

News Briefs

General Developments

Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 101, Room E215, Gaithersburg, MD 20899-2500; telephone: (301) 975-3577.

NIST'S ROLE BASED ACCESS CONTROL RESEARCH SAVES INDUSTRY \$295 MILLION

A new independent economic impact study conducted by the Research Triangle Institute (RTI) conservatively estimates that NIST's Role Based Access Control (RBAC) research has saved U.S. industry \$295 million and accelerated industry's adoption of this advanced access control method by a year. NIST's research cost taxpayers only \$2.3 million. The RTI study quantifies the benefits of RBAC and estimates NIST's impact on the development and adoption of RBAC by industry and the user community. RTI estimated that RBAC technology has saved U.S. industry a total of \$671 million, and that NIST's work was responsible for 44 % of this savings.

According to one major software company official, "This is probably one of the best examples of how an organization like NIST can help the private sector. The existence of a widely visible prototype advanced the concrete understanding of corporate IT architects so significantly that we were able to get unusually good early feedback validating and influencing our design choices. Getting educated feedback early undoubtedly saved us a significant amount of money."

A representative from another company said, "The NIST implementation was a groundbreaking and significant contribution to software technology."

Computer access control systems are designed to control which users or groups of users can invoke programs and access system resources such as databases and files. Typically, every system and application for which access control is enforced has its own proprietary access methods and system-specific meanings for oper-

ations and objects. For many organizations, the number of systems can be in the hundreds or even thousands; the number of users can range from the hundreds to the hundreds of thousands, and the number of resources that must be protected can easily exceed a million. The problem becomes even more complex with organizational hierarchies and special constraints such as conflict-of-interest rules. As a result, the management of access control data becomes a difficult, expensive, and error-prone process.

NIST's RBAC controls access to computer system networks based on the user's role in an organization, automatically handling complexities introduced by organizational hierarchies and separation-of-duty requirements. Under RBAC, users are granted membership into roles based on their responsibilities in the organization. The operations that a user may perform are based on the user's role. User membership into roles can be revoked easily, and new memberships can be established as job assignments dictate. This mechanism demonstrates the potential for enormous cost savings and better security over current methods. The website is <http://hissa.nist.gov>.

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NIST RESEARCHERS DEMONSTRATE HIGH-SPEED THERMAL IMAGING SYSTEM

NIST researchers used a high-speed thermal imaging system to study transient thermal characteristics of microhotplate structures. Their studies demonstrated the importance of the microhotplate heat-spreading layer in obtaining a uniform dynamic temperature distribution.

Researchers used a high-speed thermal imaging system to investigate the dynamic thermal behavior of MEMS based microhotplate devices. Measurements revealed delayed surface heating of the microhotplate and temperature redistribution during both the heating and cooling phases. These measurements are useful in optimizing the design of microhotplate structures.

Researchers demonstrated that the unique high-speed thermal imaging system is capable of determining various thermal time constants of the structure. These time constants include the propagation of heat from the heating element through the multi-layered, suspended membrane structure, the equalization of temperature over the upper surface, and the cooling of the entire membrane structure by convection, radiation, and conduction through the legs. It also was shown that reflected infrared radiation can be used with a normalization technique to determine the temperature of the hidden underside of the suspended membrane. The thermal time constants measured with the transient thermal imaging system can be used to optimize the thermal performance of microhotplates.

Preliminary measurements indicate that the microhotplate structure may be a useful infrared radiation calibration artifact for very small heat sources where the accuracy of the temperature measurement is affected by spatial resolution.

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ADSORPTION OF FUEL ODORANTS PROVIDES CLUE TO FADING

Sulfur compounds, specifically thiols and alkylsulfides, are added to fuel gas to give the fuel distinctive odors that alert consumers to leaks. Over time, however, odorant concentration in fuel gases may decrease below olfactory thresholds. This phenomenon, referred to as odorant fading, occurs through various mechanisms. When natural gas and liquefied petroleum gas leaks occur in distribution lines, adsorption and absorption processes between the odorants and surrounding soil media often result in diminished odorant concentration which cannot be directly compensated for by utilities. In addition, the magnitude of the interactions between the fuel gas odorants and soil media is not known. Recent research conducted by NIST furnished some answers by determining the magnitude of these sorption interactions.

The magnitude of these sorption processes was determined by measuring the heats of adsorption and interaction of the odorants on clay and organo-clay substrates. These substrates served as soil surrogates. The measurements were performed using a wall-coated, open-tubular (WCOT) column gas chromatographic technique developed by NIST researchers in Boulder. The researchers created clay stationary phases using the synthetic clay Laponite-RD. Subsequent coatings with an organic such as octadecane create an organo-clay stationary phase.

While the results to date include substrates representative of only two types of soil media, valuable insight into the magnitude of the odorant fading in soils is possible. Experimental results show that, as a class, the sulfide odorants have larger adsorption enthalpies on clay and organo-clay surfaces than the thiol odorants. As a result, the sulfides are more likely to be sequestered on soil surfaces. The difference in enthalpy values between the fuel gases and their respective odorants is valuable information from a utility operation standpoint.

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PROCESS TO REMOVE CARBONYL SULFIDE FROM LPG

A patent has been issued (Patent No. 6,334,949, Jan. 1, 2002) to two NIST scientists for a process that selectively removes carbonyl sulfide, a potentially problematic impurity, from liquefied petroleum gas (LPG). NIST scientists in Boulder discovered the carbonyl sulfide process accidentally, while researching the extraction of metals. Commercial sources of LPG are composed primarily of propane, and provide fuel gas for outdoor grills, recreational vehicles, and rural residences and businesses. In addition to propane, LPG may contain butane, ethane, but also some unfavorable impurities that can ultimately form corrosive products. One such impurity is carbonyl sulfide, or COS. This compound is of concern because in the presence of water, it can hydrolyze to form hydrogen sulfide, a deadly and corrosive gas not permitted in commercial LPG.

While conducting research on the extraction of metals with supercritical fluid extraction, the scientists discovered that the common supercritical fluid solvent carbon dioxide forms a stable complex with a macrocyclic organic molecule called *p*-tertiary-butyl-calix[4]arene. Their work with infrared spectrophotometry indicated that this might be an inclusion complex. The similarity of the molecular structures of carbon dioxide and carbonyl sulfide prompted tests with COS. As with CO₂, a very stable complex was obtained with COS.

Additional work demonstrated a new process that selectively removes carbonyl sulfide from LPG. Since calix[4]arene is a stable material, the new process has the potential of decreasing costs to LPG suppliers and processors. Current methods of removing COS from LPG all suffer from major drawbacks. For example, molecular sieve processes require large bed sizes and very short cycle times, and reactions with metal oxides result in a nonregenerable waste product. *P*-tertiary-

butylcalix[4]arene, on the other hand, can be regenerated, and has a strong interaction with COS. A process based on this material has the potential of removing COS at both low and trace levels. This is a thermally stable compound that can be used to very high temperatures.

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NIST HELPS DEVELOP FIRST IEEE WIRELESS PERSONAL AREA NETWORK STANDARD

NIST contributed significantly to a new standard for wireless personal area networks (WPANs) approved by the Institute of Electrical and Electronics Engineers (IEEE) on March 21, 2002. The approval of IEEE 802.15.1 was the long-awaited formal acceptance of the Bluetooth Special Interest Group core specification by a recognized standards body. Although the Bluetooth core specification defines all the layers from the physical layer to the application layer, only the lower layers, which are considered within the scope of the IEEE 802 Medium Access Control and Physical layers, are included. This wireless technology operates in the 2.4 GHz frequency band and provides voice communications at 64 bit/s and data transfers up to 732 kbit/s at distances up to 10 meters. The technology is meant to be inexpensive, thus positioning itself for system integration as one of the pervasive computing technologies.

NIST has been involved in this effort since the Bluetooth Special Interest Group first released the specifications and the IEEE 802.15 Working Group was formed. The work consisted of reviewing, verifying, and validating the protocols being defined. As a means to speed and ideally show the completeness and correctness of the protocols, NIST undertook the task of creating a formal description of the text prose using the ITU-T standardized formal description language called Specification and Description Language (SDL) that was included as an informative annex in the standard. SDL uses a finite state machine and describes the behavior in the form similar to a flow chart. This process generated thousands of comments and suggested changes that were incorporated in the Bluetooth specifications v1.1. In addition, NIST held editorship of several sub-clauses in the draft standard and participated in the creation of the Protocol Implementation Conformance Statement proforma.

IEEE 802.15.1 is the first in a series of new WPAN technologies being developed in IEEE 802.15.

Standards for higher (20 Mbit/s) and lower (250 kbit/s) rate WPANs are under development. See our website for more information: <http://www.itl.nist.gov/pervasivecomputing.html>.

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NIST REVIEWS SECURITY OF INDIAN TRUST MANAGEMENT, DEPARTMENT OF THE INTERIOR

On April 1, 2002, NIST's Computer Security Expert Assist Team (CSEAT) delivered the CSEAT Indian Trust Management (ITM) draft high-risk program review report to the Associate Deputy Secretary of the Department of the Interior. ITM is responsible for billions of dollars of Indian trust assets and has been a very troubled program for decades; OMB identified ITM as a high-risk program. IT security became a very large ITM issue with the advent of the Cobell class-action lawsuit.

The CSEAT ITM draft report outlines the ITM IT security deficiencies in general and those specific to the Department of Interior, the Bureau of Indian Affairs, the Bureau of Land Management, the Minerals Management Service, and the Office of the Special Trustee (an organization created to address ITM issues). The review provides a prioritized action plan to improve the IT security of ITM and presents two specific options for implementation.

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NEW STATISTIC ADDRESSES LONG-TERM STABILITY

The measurement of the long-term stability of clocks and oscillators has been a long-standing problem for science and industry. The Allan deviation and another statistic called total deviation both require data runs twice as long as the desired averaging time interval. Thus, determining the stability at one month requires a two-month-long data run.

Recently, NIST developed a new statistic that yields a measure of stability at the end-point of the data series. Moreover, the statistic retains all of the desirable features of the Allan deviation. The effect of this advance is to cut required measurement times in half, effectively cutting the cost of acquiring the most expensive data point in half.

The total deviation has long been used by manufacturers and analysts in place of the Allan deviation, where calculations of frequency stability at long-term time intervals are needed. This statistic, developed earlier, yielded a better confidence interval, but still suffered from the need for long data runs. Using the total deviation as a starting point, we found that a more complex combination of frequency sums and differences could yield an Allan-like statistic clear out to the interval of the data run itself. As a test, the statistic has been used over the past two years for measuring the performance of the NIST time scale and the primary cesium-fountain frequency standard, NIST-F1. The results, which have clearly demonstrated the efficiency of the statistic, have served to reduce the time required for evaluating the accuracy of NIST-F1 and to improve the performance of the time scale.

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LIGHT SCATTERING BY METALLIC PARTICLES ON SILICON WAFERS

Light scattering is used by semiconductor manufacturers to inspect silicon wafers for particulates, defects, and surface roughness. Since a particle's size determines its potential to cause device failure, it is critical to be able to accurately measure particle size. Polystyrene latex (PSL) spheres are often used to calibrate wafer inspection systems, but these model particles do not behave like real-world particles. For example, they scatter much less light than metallic particles do. A system, calibrated using PSL spheres, might report that a harmless metallic particle is much larger than it really is, causing more wafers to be rejected than necessary. Validated theories for light scattering are necessary to avoid such problems.

NIST, in collaboration with the University of Maryland, has developed a method for generating uniformly sized, pure copper particles and depositing them on silicon wafers. We performed measurements of the light scattered by these particles and found very good agreement with theoretical calculations. The code for these calculations has been made publicly available through the SCATMECH library of scattering codes (physics.nist.gov/scatmech). It can serve as a benchmark for testing other scattering calculations and can be used to help design improved inspection systems.

One hitch has been found, though. When we extended the theory to allow non-spherical particles, we found that the scattering by metallic particles is extremely sensitive to the shape of the particle. We demonstrated that a 1 nm dent on a 60 nm diameter

aluminum sphere at the point where it contacts a silicon wafer can change light scattering by 30 %. This sensitivity to shape will make the accurate sizing of particles on surfaces even more challenging than originally thought.

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SELF-STUDY MANUAL NOW AVAILABLE ON COMPACT DISC

The *Self-Study Manual on Optical Radiation Measurements* has proven to be a valuable and useful reference in the fields of radiometry and photometry. This series of eight documents was published from 1977 to 1985 as NBS Tech Notes 910-1 through 910-8. However, it has been out-of-print for many years.

NIST has re-released the Self-Study Manual electronically. All eight volumes are now available on one compact disc for viewing, searching, and printing. The CD-ROM presents the 600-plus page series in searchable Portable Document Format (.pdf), and includes links to the reader software. Through the Self-Study Manual, the reader can learn about the measurement of incoherent optical radiation, including definitions and terminology, measurement equations, relevant SI units, instrumentation, and sample applications.

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PHOTOASSOCIATION IN A BOSE-EINSTEIN CONDENSATE

Two colliding atoms can absorb a photon and be photo-associated into an excited, diatomic molecule. NIST researchers investigated the fundamental rate at which this process can take place in a Bose-Einstein condensate (BEC) and demonstrated that simple classical saturation arguments do not work when applied to this situation.

While the photoassociation reaction has been well-studied in a thermal gas, one can argue that the same process will run into limitations under the conditions present in a BEC. A simple classical argument says that a pair of atoms has a narrow range of internuclear separations at which the photoassociation process can be driven to take place. Once atoms with this range of separations are depleted, the population must be replenished by the movement of atoms, and that this is limited by their thermal velocity (about 0.5 mm/s in this case). On the other hand, a quantum mechanical description of the BEC has a single wavefunction for all the atoms; each atom is extended across the entire region where the

experiment is performed. The measured rate coefficient for the reaction is in good agreement with results from a quantum mechanical two-body scattering theory for all the intensities that were achievable experimentally, and can exceed the classical limit by more than four orders of magnitude.

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ULTRACOLD GASES SPIN A WINDING YARN

Experiments on quantum gases continue to yield amazing and counter-intuitive results. In autumn 2001, researchers at NIST found that a non-condensed Bose-Einstein gas could display spatial segregation of internal spin states, even though the thermal energy in the gas was more than 1000 times greater than the interaction energy of the two states. The effect was seen following the sudden preparation of all atoms in a coherent superposition of two states, after which the populations of the two states separate. This suggests an astonishingly robust effect of quantum coherence in a thermalized gas. A report of this experimental work was published in the February 18, 2002, edition of *Physical Review Letters*.

Theorists at NIST began an investigation of this phenomenon and found that it was attributable to the generation of a damped “spin wave” due to quantum interference between direct- and exchange-scattering of atoms in the two spin states. A first-principles kinetic theory that used no fitting parameters gave excellent agreement with the experimental data. The theoretical paper describing this work has been accepted for publication in *Physical Review Letters*.

The experimental and theoretical groups have since collaborated on a third paper, which reports the first spatially-resolved images of spin waves in a gas. That paper has been submitted for publication.

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NIST RESEARCHERS IMPROVE HIGH-RESISTANCE MEASUREMENT CAPABILITY

A NIST scientist has developed a new technique that enables more accurate scaling from the primary NIST quantum Hall resistance standard to high resistance levels of 1 M Ω and above. The technique exploits a unique property of the quantum Hall effect which allows a quantized Hall resistance standard to serve as a near-ideal two-terminal resistor whose resistance, 12 906.403 5 W, is essentially independent of the resistance of the room-temperature connecting leads. This will allow calibration of NIST 1 M Ω working

standards with a relative uncertainty of better than 1×10^{-7} , an improvement of about an order of magnitude. The new measurement system was recently used to calibrate a set of four 1 M Ω resistance standards for an NCSLI interlaboratory comparison. The relative uncertainty for these calibrations is about 1×10^{-6} , a factor of three improvement over the present NIST calibration service.

The property exploited by the new measurement system is unique to the quantum Hall effect and determines exactly how current flows in device leads when more than two connections are made to a single QHE device. The new NIST bridge incorporates a cryogenic current comparator, relies on superconducting leads for many connections, and is compatible with conventional guarding techniques. The ultimate result is a bridge that is significantly less susceptible to leakage errors than the series-parallel resistance networks (Hamon devices) previously used for high-resistance scaling. It may be possible to extend the range of this direct scaling measurement up to 10 Ω or even 100 Ω by using larger turns-ratio current comparators.

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NIST LEADS CREATION OF TWO NEW IEC SUPERCONDUCTIVITY STANDARDS

Two new international standards on superconductivity were recently published by the International Electrotechnical Commission (IEC) Technical Committee 90 (TC 90). The documents are:

- IEC 61788-4 Superconductivity—Part 4: Residual resistance ratio measurement—Residual resistance ratio of Nb-Ti composite superconductors;
- IEC 61788-7 Superconductivity—Part 7: Electronic characteristic measurements—Surface resistance of superconductors at microwave frequencies.

Two NIST scientists at Boulder have worked extensively on these documents and helped resolve many difficulties encountered during the development process. One of the scientists serves as Chairman of TC 90 and manages the international work. Thirteen countries participate in TC 90. The standard on surface resistance of superconductors at microwave frequencies is the first IEC standard for electronic applications of superconductivity. This brings the number of IEC TC 90 published standards to eight. Currently, six more documents are at various stages of development within TC 90.

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SPRINKLERED VERSUS NON-SPRINKLERED DORMITORY ROOM FIRES

NIST has compiled a 9 minute video of fires in two college dormitory rooms, one equipped with sprinklers and the other without sprinklers. Small fires were ignited in a wastebasket between the desk and bed in each of the two fully-furnished dormitory rooms. In the sprinklered room, the fire grew until sprinkler activation which occurred in less than 2 minutes. For the unsprinklered room, the fire continued to grow until it flashed-over the room in less than 5 1/2 minutes. The video also includes pre-fire and post-fire photographs which can be used to compare the damage caused by each fire.

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SIMULATION OF MULTIPLE FATALITY HOUSE FIRE IN KEOKUK, IOWA

This report, *Simulation of the Dynamics of a Fire in a Two-Story Duplex—Iowa*, Dec. 22, 1999 (NISTIR 6854), describes the re-creation of a fire in a two-story duplex house in Iowa which claimed the lives of three children and three firefighters. This report/CD includes the results of calculations using the NIST Fire Dynamics Simulator that were performed to provide insight on the thermal conditions that may have occurred during this fire. In addition to providing the timeline, floor plan, post-fire photographs, and model results (both still and animated), this CD also offers a video/audio clip of a NIST researcher describing the fire via photographs and computer fire model output.

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NEW STANDARD FOR THREAD WIRE MEASUREMENT

A new standard for measurement of thread wires has just been published. The standard, ASME B89.1.17, Measurement of Thread Measuring Wires, was developed under the leadership of a NIST guest researcher. The new standard will establish uniform practices for this important measurement, which is a critical step needed to properly establish pitch diameter of threaded fasteners. The new standard will promote manufacture of more uniform threaded fasteners with better fit and improved functionality.

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TEMPERATURE GRADIENT FOCUSING IN MICROFLUIDIC CHANNELS

NIST scientists have developed a new method to concentrate and separate chemical species in microfluidic channels. Microfluidic, or “lab-on-a-chip,” devices are miniaturized chemical and biochemical analysis systems that one day may replace conventional bench top instruments.

The new technique, temperature gradient focusing, balances the electrophoretic motion of analytes in a microchannel against the bulk flow of buffer solution through the microchannel while applying both an electric field and a temperature gradient along the length of the channel. For buffer solutions having temperature-dependent ionic strength, the applied temperature gradient results in a corresponding gradient in the electrophoretic velocity of a charged analyte in the channel. The bulk flow velocity can then be adjusted so that the total analyte velocity (the sum of the bulk and electrophoretic velocities) is zero at some point along the channel length, and all of the analyte in the channel will be focused, or concentrated, at that point.

The technique gives simultaneous focusing and separation of differently charged analytes in a manner analogous to isoelectric focusing of proteins, but is much simpler to implement than isoelectric focusing and has the additional advantage of working with any charged analyte, rather than just proteins. Temperature gradient focusing has been demonstrated for a variety of different analytes including fluorescent dyes, amino acids, proteins, DNA, and colloidal particles. In addition, it has been shown to be capable of greater than 10 000-fold concentration of a dilute analyte. A description of the technique has been published in the journal *Analytical Chemistry* [(David Rosso and Laurie Locascio, *Anal. Chem.* **74**(11), 2556-2564 (2002)].

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HIGH ACCURACY TECHNIQUE REVOLUTIONIZES PLATINUM GROUP METALS ASSAY

NIST researchers have developed a high performance approach to the measurement of elemental composition using inductively coupled plasma optical emission spectroscopy. This technique uses an innovative experiment design coupled with a novel drift correction approach to minimize and quantify the major sources of uncertainty in this chemical analysis. A recent

publication documents the performance of the method, high performance inductively coupled plasma optical emission spectrometry, for 64 elements where the typical relative expanded uncertainty observed is 0.1 % (*Analytical Chemistry* **73**, 4821-4829, 2001). This technique utilizes unmodified, commercially available equipment, is well suited to automation, and yields results comparable to the best attainable by classical methods.

The NIST researchers presented this technique and results at an ASTM E01 committee meeting in May 2001, and established a collaboration to implement it with a private company in North Attleboro, MA, a precious metals and advanced materials supplier. Together, methods for the determination of palladium and platinum in metallic samples were developed. NIST provided software tools to perform the calculations. Less than one year later, this technique was in routine use at the company, complementing the gravimetric wet chemical technique used in their labs. According to a company spokesman, "This technique has revolutionized our Platinum Group Metals assay lab. Assay completion times which ran 20 to 30 days, now average 4 to 6 days. Assay precisions have improved by more than an order of magnitude. Accuracy, as measured by the number of assays sent for umpire laboratory analysis, has improved to the point that we have not had to go to umpire in over three months. I am now setting up the procedure for the determination of gold at major levels."

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FABRICATING PLANAR NANOPARTICLE ASSEMBLIES WITH NUMBER DENSITY GRADIENTS

Nanoparticle-based structures are envisioned to play an important role in futuristic devices such as single-electron tunneling-based computer chips, high-density information storage devices, and other commercially-important applications such as sensors and high-efficiency solar cells. In order to position nano-components onto functional devices, it is vital to develop methods for placing them into chemically and structurally well-defined environments. To this end, scientists at NIST and their collaborators at North Carolina State University have prepared assemblies of 15 nm gold nanoparticles with continuous gradients in number density on flat silica-covered substrates. A description of the material appears as the cover story in the July 23 issue of *Langmuir* [Fabricating planar nanoparticle assemblies with number density gradients,

Rajendra R. Bhat, Daniel A. Fischer, and Jan Genzer, *Langmuir* **18**, 5640-5643 (2002)].

For surface-property optimization, it is useful to prepare samples with a continuously-varying number density of immobilized particles along the surface. Such a nanoparticle gradient surface provides a combinatorial platform for surface adsorption selectivity. The gold nanoparticle surface was created by forming a one-dimensional molecular gradient of amino groups on the substrate, and then attaching the gold nanoparticles to the amino groups by immersing the substrate in a colloidal gold solution. Atomic force microscopy demonstrated that the number density of the nanoparticles varied continuously as a function of position on the substrate from 500 particles/ μm^2 to zero particles/ μm^2 over a distance of 45 mm. Near-edge x-ray absorption fine-structure studies confirmed that the nanoparticle number density gradient was closely correlated with the concentration gradient of amino groups anchored to the substrate. As a result of this work, the number density of nanoparticles within the gradient and the length of the gradient can now be tuned by controlling the initial amino group gradient.

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SIMPLE MODEL SYSTEM FOR FABRICATION OF INTEGRATED CIRCUIT INTERCONNECTS

Electrodeposition has recently and unexpectedly evolved with a major role in on-chip metallization, which provides the interconnects between transistors, etc. in an integrated circuit. The understanding of copper deposition for this particular application has been elucidated through the Curvature Enhanced Accelerator Coverage (CEAC) model, recently developed by NIST. The CEAC is a catalyst-mediated deposition model that successfully describes the filling of submicron features with copper, and is capable of predicting conditions that produce "wires" free of porosity and other defects. This understanding is key to creating the ever-finer multi-level copper interconnects of the future that are free of current-disrupting voids and seams.

To extend and generalize the model, NIST scientists have studied silver deposition from silver-bearing electrolytes. The silver electrolyte with the addition of very low concentrations of selenium displays "brightening" and current-voltage hysteresis similar to that observed in copper baths that exhibit excellent manufacturability. The selenium concentration on the evolving surface, analyzed by x-ray photoelectron

spectroscopy as a function of deposition time, is found to track exactly with the acceleration of the silver deposition rate. Initial filling of submicron trenches and vias with silver indicates good quality interconnects.

The silver-based system offers several advantages over copper for developing a deposition model. First, the one-component additive, selenium, that accelerates silver deposition is far simpler than the three-component additive cocktail employed in copper deposition. Second, silver has a single valence in the electrolyte while copper is present in two valence states. The simplicity of the silver system will facilitate the quantification of surface coverage and its influence on silver deposition kinetics. This will make the study of metal deposition into submicron features far more straightforward, both analytically and computationally.

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SCANNED PROBE OXIDATION GIVEN UNIQUE RECOGNITION

Scanned probe oxidation (a.k.a. local oxidation or nano-oxidation), pioneered several years ago by NIST, has been given its own unique code, 81.16.Pr, in the American Institute of Physics recently updated Physics and Astronomy Classification Scheme database. Since the first report appeared in a paper entitled “Modification of Hydrogen-Passivated Silicon by a Scanning Tunneling Microscope in Air” in Applied Physics Letters in 1990, it has become the most widely used nanofabrication technique based on the scanned probe microscope. Research groups worldwide employ the technique for the fabrication of nanoelectronic devices, nano-electro-mechanical systems, electro-optical structures, and templates for chemical and biological self-assembly.

For more information see: <http://www.aip.org/pacs/pacs01/nanosci.html>.

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LEADING NIST PUBLICATION CITED

The Review of Scientific Instruments, which is widely regarded as the preeminent journal describing scientific instrumentation, is a frequent vehicle for the dissemination of NIST-developed measurement methodology. Recently, a scientist at the Institute of Scientific Information, noted that one such NIST paper was the most highly cited article appearing in this journal over the past 30 years.

The paper, “GaAs Spin Polarized Electron Source,” was written by NIST staff members in 1980. It was the

first in a series of papers that developed the measurement technology for the control and measurement of the electron spin in electron beams. Subsequently, a number of measurement methods involving the spin of free electrons were developed by NIST and applied to magnetic surfaces and films. One of these methods, SEMPA, is a type of electron microscopy used to image nanoscale magnetic domains. It has been applied productively in studies of magnetic disk storage systems, magnetic sensors, and most recently, to nanoscale magnetic random-access memory devices.

The GaAs source began the resurgence of research into spin-polarization effects that has continued to this day. There is active work worldwide on magnetic nano-devices, magnetoelectronics, and spintronics—all of which can potentially shape the future of electronics and information technology.

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NIST CONTRIBUTES TO REPORT ON PROTECTING BUILDINGS FROM TERRORIST ATTACKS

NIST participated in the development of “Guidance for Protecting Building Environments from Airborne Chemical, Biological, or Radiological Attacks.” The report, published by the National Institute of Occupational Safety and Health, was developed under the auspices of the Interagency Work Group on Building Air Protection of the Office of Homeland Security (OHS). This work group was formed in January 2002 under the Medical and Public Health Preparedness Policy Coordinating Committee of the OHS and includes representatives from many federal agencies.

The document suggests preventive measures that building owners and managers can implement promptly to protect building air environments from a terrorist release of chemical, biological, or radiological contaminants. These recommendations, focusing on short-term actions, are only the beginning of a process to develop more comprehensive guidance by the Building Air Protection Work Group. NIST’s role in this document was the development of material on building airtightness. NIST is continuing to work with the work group and will have a key role in its next document, which will address the role of particulate and gaseous filtration in protecting indoor environments from airborne threats. Copies of the report can be downloaded from www.cdc.gov/niosh, which is linked from the NIST website www.bfrl.nist.gov.

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CORRECTIVE REFRACTIVE OPTICS SHARPENS FOCUS OF SANS INSTRUMENT

Pioneering work done at the NIST Center for Neutron Research (NCNR), in collaboration with a private company, demonstrated that focusing a cold neutron beam with multiple biconcave lenses is a practical means of improving the resolution of long-flight-path, pinhole collimation, small-angle neutron scattering (SANS) instruments. Lens systems, consisting of linear arrays of up to 30 biconcave magnesium fluoride single crystals, are now used routinely on two 30 m SANS instruments at the NCNR nearly double of the size of microstructural features that can be resolved.

The lens system would be even more effective at longer wavelengths were it not for the spreading of the focal spot in the vertical direction due to gravity. A recent development has been to add large-apex-angle, single-crystal prisms in front of the lenses to correct for the gravitational aberration in the system. With the added prisms, the lenses are now effective at wavelengths up to 20 Å. The combination of lenses and prisms now enables SANS measurements to be made at Q -values as low as 0.005 nm^{-1} , a full factor of 3 improvement over the previous conventional pinhole collimation. The new optics are enabling microstructural studies that link nanoscale with micrometer-scale features in, for example, polymer-clay nanocomposites, gels, and fluxoid lattices in superconductors.

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CRYSTAL STRUCTURES AND PHASE EQUILIBRIA OF CERAMICS FOR LOW TEMPERATURE CO-FIRED CERAMICS

The primary technology drivers for wireless consumer devices and computer applications are miniaturization, higher frequency, lower operating voltages, reduction of component part count, and increased functionality. For example, multiband wireless telephones will require the packaging of two or three radios within the same cell phone format. One solution to this challenge is multilayer ceramic integrated circuit (MCIC) technology, which organizes the components into a single module containing all the passive and active components. MCIC technology requires the development of ceramic dielectrics which may be co-fired with high-conductivity metals; that is, low temperature co-fired ceramics (LTCC).

Currently available LTCC materials have relatively low dielectric constants of about 10; new ceramics with higher permittivities are required to develop integrated

filters and capacitors. As part of an ongoing program on dielectric ceramics, NIST researchers applied high-resolution electron microscopy and x-ray diffraction to determine crystal structures and phase equilibria of a new, low-sintering-temperature dielectric ceramic in the Li-Nb-Ti-O system. This material is based on the solid solution $\text{Li}_{1+x-y}\text{Nb}_{1-x-3y}\text{Ti}_{x+4y}\text{O}_3$, and exhibits excellent dielectric properties including chemically tunable dielectric constants of 55 to 78, near-zero temperature coefficients of the resonant frequency, and dielectric losses of less than 10^{-3} at frequencies above 1 GHz. Moreover, the sintering temperature of these ceramics can be reduced to 950°C by small additions of V_2O_5 , which permits co-firing with silver electrodes, thus making this material an attractive candidate for low-temperature co-fired ceramic (LTCC) technology.

NIST research on $\text{Li}_{1+x-y}\text{Nb}_{1-x-3y}\text{Ti}_{x+4y}\text{O}_3$ ceramics has demonstrated that what actually forms is not a solid solution but rather a homologous series of distinct compounds which feature intergrowths of LiNbO_3 -type blocks and corundum-type layers. Successive compounds differ in the thickness of the LiNbO_3 blocks, expressed as the number of cation (or anion) layers, n , which increases with decreasing Ti/(Li+Nb) ratio. The phase field containing these compounds was confirmed to encompass a series of such ordered commensurate intergrowths, with n ranging from 5 to 54. The structural nature of these ceramics bears important implications for their processing and properties as LTCC ceramics, since the complexity and large number of possible phases considerably extend the “tunability” of properties.

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NEW APPROACH TO FATIGUE TESTING OF CHIP-LEVEL INTERCONNECTS

Researchers at NIST, in collaboration with the Max-Planck-Institute for Metals Research, have been conducting fatigue testing of thin aluminum and copper films on silicon-based substrates through the application of high current density ac signals. This novel approach to studying cyclic deformation of chip-level interconnects depends upon Joule self-heating of the metal and the differences in coefficient of thermal expansion between film and substrate materials. Effects of frequency, waveform, encapsulant, and local interconnect microstructure are being investigated in materials systems in their actual in-use configurations, without special specimen preparation or removal of films from their substrates.

One recent result shows that soft polymeric-based encapsulants do not suppress deformation-induced topography in aluminum lines. This test method provides a means for understanding the reliability of interconnects subjected to severe repeating loads due to power cycling, energy conservation, and processor loading.

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MONTE CARLO SIMULATIONS COMPLETED FOR SEM RESOLUTION STUDY

In June, a NIST scientist completed Monte Carlo simulations of scanning electron microscope (SEM) imaging of resist lines on silicon. These represent the first part of a study being performed at the behest of International SEMATECH on the limits imposed by noise on the ability of the SEM to resolve differences between widths of lines. In the simulations, the samples are 800 nm of PAR 810 resist on 10 nm of BARC (bottom anti-reflection coating) on thick polycrystalline silicon. The resist lines edge angle (relative to the plane of the substrate) was varied from 81° to 93°, simulating a range of possible manufacturing process variation. The effect of the SEMs finite depth of field was modeled by varying the convergence angle of the incident electron beam.

These results will form the inputs for the next stage of the study, in which simulated noise will be added to the images and a number of different algorithms will be employed to determine edge positions from these simulated noisy images. By comparing the edge positions so determined with the known true edge positions, the accuracy and repeatability of the various edge detection algorithms can be determined as a function of edge shape and instrument depth of field.

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NIST EXPANDS UV OPTICAL MATERIAL CHARACTERIZATION FACILITY

NIST has completed a series of measurements to determine the damage threshold and lifetime of optical materials under intense ultraviolet (UV) radiation produced by excimer lasers. Using a capability developed by a NIST scientist and NIST guest researcher, the integrated energy density (or dose) necessary to cause

catastrophic damage of selected UV materials was determined.

These studies, part of an on-going program to develop a primary standard for 157 nm excimer laser power and energy measurements, represent the first quantitative damage studies of UV optical materials for use with excimer lasers. Long-term exposure studies were carried out in cooperation with MIT Lincoln Laboratory to simulate typical calorimeter operating conditions.

The damage measurements were performed using a beamsplitter-based calibration system in which a spatially uniform beam from an ArF excimer laser is generated using a special beam homogenizer. The beam profiles were recorded and characterized to determine the integrated dose at the sample plane. Measurement trace-ability for these measurements stems from an electrically calibrated, primary standard calorimeter developed by NIST scientists.

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LASER-FOCUSED NANOLINES LINK THE MICRO- AND NANO-WORLDS

A collaboration of scientists from NIST and the University of Nijmegen (Netherlands) has shown that two sets of nearly-identical nanolines created by laser-focused atomic deposition can interfere with each other to form a metrologically significant moiré pattern. A paper describing this work was published in the June 10, 2002 issue of *Applied Physics Letters*.

In this work, nanolines are deposited on a clear substrate (indium tin oxide) by focusing chromium atoms in a near-resonant laser standing wave, a technique first demonstrated at NIST in 1993. Each set of lines individually has a pitch of nonometer scale, which arises from the wavelength of the laser used to form them. However, by doing this twice, using two different but closely-spaced atomic transitions in chromium, a spatial beating pattern is observed with a period of 44.46 μm .

Because the nanolines are deposited in standing waves tuned to atomic resonances, their pitches can be characterized to extremely high accuracy. This accuracy is carried over into the pitch of the moiré pattern, providing a metrological link between nanoscopic and microscopic length scales.

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NIST HOSTS CONFERENCE ON DESIGNS FOR GENERALIZED LINEAR MODELS

Celebrating the 30th anniversary of the Nelder and Wedderburn paper introducing generalized linear models (GLMs), NIST hosted the Conference on Designs for Generalized Linear Models in Gaithersburg in April 2002. The primary goal of the conference was to provide a forum for interaction among researchers working on diverse areas of designs for GLMs. A main focus was on the problem of design dependency on the unknown parameters of the model. Secondary goals of the conference included the sharing of GLM design methodology across application areas and the introduction and stimulation of young researchers and graduate students in the GLM design area.

The concept of GLMs, as a unified class of regression models for discrete and continuous variables, was first introduced by Nelder and Wedderburn in their seminal 1972 JRSS/A paper. Since that time, GLMs have served as a paradigm for a large class of problems in applied statistics and have been used routinely in dealing with observational studies. GLMs have proven very effective in several application areas ranging from medicine to economics to quality control to sample surveys. Many statistical developments in terms of modeling and methodology, in the past twenty years, may be viewed as special cases of GLMs.

The conference opened with Nelder's presentation about the introduction and history of GLMs and closed with a panel discussion about the future of GLMs. Other technical sessions covered optimal and two-stage designs, sequential designs, generalized linear mixed models, comparison of designs for logistic models and industrial split-plot experiments, and designs for variance components estimation.

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PHOTOIONIZATION-OF-CO₂ DATABASE AVAILABLE ONLINE

NIST has made available on the Web a database containing photoionization efficiency, vibrational branching ratios, and asymmetry parameters in the photoionization of CO₂ in the region between 65 nm and 84 nm.

The study of CO₂ photoionization historically has attracted much attention due to the importance of the ionization of CO₂ in the photophysics of planetary atmospheres, including Earth's atmosphere. Additionally, CO₂ is an integral part of the carbon cycle for plant life and, as a consequence, the photochemistry and photophysics of this molecule has been the subject of considerable interest by a number of scientists over the

last several decades. This work was motivated because CO₂ is a prototype molecular system for complex quantum-mechanical configuration mixing of bound, excited states and states in the ionization continuum. These interactions produce distinct resonances in both energy and angle.

This data set is the most complete available anywhere for information on the ionization of CO₂ in the wavelength region within 10 eV of ionization onset. The high resolution of these data resolve individual molecular vibrational states. The graphical user interface designed for the database includes options to retrieve any subset of the data in several different formats, including graphical output (with or without errors bars displayed), HTML table output (which is fully accessible according to the Section 508 accessibility requirements), and ASCII text output (which is useful for copying and pasting into other applications).

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BLACKBODY SOURCES CALIBRATED AT THE LBIR FACILITY

The Low-Background Infrared (LBIR) facility provides means to calibrate low power thermal-infrared sources (blackbodies). Since all material gives off less infrared radiation as it gets cooler, the inner walls of the LBIR facility are cooled cryogenically to prevent them from interfering with the source being measured.

A major application of this facility is to calibrate measurement standards used in the National Missile Defense (NMD) program. (The LBIR environment simulates the cold background of space.) NIST recently characterized two blackbody sources, for two private companies, being used as NIST-traceable standards for the calibration of Exoatmospheric Kill Vehicle (EKV) infrared sensors.

These calibrations were made possible by recent improvements at the LBIR facility which increased its sensitivity by a factor of 10. A new, lower noise-floor absolute cryogenic radiometer (ACR II) became operational as the standard infrared detector. An expansion in refrigeration capacity provides a much more stable test environment. Further, automation of the calibration test sequence has allowed the acquisition of more data, thus improving statistics.

These improvements, and others planned, will assist in meeting ongoing calibration requirements of the EKV program.

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NIST RELEASES UPDATED REFERENCE DATA SET TO FIGHT COMPUTER CRIME

NIST recently released an updated National Software Reference Library (NSRL) Reference Data Set to assist the law enforcement community in fighting crimes involving computers. The NSRL encompasses a repository of all types of software, including operating systems, vertical applications, database management systems, graphics packages, games, etc. It also includes a database of file profiles, i.e., software fingerprints, that can be used to identify known and unknown files on computers, diskettes, magnetic tapes, CDs, etc., that have been seized pursuant to investigation. Containing over 8 million file signatures, the data set is available at <http://www.nist.gov/srd/nistsd28.htm>.

The NSRL can save an investigator hundreds of hours in an investigation. A single computer or hard disk drive can contain between 10 000 and 50 000 individual files, each of which must be examined for probative value. If multiple computers, disk drives, magnetic tapes, or other media are involved, staff hours could reach into the thousands and take months to finish. The NSRL reduces the time it takes to investigate each computer by allowing the investigator to weed out files that have known profiles and fingerprints in NIST's database. The investigator then can concentrate on the unknown files. NIST produces the NSRL using state-of-the-art software and computers to verify the fingerprints of each software package.

A collaboration of NIST, the National Institute of Justice, the Federal Bureau of Investigation, the Defense Cyber Crime Center, other federal, state and local law enforcement, and software vendors, the NSRL is an example of the application of technology to investigate crimes involving computers, such as child pornography, racketeering, cyber-attacks, illegal gambling, Internet fraud, and software piracy.

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NIST'S NOISE-TEMPERATURE MEASUREMENT SERVICE NOW AVAILABLE AT CONTINUOUS FREQUENCIES FROM 1 GHz TO 65 GHz

NIST has completed the testing of the 1 GHz to 4 GHz units of its coaxial radiometer (NFRad) and has reopened the noise-temperature measurement service for that frequency band. This marks the completion of a multiyear effort to restore and extend noise-temperature measurement services for coaxial and waveguide noise sources. Noise temperatures of noise sources now can be measured at virtually any frequency from 1 GHz

to 65 GHz as well as at two low-frequency points at 30 MHz and 60 MHz. Noise sources with common coaxial connectors can be measured up to 40 GHz, and wave-guide noise sources can be measured from 8.2 GHz to 65 GHz, with the exception of the 26.01 GHz to 26.5 GHz range of the WR-42 band. Noise sources with noise temperatures from about 50 K up to 15 000 K and reflection coefficients up to 0.2 can be measured. The typical expanded uncertainty (coverage factor of 2) ranges from about 0.9 % to about 1.8 %, depending on frequency and connector type, for noise sources with noise temperatures from 1000 K to about 12 000 K and a reflection coefficient of less than about 0.1.

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NIST RESEARCHERS DEMONSTRATE STACKED JOSEPHSON JUNCTION ARRAYS USING MoSi₂ BARRIERS

Researchers at NIST have demonstrated that molybdenum di-silicide is an excellent normal-metal material for junction barriers in high-density arrays of superconductor-normal-metal-superconductor (SNS) Josephson junctions. For the past few years the project has been searching for a practical barrier material to allow them to vertically stack junctions in order to make three-dimensional arrays. Higher junction density is required to increase the output voltage as well as the operating bandwidth of both programmable and ac Josephson array circuits.

A NIST researcher and a guest researcher from South Korea, have succeeded in making the first two- and three-junction stacks using molybdenum di-silicide as the normal metal and niobium as the superconductor. Precise three-dimensional control of the junctions during fabrication is critical for achieving uniformity of the electrical characteristics for the junction stacks and large high-density arrays. These new MoSi₂ circuits have demonstrated sufficient uniformity for thousands of junctions to display large quantized-voltage steps at frequencies up to 20 GHz. The barrier material has similar etch properties to niobium so that it is possible to reactively etch uniform vertical stacks and three-dimensional structures. This was not the case for previously attempted barrier materials such as palladium-gold and titanium. A contributing factor to this success is that the arrays were fabricated using a recently installed fully automated multilayer sputtering system that allows sub-nanometer control of vertical film thicknesses. Precise control of the barrier thickness, typically 20 nm to 30 nm, is essential because junction electrical

characteristics depend exponentially on barrier thickness. Reproducibility and uniformity of the fabrication process make molybdenum di-silicide the leading candidate for future lumped-array Josephson voltage standard circuits and systems.

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NEW PULSED LASER DEPOSITION TECHNIQUE SPEEDS SEARCH FOR HIGH-TECH THIN FILMS

A new rapid prototyping technique developed by two NIST scientists could speed up the search for thin films with properties optimized for next-generation memory devices and wireless communications circuits. The novel method—called dual-beam, dual-target, pulsed-laser deposition—can be used to create libraries of inorganic thin films that vary, predictably, in thickness and chemical composition. Mass producing samples of films composed of two or more compounds opens the way to high-throughput screening that can narrow the hunt for new materials.

NIST scientists demonstrated their new technique by creating libraries of barium strontium titanate (BST) films, a candidate to replace silicon-dioxide insulators in future dynamic random access memories. The technique splits a laser beam so that the high-energy light strikes two sets of starting materials simultaneously. Both target materials vaporize, creating gas plumes that mix before depositing and crystallizing on a silicon wafer nearby. By adjusting the laser's energy and the spacing between the targets, the researchers can control both the composition and thickness of the film deposited on the substrate. For example, the researchers created samples with narrow bands of uniform thickness, while the relative amounts of barium, strontium, and titanium varied systematically across each band.

Data gathered automatically after the process are used to map film thickness. Using a model that they developed, the scientists then can estimate the chemical composition at hundreds of points in a sample, eliminating a time-consuming chore. They now are extending the technique to other combinations of inorganic materials.

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ONE-DIMENSIONAL “PENCIL” DISORDER DISCOVERED FOR A CLASS OF INTERMETALLIC COMPOUNDS

Deviations of atoms from an average crystal structure caused by structural distortions, chemical ordering, or

the presence of defects cause diffuse intensity in scattering experiments. The diffuse scattering is usually difficult to detect by x-ray or neutron diffraction, but it is readily observed in electron diffraction. Recently, scientists at NIST discovered an unusual planar diffuse scattering for a group of intermetallic compounds, tetragonal Zr_9M_{11} ($M = Pd, Pt, \text{ and } Ni$) and hexagonal Co_3Y_4 , by using transmission electron microscopy. The near two-dimensional intensity planes are parallel to each other, and the inter-planar distance is incommensurate with fundamental translations of the average crystal structure.

By combining electron microscopy with powder neutron diffraction, the researchers determined that these compounds could be described as having a host periodic structure, but with one-dimensional structural channels. The channels are filled with strings of atoms, and a one-dimensional arrangement of the atoms along the strings has a periodicity that differs from the host structure. There is a weak correlation between the relative positions (phase) of the strings, i.e., the strings are largely independent of each other. In previous x-ray diffraction studies the possible existence of such disorder was proposed in order to explain unusually high temperature factors; however, the unambiguous demonstration of the phenomenon was presented in this NIST work for the first time. It is possible that the presence of the strings could impose special one-dimensional properties in a three-dimensional crystal.

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Standard Reference Materials

NIST PRODUCES TWO NEW WAVELENGTH CALIBRATION STANDARDS FOR THE WAVELENGTH DIVISION MULTIPLEXING L-BAND

NIST has developed two wavelength calibration transfer standards for the new L-band of wavelength division multiplexed (WDM) optical fiber communication systems. These standards, based on the absorption spectrum of carbon monoxide, are now available as Standard Reference Materials (SRM) 2514 and 2515.

Wavelength division multiplexing in optical fiber communication systems increases bandwidth by using many wavelength channels. Current WDM systems

typically employ 50 GHz or 100 GHz channel spacing (0.4 nm or 0.8 nm, respectively) in the 1530 nm to 1560 nm WDM C-band, but WDM is expanding into the L-band region (approximately 1565 nm to 1625 nm). Wavelength references are needed in these regions to calibrate instruments such as optical spectrum analyzers, tunable lasers, and wavelength meters that are used to characterize WDM system components and measure the channel wavelengths. To complement wavelength calibration references for the WDM C-band (SRM 2517a, acetylene, and SRM 2519, hydrogen cyanide), NIST has developed SRMs 2514 and 2515. SRM 2514 can be used to calibrate the wavelength scale of measurement equipment in the 1560 nm to 1595 nm region. The unit is a single-mode optical-fiber-coupled absorption cell containing carbon monoxide $^{12}\text{C}^{16}\text{O}$ gas, which has many absorption lines in this region. NIST has characterized the pressure-induced shifts of these absorption lines and certifies the center wavelengths of 41 lines with uncertainties ranging from 0.4 pm to 0.7 pm. SRM 2515 is nearly identical to SRM 2514, except that it contains the $^{13}\text{C}^{16}\text{O}$ isotopic species of carbon monoxide. This isotopic species has numerous absorption lines at longer wavelengths, ranging from 1595 nm to 1630 nm. NIST also certifies the center wavelengths of 41 lines for this SRM. Since they are based on the quantized vibrational and rotational motion of molecules, these SRMs provide wavelength references that are very stable under changing environmental conditions.

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The main body of NIST SP 330 gives the essentials of the current form of the SI. However, Appendix 1 provides the Resolutions, Recommendations, and Declarations put forward on units of measurement and on the SI since 1889 by the General Conference on Weights and Measures (CGPM) and the International Committee for Weights and Measures (CIPM). Further, Appendix 2 summarizes the current state of the practical realizations of some important SI units, while Appendix 3 gives a brief description of the bodies established by the Meter Convention (the CGPM, CIPM, and BIPM), which was signed in Paris on 20 May 1875 by 17 States including the United States.

The 2001 Edition of SP 330 replaces its immediate predecessor, the 1991 Edition, which was based on the sixth edition of the BIPM SI publication. Like its predecessor, the 2001 Edition of SP 330 was edited by NIST physicist Barry N. Taylor.

Single copies of the 75-page NIST SP 330, 2001 Edition, may be obtained by contacting the NIST Metric Program, 100 Bureau Drive, Stop 2000, Gaithersburg, MD 20899-2000; telephone: 301-975-3690; fax: 301-948-1416; email: metric_prg@nist.gov. NIST SP 330 is also available online at the NIST Web site entitled "NIST Reference on Constants, Units, and Uncertainty," physics.nist.gov/cuu.

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Order the following NIST publications—FIPS and NISTIRs—from the National Technical Information Service, Springfield, VA 22161.

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