Material Matters

The Quarterly Magazine of NIST's Material Measurement Laboratory

Fall 2015

Determining the Age of Fingerprints

Water Measurement Science @NIST

New Hydrogen Storage Patent

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

About NIST's Material Measurement Laboratory

Formed in 2012, 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 of matter, providing broad support for the chemical, biological, and material sciences. Our fundamental and applied measurement science research expands possibilities for determining the composition, structure, and properties of manufactured, biological, and environmental materials, and the processes that create them. In addition, MML drives the development and dissemination of tools, including measurement protocols, certified reference materials, critically evaluated data, and best practice guides, that help assure quality measurements of matter. Our research and measurement services support areas of national importance, including:

- 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 air quality, water, mitigation of climate change, the health of marine environments, and the safety of nanomaterials
- Food safety and nutrition, from contaminant monitoring to ensuring the accuracy of nutrition labels
- Health care, from clinical diagnostics and precision medicine to the reproducibility of biological data
- Infrastructure, including assessment of the country's aging buildings, bridges, and pipelines for refurbishment, reuse, and repair
- Manufacturing, from lightweight alloys for fuel-efficient automobiles to biomanufacturing, advanced electronics, and data for chemical manufacturing
- Safety, security, and forensics, from contraband residue detection to ensuring the performance of body armor materials to DNA-based human identity testing

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

MML is home to nearly 1,000 staff members and visiting scientists. Organizationally, the laboratory is divided into six divisions, dedicated to maintaining cutting edge facilities and the technical expertise required to achieve our mission in the biological, chemical, and material sciences. Our organization includes several campuses and strategic partnerships:

- NIST main campus in Gaithersburg, MD: Home of MML headquarters and five of the six MML Divisions
- NIST Boulder Laboratories in Boulder, CO: Home of MML activities including pipeline research and mechanical properties assessment
- 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
- The Institute for Bioscience and Biotechnology Research 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, proteomics, and biomanufacturing
- Brookhaven National Laboratory in Upton, NY where, in partnership with the U.S. Department of Energy, MML has established instrumentation on what will be the brightest source of synchrotron soft and tender X-rays in the U.S., which will enable unprecedented analysis of a broad range of materials technologies including flexible electronics, highly engineered nanoparticles, and next generation semiconductor structures
- The Joint Initiative for Metrology in Biology program between MML and Stanford University, where NIST staff work side-by-side with Stanford faculty, groups, and commercial affiliates to develop standards and tools that enable translation of innovations in quantitative and engineered biology to clinical and commercial practice

Cover image: Chemical imaging of a fingerprint shows the relative distribution of palmitic acid (green) and the more immobile waxy residue (red).

Credit: Muramoto/NIST

A Message from the MML Director

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

At NIST's Material Measurement Laboratory we have always valued our strategic partners for helping us meet our stakeholders' needs. Enhancing strategic partnerships is one of five goals in MML's recently released strategic plan (http://mmlstategy.nist.gov). Partnerships help MML better leverage our resources, quickly enter into technical areas with significant potential for growth, and work with academia and industry in a flexible and effective manner. In addition to the many research partnerships at our campuses in Gaithersburg and Boulder, MML jointly operates and supports research organizations in five locations, including three long-standing partnership facilities:

- The Hollings Marine Laboratory in Charleston, SC – a world-class research facility that provides science and biotechnology applications to sustain, protect, and restore coastal ecosystems, with emphasis on links between environmental conditions and the health of marine organisms and humans. HML partners include NOAA's National Ocean Service, the South Carolina Department of Natural Resources, the College of Charleston, and the Medical University of South Carolina.
- At the U.S. Department of Energy's Brookhaven National Laboratory, where MML's Synchrotron Science group develops and disseminates synchrotron measurement science and technology needed by U.S. industry to measure the nanoscale electronic, chemical, and spatial structures of advanced materials.
- At the Institute for Bioscience and Biotechnology Research in Rockville, MD, where researchers leverage the collective research strengths of the University of Maryland, College Park; University of Maryland, Baltimore; and NIST in the biological and quantitative sciences, medicine, and engineering.

More recently, MML established the Joint Initiative for Metrology in Biology with Stanford University in Palo Alto, CA, where MML staff, Stanford faculty, groups, and commercial affiliates work to develop the standards and tools needed to bring innovations in quantitative biology and engineered biology to clinical and commercial practice. In Chicago, NIST formed the Center for Hierarchical Materials Design (ChiMaD) with the University of Chicago, Northwestern University, Argonne National Laboratory, QuesTek Innovations LLC, ASM International, and Fayetteville State University, where researchers work collaboratively to realize the full impact of the Materials Genome Initiative to accelerate materials discovery and development.

Though each unique, all of these partnerships leverage valuable resources and talent across institutions to generate productive research results, and play a key role in realizing NIST's mission of advancing U.S. innovation and industrial competitiveness.

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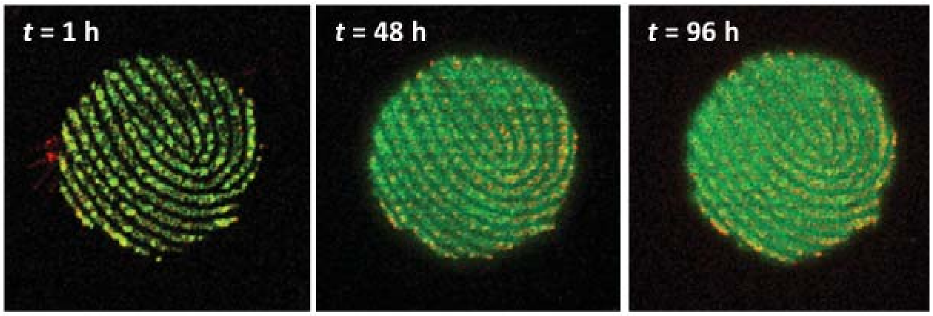

Credit: Shinichiro Muramoto, NIST

Time-of-flight secondary ion mass spectrometry (ToF-SIMS) ion images of the fingerprint on a silicon surface, showing the migration of the palmitic acid molecule (green) from the ridges of the fingerprint (1, 48, and 96) hours after deposition. The fingerprint ridge patterns are represented by a relatively immobile waxy residue (red). Images are 12 mm × 12 mm.

Who, What, WHEN: Determining the Age of Fingerprints

Note: A version of this story previously appeared in NIST's TechBeat on August 24, 2015.

Watch the imprint of a tire track in soft mud, and it will slowly blur, the ridges of the pattern gradually flowing into the valleys.

NIST MML researchers have tested the theory that a similar effect could be used to give forensic scientists something they've long wished for: A way to date fingerprints.

In a proof-of-concept paper published in Analytical Chemistry,[1] NIST MML chemists Shin Muramoto and Edward Sisco tested the idea that you could tell how old a fingerprint is by the degree to which the biomolecules, such as fatty acids, in the fingerprint's ridges have migrated down into the valleys. There have been a number of proposals for fingerprint dating in recent years, but the technique used by Muramoto and Sisco has a potential advantage in that it doesn't depend on chemical changes in the fingerprint, which are highly dependent on circumstances. Rather, it depends primarily on molecular weight and fairly well-understood models of molecular diffusion.

Even the approximate age of a fingerprint can have a critical bearing on forensic results. It can rule out some prints as being too old to be relevant to a crime scene or, conversely, to help fix the time of the crime. Military forensics experts would like to be able to date the multitude of fingerprints found on improvised bombs used by insurgents to winnow out prints of individuals who may simply have handled the components in a shop from those of the actual bombmakers. But a reliable technique has yet to be found.

"People have tried for 30 or 40 years to date fingerprints, usually by looking at changes in bulk chemical properties," says Sisco, but the specific chemical make-up of a fingerprint varies hugely from person to person, and the chemical changes with time depend heavily on the environment.

"We map chemical composition with a submicrometer resolution and look at how the compounds move," Sisco says, explaining that most of the material deposited in a fingerprint initially is in the ridges of the pattern, but over time it diffuses down into the valleys. Smaller molecules move faster, heavier ones slower, but all can be modeled with a standard function for molecular diffusion.

The NIST paper reports on aging fingerprints up to four days, but Sisco says that, in fact, "you can distinguish between the first day and a week, between a week and a month, between a month and four months."

The primary instrument, an imaging mass spectrometer, is not exactly a field device, and the NIST experiments used ideal test subjects: single prints deposited on polished silicon surfaces.

Given the promising results of their initial study, the NIST researchers say the next steps are to test the technique over longer timespans, on more realistic surfaces such as metal or paint, and under more uneven environmental conditions.

- Mark Bello

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[1] *S. Muramoto and E. Sisco. Strategies for potential age dating of fingerprints through the diffusion of sebum molecules on a nonporous surface analyzed using time-of-flight secondary ion mass spectrometry. Anal. Chem., Articles ASAP Publication Date (Web): July 17, 2015 DOI: 10.1021/acs.analchem.5b02018.*

Improvements in Analysis of Degraded Human DNA via Non-Traditional Forensic DNA Markers

NIST MML researchers have evaluated alternate forensic DNA marker types for analyzing degraded DNA samples, such as those found in mass casualty disasters. Single nucleotide polymorphisms (SNPs) are composed of a single base change in the DNA sequence, and assays composed of these marker types can be used to identify individuals. SNP assays target smaller DNA fragments than traditional forensic short tandem repeat (STR) DNA typing, and this can improve results when samples have been exposed to degrading environmental conditions such as heat or moisture. SNPs have not been widely implemented in forensic science because assays had historically proven labor intensive and limited to low throughput techniques. In recent years, next generation sequencing (NGS) technologies have greatly improved the efficiency of SNP typing. The NIST team evaluated if SNP based NGS assays may be well-suited to degraded DNA samples.

To create mock degraded DNA samples, the NIST team used an ultrasonicator to fragment high quality DNA, then used a gel system to separate the fragmented DNA into specific base pairs size ranges. For example, one mock degraded sample was size selected at fewer than 150 base pairs, meaning that no pieces of DNA larger than 150 base pairs are present. These samples containing specific fragment sizes were then analyzed with an NGS SNP assay on the Ion Torrent Personal Genome Machine, as well as via traditional STR methods. The results were measured using the statistic that is typically reported in forensic casework to convey the weight of the evidence, known as the random match probability. For various levels of degraded DNA, the NGS SNP assay yielded random match probability values many orders of magnitude higher than the traditional assays. The NIST team concluded that NGS SNP assays are expected to improve results in highly degraded DNA samples.

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K.B. Gettings, K.M. Kiesler, P.M. Vallone, Performance of a next generation sequencing SNP assay on degraded DNA, Forensic Science International Genetics, Published Online: May 25, 2015, DOI: http://dx.doi.org/10.1016/j.fsigen.2015.04.010

The NIST Mass Spectral Libraries Support Expanding Variety of Research Fields

The NIST Mass Spectral libraries, produced by a division of MML, are world renowned and widely used as a reliable comprehensive reference source of mass spectral "fingerprints" in metabolomics, environmental analysis, forensics, flavor and fragrance analysis, archaeology, art conservation, and many other disciplines where the reliable identification of specific molecules is sought. Because of the breadth of research that uses mass spectrometry for molecular identification, the subject matter of the references that cite use of the NIST Mass Spectral Library are numerous and often attention-grabbing, such as confirmation of proteins found in Tyrannosaurus Rex fossils in 2008.[1] Steve Stein, NIST Fellow and group leader of the NIST Mass Spectrometry Data Center, keeps track of NIST Mass Spectral Library citations by using a customized alert service offered by Google Scholar. Citations of the NIST Mass Spectral Library from the last year have included chemical analysis of whale breath volatiles,[2] comparison of synthetic and natural headspace molecules for marijuana, cocaine, and heroin,[3] and identification of gases emitted from the comet 67P/Churyumov-Gerasimenko.[4]

1. J.M. Asara, M.H. Schweitzer, L.C. Cantley, and JS. Cottrell, Response to Comment on "Protein Sequences from Mastodon and Tyrannosaurus rex Revealed by Mass Spectrometry", Science, 22 August 2008, vol 321, 1040c; doi:10.1126/science.1147046.

2. R. Cumeras, W.H.K. Cheung, F. Gulland, D. Goley and C.E. Davis, "Chemical Analysis of Whale Breath Volatiles: A Case Study for Non-Invasive Field Health Diagnostics of Marine Mammals", Metabolites 2014, 4(3), 790-806; doi:10.3390/metabo4030790.

3. S. Rice and J.A. Koziel, "The relationship between chemical concentration and odor activity value explains the inconsistency in making a comprehensive surrogate scent training tool representative of illicit drugs", Forensic Science International, available online 7 September 2015; doi:10.1016/j.forsciint.2015.08.027.

4. F. Goesmann et al., "Organic compounds on comet 67P/Churyumov-Gerasimenko revealed by COSAC mass spectrometry", Science, 31 July 2015: aab0689; doi:10.1126/science.aab0689.

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2015 Biomanufacturing Technology Summit

The 2015 Biomanufacturing Technology Summit was held at the Institute for Bioscience and Biotechnology Research (IBBR), a joint venture between NIST and the University of Maryland, on June 25, 2015. The summit focused on current trends, ideas, and predictions related to the determination of protein therapeutics' final quality attributes. Biotherapeutic characterization requires in-depth analysis of the final product's structure and stability. Scientists and engineers can exploit this historical knowledge to develop process models that are predictors of the quality attributes of final products. These predictive models are used during manufacturing to guide process control with biosensors, off-line measurements, and extrapolated algorithms. Industrial, federal, and academic researchers gathered to discuss the current trends in process modeling and its relation to the biotherapeutic "fingerprint."

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NIST MML Scientists Teach Forensics at STEMversity

STEMversity founder, Darrell Davis and MML scientists, Ashley B. Green and Jeanita S. Pritchett.

During the summer of 2015, NIST MML's Ashley Beasley Green and Jeanita Pritchett conducted two forensic science-based workshops for middle and high school students in the Scientific Explorers Program of STEMversity, Inc. STEMversity, Inc. is a non-profit organization that provides hands-on STEM training to students in middle school through college, as well as professionals. The Scientific Explorers Program uses forensic, pharmaceutical, environmental, and space science to stimulate students' interests and prepare them for STEM fields, and equip teachers with the resources to better teach and train students.

Pritchett's lecture focused on the basic principles of chromatography and its application in forensic science. In the "Case of the Fraudulent Check," Pritchett asked the students to identify which marker was used to commit the crime. The students used paper chromatography to separate the ink in each "suspect's" marker and compared their results to the results of the ink from the check.

The second half of the workshop focused on DNA fingerprinting and its implementation in criminal and parental identification cases. Green presented the fundamentals of deoxyribonucleic acid (DNA), the function of DNA in forensics, and the interpretation of DNA profiling data. The students used DNA profiling data to identify the suspects behind "The Veggie Mystery" and "The Murder Mystery." Green also discussed how DNA can be used for identification in the exercises, "Identification of the Unknown Soldier" and "Babies Switched at Birth."

- Ashley Beasley Green and Jeanita S. Pritchett

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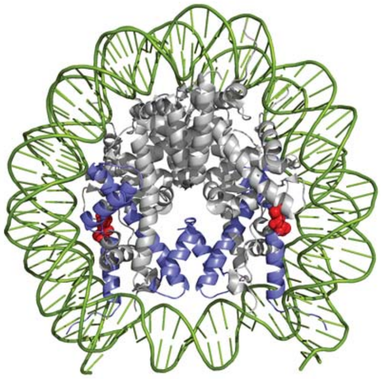
NIST MML Researchers Measure Changes in Histone Phosphorylation in Mouse Model of Alzheimer's Disease



Credit: Kyle Anderson, NIST

Structure of nucleosome with DNA shown in green, histones in gray, histone H3 in blue, and phosphorylation sites in red.

NIST MML researchers, with support from Case Western Reserve University, have measured changes in histone phosphorylation in a mouse model of neurodegeneration similar to Alzheimer's disease. Alzheimer's disease, a neurodegenerative disorder characterized by amyloid plaques and neurofibrillary tangles in the brain, is the sixth leading cause of death in the U.S. and accounts for 80% of dementia cases. Symptoms of Alzheimer's begin with impairment of memory and cognition and increase in severity until the individual loses lucidity, mobility, speech, and eventually, life. The cause of the majority of Alzheimer's cases is unknown. Currently, there is no treatment. Because people with Alzheimer's disease have a global decrease in gene expression, much Alzheimer's research now focuses on gene expression mechanisms, or epigenetics.

Histones are proteins that form a spherical assembly around which DNA is wrapped. Histone-wrapped DNA can be further condensed into chromatin, an essential higher order structure for the cell nucleus to contain the 3 billion base pairs of DNA and maintain organization. Modifications on histones can regulate how DNA interacts with them, and in turn, how genes are expressed. Phosphorylation is one type of modification on histones used to regulate gene expression. Researchers at NIST discovered two sites of phosphorylation on the surface of histones that are on the interface of the DNA-histone interaction. DNA has a negatively-charged backbone due to the abundance of phosphates. The histone surface is rich in positively-charged residues to facilitate the charge-charge attraction between histone and DNA. However, the introduction of phosphate groups on histone amino acid residue creates a negative charge, which causes repulsion between the negative charge on the DNA backbone and the negative charge on the phosphorylated histone residues. This repulsion can cause DNA and histones to favor dissociation, leaving DNA exposed and easily accessible to transcription machinery for gene expression. Mouse models that develop extensive amyloid deposits and cognitive impairment were shown to have a decrease in phosphorylation on the histone residues near the DNA backbone. Studies suggest that loss of phosphorylation favors a tighter histone-DNA assembly, which reduces gene expression. Alzheimer's disease is associated with a global decrease in gene expression, called an epigenetic blockade, which may be caused in part by the loss of histone phosphorylation measured by NIST researchers in this work.

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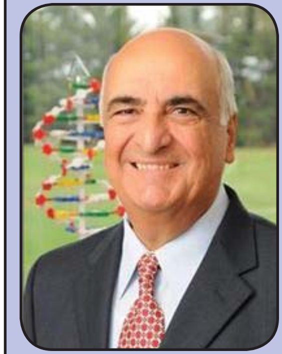
Anderson K.W., Mast N., Pikuleva I.A., Turko I.V., Histone H3 Ser57 and Thr58 phosphorylation in the brain of 5XFAD mice, FEBS Open Bio, Available online July 2015, http://dx.doi.org/10.1016/j.fob.2015.06.009.

Miral Dizdaroglu – DNA Damage and Repair



For more than 25 years, NIST Fellow Miral Dizdaroglu has developed and utilized hyphenated mass spectrometry techniques as primary analytical tools for measuring and characterizing the effects of ionizing radiation, chemical/biological carcinogens and oxygen-derived free radicals on the induction and subsequent repair of oxidatively induced DNA damage in living systems.

These ground-breaking, analytically complex measurements have led to the understanding that the specific type and accumulated level of multiple DNA lesions correlates with the incidence and progression of age-related diseases and cancers.

The 2015 Nobel Prize in chemistry (to Tomas Lindahl, Paul Modrich and Aziz Sancar) and Albert Lasker award (to Evelyn Witkin and Stephen Elledge) acknowledge work that built the foundation for emerging therapies that exploit DNA repair pathways for treatment of cancer and age-related cellular degeneration. While these researchers have been building our fundamental understanding of how these processes work, Dr. Dizdaroglu and his NIST team have been developing the analytical tools to enable reproducible measurements of the proteins involved in these pathways. The cancer therapeutics now in preclinical development based on the selective control/inhibition of DNA damage repair processes (DNA repair protein inhibition and DNA damage response modification) are enabled in part by the high-quality DNA damage measurements made at NIST.

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Genome in a Bottle Consortium Public Workshop

On August 27-28, 2015, the Genome in a Bottle Consortium held its sixth public workshop with about 170 participants from government, academia, and industry to discuss reference data and reference materials for human genome sequencing, and methods to characterize genomic reference materials. One session was devoted to exploring the use of the consortium's products for analytical validation of next generation sequencing as a clinical test, including discussion of regulatory oversight with a presentation from the FDA.

Important outcomes include:

- Since the January 2015 workshop, consortium participants have made publicly available data from 11 technologies for two father-mother-son trios from the Personal Genome Project that are candidate NIST Reference Materials. Fifteen different groups presented methods for analysis of these data for all types and sizes of variants, and the Genome in a Bottle analysis team developed plans for accepting and integrating results from these and other groups.
- Consortium members considered possible categories of Genome in a Bottle reference material products, including circulating tumor DNA/cell-free DNA, a large set of samples with difficult mutations characterized in clinically important regions, and conducting an interlaboratory study of spike-in controls that simulate somatic mutations. Although these are important projects, the Genome in a Bottle steering committee decided that these projects are out of scope with present resourcing, and that the consortium's priority should be comprehensive characterization of whole human genomes, beginning with the seven samples presently under study for reference material development.
- The next Genome in a Bottle workshop will be held in January 2016 at Stanford University. The workshop will include use of Genome in a Bottle products for measurement process optimization, including evaluation/demonstration of instrument and method performance.

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NIST MML at the International Summit on Fibropapillomatosis for Sea Turtle Research

Fibropapillomatosis, or FP, is a disease that afflicts sea turtles across the globe, resulting in large benign skin tumors that can impede the turtles' sight, foraging, and movements. More than 50% of the animals in green sea turtle aggregations inhabiting certain regions, such as the main Hawaiian islands and Florida's Indian River lagoon, have FP. The disease rates in these two areas increased during the 1900s. In Hawaii, the disease rates stabilized or began declining in the mid-1990s. The cause is likely a herpes virus, but researchers have speculated with limited scientific evidence that environmental pollution may contribute to the etiology of this disease.

Researchers representing six large geographical regions (Florida and the Southeast U.S., Caribbean, Brazil, Congo/West Africa, IndoPacific/Australia, and Hawaii) convened at the International Summit on Fibropapillomatosis, which was hosted by NOAA's Pacific Islands Fisheries Science Center at the Daniel K. Inouye Regional Center on Ford Island in Pearl Harbor, Honolulu, Hawaii, in June 2015. Their primary goal was to evaluate geographic and temporal trend data while also identifying data gaps, exchanging ideas, and recommending standardized methodology for monitoring FP.

Jennifer Lynch from NIST MML's Chemical Sciences Division gave an introductory presentation on the role that environmental pollution may play in this disease. Dr. Lynch has been studying contaminant exposure in sea turtles since 1998 and currently works for NIST's Marine Environmental Specimen Bank from a facility at Hawaii Pacific University in Kaneohe, Hawaii.

Hawaiian green sea turtle with fibropapilloma tumors on eyes and neck.
Credit: Jennifer Lynch, NIST

Dr. Lynch's presentation discussed the three-prong rationale behind the hypothesis that environmental pollutants may play a role in FP. First, FP rates are higher in habitats with degraded environmental quality, especially those with increased nutrient runoff. Second, many environmental pollutants are carcinogenic or have the ability to promote tumors through mechanisms like immunosuppression. Moreover, turtles with tumors, especially those at later, severe stages, show signs of suppressed immune systems. Dr. Lynch stated, "It is rational to suspect exogenous chemicals that are tumor promoting or immune suppressing could contribute to FP, whether they are man-made pollutants or natural products." Testing this hypothesis, however, is challenging. Where do we begin? There are over 35,000 man-made chemicals in high-volume production in the U.S. and many more natural products. Only six published scientific studies have attempted to address this hypothesis. Three of them, including one by Dr. Lynch published in 2014 in Environmental Science and Technology, provided evidence that man-made organic pollutants, like polychlorinated biphenyls and pesticides, surprisingly do not contribute to the disease onset. Natural chemicals made from organisms living in the marine environment were examined in the remaining three studies. Okadaic acid and lyngyatoxin A are known tumor promotors and are produced by marine dinoflagellates or cyanobacteria that grow on the algae that green sea turtles eat. Exposure to both chemicals was linked to higher FP rates. While several man-made chemicals have been ruled out as contributors to FP, many more pollutant classes need to be investigated. Moreover, the preliminary evidence that biotoxins or nutrient pollution could play a role warrants further studies.

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USBR-NIST Research Symposium on Opportunities in Water Infrastructure, and Water Management and Measurement

NIST MML co-hosted an exploratory research symposium with the United States Bureau of Reclamation (USBR) on August 4-5, 2015 in Colorado. The symposium focused on opportunities in water infrastructure, and water management and measurement. The 25 attendees also included guests from the National Oceanic and Atmospheric Administration and the Federal Highway Administration.

USBR is responsible for water-based infrastructure and supply in the western U.S., and NIST provides measurement science in support of standards for a wide range of technology areas. The symposium was initiated to facilitate information exchange on mission-critical research projects in two topic areas: infrastructure sustainability, and water management and measurement.

The charge of the workshop was to identify initial, high-impact focus areas of mutual interest. Participants from USBR and from MML and two other NIST labs presented technical briefs that included program overviews, summaries of specific water-related challenges, and synopses of technical capabilities. Focus areas were identified, discussed, and prioritized in discussion sessions following the briefings.

Immediate next steps include the completion and dissemination of the workshop report, and the initiation of planning discussions on high-priority focus areas.

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NIST Partners with the Bureau of Reclamation in Water Prize Competitions

NIST has recently begun collaborating with the U.S. Bureau of Reclamation (USBR), which was awarded $2 million by Congress in 2014 under the America COMPETES Act to provide prize competitions to promote innovations in science and technology that can solve our nation's most critical water problems. USBR is partnering with NIST, EPA, NASA, NOAA, USGS, U.S. Fish and Wildlife Service, and the U.S. Army Corp of Engineers to prioritize problems for the challenge competitions and to judge the solutions that are submitted. USBR will be conducting 40 to 50 competitions over the next several years in the following three focus areas: water availability, ecosystem restoration, and infrastructure sustainability. (http://www.usbr.gov/research/challenges/).

MML scientists working in the three focus areas, along with a representative from the NIST Technology Partnerships Office, have participated in challenge competition training sessions sponsored by USBR, the first of which was held in Denver in May 2015. The scientists have been actively participating in discussions to identify the top problems that must be solved in water and that would benefit from the prize competition mechanism. The first challenge, "New Concepts for Remote Fish Detection," which falls under the ecosystem restoration focus area, was recently completed. Twenty-two white paper solutions were submitted by the solver community for this ideation challenge. Of those, four were awarded cash prizes ranging from a few thousand dollars up to $11,500 for the first place winning solution. Overall, a range of potential solutions was identified, and the judging team concluded that a combination of several different proposed ideas holds the best promise for a solution.

The second challenge is currently in development and will be in the area of water availability. Mary Bedner from MML has been participating on the development team for the challenge, which seeks to find solutions for the management of waste concentrate generated from the remediation of saline and brackish waters.

This new partnership has two major benefits: it establishes collaborative efforts with other government agencies that seek to ensure sustainability of our nation's water resources, and it helps to identify measurement problems of common interest among the agencies that we can work on together.

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NIST MATERIAL MEASUREMENT LABORATORY

WATER MEASUREMENT SCIENCE

Overview

Scope

The NIST MML research in water provides measurements, standards, and data to ensure availability and sustainability of our water resources. MML supports accurate assessment of water quality, innovative water treatment technologies, and reliable water infrastructure.

Technical Focus Areas

Water Quality: Development of advanced measurement methods, reference materials, and reference data for the water measurement community to identify and measure current and emerging contaminants in water such as metals, nutrients, agricultural and industrial chemicals, disinfectants and disinfection byproducts, pharmaceuticals, antibiotics, and waterborne microbial pathogens.

Water Treatment: Development of systematic, quantitative measurements, methods, reference materials and reference data that enable manufacturers of water treatment materials, especially membranes, to improve material selection and design, for example structure-property-performance relationships for reverse osmosis membranes.

Infrastructure Renewal: Development of standard reference materials (SRMs) and data for assessing pipeline condition, enabling effective and reliable infrastructure renewal, preventing microbial-induced corrosion, and elucidating correlations between water quality and infrastructure performance.

Partners

U.S. Environmental Protection Agency
U.S. Geological Survey
National Oceanic and Atmospheric Administration
General Electric
Dow Water & Process Solutions
U.S. Department of Energy
U.S. Department of Transportation
U.S. Bureau of Reclamation
Institute of Marine and Environmental Technology (IMET)

Key Impacts

- High sales of NIST SRMs for inorganic contaminants in water to our key stakeholders including other agencies, universities, and industrial labs indicates a sustained demand for SRMs to support accurate water measurements.
- The NIST/EPA/NIH Mass Spectral Database is incorporated into the software provided by all major instrument manufacturers for GC-MS, ensuring widespread availability to spectra for organic contaminant identification.
- MML recently entered into CRADAs with two of the leading materials providers of reverse osmosis and nanofiltration membranes for water treatment.

Projects and Opportunities

Measurements and Standards for Contaminants in Water

Accurate measurements of contaminants in water is critical to ensure the usability and safety of our water supplies. NIST has developed Standard Reference Materials (SRMs) for validating or calibrating methods for inorganic contaminants including SRM 1640a, Trace Elements in Natural Water, SRM 1643f, Trace Elements in Water, and SRM 1641e, Mercury in Water.

MML is also interested in developing validated analytical methodology for characterizing the organic contaminants in water, which comprise a vast array of compounds including pesticides, herbicides, pharmaceuticals, and industrial chemicals. These methods will enable contaminant discovery in water sources with different contaminants profiles and facilitate the development of much-needed SRMs for organic contaminants in water.

Microbes also comprise a major class of potential contaminants in water, and many serious illnesses such as Legionnaire's disease have been linked to waterborne pathogens. MML is expanding its capabilities in genomics-and metabolomics-based characterization of microbiomes, which will be used to assess microbial populations in water.

Ultimately, MML's rigorous methods for inorganics, organics, and microbes will be used to provide the measurement foundation to support sensor validation for the water monitoring community.

Projects

Data to Support Water Measurements

MML provides Standard Reference Data (SRD) to support the water measurement community. The most notable collections are the NIST/EPA/NIH Mass Spectral Database and the EPA Tandem Mass Spectrometry Library, which comprise spectra to assist with the identification of organic contaminants in water. Fundamental thermophysical water data are also provided in other SRD collections, including the NIST Reference Fluid Thermodynamic and Transport Properties Database (REFPROP) and the NIST/ASME Steam Properties Database.

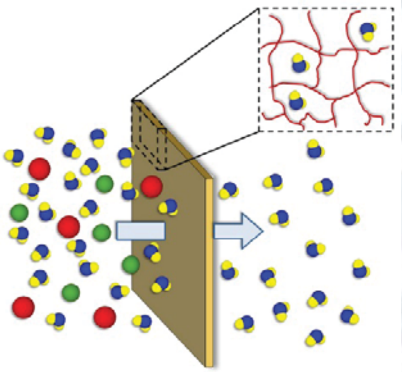
Water Prize Competitions

In addition to NIST, there are many agencies and organizations that are concerned about the sustainability of our water resources. However, even with vast research efforts in water, there are still numerous problems for which solutions remain elusive. To help find the required solutions, various water prize challenge competitions have been established to crowdsource ideas and technologies from across the globe. MML staff have been participating as subject matter experts on many water prize teams, including those offered by the USBR (http://www.usbr.gov/research/challenges/) and the Alliance for Coastal Technologies (http://www.act-us.info/nutrients-challenge/index.php).

Standards and Technology for Water Treatment

MML develops standards and technology that support the development of energy-efficient treatment technologies for water. One active area of research is the use of atomic layer deposition to tune the thickness and roughness of model, thin film composite polymer membranes. The model membranes are then well-characterized for interactions such as ion and solution diffusion and swelling. Industry can use the model membranes to design membranes in a targeted fashion to remove specific contaminants, particularly ions as part of desalination processes.

A second area of interest to MML is the development of measurement science, standards and technology for the intelligent design of sorbents for water treatment. The first step will be the determination of surface complexation models based on equilibrium constants for contaminants and adsorbent surfaces such as mineral phase, flocculants, hydrogels, and polymeric materials.



Capabilities for Infrastructure

MML has existing measurement capabilities that can be applied to assure the reliability of water pipelines and other components of our water infrastructure. These capabilities include standards for validation and calibration of fatigue detection equipment, measurement science to understand chemical and microbial corrosion processes in pipes, and computational materials science to determine properties of cement-based infrastructure materials.

Nutrient Sensor Challenge

Learn More

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PUBLICATIONS

“Tailoring the Permselectivity of Water Desalination Membranes via Nanoparticle Assembly,” Chan, EP; Mulhearn, WD; Huang, YR; Lee, JH; Lee, D ; Stafford, CM. LANGMUIR, 30, 611-616 (2014).

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NIST Calculates High Cost of Hydrogen Pipelines, Shows How to Reduce It

Note: A version of this story previously appeared in NIST's TechBeat on July 20, 2015.

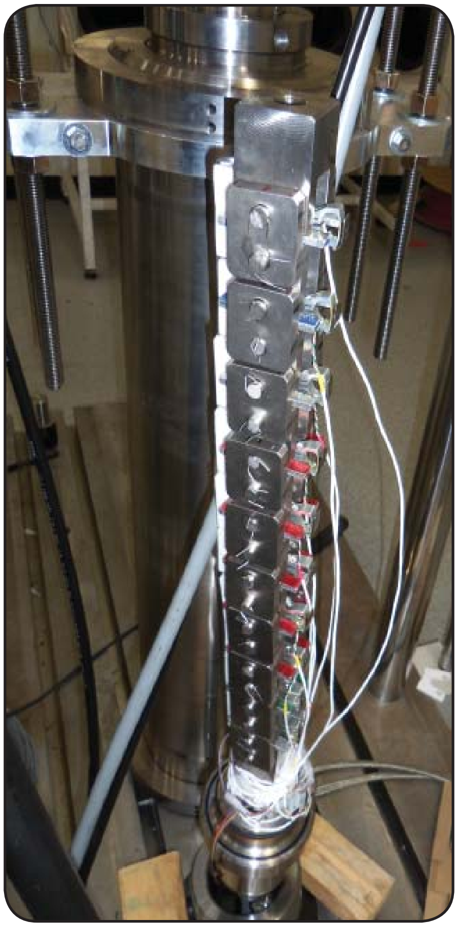
NIST MML has put firm numbers on the high costs of installing pipelines to transport hydrogen fuel—and also found a way to reduce those costs.

Pipelines to carry hydrogen cost more than other gas pipelines because of the measures required to combat the damage hydrogen does to steel's mechanical properties over time. NIST researchers calculated that hydrogen-specific steel pipelines can cost as much as 68 percent more than natural gas pipelines, depending on pipe diameter and operating pressure.[1] By contrast, a widely used cost model[2] suggests a cost penalty of only about 10%.

But the good news, according to the new NIST study, is that hydrogen transport costs could be reduced for most pipeline sizes and pressures by modifying industry codes[3] to allow the use of a higher-strength grade of steel alloy without requiring thicker pipe walls. The stronger steel is more expensive, but dropping the requirement for thicker walls would reduce materials use and related welding and labor costs, resulting in a net cost reduction. The code modifications, which NIST has proposed to the American Society of Mechanical Engineers (ASME), would not lower pipeline performance or safety, the NIST authors say.

"The cost savings comes from using less—because of thinner walls—of the more expensive material," says NIST materials scientist James Fekete, a co-author of the study. "The current code does not allow you to reduce thickness when using higher-strength material, so costs would increase. With the proposed code, in most cases, you can get a net savings with a thinner pipe wall, because the net reduction in material exceeds the higher cost per unit weight."

The NIST study is part of a federal effort to reduce the overall costs of hydrogen fuel, which is renewable, nontoxic and produces no harmful emissions. Much of the cost is for distribution, which likely would be most economical by pipeline. The U.S. contains more than 300,000 miles of pipelines for natural gas but very little customized for hydrogen. Existing codes for hydrogen pipelines are based on decades-old data. NIST researchers are studying hydrogen's effects on steel to find ways to reduce pipeline costs without compromising safety or performance.



Samples of pipeline steel instrumented for fatigue testing in a pressurized hydrogen chamber (the vertical tube). NIST researchers used data from such tests to develop a model for hydrogen effects on pipeline lifetime, to support a federal effort to reduce overall costs of hydrogen fuel. Credit: NIST

As an example, the new code would allow a 24-inch pipe made of high-strength X70 steel to be manufactured with a thickness of 0.375 inches for transporting hydrogen gas at 1500 pounds per square inch (psi). (In line with industry practice, ASME pipeline standards are expressed in customary units.) According to the new NIST study, this would reduce costs by 31 percent compared to the baseline X52 steel with a thickness of 0.562 inches, as required by the current code. In addition, thanks to its higher strength, X70 would make it possible to safely transport hydrogen through bigger pipelines at higher pressure (36-inch diameter pipe to transport hydrogen at 1500 psi) than is allowed with X52, enabling transport and storage of greater fuel volumes. This diameter-pressure combination is not possible under the current code.

The proposed code modifications were developed through research into the fatigue properties of high-strength steel at NIST's Hydrogen Pipeline Material Testing Facility. In actual use, pipelines are subjected to cycles of pressurization at stresses far below the failure point, but high enough to result in fatigue damage. Unfortunately, it is difficult and expensive to determine steel fatigue properties in pressurized hydrogen. As a result, industry has historically used tension testing data as the basis for pipeline design, and higher-strength steels lose ductility in such tests in pressurized hydrogen. But this type of testing, which involves steadily increasing stress to the failure point, does not predict fatigue performance in hydrogen pipeline materials, Fekete says.

NIST research has shown that under realistic conditions, steel alloys with higher strengths (such as X70) do not have higher fatigue crack growth rates than lower grades (X52). The data have been used to develop a model[4] for hydrogen effects on pipeline steel fatigue crack growth, which can predict pipeline lifetime based on operating conditions.

The studies at NIST's hydrogen test facility were supported by the Department of Energy and Department of Transportation.

- Laura Ost, NIST

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1. J.W. Sowards, J.R. Fekete and R.L. Amaro. Economic impact of applying high strength steels in hydrogen gas pipelines. International Journal of Hydrogen Energy. 2015. In press, corrected proof available online. DOI:10.1016/j.ijhydene.2015.06.090

2. DOE H2A Delivery Analysis. U.S. Department of Energy. Available online at www.hydrogen.energy.gov/h2a_delivery.html.

3. ASME B31.12 Hydrogen Piping and Pipeline Code (ASME B31.12). Industry groups such as ASME commonly rely on NIST data in developing codes.

4. R.L. Amaro, N. Rustagi, K.O. Findley, E.S. Drexler and A.J. Slifka. Modeling the fatigue crack growth of X100 pipeline steel in gaseous hydrogen. Int. J. Fatigue, 59 (2014). pp 262-271.

New Hydrogen Storage Formulation Patented

Note: A version of this story previously appeared on NIST's Physical Measurement Laboratory website (http://www.nist.gov/pml) on September 12, 2015.

The prospect of a "hydrogen economy"--in which vehicles powered by fuel cells travel the nation's roadways emitting nothing from their tailpipes but wisps of water vapor--was making headlines 12 years ago.

In his 2003 State of the Union address, President George W. Bush issued a challenge to the nation's scientists and engineers to overcome technical obstacles so that "the first car driven by a child born today could be powered by hydrogen, and pollution free."

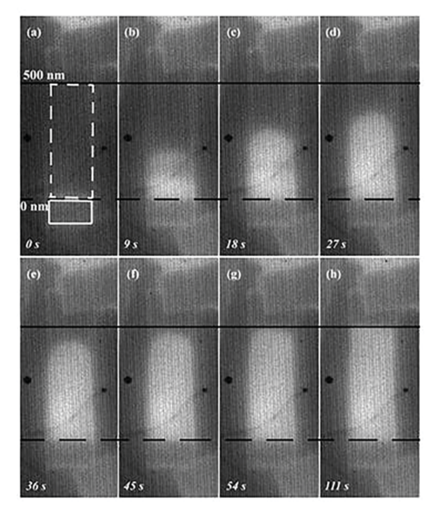
Today, that goal still remains a long way from realization because of numerous technical obstacles. One of the most persistently difficult obstacles is the question of how to store hydrogen on board a vehicle.

Current options for carrying enough hydrogen for about 300 miles of driving (5–13 kg for light-duty vehicles, according to the Department of Energy, DOE) include compressing the gas to thousands of pounds per square inch, cooling it to a liquid state, or releasing it from the surfaces of porous materials that have absorbed hydrogen. The last option has obvious appeal, but typically requires long time intervals and very high temperatures to liberate the gas from its bound state.

A team of NIST scientists that worked on this problem for years has now been issued a patent for a novel alloy formulation that can eliminate many of the drawbacks of other solid-storage materials, permit rapid adsorption/desorption of hydrogen gas, and operate at desirable temperatures. As part of NIST's hydrogen research, a team was formed a decade ago as a collaboration between the Material Measurement Laboratory and Physical Measurement Laboratory. The team focused on magnesium, a preferred material in this line of research because it is lightweight and can adsorb enough hydrogen to constitute 7.6% of its combined mass. Magnesium alone, however, is slow to take up hydrogen, so numerous experiments have explored how to improve performance by mixing it with other metals such as nickel and titanium.

The team conducted multiple comparison tests of different formulations, including magnesium and iron. Iron had not appeared particularly promising in much of the scientific literature, but there had been some indications that it could both increase the adsorption rate and perhaps reduce the temperature necessary for desorption.

"The main idea of our group," said project leader Leonid Bendersky, "was to create magnesium-based material that constitutes two components--the fast hydrogen diffuser and fine-size hydrogen absorber--which together will provide the needed charge/discharge kinetics. Utilizing thermodynamics of the magnesium-iron system, the properties of magnesium and iron, and phase separation during e-beam deposition, the desired structure was created and successfully tested for its hydrogenation properties."



Individual frames from time-resolved IR emission-measurement images for a magnesium-iron thin film in which 4 % of the atoms are iron, exposed to hydrogen gas at 100 ºC. White areas show accumulation of hydrogen over about 2 minutes at time intervals in seconds. The film is almost completely hydrogenated within 1 minute.

In 2008, Edwin Heilweil, who had experience in making the kinds of sophisticated infrared emission imaging measurements needed to track the speed and adsorption/desorption efficiency of several candidate materials, joined the team.

This approach has the advantage of rapidly screening two-dimensional combinatorial libraries for best performance by monitoring formation of hydrides in purposely grown, variable-composition films thinner than 1 micrometer (a millionth of a meter).

"Infrared imaging is extremely useful because of the telltale thermodynamic reaction involved," Heilweil said. "As hydrogen is adsorbed onto the magnesium, there is change of infrared reflectivity as the material changes from metallic to non- or semi-conductive. Looking at the sample with an infrared camera tells you how much hydrogen is being adsorbed."

When the team tested magnesium-iron thin films (about half a micrometer thick), they found that a combination in which iron makes up about 4% of the atoms in the storage material had remarkable properties: At normal atmospheric pressure, it could become fully charged with hydrogen in one minute at temperatures around 100 ˚C, and discharged at 150 ˚C.

The group pursued a patent in September of 2012. The patent was issued in June 2015.

The performance of the NIST formulation does not meet, but is not terribly far from, the DOE targets for hydrogen storage systems. The 2020 targets call for systems that can be fully filled to 5 kg of hydrogen in 3.3 minutes and operate at temperatures in the range of 40 ˚C (or up to 60 ˚C with full exposure to direct sunlight).

NIST's patented technology may make that goal more reachable.

- Curt Suplee, NIST PML

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Newly Commercialized Process Makes Better Material for Hydrogen in Titanium SRMs

Recently, NIST issued SRM 2453a Hydrogen in Titanium in pin form in support of titanium manufacturers and their customers in the aerospace, defense, and power generation industries. The material for SRM 2453a was created using a newly commercialized process based on one developed at NIST about 10 years ago. MML researchers Richard Lindstrom and Rick Paul developed a procedure for controlled dosing of known amounts of hydrogen chemically bound into the titanium alloy matrix to ensure long term stability. They used the process to prepare materials for three new SRMs, tailoring the amounts of hydrogen to match specification limits on the maximum amounts of hydrogen allowed in titanium alloys for aerospace and power generation applications. As planning began for replacements of SRMs 2452, 2453, and 2454, technical project leader John Sieber presented the project at a meeting of ASTM International Committee E01 on Analytical Chemistry of Metals, Ores and Related Materials. Subsequently, a small business owner who was developing a commercial version of the NIST process in an effort to launch a company to develop and sell certified reference materials, contacted Dr. Sieber. NIST contracted with the company and, within a year, the resulting batch of titanium pins became available as NIST SRM 2453a

The contract with the small business called for titanium alloy with a known mass fraction of total hydrogen with very low heterogeneity across the batch, plus a uniform size and mass per piece of titanium. Wire titanium alloy was chopped into pins of highly uniform mass and dimensions to make the SRM easier to use with ASTM E1447 Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method. The contractor was required to prove by analysis that the pins contained the required amount of hydrogen with the required low composition variance. Hydrogen analyses were performed after calibration of method E1447 using a special gas–dosing approach, which served two purposes. First, the calibration parameters were traceable to the mole through the use of known volumes of ultrahigh-purity hydrogen gas. Second, the quantitative results were expected to provide data of sufficient quality for material variance characterization, for demonstration of short-term hydrogen stability, and for combination with results from NIST prompt gamma-ray activation analyses for certification of the mass fraction of total hydrogen.

A little more than one year after delivery of the titanium pins, NIST issued SRM 2453a. The overall uncertainty of the certified value for H in SRM 2453a is half the uncertainty of the value for SRM 2453. This improvement will enable commercial reference materials producers and manufacturing laboratories to obtain lower uncertainties of their certified values and certified product results, respectively. The titanium-pin producing small business is continuing efforts to manufacture and to market a new line of CRMs.

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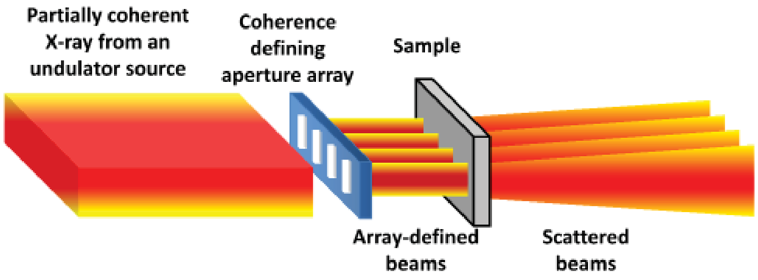
NIST MML Researchers Demonstrate Simultaneous/Multiplexed Materials Characterization Using Coherent X-ray Scattering



Schematic of coherent, multiplexed XPCS system based on ultra-small-angle scattering.

Working with NIST's partners at the Advanced Photon Source, Argonne National Laboratory, Argonne, IL, researchers in MML have successfully demonstrated simultaneous/multiplexed materials characterization using coherent X-ray scattering with a high-precision micro-slit array. This technique addresses major challenges in materials measurement science and structure determination: the need for both increased experimental throughput and for *in operando* characterization of heterogeneous materials under increasingly realistic experimental conditions.

To address this issue, MML researchers developed a novel measurement scheme that allows both simultaneous and multiplexed measurements from multiple nearby sample volumes. In this scheme, hard X-ray micro-slit arrays were fabricated by exploiting the sub-micrometer precision of photolithography. X-rays passing through each opening in the slit array serve as an independent X-ray probe. By making use of multiple probes simultaneously, this new approach enables better counting statistics in measurements of homogenous materials or the direct probing of constituent components in heterogeneous structures, dynamics, or elemental composition.

To demonstrate the possibilities, a coherent, multiplexed form of X-ray photon correlation spectroscopy based on ultra-small-angle scattering (XPCS) was applied to follow the equilibrium dynamics of simple colloidal suspensions; specifically, variations of colloidal motion relaxation times on length scale, and their relationships with diffusion constants and static structures. Through this approach, the uncertainties associated with the photon-starved XPCS technique were reduced to below those associated with the instrument resolution. More importantly, the multiplexed scheme has the potential to elucidate the dynamics of different components within a heterogeneous sample under identical experimental conditions.

This scheme is, in principle, applicable to all in-line synchrotron techniques. Indeed, it has the potential to open a new paradigm for *in operando* characterization of heterogeneous functional materials, a situation that will be further enhanced by the ongoing development of so-called multi-bend achromat based diffraction-limited storage ring designs as the next evolution (fourth generation) of large-scale X-ray synchrotron facilities around the world.

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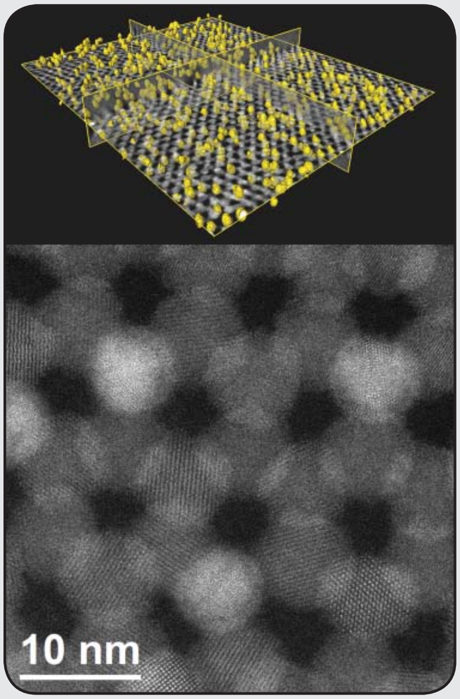
F. Zhang, A.J. Allen, L.E. Levine, D.C. Mancini, J. Ilavsky; "Simultaneous multiplexed materials characterization using a high-precision hard X-ray micro-slit array," J. Synchrotron Rad., 22, 653-660 (2015).

Doping Nanoparticle Superlattices

NIST MML researchers, collaborating with colleagues at the University of Pennsylvania, have helped to demonstrate successful substitutional doping of nanoparticle superlattices. The electrical conductivity of the superlattices is tunable over six orders of magnitude as the concentration of dopants is varied. This was the first time that substitutional doping was demonstrated to manipulate the properties of a nanoparticle superlattice.

Just as atomic doping has enabled engineering of properties in materials, this approach shows promise for control of electrical, magnetic, optical, and structural properties of nanoparticle arrays. In conjunction with controlling the size, shape, and composition of the host matrix nanoparticles, it may now be possible to produce nanoparticle superlattices with new properties.

Monodisperse nanoparticles will self-assemble into structures with periodic arrangements, much like atoms in crystalline material. In the latter case, it is well known that the addition of foreign atoms into a host's atomic lattice can significantly alter its properties. For example, the addition of boron to silicon significantly changes its electrical properties by modulating the carrier concentration. Drawing inspiration from this approach, gold nanoparticles acted as substitutional dopants in superlattices of cadmium selenide and lead selenide nanoparticles. Precisely controlling the size and surface chemistry of both the host and dopant nanoparticles was critical to prevent segregation of the dopant nanoparticles. To demonstrate that the dopant nanoparticles were incorporated randomly into the host matrix, high-angle annular dark field scanning transmission electron microscopy was used. Images produced by this technique are sensitive to composition, allowing for unambiguous identification of the dopant nanoparticles. However, these images are a two-dimensional projection of the structure and information about the spatial distribution of the dopant nanoparticles is lost in the third dimension. By acquiring a series of images while tilting the sample, a three-dimensional reconstruction of the superlattice with nanometer spatial resolution can be generated. This provided the necessary proof to establish that the dopants were truly incorporated into the host lattice rather than segregating.



A tomographic reconstruction (top) of the nanoparticle superlattice indicates the gold dopants – colored in yellow – are dispersed throughout the volume. High angle annular-dark field scanning transmission electron microscopy images (bottom) provides the necessary contrast to distinguish the gold nanoparticles, which appear brighter, from the matrix of lead selenide nanoparticles.

Credit: Aaron C. Johnston-Peck

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M. Cargnello, A.C. Johnston-Peck, B.T. Diroll, E. Wong, B. Datta, D. Damodhar, V.V.T. Doan-Nguyen, A.A. Herzing, C.R. Kagan, C.B. Murray; "Substitutional doping in nanocrystals superlattices," Nature., 524, 450-453 (2015).

NIST's Changes to the ISO Rockwell Hardness Test Method Standard Increase Availability of Calibration-Grade Hardness Indenters

Credit: Sam Low, NIST

A Rockwell hardness diamond indenter positioned to make a test in the polished surface of a steel test-block standard.

Based on NIST recommendations, the ISO standard entitled "Metallic materials—Rockwell hardness test—Part 3: Calibration of reference blocks" was recently published with significant changes on the microform geometrical specification of Rockwell diamond indenters. NIST showed that past geometrical tolerances for calibration-grade diamond indenters were overly tight, providing no benefit to the measurement of hardness, and preventing obtainment of sufficient indenters that could meet the geometrical requirements of the standard. The overly tight tolerances have caused a scarcity of these indenters throughout the world. In cooperation with the foremost diamond indenter manufacturers in the U.S. and internationally, NIST received and calibrated more than 70 Rockwell diamond indenters. Based on years of standardized Rockwell hardness and microform calibrations, and finite element simulations, NIST researchers recommended relaxing the tolerance range for cone flank straightness from "0.5 µm for the maximum deviation" to "0.5 µm for the average deviation and 0.7 µm for maximum deviation," and revising the measurement process for the geometrical calibration of Rockwell indenters. NIST researchers continually presented their research results to the Working Group on Hardness of the Consultative Committee for Mass and Related Quantities and the ISO TC 164 committee on hardness testing, as well as through conference presentations and journal publications. The NIST recommendations have now been adopted by the ISO 6508-3 standard newly issued in 2015, which should greatly improve the availability of qualified indenters without jeopardizing measurement quality. Researchers at NIST are now working on microform calibrations and hardness performance tests, and considering the initiation of a new project to support U.S. manufacturers: development of SRM Rockwell diamond indenters in conformance with the revised ISO standard.

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CHiMaD Workshop: Multivalent Interactions in Polyelectrolytes: New Physics, Biology and Materials

Credit: David Goldfeld

On October 2-4, 2015 the Center for Hierarchical Materials Design (CHiMaD), a NIST-awarded Center of Excellence, held an international scientific symposium at the University of Chicago's Institute for Molecular Engineering. As a center of excellence for advanced materials research, CHiMaD is focused on developing the next generation of computational tools, databases and experimental techniques in order to enable the accelerated design of novel materials and their integration to industry, one of the primary goals of the Obama administration's Materials Genome Initiative. This workshop, featuring more than 30 internationally recognized speakers, focused on physical, biological, and materials science opportunities enabled by highly charged macromolecules interacting with multivalent species of the opposite charge. Polyelectrolytes and ion-containing polymers remain a challenge to understand due to the long-range electrostatic interactions and charge correlations. These materials and ion-mediated effects are ubiquitous from stabilizing additives and rheological modifiers in commodity products to advanced ion-containing batteries. Many properties of polyelectrolytes are due to the solvation by water that becomes important to include in advanced molecular dynamics models. Challenges in experiment, computation and theory were discussed. In particular, theoretical and computational efforts to understand charge correlation and chirality effects on polyelectrolyte coacervates and phase diagrams, effects of dielectric media and charge valence on chain conformation and gel structure, as well as ionic transport and diffusion properties in solution and thin films that are important to application in separation by electrophoresis and ion batteries.

The workshop benefitted from the objectives of the Materials Genome program at NIST. A common theme was the development of molecular to mesoscale models and computational methods to guide and understand new materials.

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A Novel Approach for Information Discovery to Support the Materials Genome Initiative

As part of a broad array of so-called "natural language" approaches to categorizing information, a team in MML is working to build the terminology infrastructure for the Materials Genome Initiative (MGI), a multi-agency initiative designed to accelerate discovery, development, and deployment of advanced materials. Current terminology used by the MGI community is *ad hoc* and heterogeneous. To help accelerate the discovery and integration of materials data for improved design of advanced materials, MML scientists have constructed a "root" and rule-based approach that will help the community build re-useable, extensible and automation-friendly terminology to describe MGI data in an intuitive way.[1]

After reviewing many possibilities, the team adopted some of the root and rule-based concepts used by a few Indo-European languages, such as Sanskrit, Latin and German, to build MGI terminology using English words. Unlike spoken or written linguistic terminology, MGI terminology is expected to be both human and machine friendly. For this reason, the team gave special consideration for the requirements of text-mining techniques such as natural language programming, datagraphs and databases. The proposed MGI terminology effort takes advantage of our past experience in developing ChemBLAST (terminology for chemical structures) and cell image terminology (cell image data).

The project focused on producing terms that describe material properties. The terminology then could be used by database developers to develop user-friendly web interfaces to archive and distribute MGI data. The community may use these databases to get consolidated information about their product related questions which will accelerate the time to market for new products.

The next major challenge is to develop software tools that create and validate MGI terminology in a semi-automated fashion. These tools will allow the creation of index terms from tens of thousands of MGI related research papers which can serve as a user-friendly data resource of the information contained in those publications.

Contact: TN Bhat, talapady.bhat@nist.gov

1. TN Bhat, L. Bartolo, U. Kattner, C. Campbell and J. Elliott, "Strategy for Extensible, Evolving Terminology for the Material Genome Initiative Efforts", JOM, online July 2015.

Outreach and Partnering

New Agreement between PTB and NIST to Facilitate Soft X-Ray Metrology

An "annex" to the memorandum of understanding defining cooperation between the Physikalisch-Technische Bundesanstalt (PTB), the German standards agency, and NIST was recently approved and inaugurated to facilitate increased cooperation in the area of X-ray metrology. The agreement outlines a cooperative program that makes use of the PTB U49 Metrology Beamline at the synchrotron light source BESSY II, located in Berlin. PTB researchers use the beamline for a variety of programs such as the refinement of the Avogadro number and total reflection X-ray fluorescence for the characterization of thin films and silicon wafers. The new agreement adds cooperation in soft X-ray spectroscopy to the list. Inaugural experiments centered on measurements of X-ray spectra from chemical compounds of nitrogen and carbon and comparisons with theoretical calculations of their spectra. X-ray emission spectra are widely used in the microanalysis of unknown elements during examination of samples by electron microscopy. An increased understanding and computational capability will extend this type of analysis to identification of elements as well as specific chemical compounds. The PTB Metrology Beamline is a state-of-the-art source of extremely intense X-rays tunable to precise energies that irradiates a sample, combined with a spectrometer to analyze the X-rays emitted by the sample. The annex agreement promotes collaborative experiments using the beamline, facilitates the shipment of samples, proposes the short-term exchange of guest workers, enables the temporary exchange of equipment, and explores the involvement of NIST as a co-participant in projects on X-ray fundamental parameters involving multiple European standards laboratories. *Contact: Terrence Jach, terrence.jach@nist.gov*

Third International Workshop on Advanced Cobalt Base Superalloys

The third international Workshop on Advanced Cobalt Base Superalloys workshop was held June 23-24, 2015 and was attended by thirty-five researchers from industry, government laboratories, and universities. The workshop facilitated growing collaborations between experimentalists and modelers to apply integrated computational materials and engineering approaches to design this new class of superalloys. Advanced cobalt-base superalloys using a γ/γ' based microstructure have the potential to increase the operating temperatures of turbine engines by fifty degrees, thus increasing engine efficiency and reducing emissions. To achieve these benefits, researchers are working to develop cobalt-base γ/γ' alloys that have higher melting and γ' solvus temperatures than the currently used nickle-based γ/γ' superalloys. The workshop highlighted developments in determining the multicomponent phase equilibria, improvement in thermodynamic descriptions, mechanical and high temperature oxidation properties, and commercial applications. *Contact: Eric Lass, eric.lass@nist.gov*

NIST MML Hosts 19th Symposium on Thermophysical Properties

NIST MML staff in Boulder, CO recently hosted the 19th Symposium on Thermophysical Properties, held at the University of Colorado at Boulder June 21- June 26, 2015, and organized by NIST and the Joint ASME-AIChE Committee on Thermophysical Properties. Over 600 talks were presented, representing speakers from 50 countries covering six continents, and comprising a mix of industry, academia, government, and standards organizations. The triennial Symposium is the world's largest conference devoted to thermophysics. Dr. Michael Knotek, Deputy Under Secretary, Office of the Under Secretary for Science and Energy, U.S. Department of Energy, gave the keynote talk, "Lessons from the 2015 DOE Quadrennial Technology Review: The Role of Research and Development in our Energy Future." *Contact: Marcia Huber, marcia.huber@nist.gov*

NIST MML Workshop Assesses Emerging Scanning Electron Microscopy Techniques

On September 15-16, 2015, NIST MML hosted the Workshop on Analytical Transmission Scanning Electron Microscopy (tSEM) in Boulder, Colorado. The meeting featured an international group of about 40 industrial, academic, and national laboratory experts, who assessed present and future challenges and opportunities associated with the recent emergence of transmission approaches to performing scanning electron microscopy (SEM), which is a historically reflection-based measurement platform. Specific areas of instrumental and analytical R & D, as well as standardization, were identified as key to transforming these novel measurement techniques from curiosities into meaningful quantitative measurement methods. The techniques are particularly well-suited to characterization of materials that are confined to extremely small volumes, with dimensions in the range single to tens of nanometers, as well as materials composed of low atomic number elements such as carbon. tSEM is enabling the ubiquitous SEM to act as a significantly more affordable transmission electron microscopy (TEM)-like instrument, and enable screening of specimens for suitability prior to use in the more complex and specialized TEM instrument. The technique also has a unique ability to mitigate electron beam damage in sensitive materials, while creating images with sub-nanometer resolution not possible with conventional SEM. Areas where early forms of transmission SEM have begun to create impact include nanomanufacturing, semiconductors, biomedicine, geology, biology, and structural materials development. The meeting featured dialogue among attendees regarding future forms of interaction, including the formation of a consortium under a CRADA for structured collaboration in the area of transmission SEM. NIST will lead organization of both the next Workshop (targeting 2017) and a symposium at an upcoming Microscopy and Microanalysis Conference (also 2017, in St. Louis). *Contact: Bob Keller, bob.keller@nist.gov*

NIST MML Participates in Spring 2015 DIR-AG Meeting

NIST MML scientific advisor Mary Bedner recently represented NIST at the spring 2015 Meeting of the Directors of Industrial Research Analytical Group (DIR-AG), which was held June 10-12, 2015 in Cuyahoga Falls, Ohio. The DIR-AG is comprised of directors, managers, and team leaders of R&D analytical groups in major manufacturing companies, and meets three times a year. NIST is uniquely positioned as the only government member of the DIR-AG and participates regularly in meetings to foster and maintain relationships with key industry stakeholders. Avery-Denison, Lubrizol, and Sherwin Williams co-hosted the spring meeting, and representatives from Alcoa, BASF, Boeing, Clorox, Corning, Eastman Chemical, Eli Lilley, Milliken, Pepsi-Co, PPG, Sealed Air, and Stepan also participated. Many topics were discussed at the meeting, including partnering, fostering innovation, succession planning, and recruiting talent. The ensuing discussions were insightful, and the topics resonated with goals in the new MML Strategic Plan. Lubrizol, a chemical manufacturing company that specializes in additives, advanced materials, and oilfield solutions, also provided a tour of their analytical labs at their headquarters in Wickliffe, Ohio. *Contact: Mary Bedner, mary.bedner@nist.gov*

NIST Yeast Prototype Reference Material to be Used in National Guard Training Exercise

A modified yeast material developed by MML researchers was recently used as a non-threat surrogate for National Guard and first responder training related to biothreat detection in the field. The yeast was evaluated as a part of Operation Vigilant Sample IV, a full-scale functional exercise designed to demonstrate a state response to a suspicious substance and a BioWatch event with subsequent surface sampling. The BioWatch Program is funded by the Department of Homeland Security (DHS) to monitor for pathogen threats in the air in selected metropolitan areas. The yeast cells were deposited onto and sampled from 2 inch by 2 inch pieces of stainless steel that were placed at predetermined locations throughout the training area. Collected samples were analyzed by National Guard Civil Support Teams and CDC Laboratory Research Network locations. The development of a yeast reference material is supported by the DHS Science and Technology Directorate. *Contacts: Nancy Lin, nancy.lin@nist.gov; Sandra Da Silva, sandra.dasilva@nist.gov*

Selected Recent Publications

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

T. P. Forbes, E. R. Sisco, "In-source collision induced dissociation of inorganic explosives for mass spectrometric signature detection and chemical imaging" Analytica Chimica ACTA, Vol. 892, pp. 1-9, (23-Sep-2015) (PubID: 917855)

E. R. Sisco, J. L. Staymates, K. Schilling, J. G. Gillen, "Development of a Chemically Relevant Artificial Fingerprint Material" Forensic Science International, 15 pp., (16-Sep-2015) (PubID: 914626)

A. C. Johnston-Peck, M. Cargnello, B. T. Diroll, E. Wong, B. Datta, D. Damodar, V. V. Doan-Nguyen, A. A. Herzing, C. R. Kagan, C. B. Murray, "Substitutional Doping in Nanocrystal Superlattices" Nature, Vol. 524, pp. 450-453, (27-Aug-2015) (PubID: 918309)

L.F. Vargas Lara, J. F. Douglas, S. M. Stavis, E. A. Strychalski, J. C. Geist, B. J. Nablo, "Dimensional Reduction of Duplex DNA Under Nanofluidic Slit Confinement" Nanoscale , 12 pp., (19-Aug-2015) (PubID: 917678)

A. R. Montoro Bustos, E. J. Petersen, A. M. Possolo, M. R. Winchester, "Post hoc Interlaboratory Comparison of Single Particle ICP-MS Size Measurements of NIST Gold Nanoparticle Reference Materials" Analytical Chemistry, (12-Aug-2015) (PubID: 918083)

B. G. Bush, J. M. Shapiro, F. W. DelRio, R. F. Cook, M. L. Oyen, "Heterogeneity and length scale effects in PEG-based hydrogels" Soft Matter, Vol. 11, pp. 7191-7200, (10-Aug-2015) (PubID: 917666)

P. Jaruga, E. Coskun, M.M. Dizdar, N. Donley, S. Lloyd, A. McCullough, "Small Molecule Inhibitors of 8-Oxoguanine DNA Glycosylase-1 (OGG1)" ACS Chemical Biology, Vol. 10, pp. 2334-2343, (07-Aug-2015) (PubID: 918829)

S. R. Kalidindi, J. Gomberg, Z. T. Trautt, C. A. Becker, "Application of Data Science Tools to Quantify and Distinguish between Structures and Models in Molecular Dynamics Datasets" Nanotechnology, (03-Aug-2015) (PubID: 918634)

A. C. Chiaramonti Debay, B. Ozturk, A. de Luna Bugallo, E. Panaitescu, F. Liu, A. Vargas, X. Jiang, O. Yavuzcetin, M. Alnaji, Y. Zhao, N. King, M. Dubey, S. Nayak, S. Sridhar, S. Kar, "Atomically thin layers of B-N-C-O with tuneable composition" Nano Letters, pp. 1-8, (31-Jul-2015) (PubID: 917427)

J. E. Seppala, Y. Heo, P. E. Stutzman, J. R. Sieber, C. R. Snyder, K. D. Rice, G. A. Holmes, "Characterization of clay composite ballistic witness materials" Journal of Materials Science, Vol. 50, No. 21, pp. 7048-7057, (29-Jul-2015) (PubID: 917583)

P. T. Reddy, P. Jaruga, B. C. Nelson, M. S. Lowenthal, A.S. Jemth, O. Loseva, E. Coskun, T. Helleday, M.M. Dizdar, "Production, Purification and Characterization of 15N-Labeled DNA Repair Proteins as Internal Standards for Mass Spectrometric Measurements" Methods In Enzymology, (26-Jul-2015) (PubID: 918645)

J. M. Zook, A. Gargis, L. Kalman, I. Lubin, M. L. Salit, "Optimization of Next-Generation Sequencing Informatics Pipelines for Clinical Laboratory Practice" Nature Biotechnology, Vol. 33, (08-Jul-2015) (PubID: 916037)

K. W. Anderson, I. V. Turko, N. Mast, I. A. Pikuleva, "Histone H3 Ser57 and Thr58 Phosphorylation in 5XFAD Mouse Brain" FEBS Open Bio, pp. 550-556, (20-Jun-2015) (PubID: 917627)

K. D. Migler, A. R. Hight Walker, A. P. Kotula, "Mesomorphic Pathway in Early Stage Crystallization of Polyethylene" Soft Matter, Vol. 48, No. 13, pp. 4555-4561, (18-Jun-2015) (PubID: 917498)

A. C. Chiaramonti Debay, L. D. Marks , S. Rahman, M. Castell, "Transition from order to configurational disorder for surface reconstructions on SrTiO 3(111)" Nature, Vol. 114, No. 22, pp. 226101-1-226101-6, (02-Jun-2015) (PubID: 915810)

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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