Material Matters

The Quarterly Magazine of NIST's Material Measurement Laboratory

Fall 2014

Tracing the Ebola Outbreak

Improving Microscopic Batteries

Building the New Kilogram

National Institute of
Standards and Technology
U.S. Department of Commerce

About NIST's Material Measurement Laboratory

The Material Measurement Laboratory (MML) is one of two metrology laboratories within the National Institute of Standards and Technology (NIST). The laboratory supports the NIST mission by serving as the national reference laboratory for measurements in the chemical, biological and material sciences. Our activities range from fundamental and applied research on the composition, structure and properties of industrial, biological and environmental materials and processes, to the development and dissemination of tools including reference measurement procedures, certified reference materials, critically evaluated data, and best practice guides that help assure measurement quality. Our research and measurement services support areas of national importance, such as:

- Advanced materials, from nanomaterials to structural steels to complex fluids
- Energy, from characterization and performance of fossil and alternative fuels to next-generation renewable sources of energy
- The environment, from the measurement of automotive exhaust emissions and other pollutants to assessment of climate change and the health and safety aspects of man-made nanomaterials
- Food safety and nutrition, from contaminant monitoring to ensuring the accuracy of nutrition labels
- Health care, from clinical diagnostics to tissue engineering and more efficient manufacturing of biologic drugs
- Infrastructure, from assessing the country's aging bridges and pipelines to the quality of our drinking water
- Manufacturing, from lightweight alloys for fuel-efficient automobiles to biomanufacturing, advanced electronics, and data for chemical manufacturing
- Safety, security and forensics, from gunshot and explosive residue detection, to ensuring the performance of body armor materials, to DNA-based human identity testing

The Material Measurement Laboratory also coordinates the NIST-wide Standard Reference Materials® (SRM) and Standard Reference Data programs, which include production, documentation, inventory, marketing, distribution and customer service.

The Material Measurement Laboratory is home to more than 900 staff members and visiting scientists at six locations:

- NIST main campus in Gaithersburg, MD
- NIST Boulder Laboratories in Boulder, CO
- Hollings Marine Laboratory in Charleston, SC , where NIST staff work side-by-side with scientists from NOAA, the South Carolina Department of Natural Resources, the College of Charleston, and the Medical University of South Carolina to provide the science, biotechnology and standards needed to understand links between environmental conditions and the health of marine organisms and humans
- Institute for Bioscience and Biotechnology Research (formerly CARB) in Rockville, MD, where scientists from NIST, the University of Maryland College Park, and the University of Maryland School of Medicine conduct research on measurement science and standards issues associated with advanced therapeutics
- Brookhaven National Laboratory in Upton, NY where, in partnership with the Department of Energy, the laboratory has a user facility that enables researchers from industry, academia and other government agencies to apply synchrotron-based x-ray spectroscopy techniques to the development of products like oil additives and next-generation electronics
- The Advances in Biological and Medical Measurement Science (ABMS) Program at Stanford University in Palo Alto, CA, where NIST staff are working elbow-to-elbow with Stanford faculty groups and commercial affiliates to develop standards and tools that enable translation of innovations in quantitative biology and engineered biology to clinical and commercial practice

Cover image: A "nanoforest" of nanowire lithium-ion batteries.
Credit: Oleshko/NIST

A Message from the MML Director

An important part of NIST's mission is improving the quality of lives, and in NIST's Material Measurement Laboratory (MML) we are pursuing that goal through a number of important activities. Enhancing public health and safety through collaboration with other federal and non-federal partners is a key component of this, and MML is uniquely suited to support that aspect of NIST's mission in a variety of ways.

MML has unique research capabilities and strengths, and at times is optimally suited to help other organizations answer very specific kinds of questions. For instance, as you can read in this issue of Material Matters, NIST reference materials recently played a role in a study led by the Broad Institute to understand the spread of Ebola (Stephen K. Gire et al., Genomic surveillance elucidated Ebola virus origin and transmission during the 2014 outbreak, Science, 345, 1369 (2014)).

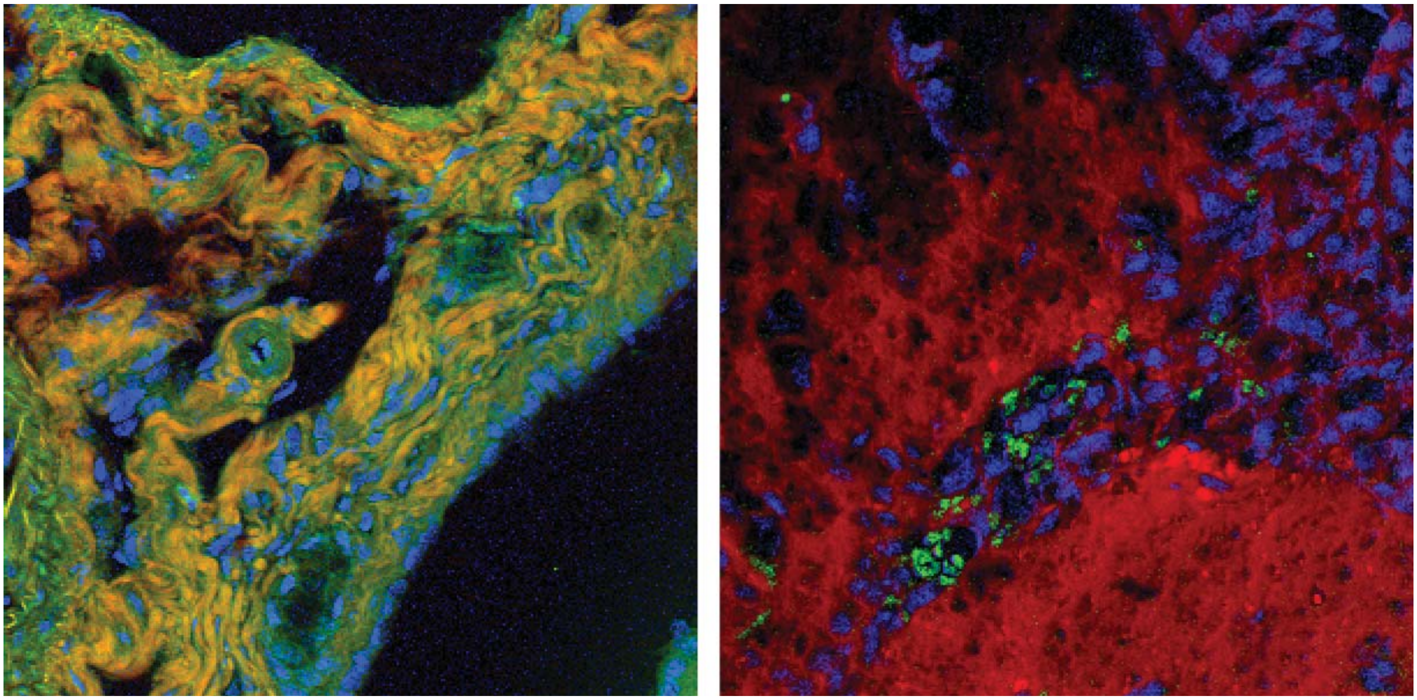
In a different area, NIST recently signed an agreement with the NFL, Under Armour and GE to collaborate on the development of advanced materials for protective equipment for athletes and other professionals. This partnership is a follow up to the "Head Health Challenge II," which has recently provided challenge prizes for different training regimens that could result in improved head protection behavior, sideline tools for assessing brain injury on the spot, biomarker measurements, and new materials systems that would result in improved head injury protections, primarily as components in helmets. More information is available at https://ninesights.ninesigma.com/web/head-health/awardees.

Finally, MML plays a role in protecting the public from attempts to transport explosives and contraband through our borders and airspace and into public venues. NIST MML works closely with federal and state agencies such as the U.S. Capitol Police, the Transportation Security Administration, and the U.S. Department of State to support their field-deployed trace explosives detection capabilities. We provide high quality test materials on an as-needed basis that allow for agency-based field tests of detector performance. We also support the continual improvement of trace detection equipment and procedures through research and development.

These are just a few examples of how MML supports and enhances public health and safety. As you can read here, MML strives to improve our lives in a wide range of interesting and important ways.

Laurie Locascio, Ph.D.
Director, Material Measurement Laboratory
NIST

Fall 2014 - Contents



High-speed BCARS allows detailed mapping of specific components of tissue samples. A false-color BCARS image of mouse liver tissue (left) picks out cell nuclei in blue, collagen in orange and proteins in green. An image of tumor and normal brain tissue from a mouse (right) has been colored to show cell nuclei in blue, lipids in red and red blood cells in green. Images show an area about 200 micrometers across.

Credit: Camp/NIST

Enhanced NIST Instrument Enables High-speed Chemical Imaging of Tissues

Note: A version of this story previously appeared in NIST's TechBeat *on July 22, 2014*

A NIST research team, working with the Cleveland Clinic, has demonstrated a dramatically improved technique for analyzing biological cells and tissues based on characteristic molecular vibration "signatures." The new NIST technique is an advanced form of the widely used spontaneous Raman spectroscopy, but one that delivers signals that are 10,000 times stronger than obtained from spontaneous Raman scattering, and 100 times stronger than obtained from comparable "coherent Raman" instruments, and uses a much larger portion of the vibrational spectrum of interest to cell biologists.*

The technique, a version of "broadband, coherent anti-Stokes Raman scattering" (BCARS), is fast and accurate enough to enable researchers to create high-resolution images of biological specimens, containing detailed spatial information on the specific biomolecules present at speeds fast enough to observe changes and movement in living cells, according to the NIST team.

Raman spectroscopy is based on a subtle interplay between light and molecules. Molecules have characteristic vibration frequencies associated with their atoms flexing and stretching the molecular bonds that hold them together. Under the right conditions, a photon interacting with the molecule will absorb some of this energy from a particular vibration and emerge with its frequency shifted by that frequency—this is "anti-Stokes scattering." Recording enough of these energy-enhanced photons reveals a characteristic spectrum unique to the molecule. This is great for biology because in principle it can identify and distinguish between many complex biomolecules without destroying them and, unlike many other techniques, does not alter the specimen with stains or fluorescent or radioactive tags.

Using this intrinsic spectral information to map specific kinds of biomolecules in an image is potentially very powerful, but the signal levels are very faint, so researchers have worked for years to develop enhanced methods for gathering these spectra.** "Coherent" Raman methods use specially tuned lasers to both excite the molecular vibrations and provide a bright source of probe photons to read the vibrations. This has partially solved the problem, but the coherent Raman methods developed to date have had limited ability to access most of the available spectroscopic information.

Most current coherent Raman methods obtain useful signal only in a spectral region containing approximately five peaks with information about carbon-hydrogen and oxygen-hydrogen bonds. The improved method described by the NIST team not only accesses this spectral region, but also obtains excellent signal from the "fingerprint" spectral region, which has approximately 50 peaks—most of the useful molecular ID information.

The NIST instrument is able to obtain enhanced signal largely by using excitation light efficiently. Conventional coherent Raman instruments must tune two separate laser frequencies to excite and read different Raman vibration modes in the sample. The NIST instrument uses ultrashort laser pulses to simultaneously excite all vibrational modes of interest. This "intrapulse" excitation is extremely efficient and produces its strongest signals in the fingerprint region. "Too much light will destroy cells," explains NIST chemist Marcus Cicerone, "So we've engineered a very efficient way of generating our signal with limited amounts of light. We've been more efficient, but also more efficient where it counts, in the fingerprint region."

Raman hyperspectral images are built up by obtaining spectra, one spatial pixel at a time. The hundred-fold improvement in signal strength for the NIST BCARS instrument makes it possible to collect individual spectral data much faster and at much higher quality than before—a few milliseconds per pixel for a high-quality spectrum versus tens of milliseconds for a marginal quality spectrum with other coherent Raman spectroscopies, or even seconds for a spectrum from more conventional spontaneous Raman instruments. Because it's capable of registering many more spectral peaks in the fingerprint region, each pixel carries a wealth of data about the biomolecules present. This translates to high-resolution imaging within a minute or so whereas, notes NIST electrical engineer Charles Camp, Jr., "It's not uncommon to take 36 hours to get a low-resolution image in spontaneous Raman spectroscopy."

"There are a number of firsts in this paper for Raman spectroscopy," Camp adds. "Among other things we show detailed images of collagen and elastin—not normally identified with coherent Raman techniques—and multiple peaks attributed to different bonds and states of nucleotides that show the presence of DNA or RNA."

- Michael Baum, NIST

Contact: Charles Camp, charles.camp@nist.gov

*C.H. Camp Jr., Y.J. Lee, J.M. Heddleston, C.M. Hartshorn, A.R. Hight Walker, J.N. Rich, J.D. Lathia and M.T. Cicerone. High-speed coherent Raman fingerprint imaging of biological tissues. Nature Photonics, Published online July 20, 2014. doi:10.1038/nphoton.2014.145.

**See for example the 2010 Tech Beat story, "Faster CARS, Less Damage: NIST Chemical Microscopy Shows Potential for Cell Diagnostics" at www.nist.gov/public_affairs/techbeat/tb20101013.cfm#cars.

First Director of New Office of Data and Informatics Joins NIST

Credit: NIST

Dr. Robert J. Hanisch has joined NIST as the Director of the new Office of Data and Informatics (ODI). Dr. Hanisch was previously a Senior Scientist at the Space Telescope Science Institute (STScI), Baltimore, Maryland, and the Director of the U.S. Virtual Astronomical Observatory, a program funded by the National Science Foundation and the National Aeronautics and Space Administration. Over the past twenty years Dr. Hanisch has led many efforts in the astronomy community in the area of information systems and services, focusing particularly on efforts to improve the accessibility and interoperability of data archives and catalogs. He was the first chair of the International Virtual Observatory Alliance Executive Committee (2002-2003) and continues as a member of the IVOA Executive. From 2000 to 2002 he served as Chief Information Officer at STScI, overseeing all computing, networking, and information services for the Institute. Prior to that he had oversight responsibility for the Hubble Space Telescope Data Archive and led the effort to establish the Multimission Archive at Space Telescope—MAST—as the optical/UV archive center for NASA astrophysics missions. He completed his Ph.D. in Astronomy in 1981 at the University of Maryland, College Park.

Hanisch is now in the process of staffing up the ODI and identifying the goals for the Office for the coming several years:

* Modernizing the Standard Reference Data, with improved user interfaces and applications programming interfaces (APIs) that allow use of SRD through programs and scripts

* Implementing data management plans and providing support for good data management practices throughout MML

* Providing consultation services to MML staff on informatics and analytic techniques

ODI intends to make NIST data products much more widely available and usable to an audience that includes the traditional industrial and research communities as well as to educators and the public. Hanisch also hopes to instill an appreciation of state-of-the-art data management—preservation, curation, interoperability—that will become the norm at NIST.

NIST MML Reference Materials Key in Tracing Ebola Outbreak

Ebola is a serious human disease, killing three-quarters of infected people. The current outbreak of the virus started in Guinea in February 2014 and is spreading through West Africa, with the number of cases doubling every 35 days. The rapid spread of this serious disease prompted an international collaboration to find the origin and track the spread of this disease through genomic surveillance, and a NIST reference material played a prominent role in assuring confidence in their results.

The results of this study were published in the August issue of Science. In this work, the Ebola virus genomes from about 100 patients were sequenced to observe the accumulation of interhost and intrahost variation, allowing the scientists to characterize patterns of viral transmission. Synthetic RNA controls, prepared and donated by NIST, were spiked-in to each individual sample so that the sequencing process could be tracked and any cross-contamination could be identified. The scientists hope that this study will help with ongoing epidemiological and genomic surveillance, the development of therapeutics, and strategic planning for health organizations.

Stephen K. Gire et al., Genomic surveillance elucidated Ebola virus origin and transmission during the 2014 outbreak, Science, 345, 1369(2014)

DOI: 10.1126/science.1259657

Contact: Marc Salit, marc.salit@nist.gov

NIST Scientists Improve Microscopic Batteries with Homebuilt Imaging Analysis

Note: A version of this story previously appeared in NIST's TechBeat *on September 26, 2014*

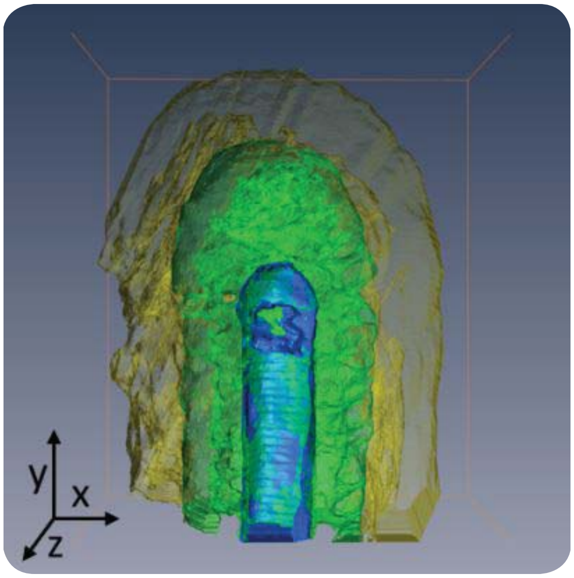
In a rare case of having their cake and eating it too, scientists from NIST and other institutions have developed* a toolset that allows them to explore the complex interior of tiny, multi-layered batteries they devised. It provides insight into the batteries' performance without destroying them—resulting in both a useful probe for scientists and a potential power source for micromachines.

The microscopic lithium-ion batteries are created by taking a silicon wire a few micrometers long and covering it in successive layers of different materials. Instead of a cake, however, each finished battery looks more like a tiny tree.

The analogy becomes obvious when you see the batteries attached by their roots to silicon wafers and clustered together by the million into "nanoforests," as the team dubs them.

But it's the cake-like layers that enable the batteries to store and discharge electricity, and even be recharged. These talents could make them valuable for powering autonomous MEMS – microelectromechanical machines – which have potentially revolutionary applications in many fields.

With so many layers that can vary in thickness, morphology and other parameters, it's crucial to know the best way to build each layer to enhance the battery's performance, as the team found in previous research.** But conventional transmission electron microscopy (TEM) couldn't provide all the details needed, so the team created a new technique that involved multimode scanning TEM (STEM) imaging. With STEM, electrons illuminate the battery, which scatters them at a wide range of angles. To see as much detail as possible, the team decided to use a set of electron detectors to collect electrons in a wide range of scattering angles, an arrangement that gave them plenty of structural information to assemble a clear picture of the battery's interior, down to the nanoscale level.



A colorized 3D side view of a battery showing the metallized silicon core and its outer layers. Credit: Oleshko/NIST

The promising toolset of electron microscopy techniques helped the researchers to home in on better ways to build the tiny batteries. "We had a lot of choices in what materials to deposit and in what thicknesses, and a lot of theories about what to do," team member Vladimir Oleshko says. "But now, as a result of our analyses, we have direct evidence of the best approach.

”MEMS manufacturers could make use of the batteries themselves, a million of which can be fabricated on a square centimeter of a silicon wafer. But the same manufacturers also could benefit from the team's analytical toolset. Oleshko points out that the young, rapidly emerging field of additive manufacturing, which creates devices by building up component materials layer by layer, often needs to analyze its creations in a noninvasive way. For that job, the team's approach might take the cake.

- Chad Boutin, NIST

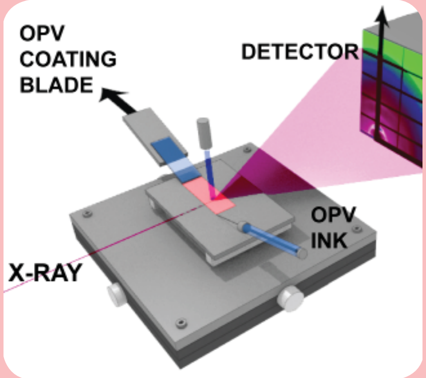
Contact: Vladimir Oleshko
vladimir.oleshko@nist.gov

*V.P. Oleshko, T. Lam, D. Ruzmetov, P. Haney, H.J. Lezec, A.V. Davydov, S.Krylyuk, J.Cumings and A.A. Talin. Miniature all-solid-state heterostructure nanowire Li-ion batteries as a tool for engineering and structural diagnostics of nanoscale electrochemical processes. Nanoscale, DOI: 0.1039/c4nr01666a, Aug. 15, 2014.

**See http://www.nist.gov/cnst/battery-032012.cfm

Watching Painted Solar Cells Dry

Electronics can now be found everywhere, from wireless tags that facilitate lean supply streams to the ubiquitous smartphone to point-of-care health monitors. Further proliferation of electronic devices requires the development of advanced manufacturing techniques for low cost, high volume, functional components: displays, sensors, and power supplies. Roll-to-roll (r-2-r) fabrication on a flexible web is emerging as an enabling manufacturing platform. One of the first applications for r-2-r manufacturing has been thin film solar cells based on the large-area printing of functional inks. A great challenge to the introduction of r-2-r techniques is the sensitivity of final device performance to the details of ink formulation and film drying. Subtle changes in the ink or drying conditions can cause radical changes in product performance.



Credit: DeLongchamp/Richter/NIST

Schematic of real-time experiment, showing custom film coater assembly, incident x-ray beam and imaging detector, and normal incidence optical probe.

In a recently-published article in Advanced Energy Materials (DOI: 10.1002/aenm.201400975), an international team of researchers, led by NIST and including researchers from King Abdullah University of Science and Technology (KAUST) and the Lawrence Berkeley National Laboratory conducted real-time, synchrotron-based X-ray scattering measurements of the development of material structure during the drying of solar cell inks in a model film coater. They studied a prototypical organic solar cell consisting of mixture of a semicrystalline polymer (poly-3-hexylthiophene) and a soluble 'buckyball' (C60). When these components are dissolved in a simple solvent, the ink produces films that exhibit poor solar cell efficiency and require a costly, high temperature annealing step. Small amounts of an additive improve the ink dramatically, resulting in high efficiency films immediately after drying. The on-line measurements established a detailed mechanism for the additive effect that includes 1) a reduction in the solvent quality, resulting in early aggregation of the polymer, and 2) a reduction in the glass transition temperature, allowing more crystal growth. Both effects improved the crystallinity of the film and improved device performance.

Contacts: Dean DeLongchamp,
dean.delongchamp@nist.gov;
Lee Richter, lee.richter@nist.gov

NIST Offers Electronics Industry Two Ways to Snoop on Self-Organizing Molecules

Note: A version of this story previously appeared in NIST's TechBeat *on October 22, 2014*

A few short years ago, the idea of a practical manufacturing process based on getting molecules to organize themselves in useful nanoscale shapes seemed … well, cool, sure, but also a little fantastic. Now the day isn't far off when your cell phone may depend on it. Two recent papers emphasize the point by demonstrating complementary approaches to fine-tuning the key step: depositing thin films of a uniquely designed polymer on a template so that it self-assembles into neat, precise, even rows of alternating composition just 10 or so nanometers wide.

The work by researchers at NIST, the Massachusetts Institute of Technology, and IBM Almaden Research Center focuses on block copolymers - a special class of polymers that under the proper conditions, will segregate on a microscopic scale into regularly spaced "domains" of different chemical composition. The two groups demonstrated ways to observe and measure the shape and dimensions of the polymer rows in three dimensions. The experimental techniques can prove essential in verifying and tuning the computational models used to guide the fabrication process development.

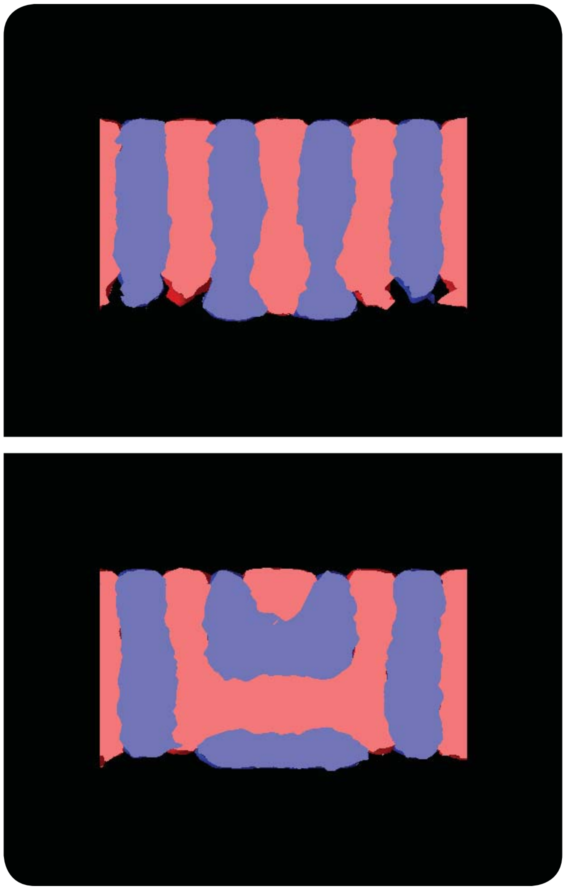
It's old news that the semiconductor industry is starting to run up against physical limits to the decades-long trend of ever-denser integrated chips with smaller and smaller feature sizes, but it hasn't reached bottom yet. Just recently, Intel Corp. announced that it had in production a new generation of chips with a 14-nanometer minimum feature size. That's a little over five times the width of human DNA.

At those dimensions, the problem is creating the multiple masking layers, sort of tiny stencils, needed to define the microscopic patterns on the production wafer. The optical lithography techniques used to create the masks in a process akin to old-school wet photography are simply not capable of reliably reproducing the extremely small, extremely dense patterns. There are tricks you can use such as creating multiple, overlapping masks, but they are very expensive.

Hence the polymers. "The issue in semiconductor lithography is not really making small features—you can do that—but you can't pack them close together," explains NIST materials scientist Alexander Liddle. "Block copolymers take advantage of the fact that if I make small features relatively far apart, I can put the block copolymer on those guiding patterns and sort of fill in the small details." The strategy is called "density multiplication" and the technique, "directed self-assembly."

Block copolymers (BCPs) are a class of materials made by connecting two or more different polymers that, as they anneal, will form predictable, repeating shapes and patterns. With the proper lithographed template, the BCPs in question will form a thin film in a pattern of narrow, alternating stripes of the two polymer compositions. Alternatively, they can be designed so one polymer forms a pattern of posts embedded in the other. Remove one polymer, and in theory, you have a near-perfect pattern for lines spaced 10 to 20 nanometers apart to become, perhaps, part of a transistor array.

If it works. "The biggest problem for the industry is the patterning has to be perfect. There can't be any defects," says NIST materials scientist Joseph Kline. "In both of our projects we're trying to measure the full structure of the pattern. Normally, it's only easy to see the top surface, and what the industry is worried about is that they make a pattern, and it looks okay on the top, but down inside the film, it isn't."



Computer simulations of two possible morphologies of a block copolymer film demonstrate the need for an accurate 3D imaging tool. Red and blue areas represent the two different phases of the polymer film, seen from the side. Each phase is about 12 nm wide. Viewed from the top, both would appear to have evenly separated rows of the "red" phase, but the bottom sample in fact has an unwanted horizontal band that will disrupt the pattern transfer. Soft X-ray scattering data can distinguish the two. Credit: Pitera/IBM Almaden Research Center

Kline's group, working with IBM, demonstrated a new measurement technique* that uses low-energy or "soft" X rays produced by the Advanced Light Source at Lawrence Berkeley National Labs to probe the structure of the BCP film from multiple angles. Because the film has a regular, repeating structure, the scattering pattern can be interpreted, much as crystallographers do, to reveal the average shapes of the stripes in the film. If a poor match between the materials causes one set of stripes to broaden out at the base, for example, it will show up in the scattering pattern. Their major innovation was to note that although the basic technique was developed using short-wavelength "hard" X rays that have difficulty distinguishing two closely related polymers, much better results can be obtained using longer wavelength X rays that are more sensitive to differences in the molecular structure.**

While X-ray scattering can measure average properties of the films, Liddle's group, working with MIT, developed a method to look, in detail, at individual sections of a film by doing three-dimensional tomography with a transmission electron microscope (TEM).*** Unlike the scattering technique, the TEM tomography can actually image defects in the polymer structure—but only for a small area. The technique can image an area about 500 nanometers across.

Between them, the two techniques can yield detailed data on the performance of a given BCP patterning system. The data, the researchers say, are most valuable for testing and refining computer models. "Our measurements are both fairly time-consuming, so they're not something industry can use on the fab floor," says Kline. "But as they're developing the process, they can use our measurements to get the models right, then they can do a lot of simulations and let the computers figure it out."

"It's just so expensive and time-consuming to test out a new process," agrees Liddle. "But if my model is well validated and I know the model is going to give me accurate results, then I can crank through the simulations quickly. That's a huge factor in the electronics industry."

- Michael Baum, NIST

Contact: R. Joseph Kline, joe.kline@nist.gov

---

*With the daunting name "resonant critical dimension small angle X-ray scattering" (res-CDSAXS).

**D.F. Sunday, M.R. Hammond, C. Wang, W. Wu, D. Delongchamp, M. Tjio, J. Cheng, J.W. Pitera, R.J. Kline. Determination of the internal morphology of nanostructures patterned by directed self assembly. ACS Nano, 2014, 8 (8), pp 8426–8437 DOI: 10.1021/nn5029289.

***K.W. Gotrik, T. Lam, A.F. Hannon, W. Bai, Y. Ding, J. Winterstein, A. Alexander-Katz, J.A. Liddle, C.A. Ross. 3D TEM Tomography of templated bilayer films of block copolymers. Advanced Functional Materials. Article first published online Oct. 2, 2014 DOI: 10.1002/adfm.201402457.

MATERIAL MEASUREMENT LABORATORY

ADVANCED MATERIALS

NIST Center for Automotive Lightweighting

Objective

Vehicles built from lightweight materials such as aluminum alloys, high-strength steels, and polymer composites are lighter, more fuel efficient, and produce fewer emissions than those built with current materials. Yet automakers lack the necessary tools for incorporation, because the data and material models that automakers need to reliably manufacture components from lightweight substitutes are currently inadequate. As a result the U.S. auto industry spends hundreds of millions of dollars per year forming dies that do not lead to the desired parts. The NIST Center for Automotive Lightweighting (NCAL) works to develop the measurement methodology, standards and analysis necessary for the auto manufacturers and base metal suppliers to transition to advanced lightweight materials for auto body components without wasteful trial-and-error development cycles, and successfully transfer this technology to the auto industry.

Impact and Customers

By directly measuring the mechanical response of sheet metal being deformed along a complex path, NCAL is providing the data needed to develop next-generation constitutive models that will accelerate the introduction of advanced lightweight materials into vehicles, lowering costs and improving fuel economy while lowering emissions. The high strain rate data being generated will ensure the safe performance of these new materials in collisions, reducing the need for costly physical crash tests. This data is critical to U.S. auto manufacturers and their Tier 1 component suppliers, as well as foreign manufacturers with a significant domestic manufacturing and development presence.

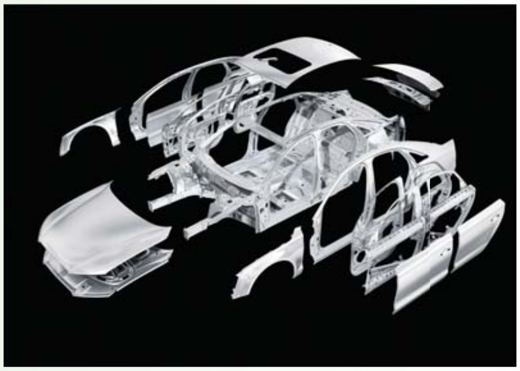
Approach

NCAL has developed a new technique for measuring the stress-strain response of sheet metal along non-linear multiaxial paths. This method provides unique data on how the multiaxial flow surface changes with plastic strain, which can be used to modify materials models used by industry. NCAL also measures the evolving crystallographic texture of the sheet to develop a microstructural understanding of the materials' deformation response.

The Center for Automotive Lightweighting includes:

- A high-rate servohydraulic testing machine – for mechanical testing at rates between quasi static (slow) to beyond crash testing.
- A next-generation metal formability system – designed to test sheet metal along any path in strain space relevant to metal forming operations. Simultaneous in-situ stress and strain mapping along with the ability to change paths in strain space without unloading the sample.
- A Marciniak testing system with two punch sizes, x-ray diffraction and digital image correlation.
- SEM, TEM, EBSD, XRD, OIM, optical, neutron diffraction.
- Computational facilities in NCAL and the Center for Theoretical and Computational Materials Science (CTCMS) cluster.
- A new sample preparation area including plasma cutting and welding.

Much of the major equipment was made possible via funding through the American Recovery and Reinvestment Act (ARRA).



Accomplishments

For the first time, the tensile multiaxial yield surface of 5754 aluminum alloy sheet was measured from initial yield up to failure. The initial shape of the surface closely approximates an ellipse, as predicted by most plasticity models of forming. However, as the strain levels increase to levels typical of forming (5 % to 20 % plastic strain), the locus evolves asymmetrically, and an apex forms in the direction of balanced biaxial (BB) straining. At the highest strain levels, the error between the predicted and the measured flow stresses exceeds 25 %.

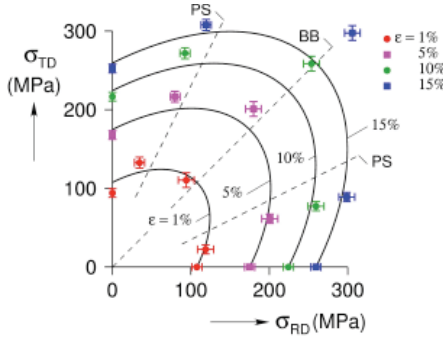


Figure 1. Flow stress of 5754 Al as a function of strain

On the same samples, the crystallographic texture evolves from a rolling texture to one of the predominant FCC deformation textures, as expected. Unexpectedly, the deformation texture that develops when the sheet is deformed in the rolling direction (RD) differs from that which develops during deformation in the transverse direction (TD). The two deformation textures are 90 degree rotations with respect to one another, but

Figure 2. Control-center and overall view of the ARRA-funded, next-generation metal formability system, based on in-situ monitoring of cruciform specimen deformation.

the twofold symmetries of the textures leads to an asymmetry in the stress needed to form the textures, which coincides with the measured asymmetry in the yield surface. These results are being modeled using a Taylor-type description of the flow behavior of polycrystalline aggregates undergoing evolving textures, the basis of many

evolving textures, the basis of many

common plasticity models, to see if this formulation adequately captures the behavior.

Advancements in standard tests and procedures have aided industry in characterizing advanced lightweight metals for the data they need for modeling. The Springback Cup test (ASTM E-2462), used by industry to screen feedstock, was analyzed for robustness and to determine the residual stress state through the thickness of the deformed sheet driving springback. Additional work is being done to measure the actual stress-strain state within the neck of an ASTM E-8 tensile specimen in order to be able to measure mechanical properties beyond instability. This high strain data is needed for forming simulations, as the constrained deformation during forming often exceeds that reached by a typical tensile test.

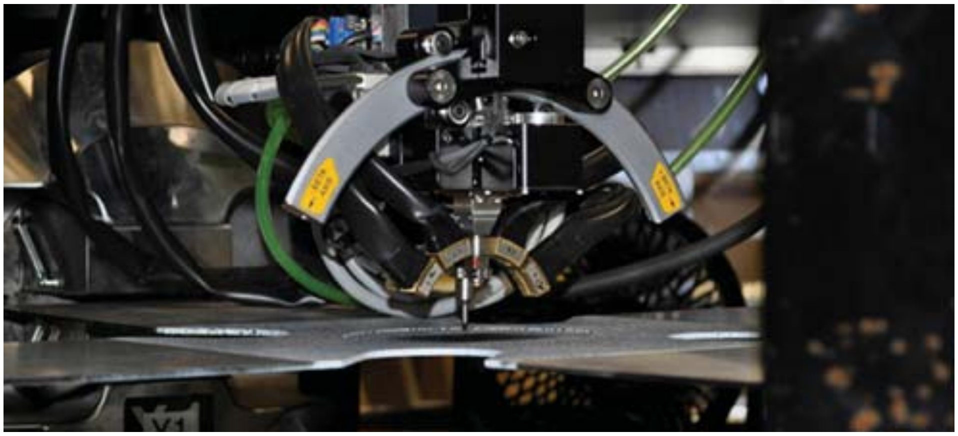


Figure 3. Close-up of x-ray stress measurement mounted on the cruciform system, positioned above a deforming sample

Learn More

Mark Iadicola, Mark Stoudt,
Thomas Gnaeupel-Herold (NCNR),
Adam Creuzige, Bill Luecke, Dave Pitchure

Tim Foecke, Group Leader
301-975-6592
timothy.foecke@nist.gov

www.nist.gov/lightweighting

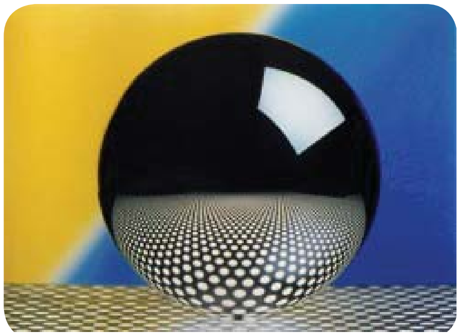
Publications

MA Iadicola, AA Creuziger, and TJ Foecke, Advanced biaxial cruciform testing at the NIST Center for Automotive Lightweighting, in Proceedings of the Society for Experimental Mechanics Vol. 8, Residual Stress, Thermomechanics & Infrared Imaging, Hybrid Techniques and Inverse Problems, M Rossi, M Sasso, N Connesson, R Singh, A DeWald, D Backman, P Gloeckner, eds., Springer, New York (2014) 277–285.

TH Gnaeupel-Herold, AA Creuziger, and MA Iadicola; A Model for Calculating Diffraction Elastic Constants J. Appl. Cryst. 45: 197 – 206 (2012)

Building the New Kilogram, One Planck at a time

The modern metric system, Le Système international d'unités, the SI, is about to undergo a major overhaul. NIST MML scientists are contributing to that change by providing critical measurements that will help establish the values for the Planck and Avogadro constants. The International Committee for Weights and Measures (CIPM) has decided that the 7 base units of the SI should all be defined by constants of nature that can be realized using "methods consistent with the laws of physics." The kilogram is the only base unit that is still defined by a physical artefact, a cylinder of Pt-Ir alloy kept in Sèvres, France. The "new" kilogram will be defined by the Planck constant h.


Credit: PTB

A 1-kg silicon sphere

The "physicist's approach" to the measurement of the Planck constant utilizes a watt balance (WB). This instrument measures h directly by an electromechanical comparison of an object's weight with the electrical current and voltage necessary to support that weight. Alternatively, the "chemist's approach" involves counting atoms. Called the X-ray crystal density (XRCD) method, the Avogadro constant NA is measured directly using a spherical, purified, ^{28}Si enriched, single-crystal, silicon ball. The ratio of the sphere's molar volume to its atomic volume fixes NA. The Planck constant h is then derived from NA via the molar Planck constant (NAh). The molar Plank constant, with the SI units $J\ s^{-1}\ mol^{-1}$, is expressed as the following: $N_Ah = cA_r(e)\alpha^2M_u/2R_\infty$ where c is the exactly known speed of light in vacuum, $M_u = 10^{-3}$ kg mol^{-1} is the exactly known molar mass constant, $A_r(e)$ is the relative atomic mass of the electron, α is the fine-structure constant, and R_∞ is the Rydberg constant.

The XRCD investigations, guided by the International Avogadro Coordination, represent a truly international effort. They involve meticulous measurements of the silicon lattice unit cell dimensions, the sphere volumes and masses, as well as the molar mass of each sphere. The molar mass measurements have been particularly troublesome, and this is where NIST has made a significant contribution.

To date, NIST and three other national metrology institutes (NMIs) have reported results of their silicon molar mass measurements on the spheres using multi-collector inductively coupled plasma mass spectrometry. However, NIST scientists reduced the uncertainty of their measurements by more than a factor of 8 compared with data from the other NMIs by using a radical sample chemistry and measurement protocol. This enabled them to detect, for the first time, a small silicon isotopic variation within the sphere samples. Fortunately, this variation was small enough to have an insignificant effect on the averaged molar mass.

The NIST results agree within measurement uncertainty with results from two of the other NMIs. The combination of these measurements has reduced the relative standard uncertainty for the derived Avogadro and Planck constants to $\approx 3\times10^{-8}$.

This international effort involving chemists and physicists is now at a point that WB and XRCD determinations of the Planck and Avogadro constants agree and will be the subject of high-level discussions at the BIPM this fall and beyond.

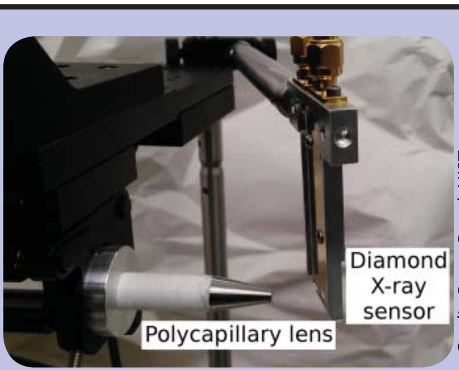
- Robert Vocke, NIST

Contact: Robert Vocke, robert.vocke@nist.gov

Compact Microfocussing for X-ray Absorption Spectroscopy using a Polycapillary Lens and a Diamond X-ray Sensor

NIST MML researchers, along with an interdisciplinary research team, have helped develop diamond sensors for use in energy-scanning X-ray experiments, such as X-ray Absorption Spectroscopy (XAS).

The research at the NIST high-energy X-ray beam line at the National Synchrotron Light Source (NSLS) in Brookhaven, New York, supported the work of a team including researchers from the Stony Brook University, Brookhaven National Laboratory, and Case Western University. (Review of Scientific Instruments, 84, 103106, 2013, doi:10.1063/1.4824350)



Credit: Bruce Ravel, NIST

X-ray Absorption Spectroscopy photon delivery system. The polycapillary lens focuses the large X-ray beam to a 30 micron spot at a distance of 6 millimeters. The diamond sensor, in operation, is rotated to a position adjacent to the outlet of the lens, leaving room to place the sample at the focal spot of the X-rays.

X-ray absorption spectroscopy (XAS) is a technique used in many areas of science, from biology to materials science, which yields information on the atomic and electronic structure of materials. The beam monitor in an XAS measurement is typically an ionization chamber, which consists of a gas-filled chamber between charged capacitor plates. As X-rays pass through the gas, they ionize gas molecules, generating a measurable current which is used to quantify X-ray flux.

Single-crystal diamond sensors have many advantages over ionization chambers, including faster response times, less leakage current, and smaller size. Although electronics-grade diamond is now readily available, diamond sensors are uncommon in XAS because they respond poorly during experiments that require scanning over a large energy range. The range of the scan usually includes multiple energies at which Bragg diffraction in the diamond contaminates the signal required for the XAS measurement.

By coupling the diamond sensor to an X-ray focusing optic known as a Kumakov lens, the diffraction problem is mitigated. The lens consists of a polycapillary bundle of glass tubes encased in a steel cylinder, with one end of the tubes drawn to a taper. The convergence angle of the X-rays focused by the Kumakov lens distribute the effect of the Bragg contamination uniformly over a broad energy range. Data gathered using the diamond sensor and the lens are of comparable quality to data measured using ionization chambers.

This combination of diamond lens and sensor has broad application to synchrotron radiation, including high energy resolution X-ray spectrometry, measurement of XAS in diamond anvil cells, and micron-scale X-ray fluorescence imaging.

Contact: Bruce Ravel, bruce.ravel@nist.gov

World's Smallest Reference Material is Big Plus for Nanotechnology

Note: A version of this story previously appeared in NIST's TechBeat *on September 24, 2014*

If it's true that good things come in small packages, then NIST can now make anyone working with nanoparticles very happy. NIST recently issued Reference Material (RM) 8027, the smallest known reference material ever created for validating measurements of these man-made, ultrafine particles between 1 and 100 nanometers (billionths of a meter) in size.

RM 8027 consists of five hermetically sealed ampoules containing one milliliter of silicon nanoparticles—all certified to be close to 2 nanometers in diameter—suspended in toluene. To yield the appropriate sizes for the new RM, the nanocrystals are etched from a silicon wafer, separated using ultrasound and then stabilized within an organic shell. Particle size and chemical composition are determined by dynamic light scattering, analytical centrifugation, electron microscopy and inductively coupled plasma mass spectrometry (ICP-MS), a powerful technique that can measure elements at concentrations as low as several parts per billion.

"For anyone working with nanomaterials at dimensions 5 nanometers or less, our well-characterized nanoparticles can ensure confidence that their measurements are accurate," says NIST research chemist Vytas Reipa, leader of the team that developed and qualified RM 8027.

Silicon nanoparticles such as those in RM 8027 are being studied as alternative semiconductor materials for next-generation photovoltaic solar cells and solid-state lighting, and as a replacement for carbon in the cathodes of lithium batteries. Another potential application comes from the fact that silicon crystals at dimensions of 5 nanometers or less fluoresce under ultraviolet light. Because of this property, silicon nanoparticles may one day serve as easily detectable "tags" for tracking nanosized substances in biological, environmental or other dynamic systems.

RM 8027 maybe ordered from the NIST Standard Reference Materials Program by phone, (301) 975-2200; by fax, (301) 948-3730; or online at http://www.nist.gov/srm.

- Michael Newman, NIST

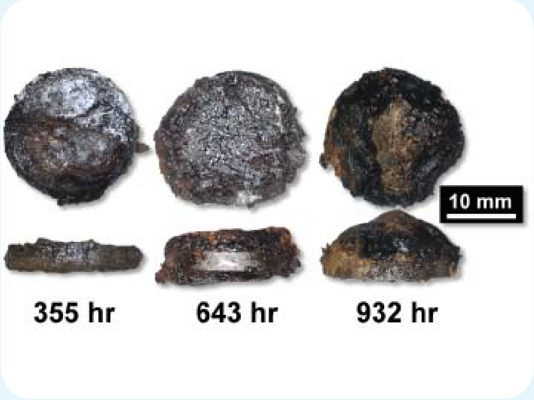
Contact: Vytas Reipa, vytautas.reipa@nist.gov

NIST MML Corrosion Lab Tests Suggest Need for Underground Gas Tank Retrofits

Note: A version of this story previously appeared in NIST's TechBeat *on July 29, 2014*

A hidden hazard may lurk beneath many of the roughly 156,000 gas stations across the United States.

The hazard is corrosion in parts of underground gas storage tanks—corrosion that could result in failures, leaks and contamination of groundwater, a source of drinking water. In recent years, field inspectors in nine states have reported many rapidly corroding gas storage tank components such as sump pumps. These incidents are generally associated with use of gasoline-ethanol blends and the presence of bacteria, Acetobacter aceti, which convert ethanol to acetic acid, a component of vinegar.



Optical micrographs of severe corrosion on steel alloy samples exposed to ethanol and acetic acid vapors -- conditions typical of underground gas storage tanks -- after 355 hours, 643 hours, and 932 hours. Credit: NIST

Following up on the inspectors' findings, a NIST laboratory study* has demonstrated severe corrosion—rapidly eating through 1 millimeter of wall thickness per year—on steel alloy samples exposed to ethanol and acetic acid vapors. Based on this finding, NIST researchers suggest gas stations may need to replace submersible pump casings, typically made of steel or cast iron, sooner than expected. Such retrofits could cost an estimated $1,500 to $2,500 each, and there are more than 500,000 underground gas storage tanks around the country.

The NIST study focused only on sump pump components, located directly below access covers at filling stations, just above and connected to underground gas storage tanks. The sump pumps move fuel from underground tanks to the fuel dispensers that pump gas into cars. These underground tanks and pipes also may be made of steel and could be vulnerable, too. "We know there are corrosion issues associated with the inside of some tanks. We're not sure, at this point, if that type of corrosion is caused by the bacteria," NIST co-author Jeffrey Sowards says.

Much of the U.S. fuel infrastructure was designed for unblended gasoline. Ethanol, an alcohol that can be made from corn, is now widely used as a gasoline additive due to its oxygen content and octane rating, or antiknock index. A previous NIST study found that ethanol-loving bacteria accelerated pipeline cracking.**

For the latest study, NIST researchers developed new test methods and equipment to study copper and steel alloy samples either immersed in ethanol-water solutions inoculated with bacteria, or exposed to the vapors above the medium—conditions mimicking those around sump pumps. Corrosion rates were measured over about 30 days.

The NIST study confirmed damage similar to that seen on sump pumps by field inspectors. The worst damage, with flaky iron oxide products covering corrosion, was found on steel exposed to the vapors. Copper in both the liquid and vapor environments also sustained damage, but corrosion rates were slower. Steel corroded very slowly while immersed in the liquid mixture; the NIST paper suggests bacteria may have created a biofilm that was protective in this case.

Although copper corroded slowly—it would take about 15 years for 1.2-millimeter-thick copper tube walls to develop holes—localized corrosion was observed on cold-worked copper, the material used in sump pump tubing, NIST co-author Elisabeth Mansfield notes. Therefore, stress-corrosion cracking is a concern for bent copper tubing because it would greatly reduce tube lifetime and result in leaks.

The NIST test equipment developed for the study could be used in future investigations of special coatings and biocides or other ways to prevent sump pump failures and leaks.

NIST held a workshop in July 2013 on biocorrosion associated with alternative fuels. Presentations and information from this workshop can be found at www.nist.gov/mml/acmd/biocorrosion.cfm.

- Laura Ost, NIST

Contact: Elisabeth Mansfield,
elisabeth.mansfield@nist.gov

*J.W. Sowards and E. Mansfield. Corrosion of copper and steel alloys in a simulated underground storage tank sump environment containing acid producing bacteria. Corrosion Science. July, 2014. In press, corrected proof available online. DOI: 10.1016/j.corsci.2014.07.009.

**See 2011 NIST Tech Beat article, "NIST Finds That Ethanol-Loving Bacteria Accelerate Cracking of Pipeline Steels," at www.nist.gov/mml/acmd/201108_ethanol_pipelines.cfm.

Latest NIST Mass Spectral Library: Expanded Coverage, Features

Note: A version of this story previously appeared in NIST's TechBeat *on July 29, 2014*

The world's most widely used and trusted resource for identifying mass spectra, the "fingerprints" of molecules, has undergone a major expansion, according to its managers at NIST. NIST 14, containing the newest edition of the NIST/EPA/NIH Mass Spectral Library, boasts significantly increased coverage within each of its three components: the NIST/EPA/NIH Mass Spectral Library of electron ionization (EI) spectra, the NIST Tandem Mass Spectral Library, and the NIST Library of GC Methods and Retention Index Data. These updated libraries were recently released to more than 30 distributors worldwide.

Mass spectrometry (MS) is routinely used to quickly and accurately identify chemical compounds for a wide range of applications such as drug detection, pollution monitoring, petrochemical processing and disease diagnosis via biomarkers. To use the method, a target molecule is first converted to an ionized gas. Fragmenting these ions into their components produces other ions whose intensities (abundances), when sorted according to mass and charge, produce a pattern known as a "mass spectrum." This spectrum serves as a fingerprint for identifying the target molecule. To ensure an accurate ID, the spectrum should be compared with a verified reference spectrum from a database such as the NIST/EPA/NIH Mass Spectral Library.

NIST 14 is made up of three databases and accompanying search software. The tool provides researchers with:

- EI (electron ionization) spectra, derived from traditional gas chromatography/mass spectrometry (GC/MS) methods;
- tandem mass (MS/MS) spectra, where the precursor (initial) ions formed in the mass spectrometer are fragmented in a separate step involving a second mass spectrometer (hence, the "tandem")—a common practice in liquid chromatography/mass spectrometry (LC/MS); and
- gas chromatography (GC) method/retention index data, which provide another means of confirming the identity of molecules.

Each spectrum in the new library has been critically examined by experienced mass spectrometrists at NIST and collaborating organizations, and each chemical structure has been scrutinized for correctness and consistency.

"This release provides one of the most significant enhancements in the long history of the Mass Spectral Library, with major increases in coverage for both the EI and tandem collections along with a higher level of organization of the data within them," says Stephen Stein, director of the NIST Mass Spectrometry Data Center and one of the researchers responsible for the development of NIST 14.

Among the other improvements to NIST 14 are a greatly expanded variety of precursor ions for tandem mass spectra; integration of retention index and mass spectral data; a new way to display the structures of molecular derivatives and the reagents that yield them; new search methods for high mass accuracy data; and the use of the International Chemical Identifier (InChI) standard for representing chemical structures (developed by NIST and the International Union of Pure and Applied Chemistry), which makes it easier to find additional information about molecular IDs made by the Mass Spectral Library.

For more information and a list of vendors offering NIST 14, go to www.nist.gov/srd/nist1a.cfm.

- Michael E. Newman, NIST

Contact: Stephen Stein, stephen.stein@nist.gov

Credit: Newman/NIST

Photo Courtesy of the Chemical Heritage Foundation Collections

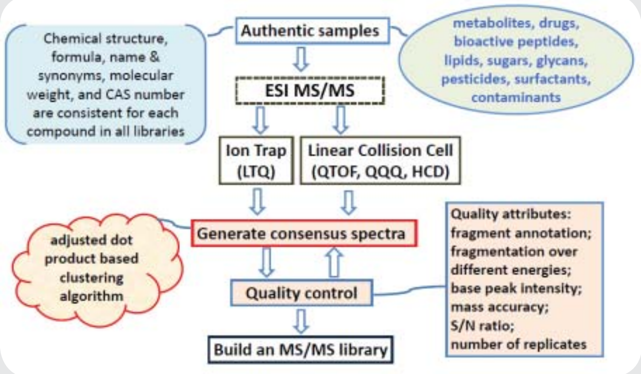
Modern mass spectrometry has advanced dramatically since the 1970s when the first spectrometers (bottom) were built, creating a demand for ever-improving mass spectral reference standards to ensure accurate chemical identifications. NIST 14, containing the newest edition of the NIST/EPA/NIH Mass Spectral Library, is now available.

NIST MML Researchers Develop Methods for Building an Electrospray Ionization Tandem Mass Spectral Library

The researchers in the NIST Mass Spectral Data Center are the creators and curators of the NIST Mass Spectral Libraries, such as the NIST/EPA/NIH Mass Spectral EI Library that contains spectra for over 200,000 compounds and is intimately integrated into many gas chromatography/mass chromatography data systems.

Electrospray ionization (ESI) followed by tandem mass spectrometry (ESI MS2), in conjunction with liquid chromatography (LC) is a common technique for identifying and quantifying compounds in complex mixtures of non-volatile compounds. The identification step can be aided by matching acquired tandem mass spectra (MS2) against reference library spectra. It has been a significant challenge for the researchers in the NIST Mass Spectral Data Center to generate library quality ESI MS2 data for target analytes due to complex ESI chemistry. Depending on the compound, a large number of different precursor ions can be generated by the ESI process each with its own unique fragmentation profile. Because of this, ES MS2 spectra require unprecedented processing methods in order to obtain complete and high quality reference spectra for individual analytes.

The team of Yang, Neta and Stein developed a foundation of processing methods that have been used to build a library of ESI MS2 data from >9000 compounds, yielding 230,000 spectra. The method (outlined pictorially) was tested on spectra obtained for >1000 compounds, and included the following steps: Consensus Spectrum Building, Evaluation of Spectral Similarity, Clustering of Spectra, Creating 'Consensus' Spectra, Quality Control, Noise Removal, Peak Annotation, and evaluation of Collision Energy Dependence. The data is included in NIST Tandem Mass Spectral Library released in July of 2014.



Procedure for building an ESI MS2 library

Reference: Yang, X., Neta, P., and Stein, S.E., Quality Control for Building Libraries from Electrospray Ionization Tandem Mass Spectra, Analytical Chemistry, 2014, 86, 6393-6400. http://dx.doi.org/10.1021/ac500711m

Contact: Stephen Stein, stephen.stein@nist.gov

NIST MML Researchers Develop New Method for Reconstructing Destroyed Firearm Serial Numbers

NIST MML researchers have recently demonstrated a novel technique, which may allow for the retrieval of defaced serial numbers on firearms, vehicles, and other objects of forensic interest. Often serial numbers are the primary method of tracking ownership and possession, leading to intentional destruction by criminals. Current methods of restoration require acid etching, and are generally unreliable and are easily defeated by methods such as overstamping or heat treatment.

In the new technique, residual sub-surface deformation beneath a die-stamped serial number imprint is visualized using electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM). In initial experiments, the stamped imprint only reached a depth of about 170 µm, but deformation of the metallic crystal structure reached about 740 µm beneath the surface. Complete superficial destruction of a stamped serial number imprint still leaves significant sub-surface deformation, which can be measured by the diffraction-based SEM technique.

Since EBSD is sensitive to the underlying crystal structure, the technique is applicable to any metallic alloy and any marking method which results in a change in the underlying crystal structure, including (but not limited to) die stamping and laser engraving. It is estimated that, with process optimization and automation, reconstruction of a full 8-character serial number could be completed in less than one hour.

A description of the technique is to be published in a leading forensic science journal: "Reconstruction of firearm serial numbers with electron backscatter diffraction (EBSD)," R. M. White, R. R. Keller, Forensic Science International (2014).

Contact: Ryan White, ryan.white@nist.gov

NIST MML Contributes to NFL Head Health Challenge

NIST MML Deputy Director Michael Fasolka observed the judging session of the NFL Head Health Challenge II, held at the Under Armour Corporate and R&D Headquarters in Baltimore, MD on July 29, 2014. Dr. Fasolka's participation was aimed at identifying the key themes and ideas of interest in this second challenge, which is co-sponsored by the NFL, Under Armour, and GE. During the full day session, Dr. Fasolka learned that the Challenge was open to proposals about different training regimens that would result in improved head protection behavior, sideline tools for assessing brain injury on the spot, biomarker measurements, and new materials systems that would result in improved head injury protections, primarily as components in helmets. Dr. Fasolka's recommendation to the group was that more can be done to attract state-of-the-art materials solutions tapping into relevant cutting edge materials technologies, including complex fluids, particulate fluids, and advanced foam designs.

Subsequent discussions between MML Director Laurie Locascio and Fasolka with the NFL, Under Armour, and GE have focused on development of a joint Challenge Prize for discovery of new energy absorptive or dispersive materials suitable for use in protective gear. On Oct. 3, 2014 NIST MML hosted leadership and scientists from the NFL, Under Armour, and GE to discuss the first steps in a Challenge Prize which they would cosponsor. This was the first activity held in support of a recent Memorandum of Understanding signed by NIST, NFL, Under Armour and GE in which these parties agreed to collaborate to foster the development of advanced materials for protective equipment for athletes and other professions. The goal of the meeting was to determine the materials properties required for enhanced impact protection, which will be used to form base criteria for the Challenge Prize.

Contacts: Michael Fasolka, michael.fasolka@nist.gov
Laurie Locascio, laurie.locascio@nist.gov

NIST MML Researchers Advise on Durability of Eisenhower Memorial

NIST MML researchers advised the National Capital Planning Commission (NCPC) on the long-term durability of materials proposed for the Dwight D. Eisenhower Memorial, just south of the National Mall. The Commemorative Works Act requires that "a commemorative work shall be constructed of durable material suitable to the outdoor environment." Contrary to other monuments in the Nation's Capital, which tend to have used materials that have demonstrated their durability over time, the proposal for the Eisenhower Memorial incorporates innovative welded stainless steel tapestries approximately 80 feet high and up to 447 feet in length. Because of "the untested complexity of the tapestries and the scale and extent at which they are proposed," the NCPC engaged experts from NIST, along with the Department of Defense and the Smithsonian Institution, to assist in reviewing and interpreting tests results from the applicant and to provide recommendations on the durability of the proposed structures.

NIST staff examined the weld integrity and corrosion resistance of tapestry mockups provided by the design team and provided expert analysis of the environmental and mechanical tests performed by the applicant. Review of an early design raised a number of concerns about both pitting corrosion and weld integrity. These findings led to a redesign, including a more corrosion resistant steel alloy, which was found to have adequate corrosion and fatigue resistance. NIST's assistance lent confidence in the durability of the proposed materials and contributed to NCPC provisional approval of the project on Oct. 2, 2014.

Contacts: Tim Foecke, timothy.foecke@nist.gov;
Adam Creuziger, adam.creuziger@nist.gov

Milligan's Legacy Honored at NOBCChE Annual Meeting

Samantha Maragh (right, MML), Jeanita Pritchett (second from right, MML), Brandi Toliver (left, NIST SURF Director), and Marlon Walker (center, MML) at the NIST Exhibit booth at the NOBCChE Annual Meeting in New Orleans.

Dolphus E. Milligan, a preeminent NBS/NIST scientist, was internationally recognized as a trailblazer in the area of free radical spectroscopy. NIST honored his scientific accomplishments this year by sponsoring a symposium at the 41st Annual Meeting of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers (NOBCChE) on September 25, 2014 in New Orleans, LA. The session featured former winners of the Milligan Graduate Fellowship, an award competitively given to an incoming minority graduate student in the Department of Chemistry and Biochemistry at the University of Maryland – College Park.

The session opened with video-recorded remarks by Acting NIST Director Willie May on the impact that Milligan had on him, other scientists of color, and on the founding of NOBCChE. Professor and Chair Janice Reutt-Robey stressed the importance of the Milligan Fellowship in increasing minority participation in her department, and in the technology pipeline in general. The presentations by the fellowship winners Geraldine Escheberi, Christopher Sims, and Julia Greenfield, Professor Herman Sintim, and NIST researchers Jeanita Pritchett, Samantha Maragh, Bryant Nelson, and Marlon Walker positively reflected on the high scientific standards exemplified in Milligan's achievements

Additionally while at the conference, Brandi Toliver, Samantha Maragh, Jeanita Pritchett, and Marlon Walker served as NIST ambassadors for the booth at the Career Fair and Trade Show. Information about NIST regarding potential opportunities for undergraduates, graduates, and post-doctoral candidates was disseminated. The exhibit served as an excellent platform for NIST to directly engage with future scientists from underrepresented groups.

- Jeanita S. Pritchett and Marlon L. Walker, NIST

NIST MML Researchers Develop Rapid PCR Protocols for Forensic DNA Typing

NIST MML researchers Erica L. R. Butts and Peter M. Vallone have developed and evaluated Rapid PCR protocols for forensic DNA typing on six commercially available thermal cycling platforms. Rapid DNA typing is a technique developed by Butts and Vallone that helped launch the development of point of collection DNA analysis instrumentation and Rapid PCR protocols for forensic laboratory use.

Rapid DNA typing uses the same general workflow as traditional DNA typing, that includes DNA extraction, quantitation, multiplex PCR amplification, and fragmentation separation and detection, but the PCR amplification time is greatly reduced. Butts and Vallone have been able to reduce the time of PCR amplification by using rapid novel polymerases (an enzyme that synthesizes DNA) and optimized thermal cycling conditions, reducing amplification times from 3 hours down to as little as 14 min.

Evaluation of the Rapid PCR protocols on six different thermal cycling platforms was the focus of a recent publication by Butts and Vallone in the journal, Electrophoresis. The presented amplification protocol and performance characteristics of the six thermal cyclers provides the basis for labs wishing to adopt faster PCR methods. When employed in fully integrated automated DNA typing systems the applications include booking stations, border crossing stations or airport security stations wishing to adopt faster DNA typing workflows.

Butts, E. L. R., and Vallone, P. M., Rapid PCR protocols for forensic DNA typing on six thermal cycling platforms, Electrophoresis, Available online 22 August 2014, http://dx.doi.org/ 10.1002/elps.201400179.

(http://onlinelibrary.wiley.com/doi/10.1002/elps.201400179/abstract;jsessionid=B-5C80ABA6DC8893AD04137079252BC87.f02t04)

Contact: Peter Vallone,
peter.vallone@nist.gov

Recent NIST MML Awardees

The American Association for Dental Research Honors Rafael "Ray" Bowen with Distinguished Scientist Award

Rafael "Ray" Bowen, of the ADA Foundation, Dr. Anthony Volpe Research Center, NIST, received the 2014 American Association for Dental Research Distinguished Scientist Award March 19, 2014 at the AADR 43rd Annual Meeting & Exhibition. Dr. Bowen is perhaps best known for his development of dental composites, patented in the 1960s.

NIST Fellow Robert F. Cook Awarded American Ceramic Society Sosman Award

The American Ceramic Society has awarded MML Researcher and NIST Fellow Robert F. Cook the Robert B. Sosman Award and Lecture. The Robert B. Sosman Award is the highest recognition of scientific accomplishment given by the ACS Basic Science Division and is given in recognition of outstanding achievement in basic science of an area that results in a significant impact to the field of ceramics.

NIST MML Tytus Mak Honored by Georgetown University Medical Center

Tytus Mak, a recently hired member of NIST MML's Mass Spectrometry Data Center, has won the prestigious Robert B. Dickson Graduate Prize for his Ph.D. thesis titled "Development of Novel Computational Methodologies for Analyzing Post-Processed Untargeted LC-MS Metabolomics Data." The award was presented to him by the Lombardi Cancer Center at the Georgetown University Medical Center at the annual Robert B. Dickson Memorial Lecture held October 10, 2014.

NIST MML Scientist Stephen Wise Receives 2014 Technical Division on Reference Materials Award

Stephen Wise is being recognized with the 2014 Technical Division on Reference Materials (TDRM) Award for his career dedication to development and provision of reference materials for the environmental, clinical, and nutrition fields. The TDRM award recognizes contributions and encourages active participation of members of the TDRM in the pursuit of the goals and objectives of AOAC International.

NIST MML/EL Team Receives International Journal of Refrigeration Best Paper Award

NIST MML researchers Mark McLinden and Andrei Kazakov, together with NIST Engineering Lab (EL) researcher Steven Brown and Piotr Domanski from Catholic University of America, have received the Best Paper Award for 2013/2014 from the International Journal of Refrigeration, the official journal of the International Institute of Refrigeration, for the paper "A Thermodynamic Analysis of Refrigerants: Possibilities and Tradeoffs for Low-GWP refrigerants."

Haynes Honored with European Conference on Thermophysical Properties Lifetime Achievement Award

At the 20th European Conference on Thermophysical Properties (ECTP), recently held in Portugal, NIST MML Scientist Emeritus and former Chief of the Physical and Chemical Properties Division, William M. "Mickey" Haynes, was presented the Lifetime Achievement Award by the European Conference on Thermophysical Properties for his original scientific achievements in thermophysics. Dr. Haynes was honored for his work in thermophysical properties, involving natural gas systems, cryogenic fluids, air, alternative refrigerants, and ammonia/water systems, as well as for the development of state-of-the-art experimental apparatus, such as magnetic suspension densimeters and torsional crystal viscometers.

NIST MML Researcher Michael Frenkel Receives Rossini Lectureship Award

The 2014 Rossini Lectureship Award from the International Association of Chemical Thermodynamics (IACT) was presented to Michael Frenkel, director of NIST MML's Thermodynamics Research Center. Frenkel was cited for his contributions to thermodynamics, including his leadership in (1), the development and the first implementation of the concept of global information systems in science in application to the field of thermodynamics; and (2), the establishment of new concepts and models for thermodynamic properties of dynamic chemical systems such as stereoisomers, conformers, tautomers, and plastic crystals based on experimental equilibria and calorimetric studies, as well as applications of statistical thermodynamics.

NIST MML Scientist Jeanita Pritchett Selected as U.S. Embassy Science Fellow

NIST MML scientist Jeanita Pritchett has been selected as a 2014-2015 U.S. Embassy Science Fellow. The fellowship is awarded by the U.S. Department of State and gives scientists the opportunity to provide U.S. embassies with access to the expertise of U.S. Government officers in science and technology fields. This fellowship will take place from January to March 2015 at the U.S. Consulate in Cape Town, South Africa.

Selected Recent Publications

MML researchers publish over 400 journal articles each year. Here are a few recent examples:

J. C. Gigault, J. M. Pettibone, C. E. Schmitt, V. A. Hackley, "A rational strategy for characterization of nanoscale particles by asymmetric flow field-flow fractionation" Analytica Chimica ACTA, Vol. 809, pp. 9-24, (16-Nov-2014) (PubID: 914192)

S. D. Hudson, P. S. Sarangapani, K. D. Migler, J. A. Pathak, "A Microliter Capillary Rheometer for Characterization of Protein Solutions" Journal of Pharmaceutical Sciences, 11 pp., (10-Oct-2014) (PubID: 914754)

E. J. Petersen, V. Reipa, S. S. Watson, D. L. Stanley, S. A. Rabb, B. C. Nelson, "The DNA Damaging Potential of Photoactivated P25 Titanium Dioxide Nanoparticles " Chemical Research in Toxicology, (30-Sep-2014) (PubID: 914280)

G. Garberoglio, K. Patkowski, A. H. Harvey, "Fully Quantum Cross Second Virial Coefficients for the Three-Dimensional He-H2 Pair" International Journal of Thermophysics, Vol. 35, pp. 1435-1449, (21-Sep-2014) (PubID: 915991)

S. Avgeri, M. J. Assael, M. L. Huber, R. A. Perkins, "Reference Correlation of the Viscosity of Benzene from the Triple Point to 675 K and up to 300 MPa" Journal of Physical and Chemical Reference Data, Vol. 43, No. 3, pp. 033103-1-033103-13, (29-Aug-2014) (PubID: 916175)

J. W. Sowards, E. Mansfield, "CORROSION OF COPPER AND STEEL ALLOYS IN A SIMULATED UNDERGROUND STORAGE TANK SUMP ENVIRONMENT CONTAINING ACID PRODUCING BACTERIA" Corrosion Science, 10 pp., (29-Aug-2014) (PubID: 914581)

P. M. Vallone, E. L. Butts, "Rapid PCR Protocols for Forensic DNA Typing on Six Thermal Cycling Platforms" Electrophoresis, (22-Aug-2014) (PubID: 916080)

D. F. Sunday, M. R. Hammond, C. C. Wang, W.L. Wu, D. M. DeLongchamp, R. J. Kline, M. Tjio, J. Cheng, J. W. Pitera, "Characterization of Buried Structure in Directed Self Assembly Block Copolymers" ACS Nano, Vol. 8, No. 8, pp. 8426-8437, (30-Jul-2014) (PubID: 915847)

W. N. Vreeland, R. R. Hood, D. L. DeVoe, "Microfluidic Remote Loading for Rapid Single-Step Liposomal Drug Preparation" Lab on A Chip, Vol. 14, No. 17, pp. 3359-3367, (29-Jul-2014) (PubID: 915563)

C. H. Camp, Y. J. Lee, J. Heddleston, C. M. Hartshorn, A. R. Hight Walker, J. N. Rich, J. D. Lathia, M. T. Cicerone, "High Speed Coherent Raman Fingerprint Imaging of Biological Tissues" Nature Photonics, Vol. 8, pp. 627-634, (20-Jul-2014) (PubID: 914850)

D. Moon, J. Obrzut, J. F. Douglas, T. F. Lam, K. Koziol, K. D. Migler, "Three Dimensional Cluster Distributions in Processed Multi-wall Carbon Nanotube Polymer Composites" Soft Matter, Vol. 55, No. 15, pp. 3270-3277, (25-Jun-2014) (PubID: 914833)

G. A. Myers, S. Hazra, M. de Boer, C. A. Michaels, S. J. Stranick, R. P. Koseski, R. F. Cook, F. W. DelRio, "Stress mapping of micromachined polycrystalline silicon devices via confocal Raman microscopy" Applied Physics Letters, Vol. 104, pp. 191908-1-191908-5, (15-Jun-2014) (PubID: 915909)

K. E. Sharpless, D. L. Duewer, K. A. Lippa, A. L. Rukhin, "The ABCs of Using Standard Reference Materials in the Analysis of Foods and Dietary Supplements: A Practical Guide" Special Publication (NIST SP) 260-181, (02-Jun-2014) (PubID: 912374)

K. W. Phinney, G. D. Carter, "Assessing Vitamin D Status: Time for a Rethink?" Clinical Chemistry, Vol. 60, No. 6, pp. 809-811, (19-Feb-2014) (PubID: 915458)

Full text versions of many papers and a full list of MML publications can be accessed through the NIST Publications Database at www.nist.gov/publication-portal.cfm

To learn more, contact:

Material Measurement Laboratory
100 Bureau Drive, M/S 8300
Gaithersburg, MD 20899-8300
Tel: 301-975-8300
Fax: 301-975-3845
mmlinfo@nist.gov

or visit http://www.nist.gov/mml

National Institute of
Standards and Technology
U.S. Department of Commerce