

# 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, Administration Building, A635, Gaithersburg, MD 20899; telephone: 301/975-3572.*

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### **ELECTRICAL POTENTIAL: ISRAEL, UNITED STATES AGREE ON STANDARD**

The unit of electrical potential, the volt, is the same in Israel and the United States, according to an agreement recently signed by representatives of the two countries' national standards laboratories. The agreement formally recognizes that the U.S. volt, as measured by NIST, and the Israeli volt, as measured by the Israeli National Physical Laboratory, are equivalent to at least one part in 10 million. Both countries measure volts in accord with the definitions of the International System of Units (SI). The agreement between NIST and INPL is the latest in a series of NIST collaborations with international standards organizations in order to facilitate trade.

### **CRADA PARTNERS FOCUS ON IMPROVING LASER LENSES**

A significant obstacle blocking the development of new, more powerful lasers may soon be removed, thanks to a collaborative effort between NIST and a private company. In recent years, physicists have been trying to generate more focused laser beams through techniques such as frequency doubling, frequency summing, and optical parametric amplification. These techniques would be more efficient if researchers could change the geometry of the

beams using cylindrical lenses for improved focusing. However, there currently is no way to measure lens quality and determine whether a beam is focused properly. The private company has produced an instrument which can perform such measurements by characterizing a beam's integrated errors. Representatives from the private company will work with a NIST physicist at the Joint Institute for Laboratory Astrophysics, to explore the use of this machine for evaluating cylindrical and other non-spherical lenses for applications in non-linear optics. For more information, contact Linda Wilbourn at (916) 888-5017 or John Hall at (303) 492-7843. JILA is jointly operated by NIST and the University of Colorado at Boulder.

### **NIST OBSERVES ONE ELECTRON IN 10 BILLION**

For more than 2 decades, advances in superconductivity have been made by studying the behavior of bound pairs of electrons. But if there is an odd number of electrons, what does the unpaired, "odd-man out" do? NIST researchers have taken the first step in answering this question. NIST scientists and a guest worker from the Center for Nuclear Studies, Saclay, France, are attempting to count individual electrons in order to improve electrical standards for capacitance or current. An early success for the project is the detection of a single excited electron above a condensed ground state of 10 billion superconducting electrons. To make their discovery, the researchers used an electrometer composed of two ultra-small tunnel junctions on a superconducting "island" flanked by two normal-state outer electrodes. The dimensions of the "island" were used to determine the 10 billion figure for the number of superconducting electrons involved.

### RESEARCH PARTNERS SEEK BETTER INFRARED RADIOMETRY

NIST and a private research company are working together to improve infrared radiation measurements for the aerospace and defense industries. These measurements ensure accuracy in sensors from simple light meters to complex missile detectors. Under terms of a recently signed cooperative research and development agreement, the private company will provide data enabling NIST scientists to improve the institute's Low Background Infrared Calibration Facility. The facility calibrates blackbodies, objects which absorb all incident radiation and reflect none. At a fixed temperature, blackbodies emit radiant energy within a specific spectrum. Aerospace and defense manufacturers can calibrate infrared detectors with these constant radiant emissions, so the LBIR Facility serves as a site to check the accuracy of their measurements. Another part of the agreement involves a absolute cryogenic radiometer manufactured by a private company, present in the LBIR Facility. The device measures radiant energy in a chamber maintained at a temperature of 20 K. Personnel from the private company will assist NIST scientists in modeling how changes in the instrument may improve its future performance.

### CRADA PARTNERS TO EVALUATE ADVANCED INSULATIONS

Innovative insulation systems are being developed to replace foam insulations manufactured with chlorofluorocarbons and to meet more stringent energy-use standards. These insulation systems include vacuum powder-filled panels, low-conductivity gas-filled panels, and vacuum insulation panels. However, current test methods cannot properly measure their thermal resistance or R-value. Under a new cooperative research and development agreement, NIST and a private U.S. company are collaborating on the creation of the necessary thermal resistance tests. The company will supply NIST researchers with evacuated insulation panels of varying sizes and materials. The project is expected to take about 3 years. For more information, contact Hunter Fannery, B322 Building Research Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5864.

### STANDARD ADDRESSES CNC PERFORMANCE EVALUATIONS

Consistent and reliable performance by computer numerically controlled machining centers is critical in manufacturing products for the defense, aero-

space, automotive and other industries. With assistance from NIST, a new standard for assessing the effectiveness of CNC machining centers has been developed by the American Society of Mechanical Engineers. The standard, "Methods for Performance Evaluation of Computer Numerically Controlled Machining Centers" (ASME B5.54-1992), establishes requirements and methods for specifying and testing machining centers. It also attempts to facilitate performance comparisons between machines by unifying terminology, general machine classification and the treatment of environmental effects. For technical information on the standard, contact Denver Lovett, Chairman, ASME Standards Committee B5, 136 Shops Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3503. A printed copy of ASME B5.54-1992 may be obtained by writing ASME, 22 Law Drive, Fairfield, NJ 07007, or by calling (800) 843-2763. Cost is \$44 prepaid; order by N14492.

### INTEGRATED OPTIC LASER EMITTING AT 905, 1057, AND 1356 nm

NIST scientists, working with private industry researchers, have developed a new integrated optic laser. NIST and the private industry have signed a cooperative research and development agreement to develop new integrated optic lasers and amplifiers for optical telecommunication networks. Another NIST scientist is providing materials characterization. The joint research has already produced the first neodymium integrated optic laser that produces light at 905 nm. The laser emits over 200 mW of light at 1057 nm, which is the highest power achieved in such a device. One of the scientists reported these results in a post-deadline paper at the Optical Society of America Advanced Solid State Lasers Topical Meeting held early in February. The private company has filed a patent application for the glass used in the laser. This application includes a disclosure of the joint NIST/private company laser and protects the foreign and domestic patent rights of both NIST and the company to this device. A joint patent application for the laser is in progress.

### TRAPPED NEUTRAL ATOM METHODOLOGY APPLIED TO HIGH-ACCURACY WAVELENGTH REFERENCE FOR OPTICAL COMMUNICATIONS

NIST scientists are pursuing the application of techniques for cooling and trapping neutral atoms to the development of high-accuracy wavelength standards. NIST played a major role in the develop-

ment of the cooling and trapping methodology, and this wavelength standard work is one of the first applications for trapped neutral atoms. Accurate wavelength standards are important for many of the proposed optical communication schemes involving NIST multiplexing. A goal of the NIST research on optical communication wavelength standards is to produce a highly stable and reproducible primary wavelength reference in the 1.5  $\mu\text{m}$  fiber laser stabilized to narrow resonances in laser-cooled rubidium. In the experiments, neutral rubidium atoms were laser-cooled and confined in a vapor-cell optical trap using 780 nm diode laser light and a weak magnetic field gradient. This arrangement produced a dense sample of cold atoms and reduced the Doppler broadening of the optical transitions to less than the natural linewidth. Spectroscopy of the rubidium  $5P_{3/2} \rightarrow 4D_{5/2}$  transition near 1.529  $\mu\text{m}$  was performed using a single-longitudinal-mode erbium-doped fiber laser. Transition linewidths of 10 MHz were observed, and the fiber laser was actively stabilized to the rubidium line.

#### **INDUSTRY, NIST COLLABORATE TO STUDY OVERLAY MEASUREMENTS**

Private industry and NIST are collaborating to study overlay measurements. The private company is making space available for a NIST test structure in a “drop-in” test chip to be fabricated along with product on wafers at its Manassas, VA, facility. NIST is supplying the software to produce the test structure and conducting measurements on completed test chips and analysis of the resulting data. Determining accurate registration of multiple lithography steps in semiconductor fabrication is a major problem as minimum device sizes continue to shrink: the Semiconductor Industry Association views overlay as “expected to continue to be the most difficult technical challenge in lithography” through the year 2007. The Modified Offset Alignment Test Structure (MOATS) used in the work was developed by NIST to provide extremely precise measurement ( $\sim 5$  nm) of relative locations of parallel features on different mask layers of an integrated circuit wafer.

NIST recently received for evaluation several 100 mm CMOS/bulk wafers from the private company containing the chip. These wafers are unique in that they were fabricated using a “mix-and-match” x-ray lithography technique. Two dimensionally critical layers, diffusion and polysilicon, were patterned using the state-of-the-art x-ray synchrotron source at the private company. All

other steps were performed using traditional photolithography techniques in Manassas. Also incorporated in the drop-in test chip (refers to sites on a wafer at which the product die can be replaced by a test chip) are several other optical and electrical overlay test structures which will allow the group to perform side-by-side comparisons of MOATS with competing techniques. NIST has applied for patent protection for the MOATS (Case Reference 91-0020), which takes the form of a so-called “sliding-wire” potentiometric structure.

#### **NIST PROVIDES LEADERSHIP FOR DARPA INFRARED DETECTOR MATERIALS PROGRAM**

A NIST scientist has been asked by the Defense Advanced Research Projects Agency (DARPA) to lead and coordinate the DARPA technical effort in optical characterization of infrared materials. NIST was chosen to lead this effort because of its demonstrated capability to perform high-accuracy semiconductor measurements, to produce Standard Reference Materials, and to function in an unbiased technical role. As one of the first steps, some 20 industry engineers and scientists participated in a workshop held early this year at NIST on techniques being developed for the noninvasive analysis of infrared materials. The participants represented the major contractors in the program. The workshop helped to identify specific metrology needs and requirements; provided a working review of the optical techniques in use such as photorefectance, photoluminescence, and infrared absorption; and identified needs for measurement criteria, calibration procedures, and analysis techniques. The participants placed considerable emphasis on needs for improvements in the absolute accuracy of measurements. In related efforts, NIST is working with DARPA to advance the state of the art in nondestructive analysis and to develop new characterization techniques to address future infrared material needs.

#### **YOUNG'S INTERFERENCE EXPERIMENT USING TWO ATOMS**

NIST scientists in collaboration with the Universität Freiburg, have reported the first observation of interference effects in the light scattered from two trapped atoms. The visibility of the fringes can be explained in the framework of Bragg scattering by a harmonic crystal and simple “which path” considerations of the scattered photons. If the light scat-

tered by the atoms is detected in a polarization sensitive way, then it is possible to demonstrate selectively the particle nature or the wave nature of the scattered photons. This is a very vivid demonstration of principles underlying the foundations of quantum mechanics.

The experiments are performed with two trapped  $^{198}\text{Hg}^+$  ions. The separation of the ions can be adjusted by changing the potential well of the trap. "Which path" a given photon takes in the experiment (that is, which atom scatters the photon) can, in principle, be determined when  $\sigma$ -polarized light is detected, since a photon with this polarization, when scattered, leaves one of the atoms in a different state. Therefore, when  $\sigma$ -polarization light is detected, no interference is seen because the experimental conditions present the opportunity to determine the photon path. On the other hand, the detection of  $\pi$ -polarized light involves a transition where the initial state and final state of the atom are identical. There is no way to determine which atom scattered a given photon, and in this case interference is observed.

#### **HOLOGRAPHIC MEASUREMENTS OF ATOMIZATION AND SPRAY FORMING**

Scientists at NIST recently applied advanced holographic techniques to measure key features in atomization and spray forming processes. The velocities and sizes of droplets formed from the break-up of a molten metal stream of a nickel-based superalloy, Inconel 625, interacting with high-velocity argon gas in the SIGMA system (supersonic inert gas metal atomizer) were investigated. This work was done in collaboration with a private company, which is developing special holographic systems under an SBIR grant from DOC.

Double-pulse techniques with laser pulses, each of 20 ns duration, separated by 3  $\mu\text{s}$  in time allowed direct measurement of droplet velocities soon after droplet formation. This process required superimposing two holographic pictures onto a single holographic plate. Velocities in the range of 50 to 90 m/s were measured for droplet sizes in the range of 20 to 100  $\mu\text{m}$ . Three-dimensional visualizations were obtained both during the initial droplet formation period and near the time of droplet impact onto a substrate.

#### **AMRL SUPPORTS THE METRIC CONVERSION OF AASHTO MATERIALS STANDARDS**

The American Association of State Highway and Transportation Officials (AASHTO) is actively pursuing the conversion of its documents to use the International System of units (SI units). This is in coordination with federal government agencies who are implementing Section 5146 of the Omnibus Trade and Competitiveness Act of 1988. The AASHTO Materials Reference Laboratory (AMRL) located at NIST, in its role of providing technical support to the AASHTO Subcommittee on Materials, has completed the task of converting to SI 416 standards for construction materials. These documents will be reviewed by the responsible technical sections of the subcommittee and processed as AASHTO standards. This AMRL support to the AASHTO standards process will speed the conversion and ensure consistency in bringing the standards into compliance with the act.

#### **NEW SRM FOR CALIBRATING INFRARED SPECTROPHOTOMETERS**

Spectrophotometers are instruments that spectrally characterize the optical properties of materials. To obtain useful information from these instruments, both the photometric and wavelength (or frequency) scales must be calibrated. Standards are currently available that allow spectrophotometer users to perform such calibrations in the ultraviolet, visible, and near-infrared spectral regions. However, standards have not been readily available for the mid-infrared (IR) spectral region (2–20  $\mu\text{m}$ ), in which large numbers of instruments are currently utilized.

Recently, NIST has developed a polystyrene Standard Reference Material (SRM) for calibration of the wavelength scale of spectrophotometers operating in the mid-IR. The SRM provides seven primary reference wavelengths, each with a total uncertainty of less than  $0.5\text{ cm}^{-1}$  for the calibration of Fourier transform infrared instruments. In addition, six more secondary wavelengths, each with a total uncertainty of  $\approx 1\text{ cm}^{-1}$ , are provided.

### **NIST SPONSORS MAMMOGRAPHY WORKSHOP**

NIST held a workshop recently to investigate the issues involved in providing a kilovoltage (kV) standard for diagnostic x-ray systems used in mammography. Mammographic x-ray units operate at a peak voltage of 24 to 32 kV, depending upon the thickness of the compressed breast. The relative attenuation of soft tissue is greater for low-energy x rays, therefore providing more contrast between subtle differences in breast densities. This contrast is needed for detecting calcifications and soft-tissue abnormalities. The need for a kV standard in mammography exists because a change of as little as 1 kV at these low energies affects mammographic film quality, as well as radiation exposure.

Participants in the workshop included representatives of the Center for Devices and Radiological Health of the USFDA, state regulatory agencies, manufacturers of x-ray equipment and kV measuring devices, secondary calibration dosimetry laboratories, and both U.S. and German national standards laboratories. The workshop focused on the present status of, and the impact of future developments on, kV measurements in commercial mammography systems. Kilovoltage measuring field devices were reviewed, as well as the approaches used in laboratories to ensure the quality of the field measurements. Accurate measurement of the kilovoltage is a key step toward improving the quality of diagnostic radiology for the 26 million mammograms taken annually in the United States.

### **THERMAL CONDUCTIVITY OF HCFC-FILLED POLYMER FOAMS**

Polymeric foams expanded with chlorofluorocarbon (CFC) gas are used extensively as thermal insulators in applications ranging from refrigerators and freezers to cryogenic-fuel tanks. New insulation materials are being developed that use more environmentally acceptable hydrogenated chlorofluorocarbon (HCFC) gases. A program to determine the thermal resistance of these foams as a function of temperature, 20 to 330 K, and density, 34 to 50 kg/m<sup>3</sup>, has been completed at NIST. The effort was supported by a private company and was executed with the aid of a guest researcher. Early results indicate that the temperature dependence of the HCFC-filled foams is similar to that for CFC-filled foams, as expected. This characteristic dependence is a linear rise from nearly zero conductivity at 0 K to a local maximum

at about 220 K, then a drop to a local minimum at 273 K, followed by a linear rise at higher temperatures. The magnitude of the conductivity of the HCFC foams is approximately 5 percent higher than that for CFC foams of the same density and matrix material.

### **COMPUTER GRAPHICS METAFILE GENERATOR TEST SERVICE INITIATED**

In January 1993, NIST expanded its Computer Graphics Metafile (CGM) Validation Test Service to include CGM generator testing. Focusing on metafile conformance testing since its inception in 1991, the service now tests either metafiles or generators for conformance to Federal Information Processing Standard (FIPS) 128, Computer Graphics Metafile (CGM), and the CALS CGM Application Profile (MIL-D-28003A).

Metafiles testing consists of analyzing binary encoded CGM files and verifying that the data stream is syntactically correct. A certificate of validation is issued for conforming metafiles. The cost of testing CGMs varies with the number of metafiles tested. Generator testing uses a NIST-developed test suite from which a set of binary encoded CGMs is generated. The CGMs are analyzed for syntactic and semantic correctness as well as preservation of graphical primitives. Conforming generators receive a certificate of validation. If errors are detected during the validation, a registered report is issued. The base price for generator validation is \$9,000.

All certificates and registered reports are listed in the Validated Products List, which NIST issues quarterly; NISTIR 5103, dated January 1993, is the current publication. Also available is a CGM information pack, which describes the test service and gives pricing information.

### **NIST/ASA/NSF FELLOW DEVELOPS NEW MODEL FOR CONTROLLING MEASUREMENT PROCESSES**

A visiting NIST research fellow has developed a computationally intensive surveillance scheme geared toward quick detection of medium-size changes in ongoing processes. In contrast, Shewhart-type control schemes are geared toward detection of large changes, and CUSUM procedures, while optimal in terms of speed of detection, cannot handle dependent data as easily. Furthermore, the new scheme has the advantage that the baseline for the process need not be known because

nuisance parameters, such as the mean and variance, are eliminated via exploitation of invariant structures inherent in the problem. This work, which included a successful application to data from the mass calibration process at NIST, was performed under the NIST/ASA/NSF Fellowship Program, administered by the American Statistical Association.

#### **NIST PUBLISHES IGES HYBRID MICROCIRCUIT APPLICATION PROTOCOL**

NIST has published Technical Note 1295, Initial Graphics Exchange Specification—Hybrid Microcircuit Applications Protocol. This document defines how users, such as developers of design and manufacturing tools, supporting design, production, and marketing functions, are to encode hybrid product data in the Initial Graphics Exchange Specification (IGES) format for exchange among computer-aided design systems and manufacturing systems designed to run in a numerically controlled mode directly from digital data. It also presents proposed extensions to the IGES standard. The use of a neutral data format such as IGES serves as a data “hub” and greatly reduces the number of translators required to interpret data from one system to another, from a maximum of  $n(n-1)$  to a maximum of  $2n$ , where  $n$  is the number of systems. Publication of the document culminates a 3 year standardization effort led by NIST within the Navy MicroCIM program. Industry involvement throughout the MicroCIM program has led to the development of an exchange specification that can be efficiently mapped to a variety of computer-aided design systems in use. Three public forums were held, which resulted in the selection of IGES as the target standard for data representation and exchange. Driven by industry needs, the scope of the application protocol was defined to include information needed for hybrid documentation and manufacturing automation, including relevant military specifications and data required by systems used to design and fabricate hybrids. NIST expects this application protocol to form part of the underlying methodology needed by the electronics industry in the evolution of fully automated design and manufacturing systems for complex products. Technical Note 1295 constitutes the second IGES application protocol and the first one developed supporting electronics. It is available from the National Technical Information Service (order PB93-175404/AS).

#### **IMPROVED METHOD DEVELOPED FOR CHARACTERIZING HgCdTe INFRARED DETECTORS**

NIST scientists have developed a new method for measuring and analyzing electrical parameters of passivated layers of HgCdTe infrared detectors. Among other applications, these detectors are the “eyes” of weather satellites such as the National Atmospheric and Oceanic Administration’s Geostationary Observational Environmental Satellites (GOES) series. The method requires access to only the two terminals of each detector element and provides a unique signature. Commercial suppliers of detectors use various methods to passivate the top and bottom surfaces of a detector to produce the thin accumulation layers needed for high performance and stability. The density and mobility of electrons in these layers directly affect the performance of the detector. The method involves exposing a detector to a high magnetic field, which causes splitting of energy levels, in turn leading to detectable quantum magnetotransport phenomena, such as Shubnikov-de Haas oscillations in the magnetoresistance of the accumulation layers. Carrier densities are proportional to the frequencies of these oscillations, periodic as a function of inverse magnetic field. The mobilities are proportional to the relaxation times, which can be obtained from the field dependence of the amplitude of the oscillations. Electron effective masses can be obtained from the temperature dependence of the amplitude of the oscillations.

The scientists found excellent agreement between theoretical predictions and empirical data for a detector having anodically oxidized surfaces. However, they found poor agreement for detectors having heavily accumulated surfaces produced by differing passivation processes. The predicted effective masses were about twice the measured values, and the relaxation times, normally assumed constant below 30 K, were found to vary with temperature. Correcting for this variation brought the measured masses into agreement with the predictions. The scientists suspect that the passivation processes damaged the surfaces.

#### **NIST DEVELOPS IMPROVED SENSOR FOR HIGH AC AND PULSED CURRENTS**

A NIST scientist has conceived, implemented, and tested a new design of a sensor for high currents in the form of a machinable Rogowski coil. His design addresses a principal factor limiting accuracy in

conventional Rogowski coils, sensitivity to position of the current-carrying conductor. Rogowski coils are air-core mutual inductors having mutual inductance of  $1 \mu\text{H}$  or less and are commonly used to measure very high ac and pulsed currents, generally greater than 1 kA. It is not unusual for the output of commercially available coils to vary several percent with different positions. They also suffer from relatively large temperature coefficients. Although these coils find application where accuracy requirements are not demanding, coils with much better performance are needed. The new machinable Rogowski coil has extremely low positional sensitivity, on the order of  $\pm 0.05$  percent, and a temperature coefficient of only  $+9 \text{ ppm}/^\circ\text{C}$ . The coil is made by laser scribing a coil-turns pattern on a silver-coated precisely machined ceramic torus. Turns symmetry is obtained by the machining process and the compensation turn required in a single-layer Rogowski coil can be precisely located. The coils' excellent symmetry and precisely located compensation turn result in its improved performance in comparison with other Rogowski coils. The coil has a nominal mutual inductance of  $0.265 \mu\text{H}$  (a reactance of  $100 \mu\Omega$  at a frequency of 60 Hz).

#### **INDUSTRY FOCUSES ON TRACEABILITY NEEDS AT NIST/DOE GEAR METROLOGY WORKSHOP**

A workshop was held recently to discuss the development of an improved national infrastructure for gear metrology. The workshop, which was held in Oak Ridge, TN, was attended by some 50 representatives from business and government agencies. One purpose of the workshop was to focus on the needs of the gear industry regarding the re-establishment of traceability to NIST for dimensional measurements of precision gears.

As a result of a previous NIST workshop which identified the need for NIST-traceable gear measurements, NIST and DOE have entered into a collaboration to provide new traceability of gear measurements to NIST through a joint NIST/DOE center being developed at the DOE Y-12 Facilities in Oak Ridge. The 2 day gear workshop gave potential users of the center the opportunity to make their needs known and to interact with industry, university, DOD, Y-12, and NIST principals in gear manufacturing and measurement. A major result was agreement by participants to establish an ad hoc industry-driven council on gear metrology. This council will be charged with developing prior-

ity gear measurement needs to be considered by NIST and Y-12 as part of the proposed gear metrology program.

#### **PHOTOPATTERNING OF ALKYLTHIOL SELF-ASSEMBLED MONOLAYERS**

Self-assembled monolayers (SAMs) currently represent the most well-defined organic thin-film system for controlling the molecular architecture of surfaces and interfaces. The ability to control the spatial distribution of these molecular monolayers on surfaces is expected to impact such diverse areas as biosensing, immunoassay diagnostics, DNA sequencing, and molecular electronics. NIST scientists have developed a new, versatile method for photopatterning an alkylthiol SAM on gold and silver surfaces. The patterning method is derived from two recent studies which showed that: 1) adsorbed alkylthiolate molecules ( $\text{RS}^-$ ) in the monolayer are oxidized to the corresponding alkylsulfonate ( $\text{RSO}_3^-$ ) when they are uv irradiated in air; and 2) alkylsulfonates are weakly bound to gold and silver surfaces and, thus, are easily displaced from the surface by subsequent immersion of the sample in an alkylthiol solution. The photopatterning method uses knowledge gained from these two observations.

A pattern of alkylsulfonates is first formed in a SAM by uv irradiation through a mask. The sample is then immersed in a dilute solution of a different alkylthiol. The alkylsulfonates in the exposed areas of the original SAM are displaced, incorporating the second type of alkylthiol into the monolayer. This results in a single monomolecular film composed of two types of alkylthiol in a pattern determined by the mask. Maps of the molecular composition of the films have been made by a NIST scientist. Secondary ion mass spectrometry (SIMS) images confirmed the efficacy of the photopatterning strategy. The advantages of the SIMS method are that physical contact with the sample is not required, and it is generally applicable to mono-layers of any alkylthiol molecule. Pattern features of approximately  $10 \mu\text{m}$  have been demonstrated, and work is continuing to determine the ultimate resolution of the photopatterning method.

#### **ATMOSPHERIC SAMPLING IN SAUDI ARABIA**

NIST and the Saudi Arabian Ministry of Defence and Aviation (MODA) recently collaborated to collect ambient particulate and gas samples at several

locations in Saudi Arabia. The interest grew from the recent degradation of air quality in the region as a result of the Kuwaiti oil well fires. The sampling team consisted of two NIST researchers and three MODA personnel. The Saudis were trained in collection methods at a 2 week Ground-Based Smoke Sampling Techniques Training Course held at NIST in August 1992.

The team collected samples to demonstrate the operation of portable gas and particulate-sampling instrumentation, including gravimetric measurements, light-scattering cells, size classifiers, battery-powered gas analyzers, and filter sampling trains. The aerosol samples will be used to determine the particulate-size distribution, inhalable particulate fraction, and total particulate mass concentration. Filter and sorbent tube samples will be analyzed to determine polycyclic aromatic hydrocarbon concentrations. At each collection site, the concentrations of carbon dioxide, carbon monoxide, hydrogen sulfide, nitrous oxide, and oxygen were monitored. Approximately half of the samples will be analyzed by the Saudi personnel in Saudi Arabia, while the others were returned to NIST for analysis. Some of the NIST particulate samples will undergo more specialized examination, including electron microscopy and laser microprobe analysis.

#### **COLLABORATIVE RESEARCH ELUCIDATES CARBON MONOXIDE AND SOOT OXIDATION**

A joint effort by a NIST scientist and university researchers has explained why soot, always produced inside luminous flames, does not always escape as smoke. The group measured the concentrations of OH radicals in the soot oxidation regions of diffusion flames of methane, a methane/butane mixture, and a methane/1-butene mixture in air at atmospheric pressure. The large, super-equilibrium values observed in the high-temperature reaction zones in the absence of soot particles are greatly reduced in the presence of soot. Their computations of the soot oxidation rates using the concentrations of the pertinent flame species showed that this diminution is primarily attributable to OH reaction, with O<sub>2</sub> making only a small contribution, and their comparison of the soot and CO oxidation rates showed that the soot (with its high-collision efficiency with OH) successfully competes with CO for OH. Thus, large soot concentrations are often accompanied by significant CO emissions. A paper on this research has been submitted to *Combustion and Flame*.

#### **NIST SUPPORTS COMPUTER-AIDED ACQUISITION AND LOGISTIC SUPPORT (CALS) INITIATIVE IN COMPUTER GRAPHICS**

In its continuing support of the Department of Defense CALS program, NIST developed a detailed design specification for determining conformance of Computer Graphics Metafile (CGM) interpreter products to the requirements of Federal Information Processing Standard (FIPS) 128, CGM, and the Military Specification MIL-D-28003A. In a three-part effort, researchers first initiated a conformance testing service for CGM metafiles in May 1991. The next phase focused on developing procedures for the testing of CGM generator (writer) products to verify that a product produces conforming metafiles that accurately and correctly define the intended picture; the CGM generator product conformance testing service is scheduled to begin soon.

The last part of the total CGM conformance testing environment is to ensure that a CGM interpreter (reader) product can correctly and completely parse an CGM file and produce the intended picture. NISTIR 5146, Detailed Design Specification for Conformance Testing of Computer Graphics Metafile (CGM) Interpreter Products, describes the CGM interpreter product testing program and makes recommendations for further CGM testing in support of CALS.

#### **SPOKEN LANGUAGE TECHNOLOGY AND APPLICATIONS DAY HELD**

Recently NIST cooperated with the Defense Department's Advanced Research Projects Agency (ARPA) to sponsor a showcase event aimed at unveiling spoken language understanding technology. Held at the National Academy of Sciences, the event attracted more than 250 attendees from government, industry, and academia. The goal of the seminar was to catalyze the rapid transfer of the technology into real human-computer interaction systems, including government and military applications. Participating in ARPA spoken language research programs since 1984, NIST developed the benchmark tests that have been used to track the progress of spoken language technology.

#### **"TRAPPED" IONS PROVIDE FIRST VIEW OF LIGHT PROPERTY**

In an experiment that demonstrates the wave-particle duality of light, NIST scientists reported in the April 19 *Physical Review Letters* that they had



made the first observation of interference effects in light scattered by two trapped atoms. Quantum theory states that light can act either as a wave or as a particle. Researchers at NIST surmised that if two or more atoms are localized (kept relatively still and close to each other), polarized laser light striking them would be absorbed and then re-radiated by each. Then, a polarization-sensitive analyzer would detect the re-radiated light and measure how the photons (packets of light) interfere. This would indicate if the photons were acting like particles or waves. Most other experiments of this type were unable to adequately localize atoms to within 50 nm (a quarter length of a light wave), and therefore were unable to observe this phenomenon. The NIST team was able to trap two mercury ions in an electromagnetic “well” and selectively demonstrate both the particle nature and the wave nature of the photons. Since publication of the paper describing the experiment, the researchers have observed the interference pattern of three ions. For a reprint of the Physical Review Letters article, contact Sarabeth Moynihan, Div. 104, NIST, Boulder, CO 80303-3328, (303) 497-3237. Ask for paper no. 12-93.

#### **PARTNERS PLAN DEVICE FOR CLEARER PROTEIN IMAGES**

The Center for Advanced Research in Biotechnology (CARB) and a Rockville, MD firm, are collaborating on a new system to simplify the study of three-dimensional shapes of protein molecules. Defining these complex structures helps pharmaceutical companies design more effective drugs. The new imaging system will be developed under a 2-year cooperative research and development agreement, with support coming from the National Institute of General Medical Sciences. Current imaging techniques involve bombarding a protein crystal with x rays and using the resulting diffraction patterns to decipher its three-dimensional structure. CARB and the Rockville firm seek to develop and test a prototype system that will collect x-ray diffraction patterns more accurately and quickly than commercially available methods. CARB was established in 1984 by NIST, the University of Maryland and Montgomery County, MD, as a unique center for government, academic and industry scientists.

#### **CRADA SEEKS BETTER FACTORY SYSTEMS INTEGRATION**

Despite years of product development, truly modular and flexible integrated manufacturing systems are still not prevalent in U.S. industry. Problems include the inability of factories to share information on engineering, production management and control systems, as well as a lack of standards specifying the interactions between such systems. Addressing these problems is the goal of a recently signed cooperative research and development agreement between NIST and a private company. The multiyear collaboration involves the development of computer-aided process planning and integrated manufacturing control systems technology. Specifically, the partners will design or implement information models, communications protocols and prototype software. For more information, contact Steven Ray, A127 Metrology Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3524.

#### **U.S./HUNGARIAN TEAM TO REFINES pH STANDARDS**

NIST and its sister agency in Hungary, the Hungarian National Office of Measures, have been awarded a grant to work toward establishing a more universal pH scale (the acid/base scale). The Board of the U.S.-Hungarian Science and Technology Joint Fund recently approved a 3-year grant for studies on pH and electrolytic conductivity. The grant formalizes more than a decade of cooperation between scientists at NIST and the Hungarian National Office of Measures. Chemists in the two agencies plan to assess similarities in U.S. and Hungarian pH and conductivity standards. Their work will form a foundation for an improved international pH scale and will refine measurements for international conductivity standardization. Such measurements are crucial to diverse industries such as foods, agriculture, medicine, fuel, materials, and semiconductors.

#### **ELEVEN INVENTIONS READY FOR LICENSING**

NIST recently announced that the following 11 government-owned inventions are now available for licensing:

- Intermetallic Ti-Al-Nb Alloys Based on Strengthening of the Orthorhombic Phase by Mega-type Phase (Docket No. 90-032);

- High-Speed, Amplitude-Variable Thrust Control (Docket No. 90-035);
- Automated Recognition of Characters Using Optical Filtering with Positive and Negative Functions Encoding Pattern and Relevance Information (Docket No. 92-004);
- Automated Recognition of Characters Using Optical Filtering with Maximum Uncertainty-Minimum Variance (Docket No. 92-005);
- Synthetic Perturbation Tuning of Computer Programs (Docket No. 92-010);
- Method and Materials for the Assay of Several Classes of Enzymes by Light-Scattering Techniques Using Substrate-Coated Colloidal Particles (Docket No. 92-011);
- A Procedure for Digital Image Restoration (Docket No. 92-028);
- Micro-Hotplate Devices and Methods for Their Fabrication (Docket No. 92-045);
- Temperature-Controlled, Micromachined Arrays for Chemical Sensor Fabrication and Operation (Docket No. 92-046);
- Application of Microsubstrates for Materials Processing (Docket No. 92-047); and
- Method and Apparatus for Precisely Measuring Accelerating Voltage Applied to X-Ray Sources (Docket No. 93-023).

For technical and licensing information on these inventions, contact Bruce E. Mattson, B256 Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-3084.

#### **WALL DESIGNERS GET HELP AVOIDING THE "DRAFT"**

Poorly designed or constructed exterior walls often leak air and moisture into and out of office buildings. This can lead to uncomfortable occupants, poor indoor air quality, increased energy consumption and even structural safety problems. NIST has developed a set of practical guidelines to help building designers understand air leakage and other thermal performance problems, and, more importantly, learn how to avoid them. The guide is divided into three easy-to-follow sections. The first explains how heat, air and moisture transfer between the inside and outside of a building. This section also explains where and how defects can

occur, and how they can be prevented. The second describes basic design principles and techniques for avoiding problems. The third describes a variety of wall systems and design features crucial to achieving good thermal performance. Envelope Design Guidelines for Federal Office Buildings: Thermal Integrity and Airtightness (NISTIR 4821) is available from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650 for \$27 prepaid. Order by PB 93-183770.

#### **NIST INTENDS TO GRANT EXCLUSIVE DSA LICENSE**

In a June 8 Federal Register notice, NIST announced its intent to grant an exclusive worldwide license to a private company for the "Digital Signature Algorithm." NIST has filed a patent application for the algorithm, which forms the basis for a proposed federal information processing standard that will allow federal agencies to verify the integrity of electronic data and the sender's identity. The proposed standard, known as the Digital Signature Standard, adopts a system that uses mathematical formulas to create and verify a digital value called a signature. The prospective license is expected to resolve a patent dispute with the private company concerning the algorithm. The company intends to permit royalty-free use of the algorithm for noncommercial purposes and by U.S. federal, state, and local governments.

#### **AEROSPACE ALLOYS CONSORTIUM LAUNCHED**

Seven manufacturers, seven universities, three federal agencies and one industry technical society have committed to join an industry/government program to improve the precision casting of metal alloys commonly used in the aerospace industry. The 18-member consortium will be the largest cooperative research and development program in materials at NIST. The overall goal of the program is to plan and undertake research on high-technology casting so that the planning, research, and technology transfer can be coordinated and brought quickly to manufacturers. The consortium is a cooperative, distributed effort by participants; research will be carried out in-house by members and by NIST. The American Foundrymen's Society will represent the U.S. casting industry. For information, contact Robert J. Schaefer, Office of Intelligent Processing of Materials, B344 Materials Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5727.

**CONSORTIUM TO ADVANCE POLYMER BLENDS PROCESSING**

Four manufacturers that produce and use engineering resins and a U.S. national laboratory are joining NIST in a cooperative research and development program to improve the processing of polymer blends and alloys. The consortium is sponsored by NIST to help the U.S. polymers industry retain its technological lead in the international marketplace. The goal of the consortium is to use NIST measurement tools to develop the data and processing models industry needs to produce new and more economical resins. New information will enable producers to make critical in-process measurements that are not now possible. For information, contact H. Thomas Yolken, Office of Intelligent Processing of Materials, B344 Materials Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-5727.

**CRADA MAY IMPROVE MICROWAVE MEASUREMENTS**

A long-standing problem for measuring microwaves has been providing a meaningful traceability between NIST's primary (six-port) microwave measurement system and automated vector network analyzers used extensively in government and private industry. Existing methods require numerous connections to the six-port system; these connections are time consuming and a major source of measurement error. A new NIST cooperative research and development agreement with a private company calls for the partners to use programmable multistate tuners as a transfer standard. This requires only one connection and should reduce significantly the amount of operator effort needed. NIST will initially evaluate the stability of the company's tuner and then work with the company to develop state-of-the-art statistical methods for uncertainty analysis. For more information, contact Bob Judish of NIST at (303) 497-3380 or Mike Fennelly of ATN Microwave at (508) 667-4200.

**ACTIVITIES REPORT HIGHLIGHTS 1992 FOR NIST LAB**

From measurement standards for nuclear medicine to ultra-precise time and frequency measurements for telecommunications, NIST provides U.S. industry with technologies that are vital to the nation's health, safety and economic competitiveness. Scientists at NIST frequently collaborate with industry, academia, and other government agencies. A

new report on the technical activities at one of NIST's principal laboratories summarizes these collaborations as well as its recent research and accomplishments. The Physics Laboratory Technical Activities Report for 1992 is available for \$36.50 (print) or \$17.50 (microfiche) prepaid from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650. Order by PB 93-178648.

**NEW CRADA TO IMPROVE ON-LINE CVD MONITORING**

Many manufacturers use a process known as chemical vapor deposition to fabricate semiconductor devices and other advanced crystalline materials. One such manufacturer and NIST are collaborating to develop an on-line monitoring system that would improve quality control and reduce defects during manufacture. This joint effort is the result of a recently signed cooperative research and development agreement between NIST and the private manufacturer. The partners are developing a system that will identify and measure metalorganics flowing into a CVD reactor. In the CVD process, metalorganics are reacted on a heated substrate to form semiconductor chips and other advanced electronics materials. Based on Fourier transform infrared spectroscopy, the system will provide the ability to continuously monitor the process. Such on-line monitoring will enable reactor operators to adjust the CVD system for optimum quality.

**REPORT REVIEWS 1992 ACHIEVEMENTS OF NIST LAB**

Whether the need is to find environmentally safe refrigerants or identify contaminants in semiconductor wafers, U.S. industry is increasingly turning to NIST for answers. One of the principal laboratories at NIST provides industry with a variety of collaborative opportunities in areas such as biotechnology, analytical chemistry, chemical kinetics, thermophysics, surface science and process measurements. A complete report covering this laboratory's scientific research and industry connections is now available from the National Technical Information Service. The report, Chemical Science and Technology Laboratory Technical Activities Report for 1992, number PB 93-173482, may be ordered for \$44.50 (print) or \$19.50 (microfiche) prepaid from NTIS, Springfield, VA 22161, (703) 487-4650.

**“HASH” STANDARD FOR DIGITAL SIGNATURES APPROVED**

Secretary of Commerce Ronald H. Brown recently approved a federal information processing standard that can be used to help verify the integrity of electronic data and the sender's identity. The Secure Hash Standard (FIPS Publication 180) is used to condense a long message or data file to 160 bits. A mathematical technique is then applied to this message “digest” to produce a digital signature. Like a handwritten signature, a digital one can be used to identify and authenticate the originator of the information and to verify that the information has not been altered after it is signed. A hashing function is used because it is faster and more efficient to sign the 160-bit digest than a message that could be thousands of bits. The standard is required for federal government use with the proposed Digital Signature Standard. Copies of FIPS 180 will soon be available from the National Technical Information Service, Springfield, VA 22161, (703) 487-4650.

**NEW ALGORITHM SHARPENS IMAGES, DAMPS NOISE**

A NIST mathematician has developed an improved algorithm for reducing fuzziness and blur in digital images. The procedure could potentially improve quality for a host of imaging applications, including medical diagnostics, astronomy, satellite mapping, industrial imaging, and low-light imaging. While not applicable to all possible sources of image blurring, the new algorithm is effective at reducing the very broad class of “Gaussian-like” blurs—such as those produced by atmospheric turbulence in a satellite photograph, or x-ray scattering in radiography. Older algorithms exist that technically reduce Gaussian blurring, but with a side effect. The same operations that reduce the blurring tend to magnify any random noise in the image—and there's always noise. Experiments on images that have been artificially blurred confirm theoretical predictions that the improved algorithm achieves better results than the older procedures, while sharply limiting the effects of noise. NIST has applied for a patent on the procedure.

**A STEP TOWARD COMPETITIVE “MADE-TO-MEASURE”**

A new cooperative research and development agreement could help make tomorrow's custom-designed clothing competitive with today's “ready-to-wear” garments. Specialists from NIST and the Fashion Institute of Technology have joined forces to develop an information model for made-to-measure pattern making that expresses pattern styles and individual body measurements in digital, standardized computer format. FIT, a research leader in the area of apparel design and manufacture, will provide NIST with data requirements for the representation of made-to-measure patterns. Four types of data will be considered for the informational model: two dimensional pattern data, grading information, anthropometric data and constraint information. NIST will develop the information model as a STEP (Standard for the Exchange of Product Model Data) application protocol. The model developed will be incorporated by NIST, FIT and a private company into STEP-based computer software that will modify patterns to reflect individual body measurements. NIST is working with clothing manufacturers and the Defense Logistics Agency to extend STEP into the apparel industry.

**PRIMER TELLS WHAT'S COOL (AND TRAPPED) WITH LASERS**

Scientists interested in a basic primer on laser cooling and trapping of neutral atoms will want a copy of a new paper by NIST and University of Colorado researchers. Laser Cooling and Trapping for the Masses describes the current techniques for cooling and trapping atoms, offers a history of the research, discusses recent simplifications made possible by the use of diode lasers and the vapor-cell magneto-optical trap, and speculates on future applications for this technology. These include improved atomic clocks, wavelength standards for optical communications, and solutions to problems in basic physics (such as a study of the fundamental weak interaction between quarks and electrons). For a copy of paper 22-93, contact Sarabeth Moynihan, Div. 104, NIST, Boulder, CO, 80303-3328, (303) 497-3237.

**EXAMINERS NEEDED FOR  
1994 BALDRIGE AWARD**

NIST is seeking examiners from all sectors of American business to review and evaluate applications submitted for the 1994 Malcolm Baldrige National Quality Award. Applicants for the award's board of examiners must be experts in quality management and capable of evaluating large/small manufacturing and service businesses. Those selected must take a three-day preparation course based on the Baldrige award examination items, the scoring criteria, and the examination process. The board of examiners currently has 265 quality experts. This number is expected to increase slightly for the 1994 board. Applications will be available in September from the Malcolm Baldrige National Quality Award Office, A537 Administration Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2036, fax: (301) 948-3716. Completed applications are due Nov. 2, 1993.

**NIST MEASUREMENTS CONFIRM  
QUANTUM HALL DEVICES AS INTRINSIC  
STANDARDS OF RESISTANCE**

NIST scientists have verified that quantum Hall devices can be used as an intrinsic standard of resistance that is independent of the specimen itself to within an accuracy of 2 parts in  $10^9$ . These measurements were carried out on the quantum Hall effect research system, which permits long-term observations of specimen performance, providing a unique tool for investigating fine-scale effects. The quantum Hall effect has been used as the international practical standard of resistance since Jan. 1, 1990.

The scientists compared specimens made from GaAs/AlGaAs heterostructures by AT&T Bell Laboratories, specimens made from GaAs/AlGaAs heterostructures by Philips LEP for the European Metrology Organization, a silicon MOSFET specimen made at the University of Southampton in Great Britain, and NIST specimens prepared from GaAs/AlGaAs heterostructures grown by NIST's molecular-beam epitaxy system. These measurements were made with a superconducting magnet at temperatures below 0.4 K using an automated high-precision potentiometric comparator. The resistances measured at the  $6453.2 \Omega$  resistance plateau on all of the devices were found to be the same to within 2 parts in  $10^9$ .

These results represent an order of magnitude improvement in NIST capabilities and are in agree-

ment with measurements made at other national laboratories, including the National Physical Laboratory in Great Britain, the Bureau International des Poids et Mesures and the Laboratoire Central des Industries Electriques in France, and the Swiss Eidgenossisches Amt Für Messwesen.

**NIST WORK ENABLES MICROMACHINE  
DESIGNERS TO FABRICATE DEVICES  
THROUGH FOUNDRY PROCESSING**

NIST scientists have developed a framework and procedures for producing micromachined semiconductor devices through the MOSIS silicon foundry service. MOSIS (Metal Oxide Semiconductor Implementation Service) is the integrated-circuit fabrication service established by the National Science Foundation and the Advanced Research Projects Agency in support of government research. Considerable technical excitement is being expressed for the potential offered by so-called "micromachined" parts—tiny mass-produced artifacts made in silicon using conventional semiconductor processing technologies until the last step, an etching process to complete the desired electrical and mechanical structures. A single silicon chip can incorporate both the micromachine and an integrated circuit, for example a sensor and signal-processing electronics.

The NIST development permits a micromachine designer to fabricate devices by using a commercially available standard process, followed by an in-house custom etching step requiring only simple, inexpensive equipment. Because their fabrication depends on tested, reliable methods, micromachines offer the promise of low cost. Parts proposed to date include a variety of sensors, electric motors, gear trains, lever mechanisms, and in vivo surgical tools, in addition to NIST projects, including thermal targets, ac-to-dc thermal converters, and chemically specific sensors. To implement the potential for micromachine development which they realized lay in commercially available processing, the NIST scientists added two layers to the MOSIS CMOS process to define a region to be micromachined by customers upon receipt of their MOSIS chips. The "open" layer defines a region of bare silicon, the region to be exposed to the post-processing etchant. The "etch-stop" layer defines an ion-implanted frame around the open layer regions within which the etched silicon is confined. The new capability has been announced to the MOSIS community.

### **“LIFT-MODE” MAGNETIC-FORCE MICROSCOPY DECONVOLVES MAGNETIC INFORMATION FROM SURFACE TOPOLOGY**

NIST scientists have developed a dual-scan form of atomic-force microscopy that solves a problem which has plagued researchers attempting to study the magnetic properties of surfaces at near-atomic levels of resolution. The problem is that the topography of the surface typically affects the measurement. In the new NIST method, the sensing tip, or cantilever, of an atomic-force microscope is coated with a magnetic film, so that the tip will respond to magnetic domains and other magnetic structures on the specimen surface. The tip is first scanned in contact with the surface, and the line-trace signal recorded. A second scan over the same path is taken with the tip lifted a small distance from the surface; a piezodrive using information from the first scan is used to keep this separation distance constant. Magnetic forces cause small deflections of the tip from the predetermined path and are recorded during the second line scan, referred to as “lift mode.” A series of paired contacting and lift-mode scans generates simultaneous topographic and magnetic images of the surface being examined.

The NIST method uses a cantilever that is non-vibrating and responds directly to the force it experiences, rather than to the derivative of force as was the case with previous methods that used vibrating non-contact cantilevers. In addition, since the positioning mechanism can place the tip within a few nanometers of the surface during the lifted scans, resolution and sensitivity are further enhanced. The capability of topographic imaging having a vertical resolution of 0.1 nm and a lateral resolution of 1 nm, with simultaneous magnetic imaging having a force sensitivity of  $10^{-12}$  N and lateral resolutions approaching 10 nm, will provide industry with a valuable tool for developing magnetic nanostructures and microelectronic circuits depending on magnetic phenomena, including memory.

### **NIST DEVELOPS REFERENCE SPECIMENS TO IMPROVE CRITICAL-CURRENT MEASUREMENTS ON HIGH-TEMPERATURE SUPERCONDUCTORS**

A NIST scientist has developed a methodology for producing reference specimens of high-temperature superconductors that yield consistent results for measurements of critical current. By providing a “standard” test specimen to evaluate the perfor-

mance of a measuring system and associated techniques for handling and preparing specimens for measurement, this work addresses the great variability of measurement results experienced by researchers in the high-temperature superconductor field. Confusion surrounding conflicting claims for performance impedes both scientific development and commercialization. Some of this confusion results from the fact that high-temperature superconductors are very susceptible to degradation as a result of mechanical stress, the presence of moisture, thermal cycling, and aging. These factors can create differences between two consecutive measurements of critical current that are as large as 40 percent.

The scientist’s methodology, together with his methods of sample preparation and mounting, can reduce these differences to less than 4 percent—a reduction in variation by a factor of 10. The approach developed by the scientist has demonstrated high repeatability: one specimen was thermally cycled nine times; two of these runs included remounts of the pressure contacts for voltage and current. The overall change in critical current was less than 4 percent from the first run to the ninth run. The NIST reference specimen, also known as the high-temperature superconductor standard reference device (HTS-SRD), is the first such HTS-SRD to be developed.

### **WORKSHOP ON THERMOCHEMICAL DATA NEEDS FOR CVD MODELING**

The proceedings from a joint SEMATECH/NIST Workshop on Chemistry of Chemical Vapor Deposition (CVD) Processes in Semiconductor Manufacturing have been published by SEMATECH. This workshop was held at NIST and was organized by several NIST scientists. There were approximately 50 participants from industry, universities, and other government agencies.

The workshop focused on the chemical kinetic and thermodynamic data requirements for process simulators used in semiconductor manufacturing. These simulators rely on models for the gas phase and surface chemical reactions controlling thin-film deposition processes. Participants in the workshop reviewed the current state of development in gas-phase/surface kinetics modeling, data requirements, and methods to obtain the data necessary to model reliably the chemical reactions controlling CVD. The current state-of-the-art for both experimentally generated and computationally generated thermochemical data were reviewed and discussed,

as were database evaluation and dissemination needs. The rapid advancements in *ab initio* calculational methods provide a means for data estimation of sufficient accuracy to be competitive with experimentally produced values. Recommendations were made for four topical areas: (1) application and use of data by industry, (2) measurement of fundamental thermodynamic and kinetic data, (3) status of present and future data estimation methods, and (4) standardization of input databases.

This workshop was an outgrowth of a previous meeting sponsored by SEMATECH, Sandia, and NIST in 1990 on CVD Reactor Modeling. Two primary needs identified by the 1990 workshop were: (1) reliable fluid dynamic models of reactor geometries and (2) reliable data on the kinetics and thermodynamics of pertinent chemical systems. Future joint workshops in the area of thin-film processing needs of the semiconductor industry are under consideration.

#### **NEW MONOGRAPH OF THERMOCOUPLE REFERENCE FUNCTIONS AND TABLES BASED ON THE ITS-90 NOW AVAILABLE**

Temperature-Electromotive Force Reference Functions and Tables for the Letter-Designated Thermocouple Types Based on the ITS-90 (NIST Monograph 175, which supersedes NBS Monograph 125) presents reference functions and tables based on the International Temperature Scale of 1990 (ITS-90) for the eight, ISA letter-designated thermocouple types: noble-metal types B, R, and S and base-metal types E, J, K, N, and T. Also, for these thermocouple types, reference functions and tables of their thermoelements versus the NIST platinum thermoelectric reference standard, Pt-67, are included. The 600-page monograph, which was prepared by several NIST staff members, contains 118 tables, giving tabular values of temperature and thermoelectric voltage in several different formats to satisfy the requirements of various users. The monograph also covers such topics as history of development, special precautions regarding usage, recommended temperature ranges of use, industrial tolerances, and nominal chemical compositions of each thermocouple type. Information

from Monograph 175 was the basis for reference functions and/or tables prepared for: Standard Handbook for Electrical Engineers, ASHRAE Fundamentals Handbook, IEC Standard Publication 584-1, Thermocouples, Part 1: Reference Tables, CRC's Handbook of Chemistry and Physics, ASTM Manual 12 On the Use of Thermocouples in Temperature Measurement, and various ASTM standards.

#### **TECHNOLOGY TO FABRICATE NANOMETER-SIZE SURFACE TEST STRUCTURES DEVELOPED**

A number of new techniques have been developed recently that are useful for visualizing surface topography with nanometer or sub-nanometer resolution. Techniques such as scanning tunneling microscopy, electron microscopy, optical interferometry, near-field optical microscopy, and scanning scattering microscopy produce very detailed images of surface topography. Different properties of a surface are probed by each technique, however, and the images can be difficult to compare or interpret quantitatively.

A small company, working with a NIST scientist, has successfully completed work supported by a Department of Commerce Small Business Innovation Research Phase I contract. The company has investigated three test structures that could be used for evaluating the performance of instruments measuring surface topography on the nanometer scale. One test structure, consisting of etched steps in silicon dioxide on silicon, showed particular promise. The company showed that 20 nm wide lines with spacings of 20 nm could be produced on the 15 nm thick thermally grown oxide. Procedures were developed to form steps in increments of 1 nm height by controlled etching. A thin metal layer was deposited over the stepped oxide. Tests made with scanning tunneling microscopy and transmission electron microscopy showed that useful devices with steps of 10 nm height had been fabricated. Improvements in the fabrication procedure have been proposed to produce structures with smaller step heights.

**OBSERVATION OF STRAIN-INDUCED  
MICROCRACKS IN HIGH  $T_c$   
SUPERCONDUCTOR COMPOSITE WIRE**

The use of new high-temperature superconductors in a number of applications is challenged by the difficulty of fabricating wire from a brittle material. One promising cable fabrication process embeds the superconductor as fine filaments in a metal matrix. However, strains in the cables due to bending, or to Lorentz forces associated with large magnetic fields can significantly reduce critical currents.

To investigate the effects of strain on current carrying capability, scientists at NIST, in collaboration with a private company, recently obtained radiographs of strain-induced microcracks in superconducting wires using an x-ray microscope developed and patented by NIST. This unique facility is located on NIST beamline X23A3 at the National Synchrotron Light Source. Microcracks as small as  $1\ \mu\text{m}$  were observed non-destructively. The effect of increasing strain was clearly visible by the number and size of cracks. Detailed studies are planned to clarify further the nature of the microcracks.

**INCOMMENSURATE SPIN DENSITY WAVES  
IN METALLIC  $V_{2-y}O_3$** 

Scientists from NIST, another government laboratory, a number of universities, and a private company, in a joint collaboration, have characterized the static and dynamic properties of metallic  $V_{2-y}O_3$  in a region of composition, temperature, and hydrostatic pressure near the metal-insulator transition (MIT). Many materials are known to possess such transitions, and various theories and models have been successful in clarifying several mechanisms for their behavior. That several mechanisms are necessary to describe the MIT in different materials reflects the variety of circumstances which the electrons (and holes) experience due to crystalline structure (or lack of it as in amorphous materials), doping conditions, and in general energetics of the interactions. In several materials that exhibit antiferromagnetism in the insulating phase, doping transforms them into a highly correlated metal. Recent theories have suggested the possibility that the metallic state near the MIT

exhibits spiral magnetic order. Until now the extensive search for this effect has been unsuccessful.

In the recent work at the NIST research reactor, the sought-for ordering has been discovered. The static ordering of the moments, measured with neutron diffraction, has been determined to be an incommensurate spin density wave. Utilizing inelastic neutron scattering, the dynamic properties of the moment fluctuations also have been characterized. In addition to providing definitive results that confirm recent theoretical models, the existence of such ordering could be significant in understanding the cuprate superconductors which are related to this general class of materials.

**HIGH-SPEED SPATIAL SCANNING  
PYROMETER DEVELOPED**

An accurate high-speed spatial scanning pyrometer has been designed and developed by NIST scientists. The pyrometer measures spectral radiance temperatures at multiple target points along the length of a rapidly heating/cooling specimen in dynamic thermophysical experiments at high temperatures. The design, which is based on a self-scanning linear silicon array containing 1024 elements, enables the pyrometer to measure spectral radiance temperatures (nominally at  $0.65\ \mu\text{m}$ ) at 1024 equally spaced points along a 25 mm target length. The elements of the array are sampled consecutively every  $1\ \mu\text{s}$ , thereby permitting one cycle of measurements to be completed in approximately 1 ms. The pyrometer output is recorded digitally with a full-scale resolution of 0.025 percent every  $1\ \mu\text{s}$ . The estimated total uncertainty of radiance temperature measurements is about 4 K at temperatures above 2000 K. This pyrometer, the only one of its kind in the world, has been used successfully in the NIST laboratory as part of a subsecond rapid heating technique for measuring thermal conductivity of tungsten in the range 1800 K–3200 K. Although the pyrometer is designed specifically for thermophysical measurements, it has the potential of having broad applications in various high-temperature technologies related to materials processing and characterization, systems performance, heat transfer diagnostics, and high-temperature research in general.



### **COMPUTER GRAPHICS METAFILE (CGM) AND POSIX FIPS REVISED**

The Secretary of Commerce approved a revision to FIPS 128, CGM, which will be published as FIPS 128-1. The revised standard adopts the redesignated version of the CGM standard known as ANSI/ISO 8632.1-4:1992; adds a requirement for the use of profiles that define the options, elements, and parameters of ANSI/ISO 8632 necessary to accomplish a particular function and to maximize the probability of interchange between systems implementing the profile; and adopts the first such profile as a requirement, the military specification MIL-D-28003A, Nov. 15, 1991, known as the CALS (Computer-aided Acquisition and Logistics Support) CGM Application Profile.

Effective Oct. 15, 1993 FIPS 128-1 is a graphics data interface standard that specifies a file format suitable for the description, storage, and communication of graphical (pictorial) information in a device-independent manner. The standard will facilitate the transfer of graphical information between different graphical software systems, different graphical devices, and different computer graphics installations.

Also revised was FIPS 151-1, POSIX: Portable Operating System Interface for Computer Environments, which will be published as FIPS 151-2. The revision adopts International Standard ISO/IEC 9945-1:1990, Information Technology—Portable Operating System Interface (POSIX)—Part 1: System Application Program Interface (API) [C Language], which defines a C programming interface to an operating system environment. Effective Oct. 15, 1993 FIPS 151-2 will promote the portability of useful computer application programs at the source code level and maximize the return on investment in computer programs by ensuring operating system compatibility.

### **OVERVIEW OF FIRST TEXT RETRIEVAL CONFERENCE PUBLISHED**

NIST Special Publication 500-207, The First Text REtrieval Conference (TREC-1), presents an overview of this conference held recently and attended by about 100 people involved in 25 participating groups. TREC-1 brought research groups together to discuss their work on a new large test collection. Participants reported on a large variety of retrieval techniques, compared the effectiveness of different techniques, and discussed how differences between the systems affected performance. The conference is expected to become an annual event.

### **NIST SPONSORS USERS' FORUM ON APPLICATION PORTABILITY PROFILE (APP) AND OPEN SYSTEM ENVIRONMENT (OSE)**

NIST sponsored the 11th in a series of semi-annual APP/OSE workshops recently, which was attended by about 150 participants. The APP defines a common set of standards and specifications that address the broad functional areas of applications portability and interoperability. The forums provide users and vendors the opportunity to exchange information on NIST's proposals on the evaluation and adoption of an integrated set of standards to support the APP and OSE. A new feature of the users' forum is an introductory half-day tutorial for users with little or no experience with the APP and OSE. The next APP/OSE workshop will be held Nov. 17–18.

### **NIST EXPLAINS ROLE OF MEASUREMENTS IN THE COMPETITIVE STANCE OF THE U.S. ELECTRONICS INDUSTRY**

NIST has explained why measurement capability has such high leverage on the competitiveness of the U.S. electronics industry in a new publication, Measurements for Competitiveness in Electronics, NISTIR 4583. This publication describes the role that measurements play in manufacturing industries, the role that NIST plays in the development of measurement capability for U.S. industry, and the specific measurement needs that are impeding U.S. competitiveness in nine fields of electronics. The nine fields addressed are semiconductors, magnetics, superconductors, microwaves, lasers, optical-fiber communications, optical-fiber sensors, video, and electromagnetic compatibility. This publication is designed to serve both technical and policy-making audiences in Government and industry.

Each chapter on a field of electronic technology contains four basic types of information: (1) a review of the technology; (2) a discussion of world markets and U.S. competitiveness; (3) an outline of the goals that U.S. industry is pursuing to improve its competitiveness; and (4) an assessment of the new measurement capability that U.S. industry needs to meet its own goals. The focus is on measurement capability that is needed widely in the U.S. electronics industry, that will have especially high impact if provided, and that is beyond the resources of the broad range of individual companies to develop. This analysis was developed in cooperation with U.S. industry as a consensus statement of the principal needs. The publication provides the framework for an action plan to correct the shortfall and to advance U.S. competitiveness.

### **NIST-INDUSTRY COLLABORATION DEVELOPING NEEDED FINE-SCALE TEST STRUCTURES**

NIST scientists are leading a collaboration with industry that is intended to extend the design criteria of practical electrical test structures to the subhalf-micrometer linewidth region. At 0.5  $\mu\text{m}$  and below, applying conventional test-structure methodology to make dimensional measurements becomes extremely difficult, as the effects of what were ignorable as perturbations at larger dimensions now need to be understood in detail and accounted for. Industry needs test structures to support lithography advances (such as the use of deep ultraviolet excimer laser wafer steppers) enabling production-line fabrication of features to half-micrometer and below design rules, required for example by next-generation digital logic, including microprocessors. The companies that have collaborated with NIST are a major semiconductor manufacturer, and a manufacturer of test equipment for semiconductor manufacturing.

The semiconductor manufacturer has applied its most advanced developmental line to fabricate wafers bearing test structures designed by the three collaborators. Test structures on these wafers have been used as the basis for an evaluation of electrical, optical, and scanning electron microscope methods for measuring fine-scale linewidth. An important goal was to establish traceability to national standards of length for linewidth-measurement methods in commercial use, work carried out by NIST. Each test chip is composed of a number of areas. Linewidths on the key structures were measured by the three methods: transmitted-light optical microscope, electrical test system (cross-bridge test structures), and scanning electron microscope. The results have been described in two papers presented at SPIE conferences: *Extending Electrical Measurements to the 0.5  $\mu\text{m}$  Regime*, SPIE Vol. 1464 (1991) and *Comparisons of Measured Linewidths of Sub-Micrometer Lines, Using Optical, Electrical, and SEM Metrologies*, SPIE Vol. 1926 (1993), to be published.

### **PRIVATE LABORATORIES TO USE NIST PRINCETON ENGINE VIDEO SUPERCOMPUTER**

NIST and a private laboratory have initiated a new joint research program through the execution of a Cooperative Research and Development Agreement. In this program, the private laboratory will

access the Princeton Engine real-time video supercomputer at NIST to evaluate the efficacy of new image-processing algorithms applied to real-time National Television Systems Committee (NTSC) color video sequences. The Princeton Engine, designed and developed by the David Sarnoff Research Center and provided to NIST by the Department of Defense Advanced Research Projects Agency to promote digital imaging developments, is uniquely suited to this research and is the only one of four similar machines that is accessible to industry and government. The Engine provides real-time video processing capability, accepting and producing a variety of video formats over wideband input and output channels. Because the Engine is programmable, it can be used to evaluate software prototypes of video processing components rapidly at a cost below that of building hardware.

### **NIST PROVIDES LEADERSHIP IN INTERNATIONAL VAMAS SUPERCONDUCTOR STANDARDS ACTIVITIES**

A NIST scientist will serve as the primary U.S. contact for a new VAMAS project on "Characterization and Evaluation of High-Temperature Oxide Superconductors." VAMAS—Versailles Agreement on Advanced Materials and Standards—is an acronym identifying the site at which the program of international standards development was formally established. In addition to NIST, the U.S. organizations participating in the new VAMAS project include two private companies and two universities.

The NIST scientist will carry out nondestructive evaluation (NDE) of the critical current of specimens used in the interlaboratory comparisons. Critical current—a measure of how much current a given superconductor material can carry and remain in the superconducting state—is the single most important parameter determining large-scale application of the high-temperature superconductor materials. The NDE methods developed by the NIST scientist will allow the participating laboratories to measure specimens using their own techniques with a minimum of residual influence from the pre-testing. Other topics which will be explored in the VAMAS project include the evaluation of reliable measurement methods for critical temperature, upper critical field, and critical-surface; physical properties; and the evaluation of existing theories and terminology.

**NIST LEADS TEAM DEMONSTRATING  
CONCEPT OF ELECTRONIC BUSINESS  
REPLY CARD**

NIST is leading a government/industry team formed to promote, develop and demonstrate implement the electronic transfer of electronic component product data. The team recently conducted a demonstration at the 1993 Design Automation Conference to provide engineers with a hands-on introduction to the concept of the "electronic business reply card." Through the use of one of several computer terminals, conference attendees were asked first to identify themselves and their affiliation and then to indicate their interest in receiving information on electronic components selected from the short demonstration list. If they were interested in learning more about a component than provided by the information appearing on the terminal screen in response to their initial inquiry, they could request data at a finer level of detail. Replacing paper transactions with electronically transmitted messages and information—an electronic marketplace—provides a number of powerful advantages to the potential buyer, as well as reduced costs borne by the potential seller. These include virtually instantaneous response to requests, assurance that the latest data is being presented, and access to a wider range of products.

Other planned features include the potential for placing orders electronically and for downloading engineering data and information directly into the customer engineer's computer-aided design system. Implementing these capabilities will involve both technical and political-administrative issues that will be addressed by the Electronic Commerce of Component Information (ECCI) Program, formed in the context of the industry-government National Initiative for Product Data Exchange in response to urgent needs of the electronics sector. The work led by NIST is part of the ECCI Program; the team plans a major demonstration of the electronic business reply card concept for the 1993 CALS Expo in Atlanta in December.

**SENSING SURFACE FINISH  
ULTRASONICALLY**

The NIST Quality in Automation program has been active in the development of sensor techniques to assure the quality of manufactured parts. One sensor technique under development has been an ultrasound-based approach to assess the surface finish of turned metal parts.

The basic concept is to direct ultrasound to the part surface, and then monitor the reflected/scattered wave signature in a "pulse-echo" mode. The machine coolant and lubricant fluid provides a convenient coupling medium to support the high frequency waves (nominally 10 MHz and above) between the sensor and part surface. Surface features such as nicks, scratches, and the periodic tool marks associated with the cutting of turned parts will produce echo signatures that may be distinguished from those for a smooth surface.

It is an on-going effort to work at shorter wavelengths (higher frequencies) to enhance system sensitivity to finer surface features. It is a significant challenge to simultaneously define and control the ultrasonic beam orientation relative to the surface normal of various part curvatures including cylindrical, tapered, and spherical.

**CHROMIUM IN COAL FLY ASH USING  
MICROWAVE**

A microwave oven-based decomposition method for the determination of chromium has been developed by NIST analysts as part of a collaborative effort with a private company and a guest scientist. Chromium is one of the elements considered to be toxic by the EPA. This new method combines the use of sealed vessels, oxidizing acids and a relatively rapid sample decomposition. The procedure yields complete recovery of chromium in NIST SRM 1633a, Coal Fly Ash and its replacement, SRM 1633b. This is a significant improvement over the traditional wet-ash decomposition procedure that can yield low values for total chromium because chromium is either volatilized, especially in samples containing organic matter, or is not completely dissolved.

In this new procedure, sulfuric, nitric and hydrofluoric acids are added to the sample in a sealed vessel, which is then exposed to microwaves. The sample is then evaporated in an open-beaker to remove the nitric and hydrofluoric acids. The chromium values obtained for SRM 1633a using this new technique agreed with the certified value of  $(196 \pm 6)$   $\mu\text{g/g}$ , and a new certified value of  $(202 \pm 7)$   $\mu\text{g/g}$  was determined for its replacement, SRM 1633b. In comparison, previous open-beaker decomposition procedures had yielded a low Cr value of 80  $\mu\text{g/g}$  for SRM 1633a. Further work is being carried out to use this new method for determining chromium in other environmental samples.

### **NIST/SEMATECH GAS FLOW ROUND ROBIN PROGRAM**

NIST scientists have completed a SEMATECH-sponsored round-robin program to ascertain measurement discrepancies that exist among the gas flow calibration facilities used in the U.S. Semiconductor Manufacturing Industries (SMI). These SMI calibration facilities provide critical performance data for the Mass Flow Controller (MFC) devices used in semiconductor manufacturing processes.

SMI calibration facilities claim total uncertainties, i.e., inaccuracies, in the range 0.1 to 0.5 percent to try to achieve 1 percent meter performance in process installations. However, reports from MFC users claim performance deviations of 10 to 20 percent or more. In an attempt to resolve this problem, NIST designed, built, and characterized a gas-flow-measurement artifact and test procedure to calibrate the SMI calibration facilities for a selected set of conditions. Nitrogen gas was used for flows in the range from 300 to 800 standard cubic centimeters per minute (sccm). The artifact consists of critical nozzles arranged in tandem to provide redundancy and assurance of performance. The artifact was calibrated at NIST and the calibration data collected were used to specify details of the test procedure. The round robin involved 55 tests performed in 22 laboratories of SEMATECH member companies and related institutions that volunteered to participate.

Results showed that approximately half of the participants had calibration facility inaccuracies of 0.5 percent or less, but approximately one-fourth had inaccuracies of 1 percent or more. Maximum inaccuracies ranged as high as 8 percent. The NIST gas-flow-measurement artifact and test procedure are currently being offered as a NIST calibration service that may be requested in the same manner as that for conventional flowmeter calibrations. Retesting this artifact enables participants either to confirm that their initially satisfactory performance is constant in time or to evaluate improvements made in response to unsatisfactory results.

Successive phases of this program, currently under development, are expected to extend the flow range to  $(0.1 - 3) \times 10^5$  sccm, which is the full range of interest to the semiconductor processing industries. Once the program is completed, the expected significant improvements in SMI gas flow measurement and control should lead to enhanced productivity in these industries.

### **CRADA WITH PRIVATE COMPANY ON SPRAY FLAMES**

NIST recently entered into a cooperative research and development agreement (CRADA) with a private company to determine the feasibility of using high-power acoustic nozzles to generate well-characterized spray flames. A NIST scientist will collaborate with the private company to design more fuel-efficient and environmentally acceptable combustion systems for U.S. industry.

Over the last few years, this program has produced extensive data on droplet sizes, number densities, and velocities in spray flames using non-intrusive optical means such as laser scattering and high-speed cinematography. This work has revealed that droplet sizes and distributions produced by many commercial atomizers are not quantitatively controllable, resulting in poorly defined initial conditions. The polydisperse atomizers used in this study can produce "made-to-order" sprays with known size distributions and number densities which could lead to flames with desirable characteristics. However, although realization of this potential is the central focus of the CRADA, acoustic nozzles have not yet been employed under burning conditions. It is expected that the combination of new spray-production capabilities and NIST spray-flame measurement and control will lead to development of much improved, quantitatively characterized spray-combustion equipment for industrial applications.

### **X-RAY GONIOMETER DEVELOPED FOR USE IN ULTRA HIGH VACUUM**

X-ray diffraction using synchrotron radiation is one of the primary tools to determine the structure of materials during growth or modification in ultra high vacuum (UHV). This has created a demand for an x-ray goniometer stage that will operate under extreme conditions:  $10^{-8}$  Pa, 2 arc second precision, and no conventional lubrication.

A NIST scientist has been collaborating for the last 3 years with private industry to develop just such an instrument. The materials which go into the gears, bearings, and frame of a standard x-ray goniometer are totally unacceptable for use in UHV. New combinations of metals capable of functioning together with the required precision, and without conventional lubrication, have been incorporated into the design. The materials must not outgas significantly during operation. The

goniometer even incorporates a stepping motor that operates inside the vacuum chamber, under UHV conditions.

Most of the testing is being performed at NIST, where a vacuum chamber designed to accommodate this type of goniometer has been constructed. The prototype goniometer stages are being tested for the ability to withstand bakeout, outgassing during rest and operation, accuracy of rotation, and lifetime of the components. While there is a considerable demand for this type of instrument in the synchrotron radiation community, the required testing equipment that is available at NIST is far beyond the resources of a small company. In addition, this Group at NIST is actually doing synchrotron radiation experiments and provides extensive experience in the area of beamline design and construction.

#### **NEW TECHNIQUE REVEALS UNEXPECTED PHENOMENON IN ELECTRON-ION EXCITATION**

Excitation of multiply charged ions by electron impact enters intimately into the modeling and diagnostics of high-temperature plasmas such as those encountered in controlled fusion. Measurements of cross sections for electron-impact excitation of  $\text{Ar}^{7+}$  using a new merged-beams electron-energy-loss technique show that near threshold the inelastically-scattered electrons are ejected primarily in the backward direction. This unusual angular scattering has not been previously observed for atoms or ions and was completely unexpected. The technique has now been used by NIST scientists to observe similar behavior with  $\text{O}^{5+}$ , strongly suggesting that preferential backscattering may be the rule rather than the exception for multiply-charged ions. The backscattering observed in these measurements was confirmed in subsequent quantum-mechanical calculations and, surprisingly, the effect can be reproduced with a simple semiclassical model. Essential confirmation was obtained from simultaneous measurements of the total (integrated over all scattering angles) cross section over an energy range to 2.2 eV above threshold. To the extent that current models of

electron transport in plasmas of multiply charged ions incorporate the common assumption of strongly forward-peaked excitation, they will have to be revised, or at least revisited, to accommodate this new evidence.

#### **MATERIALS EVALUATION IN SUPPORT OF SUPERCONDUCTING MAGNETIC ENERGY STORAGE**

Under a Cooperative Research and Development Agreement (CRADA) with three private companies, NIST will assist in materials evaluation for a Superconducting Magnetic Energy Storage device (SMES). SMES technology could be used to provide power for defense purposes, electrical grid stability, and load leveling energy storage. The proposed SMES and a subscale engineering test model are essentially large superconducting magnetic coils situated in an underground trench. The coils must be supported to withstand both Lorentz forces generated by the magnetic field and thermal contraction. NIST will perform mechanical and thermal tests on candidate coil support materials (fiber-reinforced composites) in the temperature range from liquid helium (4 K) to ambient. Properties to be determined include ultimate strength, fatigue, shear, flexure, creep, thermal conductivity and thermal expansion. NIST has the world's largest mechanical test facility for research at 4 K; modifications to this system are in progress and, when completed, will allow full-scale coil supports to be tested.

#### **NEW Hg SUPERCONDUCTORS**

Since the discovery of superconductivity in the copper-oxide systems, the expectation has been that these new materials would make commercial applications of superconductivity viable. However, their anisotropic superconducting properties combined with generally unfavorable mechanical and materials properties, have severely limited progress. Hence the community is excited by the recent announcement of superconductivity in a new series of mercury-containing compounds, with onset superconducting transition temperatures as high as 140 K.

Of critical importance is the determination of the crystal structures and phase diagrams of these new materials. The unique power of neutron powder diffraction profile refinement has been used at NIST to determine the structure for the single-layer material,  $\text{HgBa}_2\text{CuO}_{4.06}$ . The crystal structure is tetragonal, and is similar in some respects to the electron-superconductor materials, with the same space group (P4/mmm). There are a number of interesting features of this new material. One aspect concerns the doping, which is accomplished by adding a small amount of extra oxygen rather than substitutional doping. The doping appears to be similar to the "superoxygenated"  $\text{La}_2\text{CuO}_{4+\delta}$  system. In the present case the extra oxygen ions go into the centered position in the Hg plane. The role that the Hg-O layers might play in enhancing the superconducting transition temperature remains to be clarified. This is particularly important considering that the unit cell contains but a single layer of Cu-O, and thus these Cu layers are separated by the rather large distance of 0.95 nm. Yet the superconducting transition temperature is 94 K.

Finally, it is noteworthy that the compound  $\text{HgBa}_2\text{CuO}_{4+\delta}$  may be considered as the first member of the homologous series  $\text{HgBa}_2\text{R}_{n-1}\text{Cu}_n\text{O}_{2n+2+\delta}$  where R is a rare earth element. With the single-layer material  $T_c$  is already 94 K, and higher transition temperatures can be anticipated in the multilayer materials. So far  $T_c$ 's up to 140 K have been observed, and the crystallography of these materials is under active investigation.

#### MEASUREMENTS FOR POLYMER PROCESSING CONSORTIUM

Members of the NIST/Industry consortium, Measurements for Polymer Processing, attended the annual consortium review meeting at NIST recently. Industry representatives from private industry participated in discussions regarding industry requirements for real-time measurements of polymer processing parameters, particularly temperature and viscosity. The NIST measurement program, which is based on optical methods of measurement, utilizes optical fiber probes to monitor the state of polymer resins at specific positions

in the process stream of polymer processing machinery. Discussions of current work focused on progress made to monitor temperature and viscosity using fluorescence spectroscopy. Research topics to be addressed over the next year were prioritized by the industry representatives. Development of a method to measure temperature gradients (the temperature profile through a thickness of polymer resin) during processing was unanimously supported by those present. Industry representatives agreed that accurate knowledge of temperature, temperature gradients, and viscosity are needed in order to understand the physics of polymer processes and to optimize productivity and product performance.

#### OPTICAL AND ULTRASONIC SENSORS FOR POLYMER PROCESSING

In collaboration with Drexel University, NIST researchers have instrumented a polymer injection molding machine with optical and ultrasonics sensors to monitor the state of the polymer as it is being molded. Optical sensors based on fluorescence spectroscopy can be used to monitor resin temperature, temperature profiles in the mold and time of solidification; ultrasonics sensors are used to measure resin stiffness and density. A pressure transducer was also used to monitor pressure in the mold cavity during the packing phase of the process. Injection molding consists of injecting hot polymer resin into a cold mold under high pressure, holding the resin in the mold under pressure until it solidifies, opening the mold and ejecting the final product, and closing the mold to begin the next cycle. Temperature of the hot resin, pressure of injection, velocity of resin injection into the mold, cooling rate, density, and crystallinity are some of the parameters that determine product quality and the productivity of the process. Controlling the process for the purpose of producing uniform high quality products requires real-time measurements of important resin-process parameters, such as the temperature and stress experienced by the resin during the process. Current practice in the industry is to monitor machine parameters such as temperature of the injection barrel, velocity of the injection ram and pressure of

the hydraulic driving the injection ram, rather than properties of the resin itself. This state of affairs exists because the technology for carrying out real-time resin characterizations does not exist. The development of optical and ultrasonics sensors addresses this technology weakness and provides new tools for the polymer processing industry.

#### **RELIABILITY OF GLASS AIRCRAFT WINDOWS**

Researchers from private industry and NIST have developed a reliability assessment methodology for predicting and assuring the safe-life of glass windows intended for use in specialized aircraft. The methodology is based on two concepts: (i) the statistical distribution on window strengths, which are a result of the distribution of the most severe defects in the window surface and/or edge; and (ii) the time-dependent growth of these defects under the simultaneous presence of moisture and in-flight tensile stresses, which result from pressure and thermal gradients across the window. The combined phenomena lead to a statistical distribution of lifetimes for the window. The objective of the analysis was to predict the safe life for specific windows at a given failure probability and confidence level, and under a variety of glass surface conditions.

The relevant materials parameters were determined at NIST from several types of experiments. Strength distributions were measured for test specimens with an as-polished surface finish like that of the actual windows and for various types of simulated in-service "damage": scratches from cleaning and handling, and dust or sand impact damage from environmental or in-flight conditions. Moisture-assisted crack growth properties were determined from the growth characteristics of simulated defects in the glass surface which were produced by controlled indentation with a pyramidal-shaped diamond. The lifetime analysis from these data required an estimate of the 95 percent confidence level that resulted from uncertainties in the experimentally determined parameters. These confidence levels were determined from a non-parametric bootstrap statistical analysis, performed in consultation with scientists from NIST and private industry.

#### **ULTRASONIC RESONANCE SPECTROSCOPY USING EMATS**

NIST has developed ultrasonic resonance techniques for measuring elastic constants and damping in cylindrical or spherical metallic samples using noncontacting electromagnetic acoustic trans-

ducers (EMATs). These techniques present advantages over conventional contacting pulsed ultrasonic techniques for the accurate measurement of elastic constants in isotropic materials and for industrial process sensing applications. The non-contacting nature of the transduction mechanism allows the possibility of performing measurements on moving and/or hot materials. By driving samples into resonance, the circuitry can be more easily implemented than with pulsed measurements. With isotropic spherical samples, the elastic constants can be determined from the resonant frequencies with a great accuracy, limited by the uncertainty in the sample diameter. The high resolution in the measurement of resonant frequencies also provides a capability for measuring small deviations from isotropy. For example, the elastic anisotropy associated with texture in wrought alloys and reinforcements in metal-matrix composites can be measured. Variations in case depth have been measured in hardened steel axles.

#### **NIST DEVELOPS BUILDING AND VENTILATION CHARACTERIZATION PROTOCOLS FOR INDOOR AIR QUALITY INVESTIGATIONS**

A NIST scientist has developed protocols for characterizing commercial buildings and their ventilation systems for use in indoor air quality investigations. These protocols were developed for use in the U.S. Environmental Protection Agency's Building Assessment Survey and Evaluation (BASE) Program. The BASE Program is a multi-year research effort to collect baseline information on indoor environmental performance of commercial buildings. The development of the protocols involved identifying parameters that characterize a building and its ventilation system and procedures for determining their values. The protocols include checklists for obtaining information in the field and instructions for completing the checklists. The checklists are divided into four areas: descriptive information on the whole building, information on the space within the building that is being tested, design information on the ventilation system serving the space being tested, and performance measurement for the ventilation system. These protocols constitute the first systematic approach to obtaining building and ventilation information, and they will be used within the BASE program and other efforts to obtain indoor air quality information in buildings. The development of these protocols was sponsored by the U.S. Environmental Protection Agency and the U.S. Department of Energy.

**EXPERT SYSTEM FOR HIGHWAY CONCRETE**

NIST has completed the development of "HWYCON" (Highway Concrete), an expert system to assist highway departments in making technical decisions concerning concrete. The computerized system provides assistance in three areas: (1) diagnosis of distresses in highway concrete pavements, bridge decks and substructures, (2) the design of concrete for new construction and reconstruction, and (3) selection of materials and procedures for repair and rehabilitation. The focus of the system is on materials-related knowledge and activities. It was developed under the Strategic Highway Research Program's project on the Optimization of Highway Concrete Technology. The system, which is designed to operate on a desktop or portable personal computer, has been tested by potential users in state highway departments and experts in the field of concrete technology. Distribution will be made by the Federal Highway Administration (FHWA) and the Transportation Research Board (TRB). HWYCON contains high-level knowledge, visual, and explanatory information.

**NIST AND THE DEPARTMENT OF THE TREASURY SIGN MEMORANDUM OF UNDERSTANDING (MOU) IN INFORMATION TECHNOLOGY SECURITY**

As a result of a new MOU with the Department of the Treasury, NIST will develop, prototype, test, and implement computer security standards and procedures to protect sensitive Treasury information from unauthorized access or modification. Areas of cooperation include the research and development of the following as they apply to Treasury's mission: accreditation and certification of sensitive automated information systems (AIS); risk management; security management guidelines; open systems; LAN security; equipment used for switching and interchange of data; security architectures; security criteria and evaluation methods; advanced authentication technology; systems integration; and public and private key cryptographic techniques as applied to electronic data interchange (EDI), electronic funds transfer, electronic mail, and other areas.

**REVISION OF FEDERAL INFORMATION PROCESSING STANDARDS (FIPS) FOR DATABASE LANGUAGE SQL**

On May 12, 1993 the Secretary of Commerce approved a substantial enhancement of FIPS 127-1, SQL. Effective Dec. 3, 1993 the revised

standard will be published as FIPS 127-2 and replaces FIPS 127-1 in its entirety. FIPS 127-2 is mandatory for all federal procurements of relational model database management systems. The revised SQL standard adds significant new features for schema definition, diagnostics management, integrity constraints, and international character set support, as well as new data types, new table operations, and enhanced data manipulation expressions. A new Information Schema makes all schema data available to applications.

FIPS 127-2 is specified in four separate conformance levels: Entry SQL, Transitional SQL, Intermediate SQL, and Full SQL. Although only Entry SQL is required, initially, for conformance to FIPS 127-2, a higher conformance level may be specified as mandatory in individual procurements.

Available for sale to the public, NIST SQL Test Suite, Version 4.0, provides conformance tests for the Entry SQL level of FIPS PUB 127-2. Future versions of the test suite will evaluate other FIPS SQL conformance levels.

**GUIDANCE ON IMPROVING SOFTWARE QUALITY ISSUED**

NIST Special Publication 500-209, Software Error Analysis, provides guidance on software error analysis which includes error detection, evaluation, and resolution. The study considers error detection techniques such as those used in software development, software quality assurance, and software verification, validation, and testing activities. Also included are statistical process control techniques and several software reliability models. The document assists the software engineering community in understanding how error analysis can improve the software development process and gives guidance for the evaluation of high-integrity software.

**CONFERENCE ON EXTREME VALUE THEORY AND ITS APPLICATIONS**

Extreme values control design and policy decisions in a large variety of technological and scientific fields. To assure adequate safety levels, land, ocean and air or space structures must be designed to withstand the effects of extreme loads, and materials and systems used in such structures must exceed certain minimum (extreme) performance levels. Pollutant levels that may be tolerable on the average may attain extremes whose effects would be unacceptable. Statistical extreme value theory provides methods for estimating the probability of extreme events on the basis of a finite set of data.



An international Conference on Extreme Value Theory and Its Applications, cosponsored with Temple University, was held at NIST recently. The Conference attracted 150 researchers from academic, government, and industrial research groups from the United States and abroad. The focus was on the mathematical theory of extremes and applications in the areas of civil engineering, materials sciences, and environmental sciences, although papers in other applications areas such as astronomy, physics, economics were presented as well. A half-day short course before the Conference was well attended and served to acquaint attending scientists and engineers with elements of the modern theory.

A principal goal of the Conference was to assist in the transfer of advances made in the theory of extremes to applications, and to expose the mathematical researchers to the needs of the engineering community. Selected Papers from this conference will be published in a future issue of this Journal.

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## Standard Reference Materials

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### TIN-LEAD COATING THICKNESS STANDARDS 60% Sn-40% Pb

The Standard Reference Materials Program announces the release of SRM 2321, Tin-Lead Alloy coating on Copper. This SRM is intended for use in calibrating x-ray fluorescence instruments for the measurement of mass per area and composition of tin-lead alloys deposited on a copper substrate. The SRM consists of a 15 × 15 mm plate of an electroplated tin-lead alloy coating on a polished copper substrate. The plate is mounted in a recessed plastic holder.

The tin-lead alloy coating was characterized using energy dispersive x-ray fluorescence and atomic absorption spectrometry. The SRM is certified for mass per area and the percent tin to within ±5% of the given value at the center of the sample and the average over the entire surface. An approximate thickness of the alloy coating can be estimated from the measured mass per area, the measured composition, and the density.

SRM 2321 is available from the Standard Reference Materials Program, Room 205, Building 202, NIST, Gaithersburg, MD 20899, (301) 975-6776.

### STANDARD REFERENCE MATERIAL 886— REFRACTORY GOLD ORE

The gold mining industry depends on highly accurate assays to set the fair market value of its ores. Additionally, selection of the most efficient process for extracting the gold from its matrix requires the determination of concentrations of key matrix elements, such as sulfur and carbon, which can exhibit a deleterious effect on some of the processing methods.

The Standard Reference Materials Program announces the availability of Standard Reference Material (SRM) 886, refractory gold ore. The ore is certified for its gold content,  $(8.25 \pm 0.13)$  mg/kg. Information values are also reported for carbon at 5.7 weight percent and for sulfur at 1.5 weight percent.

### STANDARD REFERENCE MATERIALS 2556 AND 2557—USED AUTOMOBILE CATALYSTS

Automobile catalysts required for emission control use the metals platinum, palladium, and rhodium to catalyze the conversion of pollutant gases in automotive exhaust to harmless compounds. Because of the scarcity and price of these metals, recycling of spent automobile catalysts is routinely undertaken. Highly accurate analyses of the platinum group elements are required to establish the fair market value of the materials being recycled.

The Standard Reference Materials Program announces the availability of Standard Reference Materials 2556 and 2557, used automobile catalysts of the Pellet and Monolith types, respectively. The SRMs were certified by isotope dilution thermal ionization mass spectrometry (Pt, Pd, Pb) and by inductively coupled plasma mass spectrometry (Rh). Instrumental neutron activation analysis and cooperative industry analyses provided corroboration. The certified values for the two materials are:

- SRM 2556  
Pt 697 mg/kg; Pd 326 mg/kg; Rh 51.2 mg/kg;  
Pb 6228 mg/kg.
- SRM 2557  
Pt 1131 mg/kg; Pd 233 mg/kg; Rh 135 mg/kg;  
Pb 13,930 mg/kg.

These SRMs were developed in cooperation with the International Precious Metals Institute, whose members provided collaborative analyses. The SRMs are available in units of 70 g of J m powdered material.

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## Standard Reference Data

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### RUSSIAN ROCKET-SPACE TECHNOLOGY DATA AVAILABLE

An important collection of reference data originally developed for Soviet rocket/space technology is now available for PCs from NIST. The IVTANTHERMO database includes information on the thermodynamic properties of approximately 2300 individual substances (containing 85 elements and the electron) in the standard state within a wide temperature range. The data are internally consistent within the framework of the principal laws of thermodynamics. The most distinctive feature is that the data are not taken from various references of other databases but calculated using the molecular and thermochemical constants evaluated as a result of expert analysis of all primary data. The data collection was started in the 1950s by the Soviet Academy of Sciences. Through an agreement with an international publisher, NIST is able to make IVTANTHERMO available to users worldwide. NIST Special Database 5, IVTANTHERMO-PC, Version 1.0, is available for \$950 from the Standard Reference Data Program, A320 Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: 926-0416.

### DATABASE DETAILS ATOMIC-SCALE SURFACE STRUCTURES

Scientists and researchers in materials, physics, and chemistry now have rapid access to important information on the crystal structures of surfaces. The new Surface Structure Database for PCs, NIST Standard Reference Database 42, is the only complete critical compilation of reliable information on surface crystallographic structures available. It is an important resource for assessing and comparing detailed atomic-scale structures of surfaces and interfaces obtained from low-energy electron diffraction, or LEED, and from other experiments. Nearly 600 structures are included, covering a wide variety of materials of scientific and technological interest, including catalysts. Each structure is covered by extensive tables of numeric data that help the user evaluate how it affects chemical reactivity. The database also contains three-dimensional color graphics that permit the visual inspection of all surface structures. The structures can be rotated and

magnified, and the distance between atoms can easily be determined. The database is available for \$390 from the Standard Reference Data Program, A320 Physics Building, NIST, Gaithersburg, MD 20899-0001, (301) 975-2208, fax: (301) 926-0416.