

Material Matters

The Quarterly Magazine of NIST's Material Measurement Laboratory

Summer 2015

*A Standard for Gene Sequencing
Enhanced Indentation Testing
The MML Strategic Plan*

NIST

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: The end result of a DNA sequencing process. Each color represents one of the four base chemicals that make up DNA (adenine, guanine, cytosine and thymine). NIST's new genome reference material is a standard that can help labs determine how well their DNA sequencing processes are working by evaluating the performance of the equipment, chemistry, and data analysis involved.

Credit: Gerald Barber, Virginia Tech University (with permission of the National Science Foundation)

A Message from the MML Director

NIST's Material Measurement Laboratory (MML) was established in 2012 after a major reorganization of NIST's laboratories. Since then, we have been working diligently to build our divisions and programs, and ensure we fulfill our mission. I am now very pleased to announce MML's first Strategic Plan (mmlstrategy.nist.gov) – the map that will guide our work into the future.

Our Lab's vision is to build the foundation for tomorrow's innovation in the biological, chemical and materials sciences. This plan is a blueprint for achieving that goal over the next five years. It describes MML's priorities for establishing new measurement science and services to address emerging national needs, the operational and organizational innovations necessary to achieve our mission, and the means to sustain the wide range of technical efforts in MML that are key to our nation's economic success and quality of life. Organized around five primary goals (Measurement Science Excellence, Measurement Service Excellence, Data Science and Data Management Capabilities, Strategic Partnering, and Organizational Excellence), the plan describes new and exciting directions for MML, while reaffirming areas of strength we are committed to sustaining, and identifying important areas of potential growth.

The plan is the result of an intensive process, which included extensive input and feedback from staff and stakeholders, a series of focus groups and workshops, and much careful analysis and revision. This was a group effort in every sense, and my hope is that as we move forward to put the plan into practice MML staff and stakeholders can all participate in the plan and contribute their talents. In fact, the plan can only be successful if implemented collaboratively. We designed it that way, because we know that collaboration is key to MML's success.

This plan is a window into MML's future, and a map of how to get there. I hope you will take a look, and let us know what you think (mmlinfo@nist.gov). At the beginning of a new and exciting era for MML, I'm looking forward to working with you to make our vision a reality.



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

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'Measuring Stick' Standard for Gene Sequencing Now Available from NIST

Note: A version of this story previously appeared in NIST's TechBeat on May 21, 2015.

The world's first reference material to help ensure laboratories accurately "map" DNA for genetic testing, medical diagnoses and future customized drug therapies is now available from NIST.

The new reference material, NIST RM 8398, is a "measuring stick" for the human genome, the coded blueprints of a person's genetic traits. It provides a well-characterized standard that can tell a laboratory how well its processes for determining the patterns in a person's DNA (called DNA or gene sequencing) are working by measuring the performance of the equipment, chemistry and data analysis involved.

NIST RM 8398 was created by NIST and its partners in the Genome in a Bottle consortium, a group that includes stakeholders from industry, academia and the federal government. Scientists from NIST and the U.S. Food and Drug Administration (FDA) helped organize the collaborative effort to provide the technical benchmarks (reference standards, reference methods and reference data) needed to enable widespread clinical applications of whole genome sequencing and science-based regulatory oversight of the technology by the FDA.

In fact, the prototype version of NIST RM 8398 has already been on the job. In November 2013, the FDA used the genome created during the reference material's development phase to certify and approve one of the first commercially available high-throughput DNA sequencers.

The new reference material marks a significant step forward in addressing FDA's regulatory needs for evaluating next-generation gene sequencing and genetic testing as outlined in President Barack Obama's Precision Medicine (also known as "personalized medicine") initiative.

DNA sequencers take long strings of a person's DNA and randomly chop them into small pieces that can be individually analyzed to determine their sequence of letters from the genetic code (A, C, G and T representing the four key components of DNA that code for protein production in living organisms: adenine, cytosine, guanine and thymine). The sequenced

pieces can then be compared to a defined "reference sequence" to identify differences in the two codes. The differences reveal where mutations have occurred in specific genes.

However, biases and "blind spots" for certain sequences contribute to uncertainties or errors in the sequence analysis. These biases can lead to hundreds of thousands of disagreements between different sequencing results for the same human genome.

Laboratories can now use the DNA in NIST RM 8398 (taken from a cell line—referred to as NA12878—of an individual whose genome was acquired in a 1980s research study) to identify these biases. Using the new NIST standard as a benchmark, DNA sequencing laboratories can be more confident in their reporting of true positives, false positives, true negatives and false negatives.

The reference material is the first complete human genome to have been extensively sequenced and re-sequenced by multiple techniques, with the results weighted and analyzed to eliminate as much variation and error as possible.

The method used to create NIST RM 8398, as well as the findings validating the tool, may be found in a March 2014 Nature Biotechnology paper* as well as on the Genome in a Bottle website. In addition, the Genome Comparison and Analytic Testing, or GCAT, website enables real-time benchmarking of any DNA sequencing method using these results.

The Genome in a Bottle consortium is currently developing well-characterized whole genome reference materials from two genetically diverse groups: Asians and Ashkenazi Jews. Both of the future reference materials will include sequenced genes from father-mother-child "trios" to utilize genetic links between family members.

NIST RM 8398, Human DNA for Whole-Genome Variant Assessment (Daughter of Utah/European Ancestry), is available in vials each containing 10 micrograms of genomic NA12878 DNA. All samples of the reference material have been characterized for homogeneity (ensuring that each vial contains similar DNA) and stability

(ensuring that the DNA ordered now will be comparable to samples ordered in the future).

NIST RM 8398 is available from the NIST Standard Reference Materials Program.

- Michael Newman, NIST

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*J.M. Zook, B. Chapman, J. Wang, D. Mittelman, O. Hofmann, W. Hide and M. Salit. Integrating human sequence data sets provides a resource of benchmark SNP and indel genotype calls. Nature Biotechnology (March 2014). doi:10.1038/nbt.2835

Genome-Scale Measurements Group Hosts Workshop on Standards for Synthetic Biology

NIST's Stanford University-based Genome-Scale Measurements Group hosted the kickoff meeting for the Synthetic Biology Standards Consortium (SBSC) at the Stanford University Li Ka Shing Conference Center on March 31, 2015. The Advances in Biomedical Medical Measurement Science (ABMS) program at Stanford University sponsored the meeting. A total of 123 people, including 11 remote participants, attended this open, public workshop.

The objective of the SBSC is to collectively build the metrology infrastructure to support a fully integrated, global synthetic biology enterprise. The consortium will provide safe harbor for collaborative standards development and will maintain a broad portfolio through multiple technical working groups.

The charge of this workshop was to identify several initial working groups with critical mass, leadership teams, and a clear path forward to deliver standards to support the growth of the bioeconomy. Volunteers proposed initial ideas for candidate working group activities during a series of panels, followed by working group discussions to develop draft terms of reference. Terms of reference addressed problem definition, relevance, and identified specific actions for success.

The full workshop report is publicly available here: <http://jimb.stanford.edu/sbcs-0315-workshop-report>. Immediate next steps for the consortium will be to establish NIST-hosted discussions (via email and conference call) for each working group to refine their terms of reference and begin developing metrology products.

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To Give Cancer a Deadly Fever, NIST Explores Better Nanoparticle Design

Note: A version of this story previously appeared in NIST's TechBeat on June 16, 2015.

Heat may be the key to killing certain types of cancer, and new research from a team including NIST scientists has yielded unexpected results that should help optimize the design of magnetic nanoparticles that can be used to deliver heat directly to cancerous tumors.

When combined with other treatments such as radiotherapy or chemotherapy, heat applied directly to tumors helps increase the effectiveness of those types of treatments, and it reduces the necessary dose of chemicals or radiation.

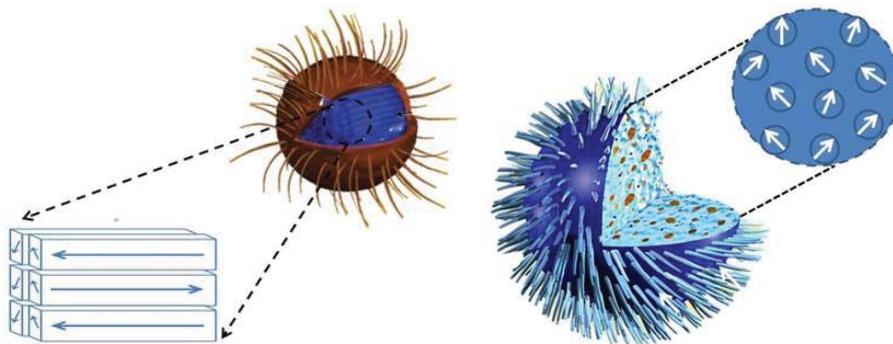
This is where magnetic nanoparticles come in. These balls of iron oxide, just a few tens of nanometers in diameter, heat up when exposed to a powerful magnetic field. Their purpose is to bring heat directly to the tumors. Materials research, performed in part at the NIST Center for Neutron Research (NCNR), revealed magnetic behavior that proved counterintuitive to the scientific team—a finding that will affect which particles are chosen for a particular treatment.

Choosing the right kind of particles is important because, depending on their structure, they deliver a different dose of heat to the cancer. Some heat up quickly at first, while others require a stronger magnetic field to get going but ultimately deliver more heat.

"You want to design your nanoparticles for the kind of cancer you're treating—whether it's localized or spread through the body," says NIST's Cindi Dennis. "The amount of electricity needed to create the field can be 100 kilowatts or more. That costs a lot of money, so we want to help engineer particles that will do the best job."

Although the magnetic field applied for hyperthermia is 100 to 1,000 times weaker than that typically used for MRI imaging, Dennis explains, it's an alternating field (the magnetic polarity switches rapidly), which requires a lot more power.

With colleagues at Johns Hopkins University School of Medicine, the University of Manitoba and in industry, the team studied two kinds of iron-oxide nanoparticles, each of which has a different internal structure. In one, iron-oxide crystals are stacked neatly, like bricks in a wall; in the other, the arrangement is more haphazard, like balls in a playpen.



Iron oxide nanoparticles with a neatly-stacked internal structure (left) need a stronger magnetic field than expected to heat up, while those with a more haphazard arrangement heat up more quickly, even under a weak field. The findings, which run contrary to expectations, could affect which nanoparticles are chosen to treat certain types of cancer. Credit: NIST

While subjecting both types to an alternating magnetic field, the team discovered that the neatly-stacked ones needed a stronger field than expected to heat up, while the haphazard particles got hot more quickly, even when the field was still weak.

It took a trip to the NCNR to figure out why these nanoparticles acted strangely. The neutron experiments showed regions of different sizes and shapes in the particles. Within each region, the so-called magnetic moments are uniform and point in the same direction. But the regions themselves did not align with each other. This unexpected behavior among regions, it turns out, profoundly affects the nanoparticles' response to a magnetic field."

"Materials often behave unexpectedly on the nanoscale, and here we have another example of that," Dennis says. "We expect it will help design better cancer treatments. A localized cancer could be treated with nanoparticles that give out lots of heat right away because the field can be focused on a small region."

- Chad Boutin, NIST

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* C.L. Dennis, K.L. Krycka, J.A. Borchers, R.D. Desautels, J. van Lierop, N.F. Huls, A.J. Jackson, C. Grüttner and R. Ivkov. Internal magnetic structure of nanoparticles dominates time-dependent relaxation processes in a Magnetic Field. *Advanced Functional Materials*. Published online June 2, 2015. DOI: 10.1002/adfm.201500405.

NIST MML Hosts Workshop on Measurement Assurance Strategies for Cell Therapy Products

On May 11-12, 2015, NIST MML hosted a workshop to examine approaches for improving confidence in the measurements that are necessary for bringing cell therapy products to market. Several industry reports have identified a lack of reliable measurements for qualifying cell therapy products as the key barrier to success in the industry. The workshop had 100 attendees, more than half of which were from cell therapy companies. FDA, NIH, Army and NIST staff were in heavy attendance, and academic and non-profits were also represented. The workshop kicked-off with a series of morning talks that set the table with the tools of measurement assurance, including traceability, validation, uncertainty, inter-laboratory studies, design of experiment, process control, reference materials and cause and effect analysis to identify sources of measurement variability. In the afternoon, participants divided into 3 parallel breakout sessions practiced applying these tools to the 3 measurements that industry felt were most in need of improved measurement assurance: cell counting, cell viability and cell potency tests. Participants expressed satisfaction and relief that the workshop brought clarity so they can improve confidence in cell measurements. Many were shocked that cell counting, the most fundamental of cell measurements, was highly variable, leading to serious concern for therapeutic dosing. It was also striking to learn that despite participation by 25+ cell therapy companies, the only reliable potency test that could be identified was cell proliferation. The need for "cell reference materials" to serve as positive controls in cell-based potency tests was clearly identified, as was the need for documents to share the language and tools of measurement assurance with the cell therapy community. The organizing committee is engaging in a number of follow-up activities, including disseminating the workshop materials, composing a white paper to summarize the tools of cell therapy product measurement assurance and discussions with key stakeholders to design impactful projects that the community may undertake to overcome these measurement challenges.

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NIST 'How-To' Website Documents Procedures for Nano-EHS Research and Testing

Note: A version of this story previously appeared in NIST's TechBeat on June 16, 2015.

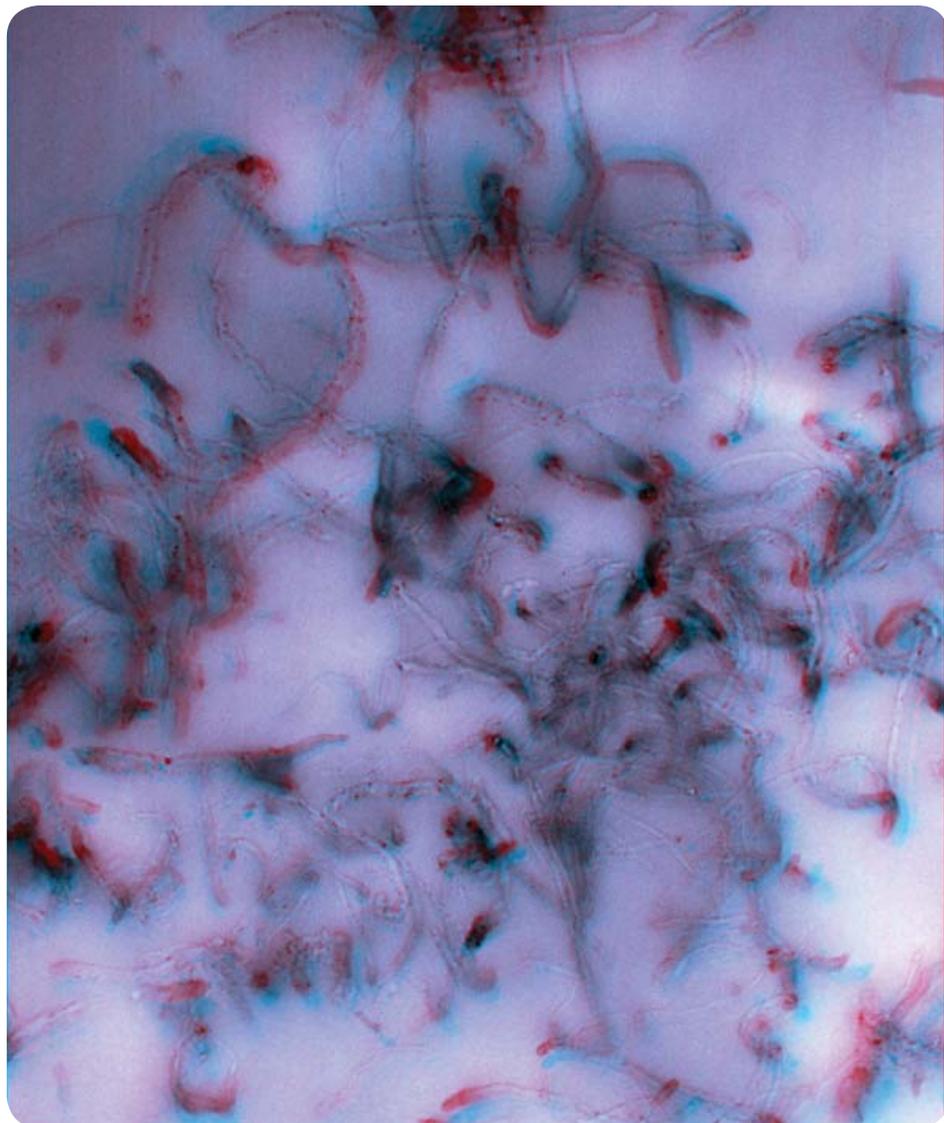
As engineered nanomaterials increasingly find their way into commercial products, researchers who study the potential environmental or health impacts of those materials face a growing challenge to accurately measure and characterize them. These challenges affect measurements of basic chemical and physical properties as well as toxicology assessments.

To help nano-EHS (Environment, Health and Safety) researchers navigate the often complex measurement issues, NIST has launched a new website (<http://www.nist.gov/mml/nanoehs-protocols.cfm>) devoted to NIST-developed (or co-developed) and validated laboratory protocols for nano-EHS studies.

In common lab parlance, a "protocol" is a specific step-by-step procedure used to carry out a measurement or related activity, including all the chemicals and equipment required. Any peer-reviewed journal article reporting an experimental result has a "methods" section where the authors document their measurement protocol, but those descriptions are necessarily brief and condensed, and may lack validation of any sort. By comparison, on NIST's new Protocols for Nano-EHS website, the protocols are extraordinarily detailed. For ease of citation, they're published individually—each with its own unique digital object identifier (DOI).

The protocols detail not only what you should do, but why and what could go wrong. The specificity is important, according to program director Debra Kaiser, because of the inherent difficulty of making reliable measurements of such small materials. "Often, if you do something seemingly trivial—use a different size pipette, for example—you get a different result. Our goal is to help people get data they can reproduce, data they can trust."

A typical caution, for example, notes that if you're using an instrument that measures the size of nanoparticles in a solution by how they scatter light, it's important also to measure the transmission spectrum of



Rigorous measurement protocols are key to unraveling the complex physical structure of carbon nanotubes [CNTs] embedded in a polymer composite, shown here in a three-dimensional scanning electron microscope image. The sizes, shapes and distribution of CNTs in the polymer can be measured from this image. Credit: Vladar/NIST

the particles if they're colored, because if they happen to absorb light strongly at the same frequency as your instrument, the result may be biased.

"These measurements are difficult because of the small size involved," explains Kaiser. "Very few new instruments have been developed for this. People are adapting existing instruments and methods for the job, but often those instruments are being operated close to their limits and the methods were developed for chemicals or bulk materials and not for nanomaterials."

"For example, NIST offers a reference material for measuring the size of gold nanoparticles in solution, and we report six different sizes depending on the instrument you use. We do it that way because different instruments sense different aspects of a nanoparticle's

dimensions. An electron microscope is telling you something different than a dynamic light scattering instrument, and the researcher needs to understand that."

The nano-EHS protocols offered by the NIST site, Kaiser says, could form the basis for consensus-based, formal test methods such as those published by ASTM and ISO.

NIST's nano-EHS protocol site currently lists 14 different protocols in three categories: sample preparation, physico-chemical measurements and toxicological measurements. More protocols will be added as they are validated and documented. Suggestions for additional protocols are welcome at nanoprotocols@nist.gov.

- Michael Baum, NIST

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Characterizing Copolymers by SEC-Raman Proven Feasible

Chemical heterogeneity, defined as the average copolymer composition as a function of analyte molar mass, has the ability to influence processing and end-use properties such as toughness, brittleness, and biodegradability. Common methods of characterizing this heterogeneity include the coupling of size-based separations such as size-exclusion chromatography (SEC) to detection methods such as infrared (IR) and nuclear magnetic resonance spectroscopy and mass spectrometry. Each of these detection methods has its own advantages and drawbacks. IR, in particular, suffers from strong water absorbance in this region of the electromagnetic spectrum, rendering difficult the characterization of water-soluble copolymers.

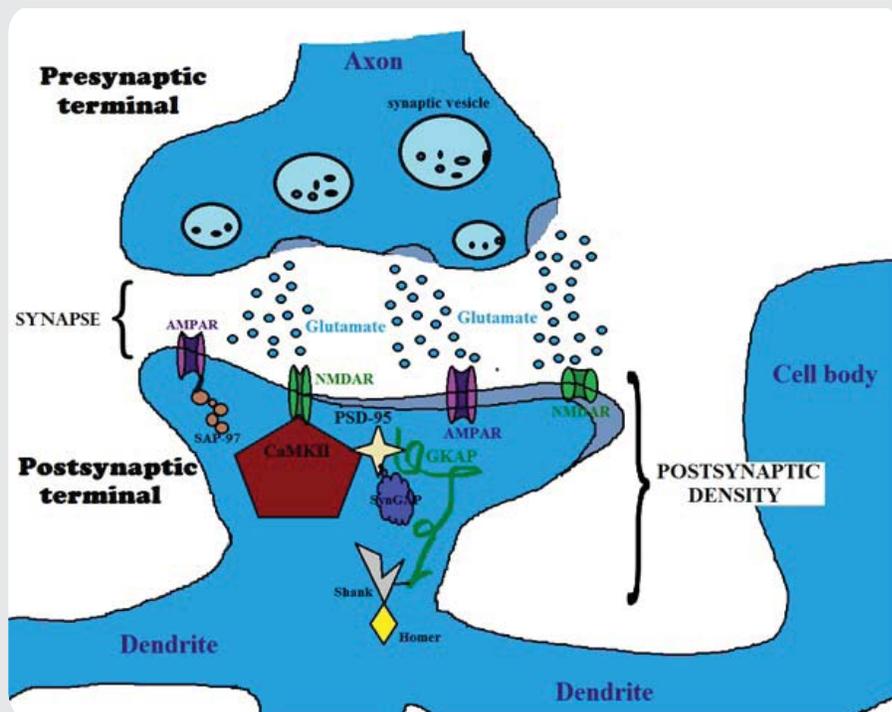
A team from NIST MML has demonstrated that the continuous off-line coupling of SEC to Raman spectroscopy is a feasible means of characterizing the chemical heterogeneity of copolymers.* The study, which involved a gradient random copolymer of styrene and methyl methacrylate, compared heart cuts from an SEC separation, subsequently analyzed by Raman, to the chemical heterogeneity of this copolymer as determined by SEC with on-line multi-angle static light scattering (MALS), differential refractometry (DRI), and ultraviolet absorption (UV) detection (SEC/MALS/DRI/UV). Not only did the SEC-Raman method rank the fractions in the correct order with respect to percent styrene (% S), but close quantitative agreement was found between this method and SEC/MALS/DRI/UV, with the % S by both methods differing from each other, on average, by less than 3%.

Raman spectroscopy provides complimentary vibrational information compared with IR, and Raman has a distinct advantage over IR for the characterization of water-soluble copolymers, as water exhibits very weak Raman scatter.

The study also determined that similar, or even the same, hardware employed for copolymer characterization by SEC with continuous off-line IR detection can almost certainly be used for the same type of coupling of Raman to SEC and other size-based separation methods such as hydrodynamic chromatography and flow field-flow fractionation.

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* L. Pitkänen, A.A. Urbas, A.M. Striegel, "On the feasibility of determining polymer chemical heterogeneity by SEC with continuous off-line Raman detection," *Polymer Chemistry* 6, 4864 (2015).



Neurons rapidly process and transmit electrical and chemical signals across synaptic junctions forming large neural networks that make up our central nervous systems. The post-synaptic density of proteins, largely unknown quantitatively, is responsible for collecting and organizing those signals via neurotransmitter receptors located in the synaptic cleft, opposing the active zone of the presynaptic terminal. This specialized protein density includes transmembrane receptor proteins (AMPA and NMDAR), cell adhesion proteins, protein kinases, and signaling proteins, among others, and facilitates rapid signaling across neural junctions.

NIST MML Researchers Measure Proteins in the Post-Synaptic Density of Rat Brains

NIST MML researchers, in collaboration with the National Institutes of Health, have measured average copy numbers of proteins in the post-synaptic density (PSD) from rat brains, helping to further illuminate the intricate structure and protein architecture of the PSD. Their findings were recently reported in an article published in the *Journal of Proteome Research*.

People may be familiar with images of synapses showing the action of drugs on neurotransmitter release or storage; the PSD functions as the intelligent receiver of these small molecules. The PSD is a large, dynamic, protein-rich and electron-dense region at the postsynaptic membrane of a neuron, on the order of tens of nanometers thick. It has various essential functions including the organization of neurotransmitter receptors, and rapid cellular signaling. Although hundreds of proteins have been identified in the PSD, determining their relative abundances (stoichiometry) is a much more challenging task, and as such, have only previously been estimated using crude, semi-quantitative approaches.

A rigorous, targeted isotope-dilution mass spectrometry approach was developed at NIST

to provide relative copy numbers of 42 proteins specific to the PSD complex. Quantification was based on the cell-free expression of eight unique stable isotope-labeled biosynthetic concatemers that were subsequently used as internal standards for precise LC-MS/MS analyses. The heavy-isotope concatemers were designed to mimic the conditions of the native full-length proteins while also including an external standard peptide (streptavidin) that could be used to normalize for the exact expression of each concatemer. PSD fractions were enriched from rat brains through dissection, tissue homogenization, and sucrose density fractionation by high-speed centrifugation. Enriched protein fractions were then digested, separated by liquid chromatography, and detected using multiple-reaction monitoring mass spectrometry.

Understanding the structure and relative composition of the PSD is essential to eventually understanding synaptic plasticity which is the basis for learning and memory. This work presents a robust quantitative analysis of PSD proteins in the ongoing effort to unravel the complex molecular architecture of this dynamic neuronal structure.

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Lowenthal, M.S.; Markey, S.P.; Dosemeci, A. Quantitative Mass Spectrometry Measurements Reveal Stoichiometry of Principal Postsynaptic Density Proteins. *J. Proteome Res.* Apr. 2015.

<http://pubs.acs.org/doi/abs/10.1021/acs.jproteome.5b00109>

NIST Impact Verification Program

Objective

The objective of the impact verification program is to evaluate the performance of impact test machines used worldwide to qualify structural steels. We offer our customers standard reference materials (SRMs) that enable certification of their impact machines to a traceable measurement system. This indirect verification of machine performance increases the accuracy of impact data, which improves predictions of the reliability of bridges, buildings, railroads and other infrastructure, as well as the safety of products manufactured from structural steel such as oil and gas pipelines, heavy trucks, mining equipment, power plants and wind turbines.

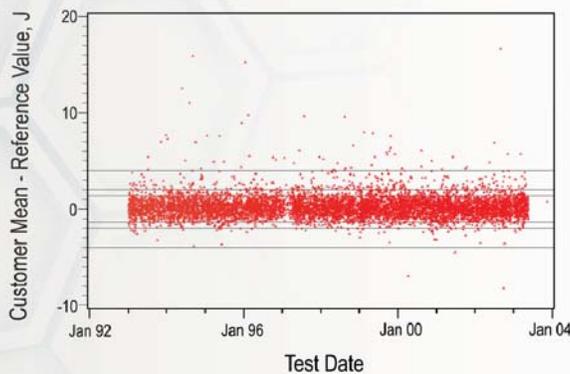


Credit: Geoffrey Wheeler

Impact and Customers

Impact testing is required for many critical applications in the construction, machinery and equipment, defense, and energy markets, which accounted for two thirds of the 91 million tons of steel shipped in 2011, valued at \$48 billion. Charpy testing provides data needed to insure the quality and reliability of these structural steel products.

Over 1,000 machines per year are evaluated against ASTM standards for our customers around the world. Machines certified through the NIST system provide results within 5%, or 1.4 J, of one another, which to the best of our knowledge is the narrowest distribution of impact results in the world today. Our customers include major steel manufacturers such as ArcelorMittal, Nucor, U. S. Steel and SSAB, as well as heavy equipment manufacturers such as Caterpillar and Westinghouse Nuclear.



Approach

Charpy impact is a high strain rate test that measures energy absorption during fracture, providing an indirect measure of fracture toughness. In order for a Charpy machine to maintain an accurate absorbed energy scale, periodic testing with certified test specimens is required. To achieve the required accuracy for these indirect verifications, NIST maintains three reference impact machines for the United States (per ASTM E23). The average absorbed energy for samples from a given lot tested on these machines is defined as the certified value.

The program provides a complete certification service. A set of five SRMs, manufactured to NIST specifications and certified on our reference machines, are sold to each customer. After customer testing they are returned to our laboratory. Based on our evaluation of the test results and the fractured specimens, NIST will either issue a certificate of compliance or provide suggestions for correction. The accumulated verification results are stored in a database, which is valuable for tracking quality of individual machine performance and for trend analysis to evaluate proposed changes to ISO and ASTM standards.



Accomplishments

The NIST impact verification program has provided 22 years of service to manufacturers and consumers and currently certifies eight SRMs that underpin quality control testing of impact toughness for structural steels. We provide SRMs used to verify the measurement of absorbed energy at three energy levels: low energy (14-20 J); high energy (88-136 J); and super high energy (176-244 J). We also provide two SRMs that are used to verify the measurement of maximum force in a Charpy impact test. SRMs are available for both Charpy and Izod impact testing (Figure 1).

The Charpy program supplies 2,000 units per year to customers worldwide. Typically, we evaluate and report on over 1,000 verification tests per year. We are available to our customers by email, fax or phone (1,500 contacts per year) and continually update our customer website and database to improve customer interactions. Most recently, the program has gone paperless, improving the already short turnaround time for our post-test evaluation to same-day service.

We recently added a means for our customers to obtain a proficiency test result for their impact test (Figure 2). When a customer purchases a set of SRMs, the data from all NIST verification tests for that lot are made available. Customers can compare their test results to this compiled data, giving them an alternate means (aside from meeting ASTM or ISO requirements) to evaluate the performance of their impact machines, and the relative performance of multiple impact machines within their organizations. This information supports

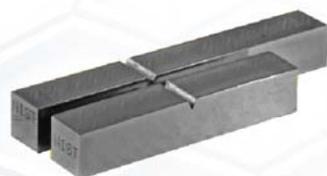


Figure 1. Examples of Charpy and Izod samples.

efforts by industry to achieve or maintain quality system certification through ISO or other standards bodies.

NIST maintains leadership positions on ASTM and ISO standards committees and has long been active in improving impact testing standards both in the U.S. and around the world. We maintain an extensive database of customer data in order to further improve measurement accuracy and support our ISO and ASTM activities. For example, we publish a guide on uncertainty analysis for Charpy tests that offers users a full explanation of the uncertainty associated with the NIST reference specimens and the customer's verification test.

We are currently working with several national metrological institutes (NMIs) to develop an approach to SRM certification that reduces the bias among the NMIs. Our focus is on standardizing the design of instrumented strikers to provide comparable load-displacement data across all types of Charpy machines. If successful, absorbed energy measured under the instrumented impact curve will be traceable to force and time, moving the underpinnings of the measurement to more fundamental quantities (Figure 3). This approach, like the current approach of measuring the energy absorbed from a pendulum swing (in Joules),

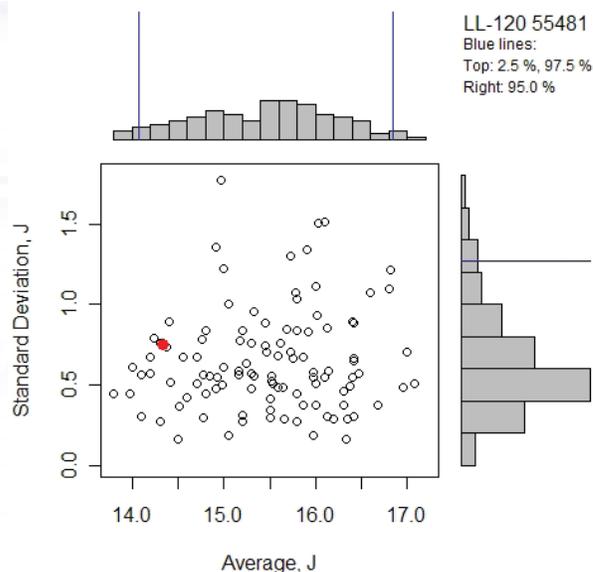


Figure 2. Example of proficiency test data available for production lots.

has many practical issues that will need to be addressed. However, we see it as the future in impact testing and the best hope for establishing a scale for measuring absorbed energy that can help to reduce bias among NMIs.

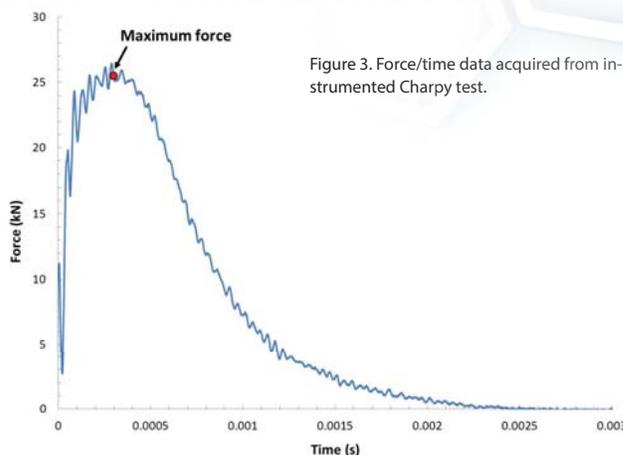


Figure 3. Force/time data acquired from instrumented Charpy test.

Learn More

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Publications

E Lucon, CN McCowan, and RL Santoyo, Certified KLST Miniaturized Charpy Specimens for the Indirect Verification of Small-Scale Impact Machines, in: Small Specimen Test Techniques: 6th Volume, STP 1576, MA Sokolov and E Lucon, Eds., ASTM International, West Conshohocken, PA (2015).

E Lucon, CN McCowan, and RL Santoyo, Overview of NIST activities on sub-size and miniaturized Charpy specimens: correlations with full-size specimens and verification specimens for small-scale pendulum machines, Proceedings of ASME 2015 Pressure Vessels & Piping Conference, PVP2015, Boston, Massachusetts USA (July 2015).

E Lucon, Certified Miniaturized Charpy Specimens for the Indirect Verification of Small-Size Impact Machines, Materials Performance and Characterization, 2, 1 (June 2013).

NIST's 'Nano-Raspberries' Could Bear Fruit in Fuel Cells

Note: A version of this story previously appeared in NIST's TechBeat on June 9, 2015.

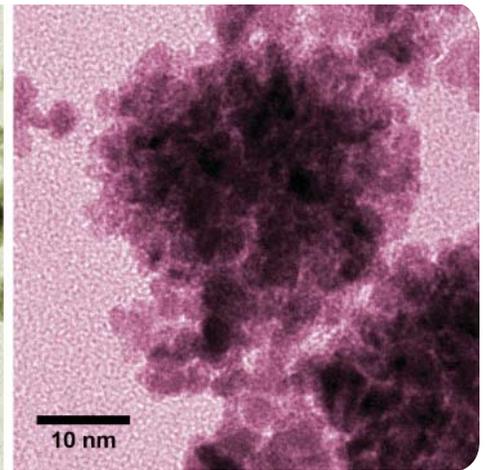
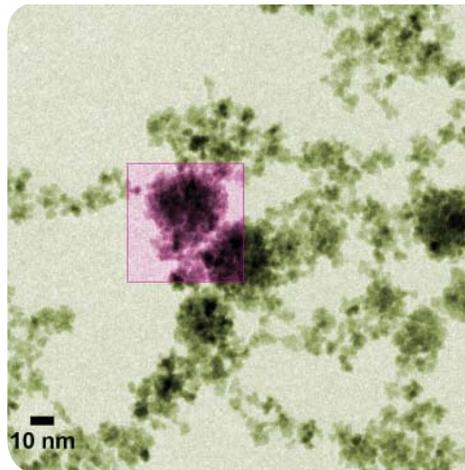
NIST researchers have developed a fast, simple process for making platinum "nano-raspberries" - microscopic clusters of nanoscale particles of the precious metal. The berry-like shape is significant because it has a high surface area, which is helpful in the design of catalysts. Even better news for industrial chemists: the researchers figured out when and why the berry clusters clump into larger bunches of "nano-grapes."

The research could help make fuel cells more practical. Nanoparticles can act as catalysts to help convert methanol to electricity in fuel cells. NIST's 40-minute process for making nano-raspberries, described in a new paper,* has several advantages. The high surface area of the berries encourages efficient reactions. In addition, the NIST process uses water, a benign or "green" solvent. And the bunches catalyze methanol reactions consistently and are stable at room temperature for at least eight weeks.

Although the berries were made of platinum, the metal is expensive and was used only as a model. The study will actually help guide the search for alternative catalyst materials, and clumping behavior in solvents is a key issue. For fuel cells, nanoparticles often are mixed with solvents to bind them to an electrode. To learn how such formulas affect particle properties, the NIST team measured particle clumping in four different solvents for the first time. For applications such as liquid methanol fuel cells, catalyst particles should remain separated and dispersed in the liquid, not clumped.

"Our innovation has little to do with the platinum and everything to do with how new materials are tested in the laboratory," project leader Kavita Jeerage says. "Our critical contribution is that after you make a new material you need to make choices. Our paper is about one choice: what solvent to use. We made the particles in water and tested whether you could put them in other solvents. We found out that this choice is a big deal."

The NIST team measured conditions under which platinum particles, ranging in size from 3 to 4 nanometers (nm) in diameter, agglomerated into bunches 100 nm



Colorized micrographs of platinum nanoparticles made at NIST. The raspberry color suggests the particles' corrugated shape, which offers high surface area for catalyzing reactions in fuel cells. Individual particles are 3-4 nm in diameter but can clump into bunches of 100 nm or more under specific conditions discovered in a NIST study. Credit: Curtin/NIST

wide or larger. They found that clumping depends on the electrical properties of the solvent. The raspberries form bigger bunches of grapes in solvents that are less "polar," that is, where solvent molecules lack regions with strongly positive or negative charges. (Water is a strongly polar molecule.)

The researchers expected that. What they didn't expect is that the trend doesn't scale in a predictable way. The four solvents studied were water, methanol, ethanol and isopropanol, ordered by decreasing polarity. There wasn't much agglomeration in methanol; bunches got about 30 percent bigger than they were in water. But in ethanol and isopropanol, the clumps got 400 percent and 600 percent bigger, respectively—really humongous bunches. This is a very poor suspension quality for catalytic purposes.

Because the nanoparticles clumped up slowly and not too much in methanol, the researchers concluded that the particles could be transferred to that solvent, assuming they were to be used within a few days—effectively putting an expiration date on the catalyst.

Two college students in NIST's Summer Undergraduate Research Fellowship (SURF) program helped with the extensive data collection required for the study.

- Laura Ost, NIST

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* I. Sriram, A.E. Curtin, A.N. Chiramonti, J.H. Cuchiaro, A.D. Weidner, T.M. Tingley, L.F. Greenlee and K.M. Jeerage. Stability and phase transfer of catalytically active platinum nanoparticle suspensions. *Journal of Nanoparticle Research* 17:230.DOI 10.1007/s11051-015-3034-1. Published online May 22, 2015.

NIST MML Releases Comprehensive Review on Strength of Silicon for Microsystems

NIST MML researchers, in collaboration with Sandia National Laboratories, have completed the most comprehensive review to date of the fracture strength behavior of micrometer- and nanometer-scale silicon structures that form the foundations of micro- and nanoelectromechanical systems (MEMS and NEMS). The miniature devices fabricated by such technologies are capable of converting electrical energy into mechanical work, and are now found in diverse consumer and military applications such as inkjet printing, accelerometers, gyroscopes, microphones, pressure sensors, micromirror displays, and energy harvesting. Since silicon is an inherently brittle material, it is imperative that these systems are not only designed and fabricated properly, but that their failure can be precisely predicted and controlled under service conditions.

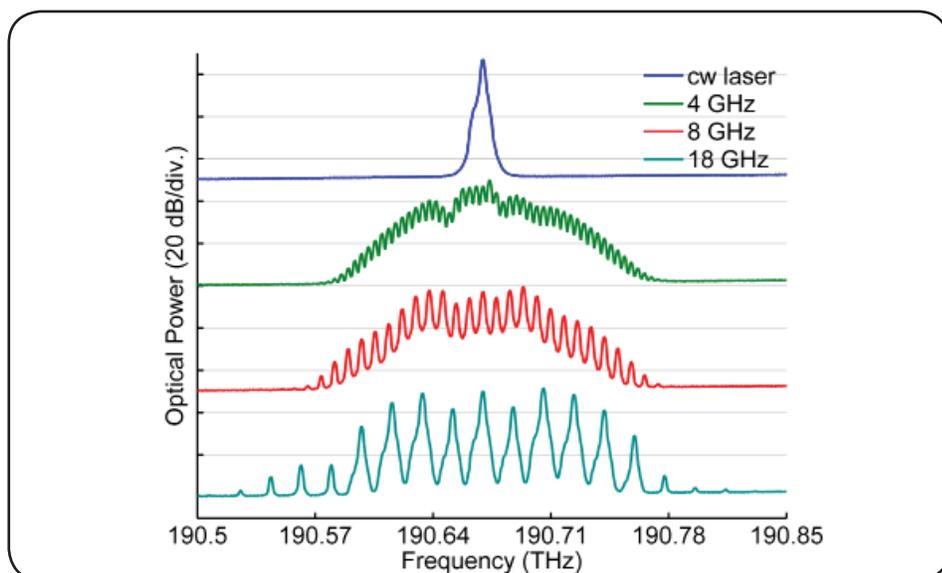
Fundamental research on strength of silicon reached maturation about five years ago, with the field now applying established materials processing-atomistic structure relationships to the production of high-performance components and devices. During that time of rapid product development, the market size has tripled, as the technologies are now found in every state of the art smartphone, automobile, and computer. This review describes in great detail how one may apply knowledge of material structure, test methods, and test results to the design and fabrication of sophisticated, reliable devices; much of this work originated within NIST. The article (FW DelRio, RF Cook, BL Boyce, *Applied Physics Reviews* 2, 021303 (2015)) is now available online (<http://dx.doi.org/10.1063/1.4919540>). Its appearance is timely, and it is anticipated to become the "standard reference" for students, researchers, and device designers who need to know what has been done in a particular sub-field of MEMS/NEMS technology.

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All-Fiber Optical Frequency Comb Generators Offer Greater End-User Flexibility

Femtosecond optical frequency combs have revolutionized the fields of optical metrology and precision spectroscopy. With exceptional precision and accuracy, frequency combs generated from the pulsed output of ultra-stable mode-locked lasers are now routinely used as frequency rulers for experiments including optical clocks, time and frequency transfer, and even molecular spectroscopy. However, not all experiments in molecular spectroscopy, particularly trace gas sensing and remote sensing, require an octave-spanning laser source. Therefore, the meticulous control of ultrafast laser pulses may not be desirable for certain applications in targeted chemical sensing.

NIST MML researchers, in collaboration with NIST's Physical Measurement Laboratory, have developed a user-friendly all-fiber frequency comb spectrometer using a single commercial laser source and specialized light modulators.* The spacing between each comb line is controlled by the end-user via simple microwave sources, and is tunable over more than three orders of magnitude. This flexibility in



Several optical frequency comb generators of increasing comb line spacing (4, 8, and 18 GHz, respectively) are shown above.

defining the comb line spacing has allowed NIST researchers to probe CO₂ transitions in an all-fiber spectrometer in only a few μ s of acquisition time. When high sensitivity is required, NIST has coupled the all-fiber frequency comb source to an enhancement cavity, which allows for trace amounts of CO₂ to be monitored with both high time and frequency resolution. Ongoing work involving these flexible fiber-based frequency combs is focused on increasing the laser bandwidth for multi-chemical sensing as well as integrating these novel laser sources into

NIST remote gas sensing programs and initiatives.

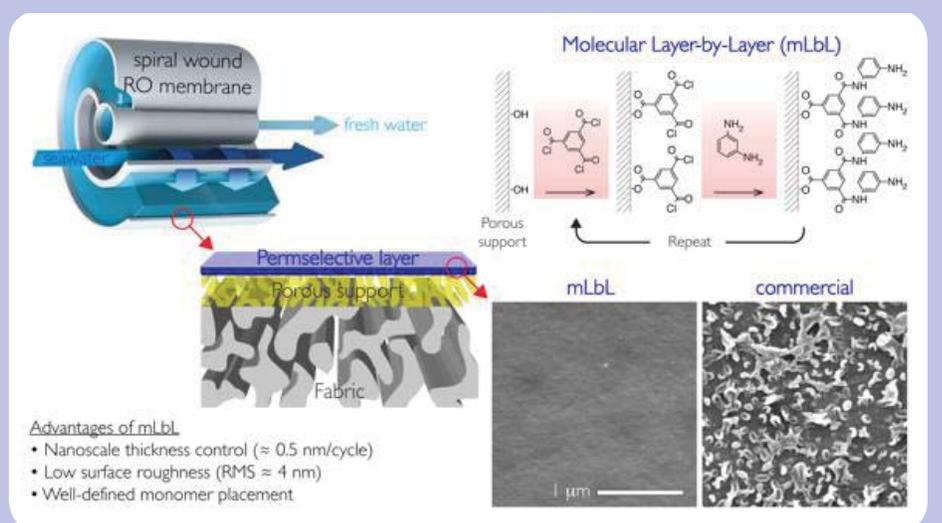
Support for this project is provided by the NIST Greenhouse Gas Measurements and Climate Research Program.

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* D.A. Long, A.J. Fleisher, K.O. Douglass, S.E. Maxwell, K. Bielska, J.T. Hodges, D.F. Plusquellic, "Multiheterodyne Spectroscopy with optical frequency combs generated from a continuous-wave laser," *Optics Letters* 39, 2688 (2014).

NIST MML Signs CRADAs with Leading Water Filtration Membrane Companies

NIST MML recently entered into CRADAs with two of the leading materials providers of reverse osmosis and nanofiltration membranes for water filtration. The CRADAs leverage a molecular layer-by-layer (mLbL) assembly process developed at NIST as a method to synthesize novel microporous membranes for water filtration. Compared with the interfacial polymerization schemes used in industry, the mLbL approach creates atomically smooth membranes with controlled and uniform thickness. This source of high quality membranes has enabled unparalleled measurements of membrane performance and opened the door for rationale structure-property-processing relationships to help design new and improved membrane materials. This led to a CRADA with Dow Water & Process Solutions and Penn State University to couple the in-silico design and modeling of new membrane materials with NIST measurement capabilities developed around the mLbL platform.



Model membrane materials for water desalination via molecular layer-by-layer (mLbL) assembly.

In a second CRADA with GE Global Research the NIST team is working collaboratively to demonstrate the scalability of the mLbL process as a manufacturing process for the roll-to-roll fabrication of water filtration membranes. Earlier this year GE received funding through the U.S. Department of Energy's Innovative Manufacturing Initiative to work

together with NIST on this effort. If successful, these membranes will greatly increase the energy efficiency of the separation process, thus reducing the overall energy demands of water purification plants.

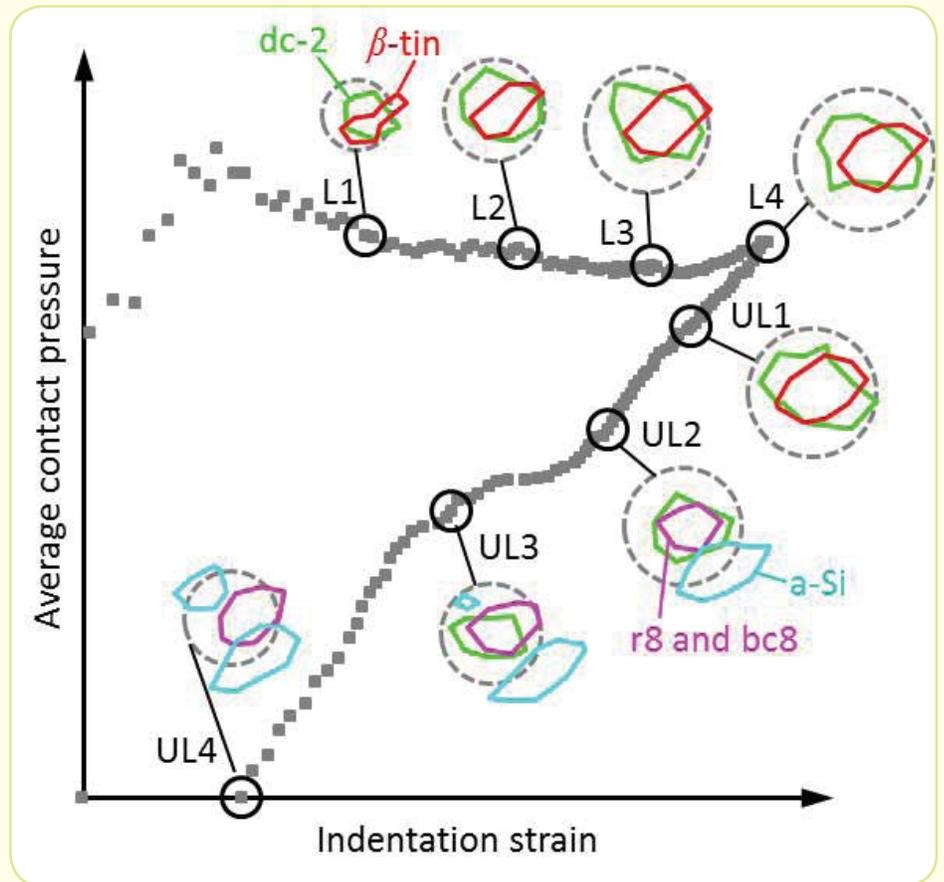
Contact: Chris Stafford, chris.stafford@nist.gov

NIST MML Researchers Demonstrate New Instrument for Raman Spectroscopy Enhanced Instrumented Indentation Testing

NIST MML researchers have developed a new instrument that combines the controlled deformation capabilities of instrumented indentation testing (IIT) with the analytical power of confocal Raman spectroscopy. The instrument enables structural changes of strained regions in transparent materials to be probed in-situ, during deformation processes.

IIT is a widely used method for fundamental studies of materials physics as well as for quantitative determination of the mechanical properties and deformation behavior of materials. However, in conventional IIT, it is only possible to characterize modifications in the material or the material stress state on residual indentation artifacts, leaving researchers without any direct measurements of the kinetic and physical processes occurring during an indentation event. The newly developed instrument overcomes this problem and enables the direct observation of such processes by employing confocal Raman spectroscopy as an auxiliary probe to identify modifications in the material nanostructure during deformation processes simultaneously with IIT in a method termed Raman spectroscopy enhanced (RSE)-IIT.

The RSE-IIT instrument can be operated in two configurations: In situ Raman microprobing and in situ Raman mapping. In situ Raman microprobing enables the in situ collection of Raman spectra at one individual spatial location. This configuration allows the direct observation of time-sensitive processes (e.g., the onset of phase transitions or ferroelastic domain switching) as only the processing of a single spectrum is required to analyze



Summary of the evolution of the phase distribution inside the contact area (dashed line) for the various loading stages during the indentation experiment represented as a strain-contact pressure curve.

spectral and related structural changes. However, in situ Raman microprobing is limited to a small fraction of the strained material region and thus provides only localized information. Therefore, recent efforts in refining the instrument design were focused on expanding the capability of RSE-IIT to in situ Raman mapping (or in situ Raman imaging) to enable the collection of Raman spectra over an entire image. The hyperspectral image can then be analyzed to yield spatial maps of material quantities, including crystallinity, grain size, type and symmetry of molecular alignment, composition, or strain.

The functionality of this new measurement tool was demonstrated in a recent study documenting the spatial and temporal evolution of the crystallographic structure of Si thin films during indentation.* The study includes the first sequence of Raman images documenting the evolution of strain fields and related changes in the phase distributions of a material as a function of mechanical load applied

under indentation conditions. The reported in situ experiments provided insights into the mechanically-induced transformation processes in Si under the influence of shear stress, confirming earlier theoretical predictions.

This new technique gives researchers the opportunity to perform in situ Raman spectroscopic studies of the evolution of structural modifications and their correlation to stress fields in materials under various types of contact loading and can thus provide critical experimental data necessary for the evaluation and refinement of new theoretical models.

Contacts: Chris Michaels, chris.michaels@nist.gov; Robert Cook, robert.cook@nist.gov

* Y.B. Gerbig, C.A. Michaels, R.F. Cook, In situ observation of the spatial distribution of crystalline phases during pressure-induced transformations of indented silicon thin films. *Journal of Materials Research / FirstView Article*, published online: 11 November 2014, DOI: <http://dx.doi.org/10.1557/jmr.2014.316>

Superball Classical Colloid Suspension Models Take a Step Towards the Real World

Non-spherical particles have been gaining more attention as manufacturing techniques advance and more anisotropic objects like biomolecules are developed for a range of applications.

Indeed most 'real world' particle solutions actually contain non-spherical particles rather than the classical spheres so often used in models. Superballs, which with the tuning of a single parameter can smoothly change from spheres to cubes, are an ideal choice of model shape to study the effects of anisotropy because of their simplicity. Prior work focused on how shape affects packing in 2D and 3D; however, the hydrodynamics of particle suspensions critically important to developing commercial fluid products, had not been considered. Using both experiments and computation NIST scientists have quantified the properties

of these suspensions, the subject of two recent Soft Matter articles.***

New techniques now make it possible to prepare large quantities of uniform silica superballs. Utilizing these colloidal superballs, experiments were carried out exploring the flow properties and micro-scale motions of these objects in 3D suspensions. By focusing on these well-controlled and well-characterized, shaped particles, these experiments were able to disentangle shape effects of isolated particles from shape-mediated particle interactions. While key properties of isolated superballs, such as their intrinsic viscosity and hydrodynamic diameter, didn't change much from comparably sized spheres, shape-mediated interactions play a significant role in the behavior of suspensions. These interactions change small-scale structure and inhibit local motion. Furthermore, the experiments revealed measurable differences in an unusual property called shear thickening that couldn't be explained simply by the non-spherical shape.

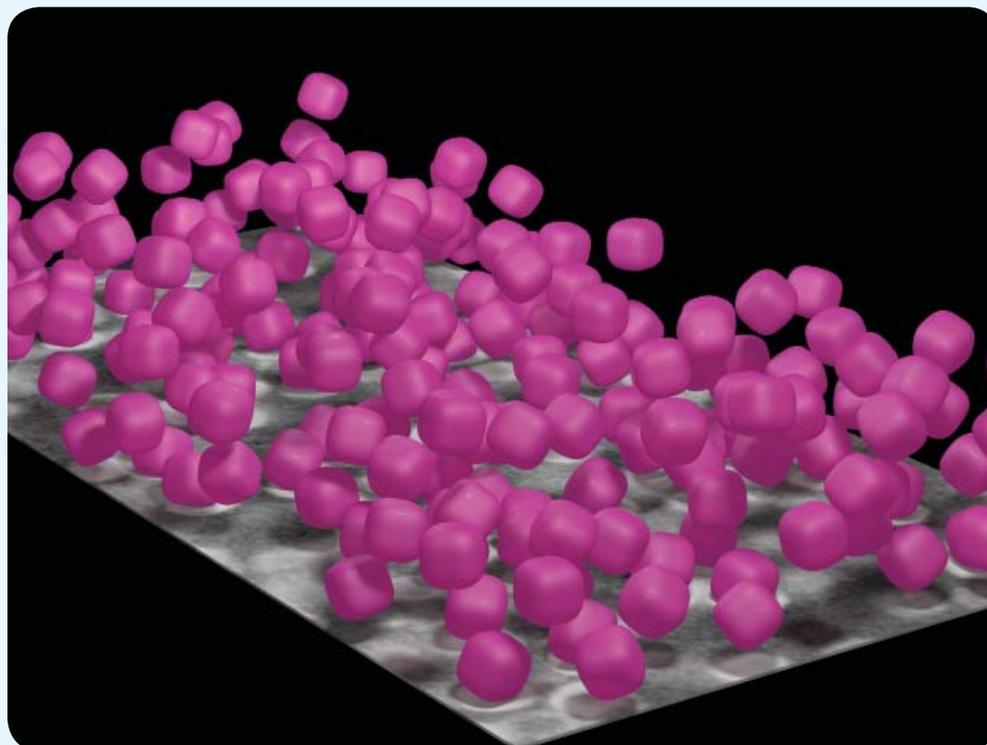
Inspired by experimental efforts, dilute hydrodynamic properties including the hydrodynamic radius, intrinsic viscosity and intrinsic solvent diffusivity were determined not only for a single super-

ball shape, but also for the entire class of particles ranging from spheres to cubes. Calculations, which were performed using three conceptually different methods, showed that the properties were only minimally affected for sphere-like particles, but as the particles became more cube-like, they started to depend strongly on shape. Additionally, it was determined that of the properties considered, the intrinsic viscosity was the most sensitive to shape. The computations were validated by NIST's experimental measurements and others from the literature. This work serves two very important goals: it adds a layer of more complex understanding to the fundamental understanding of suspensions so important in many products, and it provides additional validation and an important use case for the XENO computational tool developed at NIST.

Contacts: Debbie Audus, debra.audus@nist.gov; John Royer, john.royer@nist.gov

* D.J. Audus, A.M. Hassan, E.J. Garboczi and J.F. Douglas *Soft Matter* 11 3360- 3366 (2015)

** J. R. Royer, G. L. Burton, D. L. Blair, S. D. Hudson, "Rheology and Dynamics of Colloidal Superballs" *Soft Matter*, Vol. 11, No. 28, pp. 5656-5665, (09-Jun-2015)



A three-dimensional rendering of a superball suspension, using particle positions measured in an experiment from a confocal stack, atop a two-dimensional image from that same confocal stack. Credit: John Royer, NIST

Outreach and Partnering

NIST MML Polymerization Stress Tensometer Used in Collaborative Work with Industry

A senior materials researcher from Dentsply, a global leader in consumable dental products, visited NIST May 14-15, 2015 to conduct joint research. The goal was to better understand the performance of a new photocurable dental material under development at Dentsply using the NIST Standard Reference Instrument 6005 (Polymerization Stress Tensometer). The NIST instrument has the unique capability to capture early reaction kinetics, critical in controlling the subsequent performance of the material. Martin Chiang, Zhengzhi Wang, and Paula Baker of the MML's Biomaterials Group conducted experiments with Dr. Hui Lu of Dentsply to obtain curing kinetics measurements for several photocurable composite resins, including the material under development.

NIST MML Hosts Gross Elemental Working Group Meeting

Representatives from Shafer Laboratories, McCrone Associates, Pacific Northwest National Laboratory, NIST and the Air Force met at NIST Gaithersburg May 12-13 to discuss the state-of-the-art in automated particle analysis by scanning electron microscope with energy dispersive x-ray spectrometers (SEM/EDS). Of primary interest were discussions about how to best take advantage of higher resolution microscopes and faster spectrometers to enhance throughput. Recent advances could potentially provide an order-of-magnitude higher throughput. This improved throughput could be used to improve trace element sensitivity, to find needle-in-the-haystack-type particles or for better population statistics as the nature of the problem requires. The group discussed the costs and benefits of sacrificing throughput for a user-friendly commercial package or aiming for the highest throughput in a less refined but higher throughput custom package.

NIST MML Participates in the 2015 German-American Frontiers of Engineering Symposium

Edwin Chan and Kate Beers from MML's Materials Science and Engineering Division participated in the 2015 German-American Frontiers of Engineering Symposium, April 16-18, in Potsdam, Germany. This symposium assembled 60 outstanding engineers under the age of 45 for an intensive 2-1/2 day symposium to discuss cutting-edge developments in four areas: Nano-to-Micro Robotics, Synthetic Membranes and their Applications, Particle Accelerators and their Applications, and Protecting User Privacy in the Age of Big Data. The event facilitated international and cross-disciplinary research collaboration, promoted the transfer of new techniques and approaches across disparate engineering fields, and encouraged the creation of a transatlantic network of world-class engineers.

Recent NIST MML Awardees

Cicerone Receives Washington Academy of Science Award

Marcus Cicerone of MML's Biomaterials Group received a Washington Academy of Science Award in Physical and Biological Sciences in recognition of his groundbreaking and high-impact optical measurement methods for quantifying biological systems. The committee cited Dr. Cicerone's work for exemplifying the type of innovation in the physical sciences needed to make breakthrough advances in biology.

Becker Receives Young Scientist Award

Chandler Becker of MML's Materials Science and Engineering Division has been selected by the NIST chapter of Sigma Xi as the winner of the Katharine B. Gebbie Young Scientist Award for 2015. Becker was cited for her work in developing the NIST Interatomic Potentials Repository (<http://www.ctcms.nist.gov/potentials/>). She was also recognized for organizing a series of workshops on "Atomistic Simulations for Industrial Needs."

Hanisch Receives University of Maryland Department of Astronomy Alumnus of the Year Award

Robert Hanisch, Director of MML's Office of Data and Informatics, was honored with the Alumnus of the Year Award by the University of Maryland, Department of Astronomy in a ceremony held on April 10, 2015. Hanisch earned his Ph.D. at UMD - College Park in 1981. He was recognized for leadership in the astronomy community as well as for his recent move to NIST.

Heller Wins Patterson-Crane Award

Stephen Heller of the NIST Mass Spectrometry Group has been named the 2015 recipient of the Patterson-Crane Award for his work on the development of the IUPAC International Chemical Identifier. The Columbus and Dayton, Ohio Sections of the American Chemical Society sponsor the Patterson-Crane Award for contributions to chemical information. The IUPAC International Chemical Identifier (InChI) is a universal, open source language that uniquely defines the chemical structure of complex molecules using a string of numbers and letters. InChI has been readily adopted worldwide by chemical researchers, software developers, scientific publishers, industry, and federal agencies to exchange chemical structure information over the internet and link chemical data between diverse databases, scientific publications, patent literature, and popular news sources.

DeLongchamp Receives Flemming Award

On June 8, 2015, Dean DeLongchamp of MML's Functional Polymers Group received the Arthur S. Flemming Award at George Washington University. The Flemming Award was established in 1948 and honors outstanding federal employees. DeLongchamp was one of twelve recipients for the 2014 award cycle.

Gayle Elected as ASM Fellow

Frank Gayle of NIST's Advanced Manufacturing Program Office has been elected a Fellow of the ASM. This honor recognizes Gayle for his distinguished contributions in the field of materials science and engineering, and provides a forum for technical and professional leaders to serve as advisors to the Society. The citation reads: "For outstanding technical contributions and research management in materials measurement, applications, and manufacturing with significant contributions to light alloy metallurgy, quasicrystals, high temperature superconductivity, solder science and structural integrity."

Kattner to Receive J. Willard Gibbs Phase Equilibria Award from ASM

Ursula Kattner of MML's Thermodynamics and Kinetics Group has been selected to receive ASM International's 2015 J. Willard Gibbs Phase Equilibria Award. Her citation reads: "For contributions to the thermodynamic assessment of metallic alloys and application to metallurgical processing." The award was established in 2007 to recognize outstanding contributions to the field of Phase Equilibria.

Selected Recent Publications

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

J. M. Butler, "The Future of Forensic DNA Analysis" *Philosophical Transactions of the Royal Society B-Biological Sciences*, (22-Jun-2015) (PubID: 917722)

J. R. Royer, G. L. Burton, D. L. Blair, S. D. Hudson, "Rheology and Dynamics of Colloidal Superballs" *Soft Matter*, Vol. 11, No. 28, pp. 5656-5665, (09-Jun-2015) (PubID: 918003)

K. B. Gettings, K. M. Kiesler, P. M. Vallone, "Performance of a next generation sequencing SNP assay on degraded DNA" *Forensic Science International: Genetics*, Vol. 19, 10 pp., (27-May-2015) (PubID: 917873)

A. M. Striegel, L. M. Pitkanen, "Detection orthogonality in macromolecular separations. Role of the on-line viscometer in characterizing polymers at conditions of spectroscopic invisibility" *Chromatographia*, (21-May-2015) (PubID: 917603)

I. Sriram, A. E. Curtin, A. C. Chiamonti Debay, L. F. Greenlee, K. M. Jeerage, "Stability and phase transfer of catalytically active platinum nanoparticle suspensions" *Journal of Nanoparticle Research*, Vol. 17, pp. 1-11, (09-May-2015) (PubID: 917260)

K. B. Sebbly, E. Mansfield, "Determination of Polyethylene Glycol Surface Densities on Gold Nanoparticles with Microscale Thermogravimetric Analysis" *Analytical Chemistry*, (04-May-2015) (PubID: 908678)

N. A. Hotaling, K. Bharti, H. Kriel, C. G. Simon Jr., "Validated Open Source Nanofiber Diameter Measurement Tool" *Biomaterials*, (30-Apr-2015) (PubID: 917542)

M. S. Lowenthal, S. P. Markey, A. Dosemeci, "Quantitative Mass Spectrometry Measurements Reveal Stoichiometry of Principal Postsynaptic Density Protein Components" *ACS Journal of Proteome Research*, (28-Apr-2015) (PubID: 917230)

M. M. Schantz, B. A. Benner Jr, N. A. Heckert, L. C. Sander, K. E. Sharpless, S. S. Vander-Pol, S. A. Wise, "Development of Urine Standard Reference Materials for Metabolites of Organic Chemicals Including Polycyclic Aromatic Hydrocarbons, Phthalates, Phenols, Parabens, and Volatile Organic Compounds" *Analytical and Bioanalytical Chemistry*, Vol. 407, No. 11, pp. 2945-2954, (01-Apr-2015) (PubID: 916645)

E. R. Sisco, T. P. Forbes, "Rapid Detection of Sugar Alcohol Precursors and Corresponding Nitrate Ester Explosives using Direct Analysis in Real Time Mass Spectrometry" *Analyst*, Vol. 140, No. 8, pp. 2785-2796, (30-Mar-2015) (PubID: 917156)

K. M. Bauer, I. V. Turko, K. W. Phinney, "Quantitative performance of internal standard platforms for absolute protein quantification using MRM-MS" *Analytical Chemistry*, (26-Mar-2015) (PubID: 917718)

M.M. Dizdar, E. Coskun, P. Jaruga, "Measurement of oxidatively induced DNA damage and its repair by mass spectrometric techniques" *Free Radical Research*, Vol. 49, No. 5, pp. 525-548, (25-Mar-2015) (PubID: 917824)

A. Vaish, D.J. Vanderah, L. J. Richter, M. Dimitriou, K.L. Steffens, M. L. Walker, "Dithiol-based Modification of Poly(dopamine): Enabling Protein Resistance Via Short-Chain Ethylene Oxide Oligomers" *Chemical Communications*, Vol. 51, pp. 6591-6594, (16-Mar-2015) (PubID: 917070)

A. Y. Smolyanitsky, "Effects of Thermal Rippling on the Frictional Properties of Free-Standing Graphene." *RSC Advances*, pp. 29179-29184, (16-Mar-2015) (PubID: 916556)

J. A. Bowden, J. T. Bangma, J. R. Kucklick, "Development of an Automated Multi-Injection Shotgun Lipidomics Approach using a Triple Quadrupole Mass Spectrometer" *Journal of Lipid Research*, pp. 609-619, (12-Mar-2015) (PubID: 913825)

J. E. Schiel, C. D. Agarabi, S. C. Lute, B. K. Chavez, M. T. Boyne, K. A. Brorson, M. A. Kahn, E. K. Read, "Bioreactor Process Parameter Screening Utilizing a Plackett-Burman Design for a Model Monoclonal Antibody" *Journal of Pharmaceutical Sciences*, Vol. 104, No. 6, pp. 1919-1928, (11-Mar-2015) (PubID: 917246)

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

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