

# News Briefs

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## General Developments

*Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 820, Room 126, Gaithersburg, MD 20899-0001; telephone: 301/975-3572.*

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### **NIST SPONSORS SEMINAR ON STANDARDS INFORMATION**

On July 17-20, 1995 NIST sponsored a training seminar on standards information. The National Center for Standards and Certification Information (NCSCI) serves as a focal point in the United States for information on U.S., foreign, and international standards, technical regulations, and conformity assessment procedures in its role as the U.S. inquiry point under the WTO Agreement on Technical Barriers to Trade (TBT Agreement) and for the North American Free Trade Agreement. The Center is also the U.S. member to the ISO Information Network (ISONET).

The seminar topics included objectives and organization of a national standards information center, managing reference documents, computerized databases, sources of information and cooperation with other information centers, and responsibilities under the WTO TBT Agreement. NCSCI is a highly regarded model for these activities and served as a focus for study by seminar attendees.

Representatives from 13 Caribbean and Central American countries participated and exchanged information on their current or proposed information center activities. The seminar was presented under the auspices of the ISO Development Program, which assists developing countries in establishing their national standardization activities in all fields, including standards information, through training seminars.

### **NIST COLLABORATIVE RESEARCH PROMOTES IMPROVEMENTS IN SEMICONDUCTOR DEVICE RELIABILITY**

In collaboration with industrial and university researchers, a NIST scientist has provided new test results that integrated-circuit manufacturers can use to improve their predictions of the reliability of their products. Reliability often is determined by measuring the time to failure under extreme conditions and extending the results to predict lifetime under normal conditions. One failure mode of an integrated circuit is dielectric breakdown, in which a short-circuit current punches through a thin insulating oxide, similar to “arcing” in an electrical circuit. When the occurrence of failures is related to the duration of applied stress, the phenomenon is referred to as time-dependent dielectric breakdown (TDDB). The work showed that tests designed to accelerate dielectric breakdown in integrated circuits cannot be extended simply to normal use because there may be competing failure modes. The NIST scientist has devised a unique heated wafer chuck that permits him to carry out accelerated lifetime tests of oxides at applied electric fields representative of intended device use conditions. The resulting data have given NIST the ability to determine how TDDB depends on the applied electric field; prior workers used electric fields that were too high and deduced incorrect dependencies. A U.S. electronics company and NIST are collaborating to study TDDB in thin silicon dioxide films; the paper describing their work discusses the differences in the electric field and temperature dependencies of TDDB for oxide failure distributions that have more than one distinct failure mode. The analysis is based on data resulting from extensive characterization of oxide 20 nm thick for wafers from multiple fabrication lots.

The technical paper describing this work was chosen as the best paper at the recent IEEE International Reliability Physics Meeting.

### **NIST WORK ILLUMINATES LONG-TERM STABILITY ISSUES FOR BRAGG REFLECTION GRATINGS PRODUCED IN HYDROGEN-LOADED FIBER**

A NIST scientist and a researcher, who just received her Ph.D. from the University of Colorado through the NIST Professional Research Experience Program, have conducted a detailed study of the thermal stability of Bragg reflection gratings written in germanium-doped optical fiber. Since there is currently no theoretical model which explains this behavior, the NIST work suggests hydrogen-loaded fiber may not be a good choice for fabricating gratings if long-term stability is desired. Ultraviolet light near 240 nm induces an index change in the core of the optical fiber; this effect is used to produce Bragg reflection fiber gratings. Loading the fiber with molecular hydrogen substantially increases its photosensitivity, and fiber is commercially available in this form. Fiber gratings are important components of a variety of new devices, such as fiber lasers, dispersion compensators, and strain sensors.

In the NIST work, interference of ultraviolet light was used to induce index modulation gratings in germanium-doped optical fiber. Some gratings were kept at room temperature and others were annealed at fixed temperatures for 10 h to 20 h. For temperatures between room temperature and 350 °C, gratings in the hydrogen-loaded fiber showed significantly greater decay than those in the unloaded counterpart. For example, in one test the ultraviolet-induced index modulation in hydrogen-loaded fiber fell by 40 %, whereas it decreased by only 5 % in unloaded fiber under the same conditions. Additionally, gratings written in the hydrogen-loaded fiber which were held at room temperature for 6 months showed a decrease in index modulation of about 15 %, whereas gratings written in the unloaded fiber showed no change over the same period. The NIST annealing results show that the species responsible for the index change in the hydrogen-loaded fiber dissociate at lower temperatures than those in the unloaded fiber. The NIST work thus raises a caution with respect to using this type of fiber for Bragg gratings.

### **DISPLAY ROUND-ROBIN EXPERIMENT COMPLETED**

A round-robin exercise between NIST and a private company was requested to evaluate procedures for photometrically and colorimetrically measuring, analyzing, and reporting the capabilities of cathode-ray-tube (CRT) monitors to accurately display high-resolution, monochrome and color gray-scale imagery for both text and graphics. The experiment consisted of the private

company performing an analysis of a commercial high-resolution monitor using their standard procedures, shipping the monitor to NIST for evaluation using NIST-developed measuring instrumentation and techniques, and comparing the two sets of results. As a result of the experiment several ambiguities with the company's measurement and analysis procedures were revealed, and shifts in the monitor characteristics during shipment (particularly convergence) created measurement differences that exceeded the combined measurement uncertainties. One conclusion from the experiment was that better transfer standards are needed for this type of intercomparison procedure to be used to assess the uncertainty of the measuring instrumentation. As a result, NIST has started work on developing a more stable and robust transfer standard. A paper detailing the experiment was presented by NIST and the company at the 1995 Society for Information Display International Symposium.

### **NIST-LED TEAM SUCCEEDS IN REGISTERING IGES AS AN INTERNET MIME TYPE**

The Initial Graphics Exchange Specification (IGES) project ad-hoc team, led by NIST, successfully has registered IGES as a MIME (Multimedia Internet Mail Extension) type with the Internet Assigned Numbers Authority. One of the requirements for registering IGES was that public discussions be held on an Internet Engineering Task Force monitored mail-list and that there be a published specification for the data type being submitted. IGES registration is a significant step towards providing electronic dissemination, via the Internet, of electrical and mechanical part information. This and similar efforts will eventually allow various types of engineering data, including electronic part diagrams and two- and three-dimensional models, to be "recognized" and automatically loaded into computer-aided design and manufacturing applications. Current viewing utilities contain only image information and have no capability to represent the wide range of information which engineers use throughout the design process. The registration of IGES as a MIME type is a start towards providing the infrastructure for which industry has expressed a strong need and which would provide a user with the ability to directly access component information from any of several information providers and download the required information directly into their application. The form which documents the IGES registration and approval, can be viewed via the World Wide Web at: <http://ftp.isi.edu/in-notes/iana/assignments/media-types/application/iges>.

### **HANDBOOK BY SOFTWARE COMPANY INCORPORATES NIST WORK**

In recognition of the importance to industry of power semiconductor models developed by NIST, a software developer has incorporated in its handbook *Modeling with an Analog Hardware Description Language* a chapter discussing these models and their application. Concerns for efficient use of electrical energy in the United States are driving the development of new semiconductor devices for controlling the flow of power to components. At the same time, these concerns are emphasizing the need for design tools that permit a circuit or system designer to achieve as near optimum performance as possible. In the chapter *Electro-thermal Systems*, the authors describe both NIST's electro-thermal network simulation method and NIST's electro-thermal models.

Working in collaboration with the company, the NIST scientist developed an electro-thermal model and applied it to his insulated-gate bipolar transistor model. The company then incorporated this model into its commercial circuit simulator. The software company and another manufacturer are collaborating on the development of analog simulation models of new devices to be made available *before* devices are actually fabricated. This new approach to the design of semiconductor devices and the systems in which they are incorporated was made possible by the NIST scientist's work.

### **NIST/SEMATECH CIM APPLICATION FRAMEWORK REPORT PUBLISHED**

A NIST report to SEMATECH describes a joint effort by NIST to assist SEMATECH in defining its approach to standardization, generalization, and certification of the SEMATECH CIM Framework. SEMATECH is an industry-government consortium designed to help develop pre-competitive technologies for the U.S. semiconductor industry. One of the key areas addressed by the report is a semiconductor-domain-specific example of the more general problem of how to test object-oriented software to ensure its conformance or compatibility with the software specification (the framework). No one knows how to fully implement such a conformance testing scheme. This is becoming a significant industry problem, and a solution can have wide application beyond the semiconductor industry, as software development is moved from traditional procedural-based techniques to newer, more efficient, object-oriented coding techniques. Other topics discussed in the report include: the importance of developing a reference implementation of the framework, the high cost of certification programs and recommendations to help minimize their cost, the need to have suppliers start

developing framework-compliant programs, and the need to expand the formal description techniques in the specification.

The report was recently published both as a NISTIR (5679) and a SEMATECH Technology Transfer Document (95052825A-ENG). Entitled *Roadmap for the Computer Integrated Manufacturing (CIM) Application Framework*, the report has resulted in a request by SEMATECH for additional work from NIST.

### **PRECISION ENGINEERING SEMINARS NOW OFFERED FOR RECERTIFICATION CREDITS**

The Society of Manufacturing Engineers has approved two seminars offered by NIST for recertification credits for MECI Certified Manufacturing Engineers (CmfgE) and Technologists (CmfgT). The Gage Block Seminar (18 credits) is held each fall at NIST in Gaithersburg, MD. The three-day sessions cover all aspects of the calibration of gage blocks. The Dimensional Metrology Seminar (12 credits) that is offered in conjunction with the Measurement Science Conference each January covers the basic principles of dimensional metrology and focuses on the development of uncertainty budgets.

### **ENHANCED MACHINE CONTROLLER TECHNOLOGY TRANSFER**

NIST completed the transfer of the open architecture machine tool controller which it developed to the Defense General Supply Center, DGSC, in Mechanicsburg, PA. This open architecture controller utilizes commercially available hardware and software from multiple vendors which are integrated under a common set of application programming interfaces. NISTD staff not only transferred this technology to DGSC but also trained their staff on the installation and operation of the open architecture controller. DGSC will use this technology to rebuild and retrofit machine tools used by the Defense Department.

### **ARCHITECTS' ROUNDTABLE**

Architects' Roundtable sponsored and took an active role in a two-day meeting, July 27 and 28, 1995 of leading architects from five different national projects in manufacturing information technology. The five projects represented were: National Industrial Information Infrastructure Protocols (NIIP), Technologies Enabling Agile Manufacturing (TEAM), SEMATECH Application Framework, and two projects from NIST: the Joint Control Architecture (JCA) and Systems Integration for Manufacturing Applications (SIMA). Results will include a summary of the meeting,

a collection of electronic documents from the meeting, and a list of action items for the participants. NIST is a common point for all these efforts, but this was the first opportunity for all five to meet together specifically to discuss their technical programs.

#### **50TH CALORIMETRY CONFERENCE HELD AT NIST**

The 50th Calorimetry Conference was held at NIST July 23–28, 1995. Two NIST scientists served as conference chairman and local arrangements chairman, respectively. A total of 250 participants gave 208 oral presentations and 30 poster papers.

A symposium on the History of the Calorimetry Conference was presented on the mornings of the first and last days. Ten other symposia included topics on the stability and binding of macromolecules, model systems of biological relevance, methods in biothermodynamics, thermodynamics and industry, calorimetry of nuclear materials, fire calorimetry, advances in calorimetric instrumentation, enthalpies of mixing of non-electrolytes, aqueous electrolytes, and general topics in calorimetry.

In addition, three memorial award lectures opened the technical session on the remaining days of the conference. The Huffman Memorial Lecture awarded for continued and distinguished contributions to calorimetry and thermodynamics; the Sunner Memorial Lecture awarded to a promising young calorimetrist for high quality contributions to thermodynamics; and the Christensen Memorial Lecture awarded for the innovative development and/or use of calorimetric equipment.

#### **NEW FORM OF LITHOGRAPHY USES NEUTRAL ATOM BEAMS**

A collaboration between Harvard University and NIST has resulted in the first successful demonstration of a new form of lithography using neutral atom beams. The technique is able to reach very high levels of resolution compared to conventional photolithography. For this work an ultrathin (approximately 1 nm) self-assembled monolayer resist was patterned by a beam of neutral noble gas atoms. The normally inert gas atoms were made reactive by promoting them to an excited metastable state in a dc plasma discharge before forming the beam. Either masks or lasers were used to produce spatial patterns in the metastable beam before it impacted the resist. When the metastable atoms impacted the resist, they deposited their relatively large internal energy (up to 11 eV) and locally sensitized the resist to subsequent chemical processing. Using this technique, gold microstructures have been created on silicon with edge resolution better than 0.1  $\mu\text{m}$ . It is anticipated that there will be no limitation to pushing this resolution to

almost 100 times better, into the nm regime. Details about the first experiments were published in the Sept. 1, 1995 issue of *Science*.

#### **NIST HOSTS WORKSHOP ON ADVANCES IN BIOMEDICAL APPLICATIONS**

NIST, a private company, and George Mason University recently hosted the workshop, Ultrahigh Sensitivity Quantitation Methodologies and Instrumentation for Biomedical Applications. The purpose of the workshop, which was held at NIST, was to look at biomedical applications that require ultrahigh sensitivity and the advances in instrumentation that will be required to meet these needs. The workshop attracted more than 50 participants from companies, universities, and eight Federal agencies. Although in principle the workshop addressed all trace organic compounds, the main focus of the presentations was on the need for the Human Genome Project. Presentations were given on the need for improved instrumentation in biomedical diagnostics, the specific instrumentation needs associated with mapping the human genome, and the types of work supported by NIH, DOE, and NIST. Talks were given on advances in ultra-low-level radioactivity counting techniques and results obtained with NIST support to apply low-level counting techniques to ultrasensitive ( $\leq 10^{-18}$  mole) radioimmunoassay. Reviews were given on the sensitivity limits for other diagnostic strategies including mass spectrometry, ELISA (enzyme linked immunoassay), immunoarray technology, and long-range and quantitative PCR (polymerase chain reaction). The workshop was particularly useful in summarizing the need for high sensitivity analyses (sensitivity, specificity, affordability, speed and wide applicability) and giving some sense of the status and prospects for competing diagnostic strategies.

#### **NIST MEASURES FRACTURE TOUGHNESS FOR NUCLEAR REACTOR PRESSURE VESSELS**

Because nuclear reactor pressure vessels are large and thick walled, most laboratories cannot test full size coupons. As a result, the fracture resistance of these vessels is usually calculated from measurements made on small samples with large (deep) flaws. Several theories have been developed to explain how fracture resistance scales with sample size, flaw geometry, and material properties, but these are controversial and mostly untested. NIST's unique 52 MN force testing facility enabled NIST scientists to measure the fracture toughness of full-size samples (225 mm wide, 225 mm thick and 1.2 m long) with realistic (shallow) flaws extracted from the wall of an actual pressure vessel. These results will be used to evaluate new and existing

scaling theories and improve the reliability and safety of nuclear reactors as well as any component where full size testing is difficult or expensive.

#### **NIST AND THE UNIVERSITY OF PENNSYLVANIA FORM CRADA TO STUDY BIOMOLECULAR STRUCTURE**

A Cooperative Research and Development Agreement (CRADA) between the University of Pennsylvania and NIST has been created to combine new advances in macromolecular computer modeling with state-of-the-art neutron scattering from biologically important surfaces and interfaces. This project promises to extend our understanding of cell membrane structure and dynamics for applications to biotechnology. The agreement permits NIST to build on exciting new developments that have resulted from the study of the structure of supported lipid bilayers using neutron reflectometry in pioneering experiments by NIST and NIH scientists. This work revealed that neutron scattering was an effective tool in measuring single layers of low-dimensional systems which can emulate biological cell membranes. The CRADA brings together a team comprising experts in the fields of computer modeling, biomolecular materials, and neutron scattering. The joint effort will focus on the common goal of providing critical new knowledge about the structure and dynamics of organized membranous biomaterials which can be applied to the development of biosensor, biocatalytic, and bioelectronic devices for potential use in the chemical, pharmaceutical, and electronic industries.

The modeling activity will be coordinated by a NIST theoretical physicist and an acknowledged world leader in molecular computer modeling at the University. The research team comprises experimental and theoretical workers and students from Penn and NIST. The neutron structure studies are being led by two NIST scientists. At a later stage, NIST and NIH staff with expertise in cold neutron inelastic scattering and complementary spectroscopic methods will carry out biomolecular dynamics investigations informed by computer modeling of the systems being studied.

#### **APPLICATION OF REAL-TIME IMAGING TO THE CHARACTERIZATION OF THIN FILM DEPOSITION PROCESSES**

NIST researchers have recently adapted Intensified Charge Coupled Detector (ICCD) optical imaging technology to nanosecond time-scale monitoring of ceramic thin film deposition processes. This technique has permitted real-time detection of the key intermediate species, including atoms, ions and molecules, in

addition to particulates. For the test case of pulsed laser film deposition, temporal, spatial, and spectrally resolved data have been obtained in the form of image time sequences (or ‘‘movies’’). These observations have permitted the development and verification of new film deposition transport models. Also, the demonstrated ability to monitor instances of undesirable particulate formation in real-time has led to the more efficient production of particulate-free thin films.

#### **CHEMICALLY ASSISTED MACHINING OF CERAMICS**

The ceramic industry has identified the lack of a rapid low-cost machining technology as impeding the widespread use of ceramics. Current machining technology for ceramics is slow and labor intensive. Residual surface damage on machined surfaces is the key concern, and conventional approaches such as ultra-stiff machines, ductile grinding, and others invariably add cost. In a joint program with the Department of Energy and the Oak Ridge National Laboratory, scientists at NIST have explored the use of chemical reactions under machining conditions to assist the material removal. The concept is based on the production of dangling bonds when the ceramic surface is disrupted. These bonds undergo rapid chemical reactions. The resulting reaction products are often much softer than the ceramic substrate and, therefore, easier to remove from the ceramic surface.

To date, oxygen-containing compounds have been studied as coolants. The use of these oxygen-containing compounds led to two or three times the material removal rate on silicon nitride as compared with current commercial coolants under the same conditions.

Working closely with an industrial partner, this concept is being tested on more realistic testbeds. Plans are being made to conduct a plant trial of an experimental coolant in an actual production facility.

#### **ELECTRON CHANNELING CONTRAST IMAGING OF CRYSTAL DEFECTS IN THE SCANNING ELECTRON MICROSCOPE**

Trends towards smaller-scale microelectronics components have resulted in decreased thicknesses for the thin-film materials that comprise such structures. As a result, device performance can be degraded by a smaller number of crystal defects. One issue that has seen much attention in the semiconductor industry is that of misfit dislocation formation in heteroepitaxial films on substrates or in superlattices, line defects which can drastically reduce device performance. Most research addressing misfit dislocations has involved transmission

electron microscopy, which is a powerful materials characterization technique. However, its drawbacks include complex sample preparation that may introduce artifacts.

NIST has demonstrated an alternative, nondestructive method for characterizing misfit dislocations in an epitaxial InGaAs film on GaAs, as well as other types of crystal defects in other materials. The use of a highly tilted specimen and an efficient electron detector mounted in a low take-off angle position has resulted in the formation of images containing strong electron channeling contrast in the scanning electron microscope. This contrast is due to high-energy electron scattering and provides information about local orientation changes in materials. The system has produced channeling contrast images with spatial resolutions of approximately 80 nm to 100 nm, which are sufficient for resolving small bundles of dislocations in near-surface regions. In conjunction with backscatter Kikuchi diffraction capabilities, NIST scientists now have a non-destructive, near-surface method for characterizing crystal defects and local crystallography in materials. No sample preparation artifacts are introduced in the measurements, since the technique requires no preparation beyond routine cleaning for epitaxial film systems, and only a light polish in the case of bulk metal surfaces.

#### **CEMENT TESTING LABORATORIES RECOGNIZED BY ASTM**

NIST operates laboratory inspection and proficiency sample programs which promote the quality of testing in cement and concrete laboratories. The NIST program, which was founded in 1929, is sponsored by ASTM Committees C01 on Cement and C09 on Concrete and Concrete Aggregates. It has been called “a model of industry-Federal Government cooperation.” Remarkably, of the 630 North American laboratories that participate in the CCRL Laboratory Inspection Program, 29 have participated in the program continuously since its inception 65 years ago. All 29 were recognized for their “commitment to quality testing” by the award of certificates at the June 1995 ASTM Committee Week.

#### **NIST SIGNS CRADA WITH AIChE**

NIST has signed a Cooperative Research and Development Agreement with the Process Data Exchange Institute of the American Institute of Chemical Engineers. A central focus of this cooperation is the development of data exchange standards for process engineering.

In collaboration with the NIST SIMA program element “STEP for the Process Plant Industries,” this CRADA is supporting the development of a STEP

application protocol for the conceptual process design phase of process engineering. An international team of industry experts has been established to participate in the application protocol project and the project proposal has been submitted to ISO for formal approval as a new work item for ISO TC 184/SC4.

#### **INVITED SURVEY ON OPTIMIZATION PUBLISHED**

A major survey paper entitled “Sequential Quadratic Programming” was recently published by scientists from NIST and the University of North Carolina in the journal *Acta Numerica*. *Acta Numerica*. This journal consists only of invited survey papers by respected experts in their fields and has only one issue per year; the paper appeared in the September 1995 issue.

Sequential quadratic programming (SQP) is one of the most successful methods for solving the general nonlinear optimization problems that are ubiquitous in science, engineering, business, and management. For example, such problems arise in the control and optimization of chemical and other industrial processes, optimal scheduling and routing for business applications, maximum entropy calculations, image enhancement, and structural optimization. In their survey, the authors present the basic idea of SQP and derive the fundamental relationships that must hold for the method to be effective. They then consider the modifications and enhancements that are needed to make the method into an efficient tool for solving practical problems. Various computational approaches are discussed and an analysis of the theoretical local and global convergence behavior is given.

The scientists have been significant contributors in this area for many years and have recently successfully extended SQP to solve large-scale problems.

#### **CASE TOOL TO ASSIST EVALUATION OF HIGH-INTEGRITY SOFTWARE DEVELOPED BY NIST**

NIST developed a Computer Aided Software Engineering (CASE) tool, *unravel*, that assists the evaluation of high-integrity software by using program slices to extract computations for examination. The tool can currently be used to evaluate software written in ANSI C and is designed so that other languages can be added.

Program slicing is a static analysis technique that extracts all statements relevant to the computation of a given variable. The use of program slicing to evaluate high integrity software allows a software reviewer to focus attention on one computation at a time. After identifying a variable for further investigation, the

reviewer directs unravel to compute a program slice on the variable. By speeding up the process of locating relevant code for examination, the reviewer can inspect a larger sample of the software with greater confidence that some relevant section of source code has not been missed.

NISTIR 5691, *Unravel: A CASE Tool to Assist Evaluation of High Integrity Software*, describes the new software tool. Volume 1 relates the requirements, design and evaluation of unravel; Volume 2 contains a user manual and tutorial for the software.

#### **HANDPRINT RECOGNITION RESEARCH ADVANCES**

NISTIR 5687, *Method and Evaluation of Character Stroke Preservation on Handprint Recognition*, describes a new technique for intelligent form removal and a new method for evaluating its impact on optical character recognition. The form removal technique automatically detects the dominant lines in an image and erases them while preserving as much of the overlapping character strokes as possible. This method relaxes the recognition system's dependence on rigid form design, printing, and reproduction by automatically detecting and removing some of the lines on the form. The technique was tested on a large number of randomly ordered handprinted lowercase alphabet fields. The paper also presents the statistical analysis used to evaluate the new line removal technique and reports results.

#### **CRADA GOAL: PREDICTING SPACE RADIATION EFFECTS**

Among the many variables aerospace engineers must consider when designing spacecraft systems is the effect of ionizing radiation. Devices on communication, scientific and defense satellites can fail if not adequately protected from radiation in the near-Earth environment: protons and electrons in the Van Allen belts, solar protons and cosmic rays. Engineers who design the appropriate shielding and the use of radiation-tolerant electronic devices need reliable tools to predict radiation effects. NIST and a private company, recently signed a cooperative research and development agreement to improve software for modeling space radiation effects. NIST and the company will collaborate on integrating NIST's SHIELDOSE 2 into the company's specialized software. SHIELDOSE 2 is a computer program for rapidly calculating radiation dose in spacecraft. The company had integrated the original SHIELDOSE code into a user-friendly, comprehensive package for personal computers, which includes modeling of the radiation environment for a generated orbit, radiation penetrating into the spacecraft and dose predictions. SHIELDOSE 2 includes improved algorithms and dose

conversion to an expanded list of materials, such as silicon, silicon dioxide and gallium arsenide.

#### **HEAT PUMP MODIFICATION YIELDS POWER SAVINGS**

The sudden demand for electricity caused when tens of thousands of heat pumps collectively fail to compensate for a frigid Arctic front can cause power grid managers some anxious moments. A NIST researcher's heat pump modifications could make such worries a thing of the past. The researcher incorporated a device that separates refrigerants into the typical residential air-to-air heat pumps. The redesign better enables the heat pump to exploit the properties of non-ozone-depleting zeotropic refrigerant mixtures. The NIST-modified heat pump operates on one refrigerant until the outdoor temperature drops to a certain level. Then, instead of kicking into the auxiliary heat mode, the new device changes the refrigerant to one that performs better at colder outdoor temperatures. An experimental system employing this device has reduced seasonal resistance heat requirements by up to 5 %. Additionally, the instantaneous peak energy requirement of the test dwelling was reduced by 6 % to 9 % depending on the climate region. Although potential savings from this device would be modest for individual heat pump owners, the reduction in peak load on an electric utility can be significant. The benefit of reducing the peak energy demand is magnified if the electric power plant uses a less efficient electric generation system to meet the peak demand. For more information, contact Peter Rothfleisch, B114 Building Research Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5868.

#### **NIST DOCUMENTS ITS GATT ACTIVITIES FOR 1994**

The annual report, *GATT Standards Code Activities of the National Institute of Standards and Technology 1994* (NISTIR 5697), describes the role of NIST Applications and Assistance Program (AAP) to support industry with information on technical regulations and conformity assessment procedures that might affect U.S. trade. AAP is the U.S. inquiry point in support of the General Agreement on Tariffs and Trade (or GATT), now known as the World Trade Organization Agreement on Technical Barriers to Trade (Standards Code). Examples of AAP's 1994 include the processing of 508 notifications of proposed technical regulations, responding to 409 inquiries for GATT notification information, and participating in various bilateral and multilateral standards-related trade discussions. AAP operates a GATT Hotline on proposed foreign regulations at (301) 975-4041 and a European Community Hotline with information on draft European standards at (301) 921-4164. To obtain a

copy of NISTIR 5697, send a self-addressed mailing label to AAP, A163 Building 411, NIST, Gaithersburg, MD 20899-0001. For more information, call (301) 975-4036 or send e-mail to [overman@micf.nist.gov](mailto:overman@micf.nist.gov).

### **PARTNERS LOOK AT ULTRASOUND USE IN POWER PLANTS**

A new collaboration between NIST and the Electric Power Research Institute (EPRI) is seeking more accurate measurements of water flow rates in electric power generation plants. Utility companies want improved sensor technologies in order to enhance plant productivity, boost competitiveness and reduce the cost of electricity. One technology that EPRI has identified as needing improvement is flow metering for turbine feedwater. Power plants use internal flow meters whose accuracy is adversely affected by such things as fouling and swirling flows coming from pipe elbows. A promising alternative is ultrasonic flow metering, which places a sound transmitter and receiver at specified points on the outside of a pipe. Flow rate is determined by the time it takes sound to travel between the transmitter and receiver. EPRI and NIST recently signed a new cooperative research and development agreement with a long-range goal to develop standards of practice for installing and using ultrasonic flow meters. NIST is planning to test ultrasonic meters in ideal flows, such as those found in very long straight pipes, and in non-ideal conditions near pipe elbows and reducers.

### **INSTALLATION EFFECTS DOCUMENTED IN NEW REPORT**

A recently issued report from NIST's Flow Meter Installation Effects Consortium, Laser Doppler Velocimetry Studies of the Pipeflow Produced by a Generic Header (NIST Technical Note 1409), describes how a pipe header affects flow meters installed in downstream pipes. Scientists studied the effects of a generic header, which directs a single influent stream into two effluent streams, using laser Doppler velocimetry, a light-based technique that can detect flow velocities without inserting instrumentation into the flow. The improved flow measurements can result in significant enhancements in material accountability for continuous process industries and utilities, and can help assure equity in the exchange of commodity chemicals and fuels. NIST TN 1409 is available for \$5 prepaid from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 783-3238, fax: (202) 512-2250. Order document number 003-003-03334-1.

### **FIVE GET SITE VISITS FOR HEALTH AND EDUCATION PILOTS**

Teams of quality experts are visiting two health care and three education organizations as part of pilot programs to determine whether the Malcolm Baldrige National Quality Award should be expanded to include categories for these two important sectors of the U.S. economy. Forty-six health care and 19 education organizations submitted applications. Site visits are conducted by evaluators visiting an applicant's site to verify information in the application and to clarify questions that may have come up during the application reviews last summer. No awards will be presented in 1995 in these pilot categories. However, each applicant will receive written feedback, including strengths and areas to improve, regarding its performance management system. A Baldrige Award program for health care and education could help these organizations deliver ever-improving value to patients, students, and other stakeholders—and improve overall performance and capabilities. Whether or not to proceed with a full-scale award program depends on many factors, including a successful trial, continuing support from these two sectors, and long-term funding.

### **NIST ISSUES UPDATE OF INVENTIONS CATALOG**

Since January 1995, NIST has announced six new inventions available for exclusive and non-exclusive licenses: a new process for preparing thermoelectric alloys for alternative refrigeration systems (Docket No. 93-051); a fiber optic sensor for viewing polymer mold filling and cooling (Docket No. 93-061); a superconducting integrated circuit that could be used as the first intrinsic standard of ac voltage (Docket No. 94-008); a system of joints and clamps for holding several large objects, such as telescope or laser mirrors, in precise positions relative to each other (Docket No. 93-059); an acoustic transducer that could be used to measure case depth in automotive drive shafts (Docket No. 94-004); and a process for plating mechanical parts with a wear-resistant chromium coating that is non-toxic and up to four times harder than other methods (Docket No. 95-023D).

The most recent update of the NIST Inventions Catalog includes detailed descriptions for each of these inventions. Advantages over comparable technologies and potential applications are suggested for each invention. Both the NIST Inventions Catalog and its latest update are available from Marcia Salkeld, NIST Industrial Partnerships Program, B256 Physics Building, Gaithersburg, MD 20899-0001, (301) 975-4188, fax: (301) 869-2751.

**PAPER EXAMINES MECHANICS OF ELECTRONIC PACKAGING**

An interesting aspect of current microelectronics work is the need for increasingly more precise knowledge of the mechanical properties of materials such as thin films. As electronic circuitry gets smaller, denser, and faster, semiconductor junctions run hotter. High-performance electronic interconnect structures are made of multilayer “sandwiches” of conductors and dielectrics with different coefficients of thermal expansion. Temperature changes during manufacturing, testing, and service produce thermomechanical stresses that can split the interlayers apart, as well as fatiguing and fracturing the metal conductors. To simulate these temperature changes and thermomechanical stresses, manufacturers are creating computerized models of advanced packaging structures. This modeling and simulation effort requires an understanding of material properties and failure modes. NIST is working with industry and academia to help gain this understanding. Researchers from NIST and the University of Maryland, recently used a miniature piezoelectric-activated micromechanical testing device to obtain much needed materials property data. In particular, they studied the mechanical behavior of thin films produced by electron-beam evaporation of aluminum and copper, and deposited on silicon substrates. They found that the mechanical behavior of thin films is influenced by their thickness and grain size. To obtain a copy of their paper, contact Sarabeth Harris, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237, e-mail: sarabeth@micf.nist.gov.

**TECHNICAL ACTIVITIES REPORT AVAILABLE**

Technical programs at NIST are helping U.S. industry increase efficiency, reduce pollution and improve quality. For example, NIST researchers are assessing ways to neutralize hazardous wastes, and are developing standard reference materials to improve quality in clinical laboratories. Six technical divisions within NIST provide industry with a variety of collaborative opportunities in areas such as biotechnology, analytical chemistry, chemical kinetics, thermophysics, surface science and process measurements. A report covering these divisions’ scientific research and industry connections is now available. Copies of the 359-page Technical Activities 1994 publication are available from the Chemical Science and Technology Laboratory, A317 Chemistry Building, Gaithersburg, MD 20899-0001, (301) 975-3143, fax: (301) 975-3845.

**ARMSTRONG, CORNING DIVISIONS WIN 1995 BALDRIGE AWARD**

Armstrong World Industries’ Building Products Operation (Lancaster, PA) and Corning Telecommunications Products Division (Corning, NY) were selected as winners of the 1995 Malcolm Baldrige National Quality Award in recognition of their business excellence and quality achievement. Both companies were evaluated on their achievements and improvements in seven areas: leadership, information and analysis, strategic planning, human resource development and management, process management, business results, and customer focus and satisfaction. They will present details of their quality improvement strategies and results at the Quest for Excellence conference, Feb. 4-7, 1996, at the Washington, DC, Hilton & Towers.

**MORE ACCURATE PRICING “IN STORE” FOR CONSUMERS**

A 2 year cooperative effort between NIST, state and local weights and measures officials, and more than 500 retailers may soon benefit the nation’s consumers by improving the accuracy of retail store prices. An examination procedure, developed by officials in the NIST-sponsored National Conference on Weights and Measures and representatives from industry, soon may be used by inspectors and retail store officials to conduct price verification inspections in any type of store, including those that use “Universal Product Code” scanners and price-look-up codes at the checkout counter as a means for pricing. The system was unanimously approved at the recent NCWM 80th Annual Meeting and is now ready for adoption by the states. The procedure was developed by a working group under NCWM’s Laws and Regulations Committee. Uniform laws and regulations developed by NCWM become mandatory and legally binding when adopted by the states and local governments. To order copies of Examination Procedure for Price Verification, contact NCWM, P.O. Box 4025, Gaithersburg, MD 20855, (301) 975-4093, fax: (301) 926-0647. For information, contact Tom Coleman, (301) 975-4868, e-mail: tomc@ncwm.nist.gov.

**ADVANCE MADE IN VOLTAGE STANDARDS**

NIST scientists have made advances in low-temperature superconducting electronic technology that may lead to replacement of the direct-current voltage standard with a fast programmable alternating-current voltage stan-

ard. Such a standard has application for alternating-current metrology, precision waveform synthesis, and characterization of high-precision digital-to-analog and analog-to-digital converters. The advance is based on the Josephson effect, where an alternating current at a known frequency is applied across a Josephson junction and the current-voltage curve exhibits equally spaced constant voltage steps. Scientists at NIST fabricated and tested superconductor-normal-superconductor junctions utilizing niobium-paladium and gold-niobium. These were connected in arrays of 4096, 2048, and 1024 junctions, as well as two arrays of 512 junctions (all on the same chip). The researchers also constructed a microwave circuit that enables uniform microwave power distribution so that each junction has constant voltage steps within the same bias current range. Each array showed constant voltage steps of at least 1 mA amplitude with the same applied microwave power. When all five arrays were measured in series, the power distribution was sufficiently uniform to achieve milliampere-wide steps. With only a fourfold increase in the number of junctions, NIST scientists believe it should be possible to demonstrate a programmable voltage standard with a range of plus-or-minus 1 V, 15 bit resolution, and 30 bit accuracy. This then could be used to eventually replace the direct-current voltage standard.

### **VIDEOS HELP WITH ENERGY CONSERVATION DECISIONS**

A set of three NIST instructional videos with workbooks can help users make energy conservation investment decisions. The first video in the series, entitled “Least-Cost Energy Decisions for Buildings,” is the 60-minute “Life-Cycle Costing” (\$19) that provides tools for discounting cash flows, computing life-cycle costs and evaluating energy conservation projects. The second video, the 35-minute “Uncertainty and Risk” (\$16), helps analysts establish an economic picture of project alternatives when faced with uncertain information. The set’s third video, the 35-minute “Choosing Economic Evaluation Methods” (\$16), looks at methods such as life-cycle costing and the savings-to-investment ratio for evaluating energy conservation projects. Supporting workbooks provide descriptions of the technical material, a glossary of technical terms, and exercises in applying economic methods. The videos and workbooks can be ordered from Video Transfer Inc., 5709-B Arundel Ave., Rockville, MD 20852, (301) 881-0270.

### **NVLAP PUBLISHES THREE NEW HANDBOOKS**

The National Voluntary Laboratory Accreditation Program (NVLAP) has published three new handbooks for use by laboratories accredited by NVLAP. The handbooks, which are fully compatible with ISO/IEC Guide 25, supplement NIST Handbook 150, NVLAP Procedures and General Requirements, and replace earlier versions. A total of 13 NVLAP handbooks have now been published under the new NIST Handbook 150 series.

- NIST Handbook 150-12, GOSIP—Government Open Systems Interconnection Profile, contains the requirements for accreditation of laboratories that test products for conformance to criteria set by the U.S. GOSIP Testing Program. The handbook also identifies the requirements for proficiency testing using registered Means of Testing (MOT), the specific on-site assessment criteria, and the requirements for a quality system.
- NIST Handbook 150-15, Thermal Insulation Materials, sets forth the procedures and technical requirements for NVLAP accreditation of laboratories that perform test methods covered by the Thermal Insulation Materials program. The test method selection list for this program has been updated, with both the addition and deletion of a number of test methods.
- NIST Handbook 150-16, Commercial Products Testing, pertains to NVLAP accreditation of laboratories that perform test methods covered by the Commercial Products Testing program. This program spans test procedures for paints, paper, plastics, plumbing, and seals and sealants. Over 250 test methods are offered for accreditation in the Commercial Products Testing program.

Copies of the handbooks may be obtained from NVLAP, A162, Building 411, ext. 4016.

### **ENERGY-RELATED INVENTIONS PROGRAM RECOMMENDATIONS**

The NIST Office of Technology Innovation recommended nine innovative technologies for commercialization to its Department of Energy partner under the

Energy-Related Inventions Program during July and August:

- Vibrating Counterflow Heat Exchanger for Hot Solids—a device for exchanging heat between two counterflow streams of solid particles using vibrations to fluidize the particles and a specially contoured floor to “push” the particles in the desired flow direction.
- Flow Integrating Section for a Gas Turbine Engine in Which Turbine Blades are Cooled by Full Compressor Flow—the invention is a modification to the diffuser section of gas turbines.
- Acme-Nut’s Lubricant Retaining Grooves—a design to draw lubricant between the mating surfaces of screw threads in a valve stem/body to effect hydrodynamic lubrication during opening and closing.
- Thermal Heat and Diffusion Treatment of Bulk Powders—the invention is a process and hardware design for treating powdered metals during production for use in making parts using known and proven technologies in a new arrangement.
- Totally Automated Production System—a pump-off controller for oil and gas stripper wells primarily intended for wells utilizing downhole pumps, which are operated through sucker rods (linked in a series or string) by a prime mover (electric motor) on the surface.
- Insert Drill Having Three or More Flutes and Method of Manufacture—a three-fluted insert drill for making holes in metallic workpieces.
- Seacoaster—an air-lubricated twin hull marine vessel in which the design of the underside of each hull contains a blower-powered air cushion.
- Automated Oxygenation System for Aquaculture—the invention is a novel method, based on fish behavior, of determining low oxygen concentrations in an aquaculture environment and provides for an apparatus to begin aeration automatically.
- Helical Reaction Hydraulic Turbine—a low head, cross-flow hydraulic turbine having an open rotor with two or more airfoil profile helices in a cylindrical array around the axis of rotation, normal to the stream flow direction.

**NIST MEASUREMENTS IDENTIFY CRITICAL PROBLEM, SOLUTION, FOR REMAINING U.S. MANUFACTURER OF ELECTROLUMINESCENT FLAT-PANEL DISPLAYS**

At the request of the only domestic manufacturer of electroluminescent flat-panel displays, a NIST scientist

recently investigated a high-voltage integrated circuit suspected of causing unusually high field failure rates in the driver of the company’s displays. Flat-panel displays are a critical component in notebook computers and other portable electronic systems. NIST’s measurements on the 250 V CMOS (complementary metal oxide-semiconductor) driver showed that the devices did not meet original specifications, resulting in the high failure rates. Based upon the NIST measurements, the devices were redesigned by the circuit manufacturer to improve the margin of permissible voltages and currents handled by the device (safe operating area, SOA) with respect to the in-use operating conditions. Follow-up measurements by NIST showed a 10 % to 15 % improvement in breakdown voltage on the redesigned devices.

**NIST ADVANCES TOWARD SIA ROADMAP GOAL THROUGH ACHIEVING RESOLUTION OF 30 nm IN SCANNING CAPACITANCE MICROSCOPY IMAGES**

Researchers at NIST have demonstrated the capability of scanning capacitance microscopy to image dopant distributions in semiconductor microstructures at a resolution of 30 nm. In order to continue to advance, high-technology industries are challenged with the need to examine and measure the properties of increasingly smaller structures. A large number of scanning-probe methods have come into being in response. One of the newest of these is scanning capacitance microscopy (SCM), based on an atomic-force microscope (AFM). In SCM, the probe tip is metallized and the capacitance between probe and specimen is measured; with a two-dimensional scan, images of the capacitance variations are produced. SCM can sense quantitatively variations in carrier density in a small region beneath a conducting probe. Understanding and interpreting the resulting images or the data on which they are based is a major challenge that has been met by the NIST team through software that solves for the electric potential in the involved regions: probe, ambient (air), insulating layer (such as an oxide), and semiconductor structure. In particular, the team has computed capacitance as a function of bias voltage for uniform wafers. This modeling permits the extraction of dopant densities from the measured capacitance data.

SCM offers the potential to solve a number of measurement problems in the semiconductor industry. With respect to one application, the Semiconductor Industry Association’s National Technology Roadmap for Semiconductors has tasked NIST with the goal of developing metrological tools to measure the dopant distributions in

processed semiconductor wafers with a spatial resolution of 20 nm or less. The demonstration is a major step on the way to meeting this requirement. The NIST effort is being coordinated with a commercial manufacturer of probe instruments through a cooperative research and development agreement, and interactions are occurring with other industrial and academic researchers interested in SCM, as well as with SEMATECH.

#### **NIST PROVIDES KEY INDEPENDENT TECHNICAL EVALUATION FOR U.S. ULTRA-HIGH-PURITY SILICON PROGRAM**

NIST has provided key information to determine if a commercial contractor has met the stringent technical requirements of the first phase of the Defense Production Act Title III High-Purity Float-Zone Silicon Program. The program is aimed at re-establishing a U.S. capability for producing ultrahigh-purity silicon; the need to restore this capability had been determined to be a very high priority under the provisions of the act. NIST carried out detailed analyses of silicon specimens produced by the contractor and provided supporting technical advice to program management. Purity levels of silicon crystals of interest to this program extend to sub parts-per-billion levels of dopant atoms, well beyond the demonstrated capabilities of common analytical tests specified as test methods by ASTM and other standards organizations.

The work at NIST was carried out by scientists who performed various electrical measurements on the specimen wafers. One of the scientists then carried out infrared absorption spectroscopic measurements on the same wafers at liquid-helium temperatures to confirm the purity analysis inferred from the electrical data. The NIST team also provided significant technical input that has made possible improved processing quality control for the next phases of the program.

#### **NIST REPORT ON IMPLICATIONS OF GREENHOUSE WARMING FROM MAJOR SF<sub>6</sub> USE IN ELECTRIC POWER INDUSTRY ATTRACTS WORLDWIDE INTEREST**

NIST has carried out a study of the environmental challenge posed by the increasing dependence of the electric power industry on sulfur hexafluoride (SF<sub>6</sub>) gas and reported the results in NISTIR 5685, SF<sub>6</sub> Insulation: Possible Greenhouse Problems and Solutions. This report has drawn interest from countries all over the world.

The report assessed the present situation, including current efforts aimed at providing both short-term and long-term solutions addressing the fact that SF<sub>6</sub> is a potent greenhouse gas, some 25 000 times more effective than CO<sub>2</sub>, with a lifetime estimated to be 3200 years. Recent widespread and growing use of SF<sub>6</sub> by the electric power and other industries has led to increased concerns for global warming, since the assumption is made that all SF<sub>6</sub> being produced will eventually be released into the atmosphere. The electric power industry is the largest user of SF<sub>6</sub> in the United States; worldwide, the electric power industry uses about 80 % of the total SF<sub>6</sub> production of 7000 t. Virtually all high-voltage transmission systems and all gas-insulated substations constructed within the last decade depend on SF<sub>6</sub>; these elements are a major part of the U.S. transmission and distribution system. The present atmospheric concentration of SF<sub>6</sub> is three parts per trillion, increasing at a rate of over 8 %/yr.

As a result of the concern, the Environmental Protection Agency is seeking to work with the electric power industry and organized a conference in August at which the NIST report was distributed and two related papers presented. These were Properties of SF<sub>6</sub> and Reasons for Desirability of Use, and Chemical Substitutes and Gaseous Dielectric Research. The report points out that in the short term, an emerging consensus supports taking steps such as limiting the atmospheric release of SF<sub>6</sub> in the environment, recycling SF<sub>6</sub>, and restricting the use of SF<sub>6</sub> to those applications where the benefit from its use is the greatest, e.g., in circuit breakers. The report suggests that a long-term solution is likely to involve alternative high-voltage gaseous insulants and concomitant accurate measurements and reference data to quantify their physical, chemical, and dielectric properties.

#### **NIST FABRICATES FIRST HIGH-TEMPERATURE SNS JOSEPHSON JUNCTIONS ON SILICON WAFERS**

A collaboration between NIST and a private company has successfully fabricated high-temperature superconducting (HTS) Josephson junctions on silicon wafers. This is the first report of superconductor-normal-superconductor (SNS) junctions on silicon, and the first report of any HTS junction on silicon that uses reactive-etching (RIE) to pattern the silicon.

HTS junctions on silicon are valuable in two very different ways. First, a silicon substrate has low loss from the microwave to the infrared, making it ideal for applications such as optically coupled infrared sensors

and mixers. Second, combining superconducting and semiconducting devices on the same substrate may be valuable in the rapidly advancing area of miniaturized rf components for wireless communications. Unfortunately, the growth of HTS films on silicon has proven to be very difficult. The company has pioneered techniques to grow high quality films of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  (YBCO) on silicon using epitaxial buffer layers. This expertise has been combined with the Josephson-junction capabilities at NIST to produce junctions on silicon. The fabrication process begins at NIST by etching a step into the silicon surface using a standard RIE process. The private company then deposits a thin film of YBCO on the wafer at an angle to the step, creating a break in the superconductor. A thin film of gold is deposited over the YBCO to bridge the gap in the superconductor, making the SNS device. The patterning of the device to define the final junction geometry is done by ion milling. The resulting junctions, measured at NIST, have nearly ideal characteristics for applications requiring shunted Josephson junctions. The junctions respond to microwave radiation in the predicted manner, with excellent response from a few gigahertz to 100 GHz at temperatures higher than 70 K. Work is now under way to improve this process and to incorporate these devices in demonstration circuits.

#### **NIST APPLIES EXPERTISE IN ELECTRICAL DISCHARGES TO PREPARE REPORT FOR LIGHTNING PROTECTION COMMUNITY**

At the request of the National Fire Protection Research foundation, representing the lightning protection community, NIST has prepared a background report that provides information for members of that community interested in assessing lightning protection systems based on the early streamer emission (ESE) concept and/or developing standards for protection systems. The foundation turned to NIST as an impartial organization; the resulting report, Early Streamer Emission Lightning Protection Systems—Literature Survey and Technical Evaluation, incorporates an annotated bibliography of over 300 publications that were selected as being of direct or indirect interest to ESE technology.

Based on an examination of the literature cited in the bibliography, the authors present a discussion about the state of knowledge concerning the operation and effectiveness of ESE devices that identified issues and areas of controversy. ESE devices add an ionizing source to a conventional lightning rod, or air terminal. The intent is to attract a potential lightning stroke earlier than otherwise, by increasing the effective height of the terminal and thus decreasing the effective distance between the terminal and an electrically charged storm cloud.

The report suggests that a first-principles understanding of the mechanism of lightning has never been developed, which complicates any attempt to evaluate lightning protection systems, although air terminals have been in use for many years. The authors conclude with recommendations for research to elucidate the issues.

#### **WORKSHOP ON MODELING ELECTRON BEAM SPECIMEN INTERACTIONS**

A workshop on Electron Beam/Specimen Interaction Modeling for Metrology and Microanalysis in the Scanning Electron Microscope was organized by NIST scientists as part of the program of the Microbeam Analysis Society Conference held recently in Breckenridge, CO. During this workshop, a report was presented on a round robin of microanalysis measurements and Monte Carlo simulations for samples consisting of chromium layers on a gallium arsenide substrate. The intent of this round robin is to add significantly to the sparse database of thin film measurements. This experimental database can provide a meaningful challenge to theoretical calculations, such as Monte Carlo simulations, leading to improvements in accuracy. The chromium/gallium arsenide system provides characteristic x rays with both low and high energies, including emissions from the film as well as the underlying substrate. Experiments were performed over a wide range of beam energies from 5 keV to 40 keV. Results of the experimental measurements and theoretical calculations are currently under evaluation by the participants, who included representatives from NIST; Sandia National Laboratory; a private company; University of California at Davis; SAMx-ONERA (France); Universite de Sherbrooke (Canada); A.F. Ioffe Physical-Technical Institute, St. Petersburg, Russia; and Osaka University, Japan.

#### **NEW LUMINOUS FLUX SCALE FOR THE LIGHTING INDUSTRY**

The luminous flux (lumen) is the measure of the total light output of lamps used for lighting purposes. Lighting companies produce millions of lamps every year that rely upon the NIST luminous flux scale for specifying the output and efficiency of their lamps. The NIST luminous flux scale, last realized in 1985, was based on a blackbody radiation standard and maintained via a group of primary standard lamps. The new flux scale is tied to the detector-based NIST candela scale introduced in 1992 and a new calibration method. This method uses an integrating sphere with an opening to introduce a known amount of flux from an external source, which is

compared to the luminous flux of an internal source to be calibrated. A comprehensive and detailed spatial non-uniformity correction technique accounts for sphere variances and provides the basis for the high accuracy achieved. The new NIST luminous flux scale has a relative expanded uncertainty ( $k=2$ ) of 0.5 %. The magnitude of the new NIST luminous flux unit is 1.1 % larger than that of the 1985 scale. This change reduces the difference of the NIST scale from the world mean from 1.0 % under to +0.1 % over the mean. The new NIST luminous flux scale, effective Jan. 1, 1996, is being announced to all calibration customers.

#### REFERENCE LASERS FOR LENGTH CONTROL

A universal theme in modern equipment design is the use of servo techniques to provide accurate conformity to a planned behavior. For length control, the dual frequency interferometer is well developed but requires a reference laser. Many strategies have been used to stabilize the stand-alone HeNe red laser, but even after 35 years the achievable performance versus cost is just marginal for the desired accuracy levels.

NIST scientists have developed a universal approach for reference lasers using molecular overtone absorptions that span from the infrared to beyond the green with thousands of potential reference lines. By placing the weakly absorbing gas in an optical resonator of very high finesse ( $>10\,000$ ), the absorption of the molecular gas at the parts-per-million level gives rise to measurable resonances at nearly the percent level. The high finesse cavity also magnifies the input power level by its storage effect, generating sufficient circulating power to cause absorption saturation effects, leading in turn to narrow saturated absorption resonances. This new technique involves placing two sets of FM sidebands onto the beam incident upon the sharply resonant cavity. One set of sidebands from the optical carrier is reflected from the cavity and forms the local oscillator for sensitive locking of the laser to the cavity. The other sidebands are separated from the carrier by the cavity's free spectral range and consequently pass through the cavity with phase-shifts identical to that experienced by the carrier itself. The intracavity molecular resonances produce a differential resonant phase-shift when the carrier matches the saturated absorption frequency. Given the acronym "NICE OHMS" (=Noise-Immune, Cavity-Enhanced, Optical-Heterodyne Molecular Spectroscopy), a dozen lines around 790 nm in acetylene ( $C_2H_2$ ) have been measured and molecular constants refined as a precursor to direct optical frequency measurement. The technique is applicable to inexpensive diode lasers, which can produce highly cost-efficient length references.

#### NIST RESEARCHERS HOLD TUTORIAL ON "SMALL ANGLE SCATTERING: STRUCTURE ON THE NANOMETER SCALE"

A team of six NIST researchers presented a tutorial workshop on small angle scattering as part of the 1995 Annual Meeting of the American Crystallographic Association. Entitled "Small Angle Scattering: Microstructure on the Nanometer Scale," the tutorial was designed to assist researchers from industry and academe who wish to use neutron and x-ray scattering methods to characterize microstructures in metals, ceramics, polymers, and biological materials.

The subject matter included basic theory, instrumentation, specimen preparation, data handling, and applications. NIST operates small-angle neutron and x-ray scattering facilities that are available to outside users for proprietary or collaborative studies.

#### NIST-INDUSTRY GROUP ADVANCES ELECTRONIC PACKAGING MATERIALS METROLOGY

NIST and the semiconductor and electronic interconnect industries, in collaboration with universities and other Federal agencies, have improved the measurement of out-of-plane, or  $z$ -axis, coefficient of thermal expansion ( $z$ -CTE) of polymer thin films. The effort has determined the best-case lower limit in film thickness that can be measured reliably with existing commercial equipment and has developed a new absolute method with enhanced sensitivity and accuracy for thin polymer films. More accurate measurements of CTE are needed to meet the growing requirements for improved dimensional stability of polymer thin films within the semiconductor and electronic interconnect industries. The accomplishments are part of an effort by NIST to work jointly with leading manufacturers and users of microelectronic circuits, universities, and other government agencies to improve the methods used to characterize thin film materials used in microelectronic packaging.

In the first phase of the work, measurement round-robin and statistically designed experiments were used to study the feasibility of the thermomechanical analyzer (TMA) equipment, widely used by the electronics industry, to measure the  $z$ -CTE of a 50  $\mu\text{m}$  thick polyimide film. Experiments designed to identify factors affecting TMA baseline response, calibrate results using single crystal silicon reference samples, and measure films themselves showed the TMA is not appropriate for films less than 100  $\mu\text{m}$  to 300  $\mu\text{m}$  thick. This work establishes the foundation for improved standard test methods using the TMA equipment.

In the second phase, NIST developed and built a capacitance cell that accurately measures the distance between two parallel plate electrodes. Using the cell, the

z-CTE of single crystal silicon and of polyimide films have been measured with greatly enhanced sensitivity and accuracy over the TMA technique. Key factors that allow the method to measure film thicknesses with a relative uncertainty of less than  $10^{-6}$  are: maintaining a constant electrode area over the temperature range, eliminating stray capacitance and correcting the dielectric constant of the air in the gap for pressure, temperature, and relative humidity. NIST is using this new method in conjunction with industry to examine candidate dielectric resins for high density electrical interconnect substrates.

Packaging of integrated circuits presently accounts for roughly one-third of their delivered cost and density of electronic interconnects must be increased significantly to realize the performance being delivered by semiconductors.

#### **NIST/RUSSIAN SCIENTISTS DEVELOP PROGRAM TO MEASURE MAGNETIC DOMAIN STRUCTURE**

Scientists from NIST and the Institute for Solid State Physics in Chernogolovka, Russia, are developing a dynamic technique for the characterization of magnetic domain structure for a wide range of technologically important magnetic materials. The domain structure is imaged through the interaction of polarized light with an optically anisotropic iron garnet indicator film which is placed on top of the magnetic material. Since the polarization of light in the indicator film is affected by both the applied external field and the local magnetic field in individual magnetic domains (Faraday effect), individual domains can be identified through local changes in polarization of the indicator film using a polarizing light microscope. As part of this U.S.-Russian program, the technique is being used to study thin magnetic multilayers displaying the giant magnetoresistance effect and other types of magnetic recording media.

#### **SUMMER SCHOOL ON NEUTRON SCATTERING**

Researchers from academia, industry, and government laboratories attended NIST's Cold Neutron Research Facility (CNRF) first summer school on neutron scattering, held at NIST on Aug. 28-31, 1995. The course focused on the complementary techniques of small-angle neutron scattering (SANS) and neutron reflectometry (NR) and was intended to provide potential new users of these techniques with a basic understanding of the fundamental concepts and methodology along with training in the use of the SANS and NR facilities at the CNRF. In all, 27 "students" participated in the summer

school, the maximum number that could be accommodated.

The course consisted of lectures each morning by NIST staff, and a few invited speakers, which progressed from introductory material to applications drawn from recent research in, for example, polymer science, complex fluids, magnetism, superconductivity, and structural biology. Afternoons were devoted to demonstrations and hands-on sessions at the instruments in which participants carried out simulated experiments and analyzed sample data sets. These sessions were highly interactive and effective at stimulating discussion and will make up a larger portion of courses planned for the future. Information on the CNRF's peer-review-based experiment proposal system also was provided. The course was a success, judging from the highly favorable comments noted on course evaluation forms, and has clearly enabled this group of researchers to assess the applicability of neutron scattering to their own research interests. This was the first of a series of courses that have been planned in cooperation with the National Science Foundation.

#### **LASER-INDUCED INCANDESCENCE MEASUREMENTS IN DIFFUSION FLAMES**

Quantitative experimental measurements of soot concentrations and soot scattering are presented for a series of steady and flickering co-flowing methane, propane, and ethylene flames burning at atmospheric pressure. Flickering diffusion flames exhibit a wide range of time-dependent, vortex-flame sheet interactions, and thus they serve as an important testing ground for assessing the applicability of chemical models derived from steady flames. Acoustic forcing of the fuel flow rate is used to phase lock the periodic flame flicker close to the natural flame flicker frequency caused by buoyancy-induced instabilities. For conditions in which flame clip-off occurs, the peak soot concentrations in the methane flickering flames are measured to be 5.5 to 6 times larger than a steady flame burning with the same mean fuel flow rate, whereas the enhancement for the flickering propane and ethylene flames is only 35 % to 60 %, independent of the flicker intensity. Soot concentration profiles and analysis of the soot volume fraction/scattering results reveal significant differences in the structure of the soot fields and in the roles of soot inception, growth, and oxidation for the different hydrocarbon fuels.

The soot measurements have been made using laser-induced incandescence (LII). Since this is the only technique currently available for making time- and spatially-resolved soot concentration measurements in time-varying flowfields, considerable effort has been devoted

to developing LII for quantitative applications. Important considerations include: (1) proper calibration measurements; (2) signal detection, which minimizes interferences from C<sub>2</sub> Swain band emission and broad-band molecular fluorescence; (3) correction for the laser beam focus/depth-of-field effect in line image measurements; and (4) correction for LII signal extinction within the flame.

#### **NIST HOSTS WORKSHOPS FOR INDUSTRY ON KEY ESCROW ISSUES**

Renewing efforts to develop a key escrow encryption policy acceptable to industry and other interested parties, NIST sponsored two workshops on key escrow issues. On Sept. 6-7, 1995 about 200 industry and government representatives attended the key escrow issues workshop, which addressed two principal issues: developing the criteria for software key escrow encryption exportability and identifying the desirable characteristics for U.S. key escrow agents. A second meeting on Sept. 15, attended by about 120 people, focused on Federal standards for key escrow encryption.

#### **SOLUTION TECHNOLOGIES FOR IMPROVING SOFTWARE PRODUCTS SUBJECT OF NIST WORKSHOP**

On Sept. 21, 1995 NIST and the Center for High-Integrity Software System Assurance (CHISSA) co-sponsored a workshop on "Technologies to Improve Your Software," which was hosted by a private company in Rockville, MD. About 90 industry and government managers attended. The purpose of the workshop was to help industry understand the challenges and learn about solution technologies for improving their software products. Leaders from government and industry presented talks on the challenges of rapidly changing technology for complex, global high-integrity systems and described solution technologies such as defect data collection and analysis, formal specifications, and software inspections.

#### **NEW PUBLICATION FOCUSES ON NETWORK APPLICATION PROGRAMMING INTERFACES (APIs)**

NISTIR 5657, An Introduction to the P1003.1g and CPI-C Network Application Programming Interfaces, describes two examples of the numerous network APIs that have been developed to assist programmers in developing distributed applications. The report provides an overview of the IEEE P1003.1g and the Common Programming Interface for Communications (CPI-C) specifications, characterizing the basic functionality of each API and giving a simple programming example.

#### **NIST DEVELOPS CLASSIFICATION AUTOMATION SYSTEM FOR FINGERPRINTS**

NISTIR 5647, PCASYS—A Pattern-Level Classification Automation System for Fingerprints, describes a system that automatically classifies images of fingerprints into six pattern-level classes. The program takes gray-level images of fingerprints as input, and for each fingerprint it produces a hypothesized classification as arch, left loop, right loop, scar, tented arch, or whorl, as well as a number indicating how much confidence should be assigned to its classification decision. Automatic classification is useful in an Automated Fingerprint Identification System because it can be used to partition the database of fingerprint cards and reduce the amount of work that must be performed by the fingerprint matcher.

#### **BEEES TO OFFER TIPS TO BUILDERS**

Environmentally sensitive and cash-conscious BEEES could soon be helping designers and architects plan better buildings. BEEES, an acronym for "Building for Environmental and Economic Sustainability," is a software product being developed to identify building materials that improve environmental performance with little or no increase in cost. A NIST and U.S. Environmental Protection Agency team plans to have the building tool ready by 1997. The BEEES project leader, who is an economist at NIST, says: "The building community is making decisions today that have environmental and economic consequences. The NIST/EPA team seeks to support these decisions by gathering environmental and economic performance data and by structuring and computerizing the decision-making process. The software thus will enable users to compare the environmental and economic performance of alternative building materials." BEEES will be available either in CD-ROM format or over the Internet. For more information, contact Barbara C. Lippiatt, B226 Building Research Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-6133, e-mail: lippiat@enh.nist.gov.

#### **CONFEREES SAY IT'S EASY FOR BUILDINGS TO BE GREEN**

Chances are, most of tomorrow's buildings will be "green." Not in color, but so that they have a minimal impact on both the indoor and outdoor environments in the way they are designed, constructed, operated and even demolished. Nearly 300 building owners, developers, design professionals, interior designers, builders, contractors, materials suppliers and others met recently, to exchange green building information, technologies and practices. Highlights of the Second International

Green Building Conference and Exposition, co-sponsored by NIST and the U.S. Green Building Council, are featured in the just-published proceedings. The report cites case histories of effective green strategies for universities, office buildings, single-family residential and municipal projects. It also pays close attention to the economic benefits of green building technologies. Conference proceedings are available by faxing a request to Kim Whitter at (301) 990-4192 or by sending an e-mail message to whitter@micf.nist.gov. For information on NIST's Green Building program, contact A. Hunter Fanny, B322 Building Research Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5864, e-mail: hunter@micf.nist.gov.

### **LEARN NEW RULES FOR IMPROVING “NOISE”**

NIST scientists have developed a theoretical framework for describing excess low-frequency phase modulation (known as PM) and amplitude modulation (known as AM) noise generation processes in radio frequency and microwave amplifiers. They produced a set of design rules that work exceptionally well for circuits based on bipolar junction transistors. A key to this advance is the development of state-of-the-art systems for measuring PM and AM noise close to the carrier. This has allowed complete testing and verification of the concepts. The design rules take into account several different noise sources, including up-conversion of low-frequency base-band noise from bias currents, noise generated within the transistor and power supply noise. Application of these methods can provide substantial improvements in the fidelity of processed and amplified signals, particularly where a strong carrier frequency is involved and for high-spectral-purity oscillators. For example, they have demonstrated high gain amplifiers where the excess phase noise is less than the thermal noise, even within a few hertz of the carrier. This is an improvement of 10 db to 20 db over current systems. NIST is prepared to disseminate this new technology through intensive training sessions with limited attendance. The training will include the origin of low-frequency PM and AM noise in bipolar transistor amplifiers, the design of amplifiers with low excess PM and AM noise, implementing high-resolution PM and AM noise measurements, and bench measurements of PM and AM noise. Persons interested in the training should contact Fred L. Walls, NIST, Boulder, CO 80303-3328, (303) 497-3207, e-mail: walls@bldrdoc.gov. Session dates will be arranged to accommodate requirements.

### **COMPOSITE MATERIALS WORKSHOP PROCEEDINGS AVAILABLE**

The First International Workshop on Composite Materials for Offshore Operations, held Oct. 26–28, 1993, at the University of Houston, provided a forum for international experts from industry, government and academia to explore the role of composite materials—glass or carbon-fiber reinforced plastics—in meeting the stringent requirements of deep-water offshore structures used for petroleum exploration and production. The proceedings are now available. Consisting of 32 papers, the collection provides a comprehensive overview of composite applications in the offshore industry. Included are evaluations of state-of-the-art offshore composite technologies; a view of certification and regulatory agency perspectives toward using such technologies; and summaries by eight working groups, which assessed industry practices, identified gaps between state-of-the-art and state-of-practice, and prioritized research initiatives that might mitigate these gaps. The Minerals Management Service of the U.S. Department of the Interior, NIST, and the University of Houston sponsored the workshop. The publication, entitled Composite Materials for Offshore Operations: Proceedings of the First International Workshop (NIST Special Publication 887), can be obtained for \$24 prepaid from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, (202) 512-1800. Order by stock number 003-003-033-57-0. Additional information is available from Fitting at (303) 497-3445.

### **PUBLICATIONS HIGHLIGHT FY 1994 NEUTRON RESEARCH**

Materials researchers are investigating the structures of complex polymers, superconductors, and biological macromolecules with sensitive, penetrating neutron beams that often give otherwise unobtainable information. Industry, academic, and government researchers annually conduct hundreds of studies on a wide range of advanced materials at NIST's neutron research facilities. Summaries of activities at the NIST research reactor for fiscal year 1994 are now available from the National Technical Information Service. A 149-page report, Reactor Radiation Technical Activities 1994 (NISTIR 5583), gives descriptions of NIST technical activities as well as research programs at the NIST reactor supported by industry and academia. A more detailed 171-page report, NIST Reactor: Summary of Activities, October 1993 through September 1994 (NISTIR 5594) covers the same activities. Each publica-

tion is available for \$27 prepaid. Order NISTIR 5583 by PB 95-209888 and NISTIR 5594 by PB 95-220430 from NTIS, Springfield, VA 22161, (703) 487-4650.

### **SABIT TRAINING**

The Office of Standards Services, in cooperation with the Special American Business Intern Training (SABIT) program of the International Trade Administration, other NIST offices, and the private sector, has completed three training sessions for standards and regulatory experts from the automotive, medical equipment, and telecommunications sectors in Russia and the Newly Independent States (NIS). A fourth SABIT session, for aerospace experts, was held recently at NIST.

Follow-up meetings with past participants, held in Moscow, indicates considerable success in achieving the intended purpose of the training by reducing the impact of technical barriers to trade through a better understanding of the U.S. system of standards development and conformity assessment of products; the relationship of public and private-sector programs for ensuring health safety and the protection of the environment; and introduction to U.S. companies interested in marketing in Russia and the NIS. As a prime example of success, a senior Russian participant in the automotive session acquired enough confidence in the U.S. system to sign a Memorandum of Understanding with the Department of Transportation that provides for acceptance of U.S. safety standards in fulfillment of specific Russian regulatory requirements where equivalence has been demonstrated. Upcoming NIST/SABIT training is scheduled for experts in food processing/packaging, oil and gas equipment and services, and construction/infrastructure.

### **NIST AND ANSI CO-CHAIR SUCCESSFUL U.S. CELEBRATION OF WORLD STANDARDS DAY**

A capacity crowd of 260 people attended the 1995 World Standards Day reception and dinner on Oct. 11 at the Crystal City Marriott Hotel. The annual event is celebrated worldwide in conjunction with the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC) to further increase the awareness level of the importance of standardization among the business community, policy makers, and consumers.

The World Standards Day Planning Committee is co-chaired by NIST and ANSI and sponsored by some 50 trade associations, professional societies, standards development organizations, corporations, and government agencies.

### **LABORATORY ACCREDITATION FORUM**

ACIL (formerly American Council of Independent Laboratories), American National Standards Institute (ANSI), and the NIST Office of Standards Services co-sponsored an open forum at NIST for discussion of issues in laboratory accreditation recently. The forum provided an opportunity to discuss achieving greater compatibility, coordination, and mutual recognition of competent laboratory accreditations at the national and international levels; the problems of multiple and/or duplicate accreditations that result from overlapping responsibilities of governments and industry; and assuring reciprocity among accreditation programs.

Laboratories, accreditors, manufacturers, the National Environmental Laboratory Accreditation Conference, and government representatives, both federal and state, presented their views on the cost of multiple accreditations; conflicting requirements of users of accreditation; special programs tailored to narrow customer or supplier bases; non-uniformity of requirements and lack of reciprocity; international trade implications; and other pertinent factors. Issues discussed included: the history of efforts to build a U.S. system of laboratory accreditation dating back to a conference held at NBS in the early 1960s; needs to satisfy foreign government concerns about the competence of laboratory accreditation bodies when negotiating mutual recognition agreements. A panel discussion and open exchange of ideas explored concepts for future collaboration that may lead to "one-stop-shopping" in laboratory accreditation. All participants were urged to submit ideas for inclusion in the proceedings that will be issued later this year.

### **THREE OFFICIALS FROM MEXICO'S NATIONAL STANDARDS BODY VISIT NIST**

The Office of Standards Services sponsored three representatives from the Mexican Direccion General de Normas (DGN) at NIST to learn about legal metrology, laboratory accreditation, and standards information obligations under NAFTA. They exchanged information with corresponding NIST metrology and conformity assessment staff. Talks were given by NIST and DGN personnel, and there were visits to the U.S. Department of Agriculture Laboratory in Annapolis, and the FDA Ionizing Radiation Laboratory in Rockville. For the first time since NAFTA was ratified, the directors of the Inquiry Point Offices (DGN, NIST's National Center for Standards and Certification Information, and the Standards Council of Canada) of the three NAFTA countries met to discuss the obligations spelled out in the NAFTA and the World Trade Organization Technical Barriers to Trade agreements.

NIST will summarize the inquiry point officers' meeting and officially present a report at the next NAFTA Standards Related Measures Committee Meeting; develop a timetable of visits in support of the North American Calibration Cooperation effort; and work out details for a NIST representative to be located at DGN to facilitate cooperation on various projects, including legal metrology.

### FLASH POINT REFERENCE MATERIALS DEVELOPED

Flash point measurements have become more important with the tightening of regulations in response to fires, spills, and shipping issues. Properties of materials must be measured to ensure safety requirements are met. One common test to ensure this safety is the flash point of materials (the lowest temperature at which the vapor from a material will ignite or flash upon application of an ignition source). ASTM Coordination Committee S-15 on Flash Point, in cooperation with the NIST Standard Reference Materials Program, has coordinated a 19-laboratory effort leading to the development of a series of four flash point calibration fluid Reference Materials (RMs): 8517 n-Decane; 8518, n-Undecane; 8519, n-Tetradecane, and 8520, n-Hexadecane.

The four flash point calibration fluids and their corresponding closed cup, nominal flash point temperatures are: n-Decane (53C), n-Undecane (69 C), n-Tetradecane (109 C), and n-Hexadecane (134 C). These cover the range of temperatures called for in several ASTM standards. The flash point fluids provide a means to test the equipment in laboratories and ensure that the equipment is calibrated properly and measurements are traceable to national standards via ASTM methods.

The RMs are currently available for purchase. Each flash point material will be provided separately and includes four sealed ampoules, each containing 20 ml of the flash point fluid. As stated in the agreement made between ASTM and NIST, these fluids "will enable NIST to upgrade its existing fossil fuels standards and, at the same time, address one of the more critical measurement needs in fossil fuel research."

### ENERGY-RELATED INVENTIONS PROGRAM RECOMMENDATIONS

During September, the NIST Office of Technology Innovation recommended three innovative technologies for commercialization to its Department of Energy partner under the Energy-Related Inventions Program.

- Methods and Devices for Energy Conservation in Refrigerated Chambers—a control system to reduce the evaporator fan speed in reach-in and walk-in commercial refrigerators.
- Refrigerator with Pan Chiller System—a method for cooling food in open-top, insert-pan type refrigerators commonly used in commercial food preparation facilities.
- Laminated Ceramic Cutting Tool—a composite ceramic cutting tool formed in distinct layers of reinforced and unreinforced ceramics. Reinforcement is accomplished by strengthening with SiC (silicon carbide) whiskers.

### NIST LEADS REVISION OF RELIABILITY STANDARDS FOR IMPROVED MEASUREMENTS

As a result of data from NIST-led experiments and other NIST activities, two reliability standards have been extensively revised by a NIST researcher. Both standards relate to the failure mechanism known as electromigration in thin-film metal interconnects, which challenges the development of smaller integrated-circuit geometries. Engineers in the semiconductor industry use these standards for evaluating the reliability of metal interconnects by means of accelerated stress tests. These standards also respond to needs identified in the Semiconductor Industry Association National Technology Roadmap for Semiconductors and in the Semiconductor Research Corporation 2001 Reliability Roadmap.

One of these standards is an ASTM test-method standard for measuring electrical linewidth of metal interconnects on oxide-coated silicon wafers. Together with a value for line thickness, this parameter must be known to calculate line cross-sectional area, in turn required for the calculation of current density, one of the stress drivers of electromigration. The revision incorporates the results of an interlaboratory experiment that reported a between-laboratory reproducibility of 10 nm (0.58 %) for the method and a within-laboratory repeatability of 0.6 nm (0.05 %) for metal lines ranging from 1  $\mu\text{m}$  to 2  $\mu\text{m}$ .

The other standard is a Joint Electron Device Council standard for the measurement of both the temperature coefficient of resistance (TCR) and the joule heating in metal interconnect lines. These values are needed to determine the stress temperature of metal lines, another of the stress drivers of electromigration. The revision again incorporates the results of an interlaboratory experiment that demonstrated between-laboratory

reproducibilities of TCR and joule heating measurements of 2.5 % and 0.14 °C, respectively, and within-laboratory repeatabilities of 0.2 % and 0.03 °C. Knowledge about the expected reproducibility of the stress drivers of electromigration (current density and metal temperature) are important to evaluate the significance of any differences in the values measured in accelerated stress tests for evaluating metal interconnect reliability.

#### **NIST-DEVELOPED TUTORIAL ON MICROELECTRONIC PACKAGING MEASUREMENTS ATTRACTS INDUSTRY INTEREST**

As the microelectronics industry continues to struggle with performance constraints resulting from the packaging that connects integrated circuits with computer and telecommunications systems, engineers are increasingly realizing the need for accurate electrical measurement and modeling tools. In a survey conducted at last year's IEEE Topical Meeting on Electrical Performance of Electronic Packaging, the premier forum for discussion of these topics, participants indicated a strong interest in tutorial short courses, particularly on measurements.

In response a NIST scientist developed a half-day course entitled "Understanding, Performing, and Applying High-Frequency Electrical Package Measurements," held in conjunction with the recent Fourth Annual IEEE Topical Meeting on Electrical Performance of Electrical Packaging. The course focused on leading-edge metrology developed at NIST for network analyzer measurements of microcircuits. The NIST scientist also worked with industry experts to develop additional lectures covering time-domain network analysis and illustrating applications of the techniques to commercial packages. The course attracted students from companies in the computer industry as well as the microwave and radio frequency industry.

#### **JAPANESE AND CHINESE RESEARCHERS SUPPORT NIST'S APPROACH TO UNIFYING THE INTERNATIONAL ROCKWELL C HARDNESS SCALES**

A NIST scientist was invited to visit the National Research Laboratory of Metrology (NRLM) in Tsukuba, Japan, where he presented technical talks entitled "Surface Finish and Microform Calibrations at NIST" and "A Metrology Approach to Unifying Rockwell C Hardness Scales." The NIST scientist performed surface and hardness comparison tests and conducted discussions on the metrology approach to unifying the international Rockwell hardness scales. He

also visited the National Institute of Metrology (NIM) in Beijing, China, where he presented identical talks, performed similar measurements, and held discussions. From these discussions, it was learned that both NRLM and NIM researchers support the NIST metrology approach and want to cooperate with NIST to establish their own metrology-based Rockwell hardness system. As the first step to determine whether the small difference between a NRLM and NIST indenter is systematic, five Japanese national indenters were brought to NIST for geometric measurement and hardness tests.

Rockwell C hardness (HRC) standard is one of the most important industrial standards. Since the 1980s, the EC countries unified their HRC scales. With the development of ISO 9000 standards, the unified EC scale is forming a challenge to the HRC tests performed in other countries, including the United States. Because the EC approach was based on performance comparisons, the unified EC scale still may exhibit an unknown offset to the correct scale and may not have traceability and reproducibility.

At the ISO/TC164/SC3 meeting, NIST proposed a metrological approach to unifying the international Rockwell hardness scales. The SC3 issued a document to establish a working group to investigate the approach to unify the international Rockwell hardness standards.

#### **DEPARTMENT OF VETERANS AFFAIRS AWARDS COMMENDATIONS**

For many years NIST has been assisting the Department of Veterans Affairs (VA) by making extensive electroacoustic measurements on hearing aids and by providing related technical consulting. Much of the work involves developing new test methods necessitated by the increasing technical sophistication and complexity of rapidly evolving new products, which include custom, special purpose, programmable, and/or digital hearing aids.

Because of the number of veterans who are hearing challenged due to either war-related injuries or excessive noise exposure "on the job" and the number of those whose quality of life can be improved significantly through hearing aids, the VA is the largest single customer of hearing aids. Their need for versatile aids of the highest quality motivated the NIST development of advanced test methods and reliable data on hearing aids. The data are published by the VA in their annual Handbook of Hearing Aid Measurements for guidance to VA audiologists in fitting aids to veterans. (The data are traceable to NIST acoustical standards.) Many of the test method developments have been incorporated into ANSI measurement standards. Two acoustical engineers at NIST were awarded a Department of Veterans Affairs

Commendation from the Under Secretary for Health for their work in this area.

#### **ENHANCED MACHINE CONTROLLER (EMC) VIDEO COMPLETED**

The Enhanced Machine Controller (EMC) videotape is complete. The video outlines the evolution of the NIST Real-time Control System (RCS) and highlights the implementation of EMC at a major automotive manufacturer. The EMC effort was undertaken in cooperation with the Department of Energy's Technologies Enabling Agile Manufacturing program, and has developed a specification for interfaces to open-architecture controllers. The controller is the first production-tested controller that conforms to the interface specification and includes software from a private company for the control of the tooling and part loading systems. As depicted in the video, the controller is based on commercial off-the-shelf hardware and computer operating systems, and provides a familiar graphical user interface. The controller is now in daily use at the manufacturer's facility on a four-axis horizontal machining center. Requests for copies of the videotape should be made to Tracy Becker, ext. 3452.

#### **NIST JOINS OMG**

NIST has joined the Object Management Group (OMG) as a new end user/government member. The OMG is a consortium of more than 500 member companies and organizations with a goal of promoting object technology through mutually agreed upon and openly available specifications for object request brokers and object services.

Individuals from NIST have been attending OMG meetings as guests. This membership will give NIST's participation full impact and allow NIST to vote in all committee and subcommittee activities regarding manufacturing and information technology and also give NIST ready access to OMG electronic material and copies of publications.

#### **PETROLEUM ENVIRONMENTAL RESEARCH PROJECT MEETING ON TOXIC COMBUSTION BYPRODUCTS HELD AT NIST**

Scientists at NIST hosted a meeting of experts in petroleum environmental research recently. A total of 31 scientists and engineers representing oil and gas producers, burner suppliers, national laboratories, government agencies, the University of California at Los Angeles, and the analytical chemistry contractor met at NIST to review the results of a program designed to assess the nature and quantity of the volatile organic emissions from the gas burners used in refineries.

The program came about in response to proposed more stringent regulations on emissions from such burners and field studies that indicated that the new standards were not being met. Results from a laboratory burner at UCLA, small scale and pilot facilities at Sandia and Livermore, and analytical models were compared with each other and with earlier field measurements, with attention given to the consequences of failure. The chemical kinetics models, largely based on the work of NIST scientists, were found to be capable of predicting the formation of naphthalene, benzene, and some other aromatic organic compounds.

#### **AIR SPEED PROFICIENCY TESTING PROGRAM**

NIST is forming a consortium with industry to establish an Air Speed Proficiency Testing (ASPT) program. This program is in response to concerns expressed by representatives from industry at a NIST workshop on air speed measurements. The ASPT program will quantify the performance of anemometer calibration facilities operated by anemometer manufacturers, calibration laboratories, and users through a round-robin testing program. Testing will span the air speed range from 0.3 m/s to 15 m/s. Using a transfer standard developed as part of the ASPT cooperative research and development agreement, NIST staff will measure the air speed in the participant's wind tunnel. At the same time, the participant will measure the air speed using their instrumentation, measurement techniques, and data reduction methods. The results of this direct comparison of the entire measurement process will provide an assessment of the accuracy and a basis for developing realistic air speed measurement traceability of the process.

#### **NIST CO-SPONSORS INTERNATIONAL CONFERENCE ON ENVIRONMENTRICS AND CHEMOMETRICS**

The International Conference on Environmetrics and Chemometrics took place in Las Vegas, NV, recently, and featured speakers from Finland, Switzerland, Germany, Sweden, and America. The meeting was jointly sponsored by the National Institute of Standards and Technology, Environmental Protection Agency, Texas A&M University, University of Nevada—Las Vegas, National Institute of Statistical Science, and three private companies served as a forum on such topics as chemical mass balance and receptor modeling, regulatory issues (industrial and environmental perspectives), metrology, environmental restoration, reactions on ice surfaces, quantitative structure activity relationships, detection, and human population exposure.

A NIST scientist served as an organizer and speaker, presenting two invited talks: “Revolutions in the Development of  $^{14}\text{C}$  Metrology: a Case Study in Physical-Statistical Counterpoint” and “Detection: International Update and Some Emerging Di-Lemmas.” The first talk traced the development of modern  $^{14}\text{C}$  metrology and its remarkable impact on fields so diverse as archaeology, isotopic geophysics, and atmospheric chemistry. The evolution of this technique, which has seen an expansion in sensitivity by nearly a million-fold, was offered as a classic success story in total quality measurement. The second talk addressed recent international developments in concepts and nomenclature related to limits of detection and quantification, together with certain critical open questions that stimulated significant discussion among the statisticians and chemometricians present at the meeting, especially in the context of meaningful regulatory decisions.

The conference was a natural sequel with integrated objectives to previous conferences on chemometrics and environmetrics. The proceedings of the conference will be published in an early 1996 issue of *Chemometrics and Intelligent Laboratory Systems*.

#### **CHEMICAL IDENTIFICATION BY SCANNING TUNNELING SPECTROSCOPY**

Scanning tunneling microscopy (STM) has provided many dramatic atomic-scale images of the topography of surfaces. It is not possible from the images alone, however, to know what elemental species give rise to the topography observed. Scanning tunneling spectroscopy measurements by NIST scientists coupled with calculations of a scientist from Brookhaven National Laboratory, were reported in the Oct. 16, 1995 issue of *Physical Review Letters*. These measurements showed that in some cases it is possible to distinguish the elements present in a tunneling measurement. Tunneling spectroscopy, in which the tunneling current is measured as the tip-sample voltage is varied, probes the energy dependence of the electron states in the sample and the tip. An intense, sharp feature was found in the tunneling spectrum from iron surfaces and, at a different energy, in the spectrum from chromium surfaces. Electron band-structure calculations show that this feature is due to a surface state that is a general feature of the surfaces of body centered cubic (bcc) metals. It is interesting to note that such a surface state was observed in field emission energy distributions (tunneling into vacuum instead of a surface) of tungsten, another bcc metal, and intensely investigated both theoretically and experimentally more than 20 years ago at NBS. The measurement of this surface state in scanning tunneling spectroscopy permits chemical identification with atomic spatial

resolution on bcc surfaces. Moreover, in the case of iron and chromium, the surface state exists only in the minority electron spin sub-band and thus is well suited to test future electron spin polarized tunneling experiments, which hold the promise of magnetic measurements on an atomic scale.

#### **CALCULATIONS SUGGEST THAT $^{87}\text{Rb}$ WAS THE “RIGHT” ATOM FOR THE BOSE-EINSTEIN CONDENSATION EXPERIMENT**

Considerable excitement was generated recently when the NIST/JILA group in Boulder, CO achieved Bose-Einstein condensation using cooled and trapped  $^{87}\text{Rb}$  atoms. Collisions between the Rb atoms are crucial both in the evaporative cooling process that lead to the formation of the condensate and in determining the stability and lifetime of the condensate. NIST and JILA scientists have calculated the collision rates for the dominant collisional process that ejects atoms from the condensate and causes the condensate to have a finite lifetime, on the timescale of seconds or tens of seconds. This collisional process causes a trapped  $^{87}\text{Rb}$  atom to become an untrapped atom after a collision of two of the trapped atoms. Fortunately, the rate coefficient for this collisional loss process is small, so the condensate can live for a reasonable length of time. The rate coefficient for Rb atoms is unusually small due to a cancellation between competing effects unique to the Rb species. This small collisional loss rate may turn out to be the lowest of all the alkali species. It leads to a longer lifetime of the condensate and makes  $^{87}\text{Rb}$  an especially favorable case for studying BEC.

Researchers at NIST, Oxford University, University of Maryland, and Georgia Southern University are now using this rate coefficient to calculate the actual trap lifetimes for the condensate produced by the NIST/JILA group.

#### **WORKSHOP HELD ON CALIBRATION OF IR FOCAL PLANE ARRAYS AND IMAGING SYSTEMS**

Two-dimensional infrared focal plane arrays became commercially available during the late 1970s and, in the past 20 years, have been increasing in size and in sensitivity. They have allowed U.S. industry to produce imaging systems for astronomical and military applications, both ground and space-based. In response to the needs of this community, NIST hosted a workshop, “Standards for Testing and Calibration of IR Focal Plane Arrays and Imaging Systems for DoD/BMD Applications” at NIST recently for representatives from the military, industry, and academia. The goal was to identify the standards and calibration support required for infrared focal plane arrays and imaging systems.

### **NIST KEY PARTICIPANTS IN RADIATION PROCESSING WORKSHOP**

Experts from NIST constituted a commanding core of instructors and session leaders at the Third International Workshop on Dosimetry for Radiation Processing, held Oct. 1–6, 1995 in Ste-Adèle, Québec, Canada. With other international experts, five NIST scientists, presented plenary lectures, led workshop sessions, presented papers, and acted as instructors in hands-on dosimetry exercises.

The workshop drew 140 registrants from 24 countries, representing the growing international community involved in radiation processing dosimetry: scientists, industrial users, quality assurance personnel, dosimeter manufacturers, and regulators of irradiated products. The workshop, organized by the American Society for Testing and Materials (ASTM), was the third in a series begun in 1989 to address the growing use of radiation processing of medical products, pharmaceuticals, polymers, foods, and other products. Accurate radiation dosimetry is often critical in these applications, requiring measurement assurance to meet stringent regulatory standards. NIST has been a world leader in the development and application of radiation dosimetry in these areas, including radiochromic dye systems, alanine/ESR dosimetry, calorimetry, and calibration and measurement quality assurance programs.

### **IMPROVED UNCERTAINTY FOR NIST-7**

NIST scientists recently reported a substantial reduction in the uncertainty of NIST-7 based on an improved approach to evaluation of the systematic errors of the standard. NIST-7, an optically pumped cesium beam frequency standard, was originally designed to have an uncertainty of  $1 \times 10^{-14}$ , but the uncertainty now reported to the International Bureau of Weights and Measures (BIPM) is  $5 \times 10^{-15}$ . This improvement is the result of recently developed evaluation methods providing for substantially smaller uncertainty in the measurement of the various systematic effects.

The new methods hold the potential for further improvement, perhaps as much as another factor of five. This means that the original objective of an improvement of a factor of 10 over the accuracy of NBS-6 could be extended to a factor of 100, producing the largest performance advance for any frequency standard constructed at NIST. This gives NIST a more comfortable margin over commercial frequency standards, which are now approaching uncertainties of  $1 \times 10^{-13}$ .

### **PHASE EQUILIBRIA STUDIES OF CERAMICS-FOR WIRELESS COMMUNICATIONS**

Magnetic ceramics are essential components in a wide variety of electronic applications; e.g., nearly all wireless communications systems use coaxial circulators and isolators fabricated from ceramic magnets. The crucial properties of these ceramics include high dielectric constant (to enhance miniaturization), low dielectric loss, and high saturation magnetization. A materials system of immediate relevance is  $\text{BaO-Fe}_2\text{O}_3\text{-TiO}_2$ , which effectively combines the technically important barium titanates (used as microwave resonators in all base stations) with magnetic iron oxides. NIST scientists recently completed the experimental determination of this ternary phase diagram. The system was found to be surprisingly complex; the NIST study found that 16 ternary compounds exist, only two of which were known previously. Collaborative studies are in progress at NIST to determine the crystalline arrangements of the various compounds using neutron diffraction. Measurements of the compound's dielectric and magnetic properties are also under way at NIST.

In addition to the potential usefulness of the new compounds, the phase diagram is useful as a "processing road map." For example, the important permanent magnetic material  $\text{BaFe}_{12}\text{O}_{19}$  (barium hexaferrite) was found to exist in equilibrium with 10 other phases; the phase diagram, therefore, gives "co-processing coordinates"—exact chemical compositions at which the properties of barium hexaferrite ceramics could be deliberately modified with those of controlled amounts of selected second phases. Barium hexaferrite also was observed to dissolve substantial amounts of the higher-dielectric-constant  $\text{TiO}_2$ , thus indicating that the dielectric constant of barium hexaferrite ceramics may be increased by co-processing with  $\text{TiO}_2$ , thereby enhancing miniaturization. In addition, the observed extensive dissolution of iron in  $\text{BaTiO}_3$  indicates that  $\text{BaTiO}_3$  ceramics may be processed to contain varying amounts of magnetic ions.

### **MAGNETIC DOMAIN PATTERN COLLAPSE OBSERVED IN SINGLE CRYSTAL NICKEL THIN FILMS**

Magnetic domains in demagnetized sub-micrometer single crystal nickel films recently were observed at NIST in a magnetic force microscope (MFM) in the magnetic materials group. Surprisingly, it was found that the domains apparently could be directed into any in-plane direction, irrespective of the crystal orientation.

Normally, since materials preferentially magnetize along only certain crystal directions, the domain structure also reflects this anisotropy. The width of the magnetic domains in these films was found to be uniform across the sample and to depend upon the film thickness. In particular, the domain width was found to scale with the square root of the film thickness down to a critical value below which the striated domain pattern collapsed.

Unfortunately, the MFM does not tell the whole story, as it measures only the stray fields above the sample and provides little information about the magnetization below the surface. Consequently, to uncover the magnetic domain behavior throughout the film thickness, calculations based on micromagnetic models were performed to complement the MFM observations. Preliminary model results indicate that the domains in the nickel films are in fact canted with respect to the surface. More sophisticated models are being developed that will use the MFM data as an input to calculating and subsequently predicting the magnetization behavior at the nanometer level. As magnetic devices become more miniaturized and sophisticated, there is an increased need to understand this magnetization behavior at such high resolution.

### NIST REACTOR RESTARTED

On Sept. 27, 1995 the NIST Research Reactor was restarted with liquid hydrogen in the new cold source and was step-wise increased to full power (20 MW), achieving it on Oct. 5. All systems worked well, and routine operation, with no major scheduled shutdowns, is expected for the next 5 years. There were a number of major upgrades during the shutdown:

- **Reactor Systems:** During the shutdown, the two main tubes and shell heat exchangers were replaced by three new plate type heat exchangers, providing redundancy (the reactor operates with two in service), and a more efficient system. The new design also provides better protection against small leaks. The new heat exchangers were tested extensively during fabrication and installation, and meet or exceed all specifications for helium leak tightness, flow capacity, and other parameters. Many other items were upgraded and/or overhauled in the reactor systems to provide better long-term operation of the reactor.
- **Cold Neutron Source:** The D<sub>2</sub>O source and bismuth tip were removed and replaced by a liquid hydrogen

source and thermosiphon. This required extensive reworking of the shielding and auxiliary D<sub>2</sub>O system, as well as the installation of other components (refrigerator, hydrogen storage tank, and associated plumbing).

- **Neutron Guide System:** The neutron guides within the reactor confinement building were completely reworked, with vacuum casings installed to prevent stress on the heavily irradiated sections of the glass. The final three guides were installed (NG-1,2,4), and a new curved guide was installed at NG-0.

A particularly significant result of these upgrades for the users of the Cold Neutron Research Facility (CNRF) is the increase in cold neutron intensity that the new cold source provides. Over the 0.6 nm to 2.0 nm wavelength range, the intrinsic gain with respect to the original cold source is no less than 4.5. When the 15 MW to 20 MW increase in power is also included, the gain is between 6 and 8 over the same wavelength range (which is very close to design predictions).

Successful completion of these improvements secures NIST's position as a leading neutron research center, directly competitive with the best in the world.

### STATUTORY INVENTION REGISTRATION ISSUED FOR NIST X-RAY RESOLUTION DEVICE

The U.S. Patent Office has issued a Statutory Invention Registration for a device to measure the *z*-dimension resolution of x-ray laminography systems. The device was invented by scientists from NIST, the U.S. Army Harry Diamond Laboratory and a private company.

The device is a right triangular prism constructed from layers with different x-ray absorption characteristics. As the angled beam in a laminography system passes through the various layers, the x-ray beam intensity is modulated accordingly. The spacing of the layers measured by the laminography system can be compared to the optical calibration data furnished with the device. Although this device was designed for calibration of systems that inspect electronic circuit boards, it is expected to have wide application to laminography systems. It adds a valuable reference standard to the very few that exist for checking the performance of laminography systems. A device based on this technology is now available from the Standard Reference Materials Program as SRM 1843.

### **NIBS HONOR AWARD TO BE PRESENTED TO NIST**

The Board of Directors of the National Institute of Building Sciences has selected the NIST Building and Fire Research Laboratory (BFRL) to be the recipient of the Institute's 1995 Honor Award. NIBS' Honor Award is presented annually to an organization or individual in recognition of exceptional contributions to the nation and the building community. In this context, NIST is specifically cited for its significant contributions to the national competence in building and fire research through state-of-the-art studies, analysis of structural failures and earthquake damage, and evaluations of test methods. The board recognizes NIST's important commitment and dedication to improving the safety and quality of constructed facilities and enhancing the competitiveness of the U.S. construction industry.

### **NEW STEP PROJECT FOR CHEMICAL PROCESS ENGINEERING DATA**

ISO TC 184/SC4, Industrial Data and Global Manufacturing Programming Languages, has approved a new work item to develop a STEP application protocol for the representation and exchange of chemical process engineering data. The new work item proposal was developed under the recently initiated cooperative research and development agreement between NIST and the Process Data Exchange Institute of the American Institute of Chemical Engineering. The proposal was approved for submission to ISO by the ANSI Technical Advisory Group to TC 184/SC4. The scope of the new work item dovetails with existing work on STEP application protocols dealing with functional and physical descriptions of process plants. When completed and adopted, this new application protocol will become Part 228 of ISO 10304, Product Data Representation and Exchange.

### **NIST AND INDUSTRY DELIVER INFORMATION EXCHANGE STANDARD**

As a result of the NIST collaboration with the Plant-STEP consortium, the first STEP application protocol for the process plant industries, ISO 10303-227, was submitted to ISO TC184/SC4 for review and ballot as an ISO Committee Draft standard. The process plant industries include the chemical, pharmaceutical, and power generation industries; engineering and construction industries; and the suppliers and fabricators of components and equipment for process plants. BFRL is working with the process plant industries and the vendors of information technology for these industries to develop, test, and demonstrate information exchange

standards based on ISO 10303, commonly referred to as STEP, STandard for the Exchange of Product Model Data. ISO 10303-227, Plant Spatial Configuration, supports the exchange of the spatial configuration of plant systems with a central emphasis on piping systems. This committee draft standard supports the information required to construct piping systems, including the shape, material, and arrangement of components, and supports the physical description of other plant systems, e.g., heating, ventilation, and air conditioning systems.

NIST researchers also are building the Advanced Manufacturing and Network Testbed (AMSANT) for the Process Plant Industries. This testbed establishes an experimental computing and communication facility for the testing of the draft application protocols (APs) and the integration activities of the process plant industries. AMSANT for the Process Plant Industries provides an open facility in which companies and others can test trial implementations of the APs and proposals for integrating computerized systems without bias or unnecessary disclosure of proprietary developments.

### **INFORMATION SECURITY CONFERENCE ATTRACTS LARGE TURNOUT**

On Oct. 10-13, 1995 NIST and the National Security Agency's National Computer Security Center (NCSC) co-sponsored the 18th National Computer Security Conference in Baltimore, MD. The theme of the conference was "Making Security Real." The conference, attracted about 1900 attendees from government and industry.

The conference focused on developing and implementing secure networks, technologies, applications, and policies. Six tracks were featured: technical challenges, solutions, criteria and assurance, management and administration, the Internet and beyond, and the tutorial track. Specific topical areas included intrusion detection, WWW security, incident response, research and development for secure products, risk management, system integration, privacy, disaster recovery and business continuity, computer crime, ethics, and new security paradigms. Many NIST staff members participated in the conference as speakers, panel members, and moderators.

### **WORKSHOP SEEKS STANDARDS FOR DIGITAL MUGSHOTS**

On Oct. 23-25, 1995 NIST sponsored the Mugshot and Facial Image Standards Workshop in cooperation with the Federal Bureau of Investigation. The goal of the workshop was to identify commonalities between different systems of exchanging digital mugshots and to

develop consensus about a set of voluntary standards or guidelines for the exchange of digital mugshots. Organizations relying on digital images tend to use their own private standards and formats for capturing, processing, and exchanging images of faces. Electronic facial images are used increasingly in law enforcement, motor vehicle licensing, and passport control as a form of identification.

### **NIST ESTABLISHES NEW PUBLICATION SERIES**

NIST recently launched the new NIST Special Publication Series 881 for Federal Electronic Data Interchange (EDI) Conventions. As part of the overall federal effort to establish a single face to industry for conducting electronic commerce, NIST was designated as the organization responsible for coordinating the development of Federal Implementation Conventions (ICs) for EDI. ICs are defined by functional-area experts who create and select options from standard EDI Transaction Sets to yield the implementations to be used for practical Electronic Data Interchange. NIST makes these ICs available to Federal agencies and industry by electronic and paper means. NIST Special Publication 881-1, Federal Implementation Guideline for Electronic Data Interchange, ASC X12 003040 Transaction Set 838, Trading Partner Profile (Vendor Registration), Implementation Convention, is the first publication in the new series; the initial set of ICs to be published will consist of 11 ICs in the procurement and finance areas.

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## **Standard Reference Materials**

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### **SRM 8090 LITHOGRAPHIC SEM CALIBRATION STANDARD**

The low-accelerating voltage, scanning electron microscope metrology instrument has assumed an important role in modern semiconductor process control. As semiconductor structures are miniaturized, with geometries down to the 0.35  $\mu\text{m}$  level (and below), accurate calibration of the SEM becomes increasingly critical. A new SEM magnification calibration standard has been developed for the semiconductor industry specifically to address this need.

RM 8090 is a prototype of SRM 2090 that is projected to be available by the summer of 1996. RM 8090 is being released to obtain industry measurement experience and input to the final design specification of

SRM 2090. SRM 2090 will ensure measurement linkage to NIST and other worldwide national standards organizations.

The RM 8090 unit consists of a 10 mm $\times$ 10 mm silicon wafer with a titanium/palladium pattern, stored in a cleanroom-sealed container. The pattern was fabricated using an electron beam lithography and metal lift-off technique. This pattern has line spacings ranging from 0.2  $\mu\text{m}$  to 3000  $\mu\text{m}$ , in both the X and Y directions, useful for magnifications ranging from 100 $\times$  to greater than 300 000 $\times$ , along with patterns useful in focusing and astigmatism correction.

### **REFERENCE MATERIALS FOR THE ADVANCED ADSORBENTS INDUSTRY**

Representatives from NIST recently met with a spokesperson from the International Adsorption Society (IAS) to discuss industry needs for a standard suite of reference adsorbents. Advanced materials with unique adsorbent properties play a major role in separations and purification in the chemical industry. Current industry efforts to develop and exploit new adsorbent technologies is restricted by the absence of well calibrated reference adsorbents that will permit interlaboratory comparisons. For instance, as industries look to more complex separations and purification schemes, they are moving to more elaborate computer simulations to aid in elimination of pilot scale testing of adsorbent performance. To enable intralaboratory comparisons and computational modeling, a suite of common adsorbents standards has emerged as a critical infrastructure component.

The range of materials discussed included crystalline zeolites as well as amorphous carbons, aluminas, and silica gels. The discussions indicated that standards would be a significant benefit, both within the adsorbents community and downstream to its customers in terms of alternative separation and purification technologies. The availability of a suite of reference adsorbents would also facilitate critical comparisons between industrial, academic, and government laboratories. By removing the uncontrolled variation between current benchmark materials, the ability to evaluate advanced adsorbents with specific design parameters will be greatly enhanced.

As a result of the meeting, a detailed proposal is being assembled that will define the scope and approach for a suite of reference adsorbent materials. In the proposal, the IAS will recommend the materials to comprise the suite of reference adsorbents; identify industrial donors of the recommended materials; and conduct round robin testing using NIST statistical design and reference materials handling expertise. The final

suite of adsorbents will be issued as reference materials through NIST's SRM program.

### **STANDARD AIDS STUDY OF VITAMINS, CANCER PREVENTION**

A reissued NIST Standard Reference Material will help researchers and clinical laboratories accurately measure nutrients that may play a role in cancer prevention. NIST Standard Reference Material 968b, Fat-Soluble Vitamins and Cholesterol in Human Serum, includes certified concentrations of eight analytes, including vitamins A and E, beta-carotene, and cholesterol. Non-certified values are given for 10 additional analytes. The SRM includes three vials of freeze-dried human serum, each containing a different concentration level for vitamins A and E, and beta-carotene. NIST chemists developed the new SRM as part of an ongoing quality assurance program with the National Cancer Institute. Cancer researchers are conducting numerous studies on the role of anti-oxidant vitamins in preventing cancer. NIST's SRM will help researchers verify that their measurements are accurate. This SRM also can be used for assigning values to in-house control materials and to ensure proper interlaboratory interpretation of data. NIST has been helping NCI with analytical methods development and interlaboratory quality assurance since 1984. For ordering information, contact the NIST Standard Reference Materials Program, Rm. 204, Building 202, Gaithersburg, MD 20899-0001, (301) 975-6776, fax: (301) 948-3730, e-mail: srminfo@enh.nist.gov.

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## **Standard Reference Data**

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### **FIRST DATABASE FOR ATOMIC SPECTROSCOPY AVAILABLE**

The Database for Atomic Spectroscopy, the first computerized reference source of its kind, will give physicists, chemists, astronomers, geologists and industry researchers easy access to the most comprehensive collection of accurate and critically evaluated spectra available electronically. The DAS is now available from NIST's Standard Reference Data Program. It contains wavelengths, transition probabilities and energy levels for most ionization stages of 28 elements from hydrogen to nickel. Every element has signature spectral lines that scientists can use to identify and quantify elemental composition in nature, space or industrial research. The

DAS is an easy-to-use, menu-driven database designed for personal computers. Users can conduct searches for specific wavelengths, energy levels or elements. They also can change the default options for units, output and other parameters as desired. The first version of DAS contains 27 000 spectral lines (from 0.1 nm to 20 000 nm in wavelength) and 45 000 energy levels. Single copies of the DAS are available for \$215. For more information or to order the DAS, contact the SRDP, NIST, A320 Physics Building, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: (301) 926-0416, e-mail: srdata@enh.nist.gov.

### **NIST COMPLETES PROPERTY DATABASE ON HIGH TEMPERATURE SUPERCONDUCTORS**

Standard Reference Database 62: High Temperature Superconductors, Version 1.0, has been completed at NIST. This database provides evaluated thermal, mechanical, and superconducting property data for the class of materials commonly called high temperature superconductors. The current range of materials covers the major series of oxide compounds derived from the Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O, Tl-Sr-Ca-Cu-O, and La-Cu-O chemical families, along with numerous other variants of the cuprate and bismuthate materials that are known to have superconducting phases. The materials are described by specification and characterization information that includes processing details and chemical compositions. Physical characteristics such as density and crystal structure are given in numerical tables. All measured values are evaluated and supported by descriptions of the measurement methods, procedures, and conditions. In all cases, the sources of the data are fully documented in a comprehensive bibliography. A software interface to the database is provided for use on personal computers.

### **NIST/NASA/CARB CRYSTALLIZATION DATABASE AVAILABLE ON-LINE**

Crystal data and crystallization conditions for biological macromolecules are now available "on-line" to scientists working in x-ray crystallography. NIST Standard Reference Database 21, the NIST/NASA/ CARB Biological Macromolecule Crystallization Database (BMCD), has been redesigned by researchers at CARB, working in collaboration with the NIST Standard Reference Data Program, for on-line access over the Internet. The BMCD contains summary information, crystal data, crystallization conditions, and complete references for each biological macromolecule that has been crystallized in a form suitable for single-crystal

x-ray diffraction studies. The data are used by crystallographers to develop crystallization strategies for newly discovered biological macromolecules and to reproduce crystallization protocols for existing molecules. Recently, the BMCD was expanded to include protocols and results of crystallization experiments undertaken in space. These data are comprised of entries from the NASA Protein Crystal Growth Archive, NASA-sponsored protein crystal growth experiments, and other microgravity crystallization studies sponsored by international organizations.

The BMCD provides a sophisticated user interface that allows browsing, searching, downloading, and displaying the data. The alpha-test, on-line version of the BMCD is available at the URL address (<http://ibm4.carb.nist.gov:4400/carb.html>). Since initial testing began in February, the on-line BMCD has been accessed more than 80000 times by more than 1200 institutions worldwide. More than 1200 users have transferred data from the database to their own computers, and more than 600 users have taken data multiple times.