assisted OPS in organizing the Government/Industry Pipeline R&D Forum in Houston, TX March 22-24, 2005.

Contributors and Collaborators

F. Gayle, T. Foecke, S. Mates, R. J. Fields, C. Handwerker (Metallurgy Div.); J. Merritt, S. Gerard, R. Smith (DoT/OPS); M. Else (DoI/MMS).

Materials Reliability Division FY05 Publications

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