PROGRESS AND PLANS OF NATIONAL NANOTECHNOLOGY INITIATIVE (NNI) AGENCIES

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Summary

Advancing nanoscale measurement science, standards, and technology is an important component of NIST's mission to promote U.S. innovation and industrial competitiveness. From leading cutting-edge research, to providing world-class facilities, to coordinating the development of standards that promote trade, NIST's intramural nanotechnology research program directly impacts the Nation's economy and well-being. The nanotechnology research conducted in NIST's laboratories and user facilities results in measurements, standards, and data crucial to a wide range of industries and Federal agencies, from new measurement and fabrication methods for advanced manufacturing to reference materials and data needed to inform the responsible development and use of nanotechnology. NIST further supports the U.S. nanotechnology enterprise through its user facilities, including the NIST Center for Neutron Research (NCNR) and the Center for Nanoscale Science and Technology (CNST). NCNR provides access to a range of world-class neutron scattering tools for characterizing the atomic and nanometer-scale structure and dynamics of materials. CNST provides rapid access to state-of-the-art tools needed to make and measure nanostructures. NIST staff members contribute unique technical expertise and leadership in nanotechnology-related standards development and international cooperation activities such as the Organisation for Economic Co-operation and Development (OECD) Working Party on Manufactured Nanomaterials, the International Organization for Standardization (ISO) Technical Committee (TC) 229, the International Electrotechnical Commission Technical Committee 113, and ASTM International Committee E56. NIST's world-class expertise in measurement, characterization, and standards development helps ensure that the resulting international standards reflect the state of technology, are unbiased, and facilitate innovation, commercialization, and user confidence in the safety of nanotechnology. Interagency coordination and information sharing related to these activities is facilitated through the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council.

Plans and Priorities by Program Component Area (PCA)

PCA 1. Nanotechnology Signature Initiatives (NSIs) and Grand Challenges

1a. Nanomanufacturing NSI

Graphene and transition metal dichalcogenides (TMDs) are single atomic layers of crystalline materials that exhibit unusual electronic and quantum characteristics that hold promise for high-performance devices of the future. If these materials are to make a real impact in nanoelectronics and future computing, they must show positive manufacturability and reliability traits. This means they must be able to be fabricated into the structures needed for devices, they must display their intended physical properties of interest, and they must continue to provide those properties over the targeted lifetime of a device. Implicit to the fabrication, properties, and reliability of such materials is that all the correct atoms are in their correct places in the manufacturing and operational environments. In fiscal year (FY) 2020 NIST plans to develop testing techniques that allow the application of operational stresses such as temperature and mechanical forces to TMDs while they are observed in a scanning electron microscope. The microscope will enable researchers to determine if the TMD crystalline structure remains intact, and whether defects form and evolve with continued exposure to stresses. Such information will inform material fabrication and device design parameters.

1b. Nanoelectronics NSI

To improve production yields of electronic devices and facilitate production processes for next-generation ultra-thin films, e.g. two-dimensional (2D) materials, NIST will develop and apply *in situ* diagnostics and process control methods for chemical vapor and atomic layer deposition. Recent accomplishments, in collaboration with commercial partners, include the investigation of controlled metal precursor delivery, specifically cobalt and tantalum for cobalt metal and tantalum nitride films, and the study of precursors and deposition chemistry for metal oxide and 2D transition metal dichalcogenide films (MoS_2 and WS_2), potential materials for next-generation devices. In FY 2020, this work will focus on developing a fundamental understanding of the film growth to enable larger domain sizes and better film thickness control and to evaluate methods to control the precursor flux.

1d. Sensors NSI

NIST will perform simulations of nanoporous membranes that have varying nanopore structures with potential applications in lead ion detection (PCA 1d) and quantum computing (PCA 1b).

PCA 2. Foundational Research

NIST's foundational nanotechnology research portfolio includes research to measure and advance the understanding of functional nanostructured materials, nanoscale imaging, and nanomechanical properties of materials. NIST staff members are developing advances in spectroscopy of low-dimensional nanoscale materials and measurement science of colloidal nanomaterials. The research efforts span a variety of materials and application areas.

In FY 2020 NIST plans to develop, validate, and apply new and improved measurement science for the characterization of soft nanomaterials, particularly materials designed to detect, diagnose, and treat human disease. The vast majority of engineered nanoscale materials incorporated into medical products submitted for regulatory approval or clinical use consist, at least in part, of soft components (e.g., lipid-based laminar membrane-encapsulated particles for drug delivery—liposomes). Quantifying the properties of these components is technically challenging due to their deformable nature and inherent complexity. NIST anticipates that insights from this work will inform proposed standards development on liposome characterization in ISO TC 229's Working Group on Measurement and Characterization, which will be led by the Food and Drug Administration (FDA).

PCA 3. Nanotechnology-Enabled Applications, Devices, and Systems

NIST measurement science programs to support PCA 3 include efforts for photonic circuits, nanoscale coatings, atom-based devices, and nanoelectronics. NIST will continue to develop new methods to advance the development of single-photon detectors to harness the unique properties of graphene to develop improved reference standards. NIST will also continue its research efforts to advance the use of scanning probe microscopies to evaluate nanoscale properties of materials and to develop measurement and characterization methods that can drive advances in atomically thin two-dimensional devices for use in microelectronics.

Examples of PCA 3 activities include the following:

- NIST researchers plan to continue studies on the impact of UV exposure and outdoor weathering on the scratch resistance of coatings using a NIST-developed "nanoscratch" test to simulate scratch and other mechanisms of coatings damage.
- In FY 2020, the NIST single atom device project expects to have demonstrated manipulation, initialization, and readout of single- and two-qubit systems made from precisely placed phosphorus dopants in Si. This work is planned to be extended to develop arrays of dopant atoms (including dopants other than phosphorus) to carry out analog quantum simulations to attack hard quantum and many-body problems that cannot be dealt with by traditional computing, and to implement dopant arrays designed with novel quantum properties.
- NIST recently initiated a partnership with the Chemours Company to address nanoparticle metrology issues relevant to U.S. industry. A planned pilot study will support an internship at NIST and will likely focus on development of traceable methods to measure nanoparticle number concentration, a topic that is both technically challenging and highly germane to a wide range of nanotechnology applications and potential regulatory issues.

PCA 4. Research Infrastructure and Instrumentation

The NIST Center for Neutron Research and the Center for Nanoscale Science and Technology provide scientists from academia and industry with unique world-class capabilities that help move the state of the art forward in advanced materials, nanotechnology, bioscience, and other emerging technology areas. CNST constantly strives to improve the user experience for industry, academia, NIST, and other agency users by increasing tool uptime and process isolation so that advances in nanotechnology can continue to be realized.

In FY 2020, a currently operating NIST beamline, based on "hard" x-rays, will be joined by two NIST beamlines with "softer" x-rays at the National Synchrotron Light Source II (NSLS-II) located at Brookhaven National Laboratory. Nine end stations will take advantage of the unique broad and brilliant spectrum at NSLS-II to provide a world-leading suite of measurements of materials' atomic, molecular, and electronic structures. A unique aspect of the NIST beamlines is the ability to measure samples with multiple beams, enabling the measurement of more diverse and complex structures such as in polymer-metal composites. A "hard" x-ray beamline features both a NIST-operated x-ray spectroscopy line to examine structure at solid/solid interfaces—such as in semiconductors, glasses, and advanced metal alloys—and a second end station, built and operated in partnership with IBM to provide an x-ray diffraction capability that complements material structure measurements in crystalline materials. The other two beamlines featuring longer wavelengths, called "soft" and "tender" x-rays, will be able to measure materials across the entire periodic table, including microscopes that can probe molecular composition and orientation.

PCA 5. Environment, Health, and Safety

The NIST nanomaterial environment, health, and safety research effort is at a modest level, having declined in recent years.

Examples of PCA 5 activities include the following:

- NIST researchers are working on a project funded by the Consumer Product Safety Commission (CPSC) to develop protocols to screen, collect, and detect engineered nanomaterials released in house dust.
- NIST researchers plan to continue refining the computational tools developed to evaluate consumer exposure to airborne engineered nanoparticles by incorporating enhancements associated with transport phenomena, usability, and input data.
- NIST and the European Commission Joint Research Centre (JRC) intend to continue a collaborative effort to address the need for proven methods, standards, and reference materials to expedite the testing and regulatory approval of nanotechnology-enabled medical products. The Collaboratory for Nanomedicine resulted from a NIST-JRC workshop held in Ispra, Italy, that included scientists from key government, industry, and academic centers. Two pilot programs focused on liposomal-based drug formulations are under development. Key partners in this effort include FDA, the National Cancer Institute's Nanotechnology Characterization Lab (NCL), National Physical Laboratory (UK), and members of the European Union (EU) Nanomedicine Characterisation Laboratory.

Key Technical Accomplishments by NNI Goal

Goal 1. Advance a World-Class Nanotechnology Research and Development Program

- NIST researchers (with Purdue University) discovered a method to use electric fields to induce structural transitions in resistive random access memory (RRAM) devices. RRAM programming voltages are tunable by controlling the thickness of MoTe-based nanofilms and have achieved reproducible resistive switching within 10 ns between a high-resistant state (HRS) and low-resistant state (LRS). These findings demonstrate controlled electrical state switching in two-dimensional materials and highlight the potential of transition metal dichalcogenide nanomaterials for memory applications. This research was published in *Nature Materials*.[2]
- NIST performed simulations of a new nanoporous membrane, demonstrating that ion flow through the nanopores in the membrane is highly sensitive to membrane stretching. Potential applications of these nanoporous membranes include salinity control of solutions, nanoscale mechanical sensing, and a highly sensitive platform for studying ionic solvation. NIST also performed simulations of the nanoscale behavior of ionic liquid mixtures near charged carbon surfaces, providing insight that can be useful for the development of improved energy storage devices.
- NIST developed a new variation of atomic force microscopy (AFM), eliminating signal artifacts that made it impossible to measure physical properties of fragile substances such as biomaterials, ultrathin films, and other nanoscale structures. Generation of extremely high resonant frequencies in the AFM now allows use of soft cantilevers to probe such materials with extremely low forces, thereby preventing their being destroyed during measurement.
- NIST developed a user-programmable detector for a scanning electron microscope, working in partnership with Carl Zeiss Microscopy. The instrument can measure atomically thin materials that hold promise for diverse applications ranging from quantum computing to advanced nanocomposites.

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[2] https://www.nature.com/articles/s41563-018-0234-y

The low energy of this microscope naturally amplifies signals from extremely small volumes of material, allowing for precise determination of atomic structure and chemical composition, which inform the tailoring of properties of new advanced nanomaterials.

- Working in partnership with Cameca and the Colorado School of Mines, NIST has made dramatic improvements to the atom probe tomography technique—the most powerful analysis method available for imaging and identifying atoms in three dimensions with atomic resolution. Use of extreme ultraviolet photoionization now enables accurate, high-resolution measurements of chemical composition in a wide variety of nanoscale solids, including complex oxides and semiconductors.
- NIST's single atom device project, with support of the Department of Energy and partners at Zyvex and the University of Maryland, has now made single-atom transistors using single phosphorus dopant atoms deterministically placed with near atomic precision in Si, and aligned with gates and leads also fabricated with single-atom placement. Characterization of the device operation as a single-electron transistor shows clear transport of electrons one-by-one, stable for long periods of time, resistant to typical forms of noise because of the strong quantization provided by individual atoms. This is an essential step toward controlling individual electrons as qubits in an Si quantum computer and for advancing the development of chip-scale standards.
- In a marriage of quantum science and solid-state physics, researchers at NIST have used magnetic fields to confine groups of electrons to a series of concentric rings within graphene, a single layer of tightly packed carbon atoms. This tiered “wedding cake,” which appears in images that show the energy level structure of the electrons, experimentally confirms how electrons interact in a tightly confined space according to long-untested rules of quantum mechanics. The findings could also have practical applications in quantum computing.[3]
- NIST researchers are advancing measurements of atomically thin two-dimensional materials for use in nanoelectronics, and are using the results to both understand the fundamental behavior of these promising materials and develop methods to better control them to fabricate state-of-the-art prototypical devices. One the great challenges of using 2D materials in electronics is to create the proper polarity (electrons or holes) and number of charge carriers. NIST researchers developed a chemical modification method for controllable and wide-ranging doping of monolayer transition-metal dichalcogenides (TMDs). This method can be used to make complementary metal oxide semiconductor (CMOS) transistors out of these materials for low-energy electronics.
- NIST researchers, working in partnership with the Los Alamos National Laboratory Center for Integrated Nanotechnologies, are developing advanced spectroscopy approaches and using them to determine critical fundamental properties of nanoelectronic materials. NIST's team has used internal photoemission successfully to determine the electronic band alignment of technologically important 2D monolayer layer heterojunctions. This information is used to properly design and fabricate novel 2D tunnel field effect transistors. In another spectroscopy advancement, researchers have discovered an optical signature showing excitons bound to a single carbon nanotube (CNT) are accompanied by “intertube excitons” that involve tunneling across to closely interacting nanotubes. Excitons, which are created when light is absorbed by a material such as CNTs, effectively carry energy as tightly bound pairs of negative and positive charge (electrons and holes). The newly observed quantum tunneling action could impact energy distribution in carbon nanotube networks, with implications for light-emitting films and light harvesting applications.[4]

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[3] https://www.nist.gov/news-events/news/2018/08/novel-graphene-quantum-dot-structure-takes-cake

[4] https://www.lanl.gov/discover/news-release-archive/2018/February/0214-carbon-nanotubes.php

- NIST researchers measured the inherent electronic properties of colloidally synthesized semiconductor nanostructures, both directly by measuring custom nanofabricated test structures and indirectly by using advanced measurements such as Raman and terahertz spectroscopy. Despite much progress in the development of solution-based nanocrystal technology, including the commercialization of quantum dots, elucidating the electronic properties of these materials has remained a substantial challenge. These NIST results are helping to overcome this measurement hurdle.

Goal 2. Foster the Transfer of New Technologies into Products for Commercial and Public Benefit

In April 2018, NIST Director and Under Secretary of Commerce for Standards and Technology Walter Copan announced the Return on Investment (ROI) Initiative to unleash the innovation power of America into our economy by maximizing the transfer of Federal investments in science and technology into value for the Nation. The initiative is designed to advance the Lab-to-Market cross-agency priority goal of the President's Management Agenda, with the objective of assessing, streamlining, and accelerating the transfer of technology from the laboratory to the marketplace. In December 2018, NIST released a draft Green Paper identifying intended actions based on broad input from Federal, intellectual property, and technology transfer stakeholders.[5] After the publication of the final Green Paper in early 2019, steps will be taken to implement specific actions. Implementation of any of the intended actions that require specific policy, legislative, and/or regulatory actions will be advanced via formal proposals subject to appropriate interagency review and public comment.

Additional examples of NIST activities and accomplishments in support of Goal 2 include the following:

- NIST developed a suite of fast, reliable laboratory methods for simulating scratch damage at the nano-, micro-, and macroscales on automobile clear-coat finishes. This suite of test methods provides an improved understanding of the mechanisms behind those processes to ultimately enable development of more durable and scratch-resistant coatings.[6]
- The ability to accurately measure the size and concentration of nanoparticles underpins high-confidence risk assessment for using nanoparticles in a variety of applications ranging from commercial products to nanomedicine. In collaboration with industry and other agency partners, NIST completed major studies to (1) understand the performance of testing methods for evaluating size and concentration, and (2) demonstrate measurement assurance strategies for improving the testing methods. Ongoing conversations, including with CPSC and the EU Reference Laboratory for Alternatives to Animal Testing, are underway for further development and adoption of NIST methods.
- In June 2018, ISO published ISO/TS 21362, "Analysis of nano-objects using asymmetrical-flow and centrifugal field-flow fractionation." Developed in ISO TC 229's Working Group on Measurement and Characterization, this is the first international consensus specification on field-flow fractionation, an increasingly important group of analytical techniques used in applications where nanomaterials are present in complex liquid media (e.g., drug development, natural products, food, biologics, and environmental systems). The work was led jointly by NIST and the National Metrology Institute of Japan, and addresses NNI Goal 4 (responsible development of nanotechnology), as well as Goal 2.

[5] https://www.nist.gov/unleashing-american-innovation/green-paper

[6] https://www.nist.gov/news-events/news/2018/09/improve-auto-coatings-new-tests-do-more-scratch-surface

Goal 3. Develop and Sustain Educational Resources, a Skilled Workforce, and a Dynamic Infrastructure and Toolset to Advance Nanotechnology

The Center for Nanoscale Science and Technology has continued to provide companies with access to state-of-the-art nanofabrication resources. Users have included the following:

- Small business *Modern Microsystems, Inc.*, which plans to increase the power limits of high-power electronics by bringing thermal transport paths to within hundreds of nanometers of the power transistors. Proprietary electron devices are fabricated in other facilities and transferred into the CNST nanofabrication facility by means of packaged wafer and die carriers, which are wiped down during entry to prevent introduction of ambient contaminants from the outside world into the CNST nanofabrication facility. Carriers for the electron devices are similarly imported taking care not to damage the imported parts and also taking care to protect the cleanroom from any foreign particles that may otherwise be introduced on the outside of packaging. Samples are double-wrapped where applicable. The devices are coated for protection using spinners and/or vapor deposition systems, diced using the dicing saw, stripped and cleaned using wet and/or dry chemistries, and then bonded to carriers using wafer bonding equipment. Samples are then packaged for removal from the CNST nanofabrication facility and transferred to other facilities for further processing. Upon completion of external processing, the samples are brought back into the CNST nanofabrication facility along with heat sinks and other microdevice components, taking the same precautions as above to minimize introduction of particles. The parts are cleaned as necessary, patterned using lithography systems, etched as necessary using the reactive ion etching systems, wet chemistry, and neutral gas-phase etching, and then the components are mounted to the devices using the wafer bonding equipment. Throughout the processing sequence, CNST metrology tools are used to characterize the samples and to provide output data. The company is also using diamond and related materials for improving semiconductor electronics and x-ray optics in cutting-edge high-power applications. The company is leveraging CNST tools to develop techniques and technologies for diamond microsystems, nanofabrication, and material and system characterization in support of U.S. governmental, industrial, and academic applications. Modern Microsystems is a recipient of SBIR funding from the Department of Defense and the Department of Energy.[7]
- *ExxonMobil Research and Engineering* is using CNST equipment and expertise to study the contact mechanics and tribology of patterned engineering surfaces. Pattern feature length scales of less than ten micrometers are difficult to achieve with conventional methods but may be important in controlling mechanical performance.
- *Roche Sequencing Solutions* is evaluating various approaches to fabrication of devices for all-electronic detection and identification of biopolymers (DNA, RNA, peptides) using quantum transport of electrons by molecular recognition tunneling in metal-insulator-metal junctions. Roche relies on advanced ion beam deposition and ion beam etching tools at CNST to make prototype devices and demonstrate the feasibility of biomolecular detection. So far, the company has successfully fabricated and characterized quantum-tunneling junctions and observed significant increase in yield when compared to devices fabricated in collaboration with academic and industrial facilities using conventional deposition (thermal and e-beam evaporation) and patterning tools.

[7] https://www.sbir.gov/sbc/modern-microsystems

Goal 4. Support Responsible Development of Nanotechnology

- Nanosilver particles are commonly incorporated into kitchen products used to prepare and store food, due to their antimicrobial properties. NIST researchers, working in conjunction with FDA, have studied the effect of cutting, washing, and scratching on these nanosilver-containing materials to determine if these common consumer practices initiate or impact nanoparticle release into the environment or into food. The studies showed that, while bits of matrix polymer were released by the various methods of abrading the surface, no free silver nanoparticles were observed.[8]
- NIST researchers are in the fifth year of a multi-year collaboration with CPSC to develop testing and measurement protocols for determining the quantities and properties of engineered nanoparticles released from floor coatings and paints induced by abrasion and/or weathering.
  - Accomplishments to date include protocols and methodologies for characterizing size, morphology, and composition of engineered nanoparticles released from coatings following (1) rotary abrasion (both dry and wet), (2) environmental weathering, and (3) abrasion after weathering.
  - NIST and CPSC also conducted a study to characterize engineered nanoparticles released via dry abrasion of coatings containing an antimicrobial agent in nanoparticle form.
  - Knowledge gained from these efforts contributed to the ongoing development of ISO Technical Report (TR) 22293 "Evaluation of methods for assessing the release of nanomaterials from commercial, nanomaterial-containing polymer composites."
- NIST has developed two computational tools to evaluate consumer exposure to airborne engineered nanoparticles: The first is an online tool that provides estimates of indoor occupant exposure to airborne particles. The second tool, referred to as the size-resolved tool, includes additional physical models that account for the properties of nanoparticles that may impact their transport within the built environment. Both of these tools can be accessed as applications within a web browser.
- NIST has rigorously validated the capability of single-particle inductively coupled plasma mass spectrometry (spICP-MS) for measuring nanoparticle size distributions in comparison with high-resolution scanning electron microscopy (HR-SEM). Efforts to anchor these measurements with rigid traceability to the International System of Units (SI) are ongoing. Measurements of spICP-MS for nanoparticle number density was compared to other techniques, with generally favorable results. NIST research continues to enable microsecond time resolution for spICP-MS to overcome interference. Moreover, NIST continues to develop new calibration strategies, with a long-term view toward providing calibration reference materials for the technique.
- NIST continues to produce systematic procedures to assess the chemical and colloidal stability of nanomaterials to support the creation of a comprehensive knowledge base for evaluation of the potential risks and benefits of nanotechnology. Some engineered nanomaterials such as gold and silver nanoparticles are being intensively studied and used in nanoscale platforms for biomedical applications including diagnostics, wound dressings, therapeutics, and transfection, due in part to their general biocompatibility, antimicrobial activity, facile synthesis, and unique optical properties. A systematic evaluation of chemical and colloidal stability, particularly under physiologically relevant conditions, is critical for the efficacious and safe application of those nanoparticles in any biomedical application. To meet this need, NIST developed a protocol for the evaluation of nanomaterials' stability that was published in a NIST special publication (NIST SP1200-26)[9] as well as a series of

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[8] https://www.nist.gov/news-events/news/2018/11/do-kitchen-items-shed-antimicrobial-nanoparticles-after-use

[9] https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.1200-26.pdf

research articles in prestigious journals. Some of the gold nanoparticles developing by NIST show outstanding stability according to the protocol, and one is currently under study in the evaluation of developmental toxicity of *frog embryo teratogenesis assay-Xenopus* in collaboration with the National Cancer Institute.