

# PROGRESS AND PLANS OF NATIONAL NANOTECHNOLOGY INITIATIVE (NNI) AGENCIES

December 2019

## Department of Commerce (DOC)<sup>1</sup>

### *National Institute of Standards and Technology (NIST)*

#### Summary

An important component of NIST's mission to promote U.S. innovation and industrial competitiveness is the institute's research advancing nanoscale measurement science, standards, and technology. 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 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 advance the 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). 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 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 and 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*

NIST continues to contribute to the Nanomanufacturing NSI through many research efforts, including scientific advancements in device fabrication, imaging, and emerging areas. NIST is studying the fundamental science and advancing the practical limits of precision and accuracy of focused ion-beam (FIB) nanofabrication, a powerful approach to forming complex nanostructures for various measurement devices including nanofluidic devices for nanoparticle characterization. NIST is also developing fabrication

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processes to enable the integration of quantum emitters into nanophotonic devices such as bright, on-demand single-photon sources. Driving new capabilities in imaging and characterization of quantum emitters, NIST is developing novel instruments that implement fluorescence microscopy, spectroscopy, and photon counting. Researchers are studying DNA nanofabrication to understand and mimic the energetics of biological self-assembly on the nanoscale for pharmaceutical, chemical, and energy-harvesting applications.

#### *1b. Nanoelectronics NSI*

Nanoelectronics continues to be an area of emphasis in the NIST research laboratories. NIST is developing new capabilities in computational scanning electron microscopy (C-SEM), an emerging method in which accurate physical models of the microscopy process enable researchers to collect image data only when and where necessary, making the imaging collection more efficient and minimizing sample damage. C-SEM also uses physical models to combine nanostructure data from images acquired from multiple angles, enabling reconstruction of the three-dimensional geometry of the nanostructure with sub-nanometer fidelity. These new capabilities will improve the resolution and throughput of measurements that are essential for scaling up the fabrication of electronic devices. NIST researchers continue to explore new materials and structures for nanoelectronics, such as the intersection of spintronics, electronic structure topology, and two-dimensional (2D) materials. This fruitful area of multidisciplinary study has potential applications in next-generation electronics. NIST researchers are exploring the consequences of the topological properties of 2D materials such as transition metal dichalcogenides for the spin-dependent behavior of electrons.

#### *1d. Sensors NSI*

NIST continues to develop approaches to exploit nanoscale phenomena for sensing. For example, NIST is developing hybrid sensors that integrate plasmonic nanoparticles with nanoscale pores to enable control and study of molecular transport in confined liquid environments. Done in tandem with novel approaches to simulate biomolecular fluctuations, transport phenomena, and molecular interactions, this work has potential for applications in measuring fundamental characteristics of transport in biological media, biomarker detection, and water-quality monitoring. NIST is also developing novel materials and devices at the atomic scale to improve nanoelectronic biosensing and enable new sensors for biocompatible chemical imaging for precision medicine applications.

#### *1e. Water NSI*

NIST is engaged with representatives from the pharmaceutical industry to identify and address measurement challenges associated with water bioburden analysis.

#### *1f. Future Computing Grand Challenge*

NIST is exploring the fundamental aspects of measurements that can enable the realization of brain-like computing. For example, NIST researchers are developing approaches to measure the phase noise of injection-locked microwave spin-torque nano-oscillators, to understand how the noise affects the fidelity of match for arrays of oscillators for application in neuromorphic computing.

### PCA 2. Foundational Research

NIST's foundational nanotechnology research portfolio includes the development of cutting-edge approaches to design and accurately measure the size, shape, quantity, and physico-chemical complexity

of nanoparticles, nanostructured films, and nanocomposites. The NIST research spans a variety of nanomaterials and nanotechnologies. Some examples include the following:

- NIST is developing a suite of measurements to monitor and characterize dynamic DNA nanostructures for manufacturing. This measurement platform will enable the creation of quantitative, validated theory and models to guide the development of DNA nanotechnology by achieving molecular resolution of the critical details of structural self-assembly.
- NIST is developing novel tools and models to study the ultrafast dynamics of biomolecules, including methods to interpret and extract key dynamic and thermodynamic quantities from measurement. This work will improve the accuracy of biomolecule depictions in simulation for applications ranging from self-assembly to engineering molecular assemblies into functional devices.
- NIST is developing high-performance optomechanical devices that trap photons in nanoscale structures to strongly increase interactions between light and matter, enabling precise measurements at the nanoscale. These devices include probes, sensors, and transducers that can be used for magnetometry and emerging quantum applications as well as advancing understanding of nanoscale phenomena.
- NIST researchers are exploring atomic lattices assembled on 2D materials using scanning probe microscopes with the final goal of modelling quantum problems and systems.
- In collaboration with academic researchers, NIST is developing lens-less, time-resolved, nanoscale imaging by means of ultrafast, coherent sources of extreme-ultraviolet radiation. NIST plans to build a diffractive microscope with unprecedented lateral, depth, and time resolution capable of providing vital information about the performance of nanoscale semiconductor devices and magnetic memory while they are in operation.<sup>2</sup>
- NIST intends to expand its efforts in characterization of soft nanomaterials, particularly aimed at applications in biotherapeutics, plastics recycling, and marine debris characterization. Quantifying the properties of soft nanomaterials in biological matrices is technically challenging because of the deformable nature of cells and the inherent complexity of biological environments.

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

NIST measurement science research to support PCA 3 includes efforts for nanophotonic devices, nanoparticle tracking systems, atomically precise lithography, and nanocomposites. NIST will continue to advance research on nanoscale devices and systems for a variety of applications, from microelectronics to coatings and pharmaceutical products. Examples are listed below:

- NIST is developing multifunctional optical metasurfaces that are ultrathin and amenable to lithographic fabrication and integration, enabling new capabilities and applications in the fields of ultrafast, quantum, and nonlinear optics.
- NIST is working with the U.S. Food and Drug Administration (FDA) to develop optical microscopy and nanoparticle tracking methods to characterize complex liposomal products to support measurements for quality control and of product equivalence.
- NIST is developing optical microscopy and nanoparticle tracking methods to better measure the motion of microelectromechanical systems (MEMS). This motion is fundamental to understanding the behavior of MEMS, but existing measurement methods are not adequate.

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<sup>2</sup> <https://www.nist.gov/programs-projects/spin-dynamics-and-magnetic-microscopy>

- NIST is using MEMS to develop a new type of scanning tunneling microscope (STM) with exceptional precision for atomically precise lithography. This effort focuses on the first demonstration of atomically precise lithography using a single MEMS-STM, while providing a path towards massively parallel arrays that will enable fabrication with higher throughput.
- In the coming year, the NIST single atom device project expects to have demonstrated operation of three and four qubit systems made from precisely placed phosphorus dopants in Si to establish the manipulation of quantum information in Si dopant devices. This work is planned to be extended to other dopants to increase device functionality. In parallel, extended arrays of dopant arrays will be systematically studied to establish their operation as quantum simulators to attack quantum and many-body problems that cannot be addressed with traditional computing, as simulators for the dynamics of quantum information in complex, heterogeneous systems, and to implement in Si material regions with novel quantum properties.
- NIST is advancing nanoscale light emitters for microdisplays and virtual/augmented reality devices. NIST has recently partnered with a major U.S. aerospace company to develop light-emitting diodes for these demanding applications. In the coming year, NIST researchers will expand the range of light emission wavelengths to cover red, green, and blue, in addition to ultraviolet.
- 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.
- NIST researchers, working in conjunction with FDA and academia, are investigating the degradation of polymers containing nanomaterials that exhibit barrier properties useful in many diverse applications, including outdoor infrastructure coatings for corrosion resistance and food packaging for extended ingredient freshness. NIST researchers developed measurement protocols that assess polymer nanocomposite degradation following accelerated moisture, heat, and UV exposure to simulate degradation of products during outdoor use or upon disposal.
- NIST is advancing scanned probe microscopy to rapidly map mechanical and physical properties of nanoscale devices to reveal inhomogeneities and other manufacturing defects and to validate predictive models of device reliability and performance. NIST research is aimed at improving the accuracy, sensitivity, and speed of these methods and applying them to novel device structures such as those produced by additive manufacturing.

#### PCA 4. Research Infrastructure and Instrumentation

NIST provides scientists from academia, industry, and other government agencies access to unique, world-class facilities and research instrumentation that help move the state of the art forward in emerging technology areas and advance our understanding of nanoscale phenomena and systems. Examples include the following:

- The NIST Center for Neutron Research is a user facility on the NIST Gaithersburg campus that provides neutron measurement capabilities to the U.S. research community. It is a national center for research using thermal and cold neutrons, outfitted with a variety of instruments for neutron imaging, small angle neutron scattering, reflectometers, diffractometers, and spectrometers.<sup>3</sup>
- The Center for Nanoscale Science and Technology NanoFab on the NIST Gaithersburg campus provides researchers with rapid access to state-of-the-art, commercial nanoscale measurement and

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<sup>3</sup> <https://www.nist.gov/ncnr>

fabrication tools and methods. The NanoFab’s comprehensive toolset and dedicated technical staff enable users to process and characterize a wide range of nanoscale materials, structures, and devices.<sup>4</sup>

- The NIST Boulder Microfabrication Facility enables development of nanoelectronic devices for chip-based quantum standards, sensors, integrated and quantum photonics, and advanced computing. Planned acquisitions include state-of-the-art tools for atomic-layer deposition of coatings on ultrathin films, plasma-enhanced chemical-vapor deposition, aluminum electron-beam deposition of Josephson junctions, and atomic force microscopy.<sup>5</sup>
- NIST intends to deploy miniaturized, multiplexed, x-ray and gamma-ray superconducting transition-edge sensors at the second-generation Linac Coherent Light Source (LCLS-II) and other national x-ray light sources to support materials research in nanoscale materials dynamics, heterogeneity, and fluctuations; fundamental dynamics of energy and charge; catalysis and photocatalysis; and emergent phenomena in quantum materials.<sup>6</sup>
- NIST is expanding its capabilities at the National Synchrotron Light Source II (NSLS-II) located at Brookhaven National Laboratory (BNL), supplementing the existing NIST beamline, based on “hard” x-rays, with two additional beamlines with “softer” x-rays. 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 structure. 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 industry 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.

NIST leads and participates in a number of organizations where voluntary consensus nanotechnology standards are being developed. U.S. leadership and participation in the development of international standards ensures that the United States can shape the strategic and technical direction of nanotechnology development everywhere. The expertise that NIST scientists contribute to the development of these consensus standards helps ensure that the resulting standards are technically robust, timely, and fit-for-purpose. NIST experts’ leadership and engagement in pre-standardization activities such as those being conducted under the Versailles Project on Advanced Materials and Standards (VAMAS) helps accelerate the development of consensus standards by leveraging the outcomes of the round-robin testing efforts within VAMAS activities to inform the development of consensus standards. NIST representatives lead ASTM International’s Committee E56 on Nanotechnology. A U.S. Technical Advisory Group (TAG) (accredited by ANSI) chaired by NIST represents the United States at ISO TC 229, Nanotechnologies. The TAG is responsible for formulating positions and proposals on behalf of the United States with regard to ISO standardization activities related to nanotechnology. NIST staff also lead nanotechnology-related efforts in the Technical Committee 113 of the International Electrotechnical Commission (IEC). Examples of ongoing or planned

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<sup>4</sup> <https://www.nist.gov/cnst>

<sup>5</sup> <https://www.nist.gov/programs-projects/boulder-microfabrication-facility>

<sup>6</sup> <https://www.nist.gov/programs-projects/quantum-microcalorimeters>

NIST-led nanotechnology consensus standards development projects in several organizations are as follows:

- In ASTM E56, NIST is leading several new activities:
  - Test Method for Measuring the Size of Nanoparticles in Aqueous Media Using Dynamic Light Scattering, in collaboration with FDA.
  - Test Method for Analysis of Liposomal Drug Formulations using Multidetector Asymmetrical-Flow Field-Flow Fractionation (AF4), draft in collaboration with the EU.
  - Test Method for the Determination of the Mass Fraction of Particle-Bound Gold in Colloidal Gold Suspensions.
  - Guide for Visualization and Identification of Nanomaterials in Biological and Nonbiological Matrices Using Darkfield Microscopy with Hyperspectral Imaging Analysis: 4<sup>th</sup> draft under review; in collaboration with NIOSH and industry.
- In ISO TC229, NIST is transitioning *ISO/TS 21362:2018 Nanotechnologies – Analysis of nano-objects using asymmetrical-flow and centrifugal field-flow fractionation*<sup>7</sup> towards a full International Standard with an interlaboratory study planned in early 2020, jointly led by NIST and Japan.

#### PCA 5. Environment, Health, and Safety

The NIST nanomaterial environment, health and safety (EHS) research portfolio remains at a modest level, with projects ranging from nanoplastics and reference materials to new measurements on consumer products and real-world environments. Examples include the following:

- NIST is establishing a research effort on nanofluidic measurement devices and optical microscopy methods to characterize nanoplastics, an active area of research due to their potential role as environmental contaminants. Quantifying the structures and properties of nanoplastics is fundamental to understanding byproduct risks.
- NIST is developing nanoparticle Standard Reference Materials (SRMs) of different sizes and materials, including polystyrene, gold, and silica to enable accurate calibration of instruments used to characterize nanoparticles, whether to monitor their presence and effects on EHS, or to improve their performance in materials or biomedical applications. This work involves the production of nearly ideal nanoparticles and the certification of nanoparticle sizes through cross-validation of methods.
- NIST researchers are studying the performance of components of vinyl siding, a popular home exterior used across the United States. This siding is a composite material composed of polyvinyl chloride (PVC), and particulate additives on the micrometer to nanoscale are added to improve the durability of siding. Recently, NIST researchers have begun to investigate how different formulations of PVC and additives affect the appearance, impact performance, and flammability of composite siding materials before and after UV exposure.
- NIST researchers are continuing to refine computational tools to evaluate consumer exposure to airborne engineered nanoparticles by incorporating enhancements associated with transport phenomena, usability, and input data into their models.

Consensus standards activities play an important role in developing confidence in the safety and efficacy of nanomaterials. Through a representative from the National Institute for Occupational Safety and Health (NIOSH), the United States holds leadership of ISO TC 229's Working Group 3: Health, Safety and

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<sup>7</sup> <https://www.iso.org/standard/70761.html>

Environmental Aspects of Nanotechnologies. Recently, NIST assumed leadership of the ISO TC 229 Task Group on Sustainability, Consumer and Societal Dimensions. One example of a new EHS consensus standards activity led by NIST is the development of an International Standard to test nanomaterials phototoxicity *in vitro*.

## Key Technical Accomplishments by NNI Goal

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

- *The NIST on a Chip program.* The NIST on a Chip (NOAC) program<sup>8</sup> is focused on revolutionizing measurement services and metrology by bringing them out of the lab and directly to the user. The NOAC program continues to leverage NIST's excellence in precision measurement with a focus on creating prototypes for a new generation of ultra-compact, inexpensive, low-power measurement tools for time and frequency, mass, force, temperature, and other key quantities. Many of the NIST-pioneered technologies in the NOAC portfolio are based on nanoscale measurements and devices. NOAC is exploring and creating robust industry and government partnerships to turn NIST innovations into commercial products.<sup>9</sup> For example, working with partners, NIST recently demonstrated an experimental, next-generation atomic clock smaller than a coffee bean that is a vapor cell on a chip, containing rubidium atoms that "tick."<sup>10</sup>
- *Biosensors.* NIST, working with the National Institutes of Health, demonstrated novel dual-gated field-effect transistor-based biosensors fabricated with single atomic-layer semiconducting films that achieved an unprecedented pH resolution of  $92 \times 10^{-6}$ . These biosensors enabled precise measurements of biomolecular enzymes implicated in Alzheimer's disease at sub-physiological concentrations. This work enables diagnostic tests to speed up drug discovery for several debilitating neurological diseases.
- *Vibrational spectroscopy of biomolecules.* NIST developed an innovative approach to analyze data from ultrafast vibrational spectroscopy of biomolecules, showing that, for the first time, features of the energy landscape can be extracted from such measurements. This simultaneously identified a new physical phenomenon—nonlinear localization of vibrational modes—that is likely ubiquitous in biomolecules and thus will have repercussions for simulation and understanding of biomolecule dynamics
- *Graphene pores.* NIST modeled a novel platform based on functionalized graphene pores for quantifying optimal ionic permeation and selectivity. This will help delineate the role of dehydration and electrostatic interactions in more complex biological ion channels, as well as improve molecular simulation.
- *Transport simulation.* NIST discovered and reviewed an efficient approach to simulating transport phenomena, including filtration and selectivity, in nanoporous materials. This approach eliminates artifacts due to the finite size of atomic-scale simulations. Molecular simulations of filtration membranes and nanopore-based sensors can now properly incorporate the influence of the bulk solution-pore interface in separation efficiency and other membrane characteristics.
- *DNA nanofabrication thermodynamics.* NIST measured the thermodynamics of the essential building block of DNA origami, a single fold, as well as the relative yield of that single fold. In doing so, NIST

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<sup>8</sup> <https://www.nist.gov/noac>

<sup>9</sup> <https://www.nist.gov/pml/productsservices/nist-chip-portal>

<sup>10</sup> <https://www.nist.gov/news-events/news/2019/05/nist-team-demonstrates-heart-next-generation-chip-scale-atomic-clock>

identified a previously unconfirmed intermediate state that acts as a kinetic defect during nanostructure assembly. This is the first study to have confirmed the existence of an intermediate responsible for failed assembly in DNA origami. This work informs the design of DNA origami when scaled to a production level, where minimization of defects is critical.

- *High-throughput nucleic-acid thermodynamics.* NIST developed a new technique for data analysis that significantly reduces subjectivity in baseline and background correction of optically-reported melt data. NIST applied this system to high-throughput measurement of the thermodynamics of DNA hybridization, and hard-to-measure topological contributions to hybridization. This technique has the potential to enable biological researchers to obtain significantly more information from melt measurements that they already perform.
- *FIB nanofabrication.* NIST developed novel processes of FIB nanofabrication that advance the limits of resolution, precision, and accuracy. NIST achieved sub-nanometer vertical resolution and generalized a method of achieving lateral super-resolution by FIB nanofabrication. These capabilities enable the rapid prototyping of complex nanostructures that perform metrology functions such as spatial calibration of optical microscopes and dimensional and optical characterization of colloidal nanoparticles.
- *Quantum-emitter integration.* NIST demonstrated various nanophotonic geometries that integrate semiconductor quantum dots to produce high-efficiency triggered sources of single photons, both as stand-alone, fiber- or free-space coupled chip-scale devices, or as on-chip integrated sources for silicon photonic circuits. The processes and designs are generally applicable to other types of quantum emitters and available to all researchers. NIST has achieved a high single-photon brightness that can be used to scale up photonic technologies for quantum communication, simulation, computing, and metrology. The work also benefits single-emitter spectroscopy studies for further technological development.
- *Quantum-emitter characterization.* NIST developed optical microscopes to characterize quantum emitters at cryogenic temperatures, allowing the localization of quantum dots across a sample surface and measurement of the spectral and statistical properties of single-photon emission. NIST used such instruments to fabricate single-photon sources by characterizing and integrating single quantum dots into nanophotonic devices. Ongoing activities involve microscope automation to characterize quantum dots with high throughput, as well as characterization of other emitter types. Such instruments will improve the manufacturing yield of nanophotonic quantum devices by achieving statistical characterization of emitter production processes for the first time. Such instruments will also enable novel measurements of materials at the nanometer scale using quantum emitters as optical probes.
- *Localization microscopy.* NIST developed arrays of nanoscale apertures as multifunctional reference materials for localization microscopy. In combination with novel calibration methods, NIST has demonstrated the reduction of localization errors from a widefield optical microscope by up to four orders of magnitude. This enables the calibration of optical microscopes for both precise and accurate localization and tracking of subresolution point sources below the nanometer scale. NIST plans to transfer these technologies to the research community by applying this new capability to the production and distribution of reference materials for optical microscopy.
- *Nanoparticle tracking.* NIST developed methods of nanoparticle tracking that enable new measurements of microsystem motion in three dimensions and six degrees of freedom, with uncertainties down to the nanometer and microradian scales. These methods can rely on calibration

of ordinary optical microscopes that are broadly available, thus lowering the barrier to entry into the field of nanoparticle tracking.

- *Nanomedicine metrology.* NIST has developed a measurement device to confine single nanoparticles for tracking analysis. This multifunctional microstructure imposes top-down control of focal depth and provides built-in reference positions to correct localization errors from optical aberrations. Ultralong trajectories and ultrawide-field sampling provide unprecedented precision. Importantly, this confinement scheme facilitates correlative measurements of single particles by fluorescence and light scattering, providing specificity in analysis of active pharmaceutical ingredient encapsulation.
- *Optical metasurfaces.* NIST has developed a platform to simultaneously and independently shape the amplitude, phase, and polarization of femtosecond pulses in the near-infrared spectral range with record frequency resolution and bandwidth. Ultrafast pulse shaping has applications in pulse compression, dispersion compensation, and nonlinear microscopy. Careful design and precise patterning and placement of deep-subwavelength metasurface elements enables the ability to address individual lines of an optical frequency comb for applications in arbitrary waveform generation. NIST has also developed novel materials and lithography methods to extend the frequency range of operation of metasurfaces from the deep-ultraviolet to terahertz. Integration of metasurfaces into nanophotonic devices provides the ability to create arbitrary optical fields in the far-field in a compact platform for applications in cold-atom traps, biosensing, and light detection and ranging (LIDAR).
- *Optomechanical devices.* NIST developed nanoscale mechanical resonators as building blocks for nanomechanical sensors. High-performance sensing occurs by measuring the resonator frequency with low uncertainty, high stability, and within a short time. Thermodynamic fluctuations and the dependence of frequency on temperature are two key limitations of such sensors. NIST has developed a new way to make a common class of nanomechanical resonators insensitive to temperature change and has measured their frequency at the fundamental limits imposed by thermodynamics using a built-in photonic readout.
- *Atomic force microscopy.* NIST developed probes for atomic force microscopy that combine a nanoscale mechanical cantilever and integrated photonic resonator. The miniaturization of these instruments to the nanoscale resulted in a record combination of measurement speed and precision. These probes have been used to radically increase the throughput of atomic force microscopy and demonstrate new nanoscale measurement modalities such as mapping of chemical composition and thermal properties of nanomaterials.
- *Nanomechanical transduction.* NIST developed electro-optomechanical transducers on the basis of nanomechanical resonators that interface to optical waves, through radiation pressure or photoelastic forces, and microwaves, through electrostatic or piezoelectric mechanisms. NIST used these devices to demonstrate a mechanically mediated coherent interface between the microwave and optical domains. NIST and external stakeholders cryogenically cooled these devices to their motional ground state to demonstrate the potential for ultralow-noise microwave-to-optical quantum transduction. NIST also used these devices to demonstrate room-temperature, high-bandwidth feedback stabilization of optical modes, low-power acousto-optic modulation in which electrically-driven acoustic waves modify optical wave propagation, and acousto-optic gating in which optically-driven acoustic waves modify acoustic wave propagation.
- *Pulsed-laser interferometry.* High-frequency nanomechanical oscillators have become common components of integrated circuits for cellular communication and integrated sensors—yet the underlying physics of their operation remains poorly understood. NIST has developed a one-of-a-kind

facility, including new optical measurements such as pulsed-laser interferometry, to allow fast and precise measurement of these oscillators for the first time and enable better understanding and optimization of these essential components of next-generation communications and sensing.

- *Spintronics*. NIST researchers theoretically predicted the occurrence of a unique form of spin current present in ferromagnets. This spin current was previously unrecognized and has the unusual property that flowing spins are misaligned from the magnetization. Working with collaborators from the University of Denver, signatures of this spin current were measured experimentally. In the experiment, accumulation of these transverse spins rotate the magnetization at the sample surface, which is detected with optical methods. This novel spin current may be useful for developing magnetic random access memory technology.
- *Graphene devices*. In a marriage of quantum science and solid-state physics, researchers at NIST have used a fabrication by scanning tunneling microscope to confine groups of electrons to a series of quantum dots “sketched out” within graphene, a single layer of tightly packed carbon atoms. In turn, the STM characterization was used to map the wavefunctions and the energy level structure of the electrons, experimentally determining 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.
- *Single-atom devices*. NIST’s single-atom device project, with support from the Department of Energy and partners in industry and academia, has now made donor-quantum dot devices using single P dopants or a cluster of a few P dopants as the donor deterministically placed in Si and aligned with gates and leads, all fabricated with near atomic precision. The quantum dot acts as a sensor of the charge or spin on the donor. This setup allows for the single-shot readout of dopant-based qubits necessary for any quantum computing device. Devices with control of the donor-lead separation down to a single atomic row have provided the most detailed characterization of transport in such devices. Extended 3x3 arrays of dopants have now been fabricated, and transport through these arrays has been measured for the first time. This provides the first step towards building a quantum simulator from dopant arrays in Si.
- *Computers that emulate the human brain*. Neuromorphic computing can improve both the speed and efficiency of many computational tasks such as image recognition and Internet search. NIST’s recent work on Josephson junctions with magnetic nanoparticles in the barrier demonstrated devices with quasi-analog control of the Josephson critical current. These devices can tune the Josephson critical current by more than 1,000% by adjusting the amount of spin order between the nanoparticles. These devices can be combined with naturally spiking Josephson junctions to implement artificial neural networks directly in hardware. The single-flux-quantum pulses produced by Josephson junctions in the voltage state are analogous to the action potentials in biological neurons, and superconducting transmission lines act as axons distributing the signals with near-lossless transmission. The magnetic nanoclustered Josephson junctions form a high-speed, energy-efficient synaptic element that can control the weighting to form a spiking neural network. NIST combined these elements to make a new neuromorphic architecture with the potential to be applied to problems that are currently addressed by artificial neural networks.<sup>11</sup>
- *Cryogenic computing*. NIST completed a multiyear collaboration with industry under the auspices of the Intelligence Advanced Research Projects Activity (IARPA) Cryogenic Computing Complexity (C3)

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<sup>11</sup> <https://www.nist.gov/news-events/news/2018/01/nists-superconducting-synapse-may-be-missing-piece-artificial-brains>

program, to perform tests and measurements needed for the development of spin-based nanoscale cryogenic memory and logic for energy-efficient, exascale computing, which is critical for many national priorities and for national security. NIST developed state-of-the-art measurement tools and benchmark data and devices for IARPA, including high-speed, sub-micrometer, superconducting Josephson junctions and stacked Josephson junctions for computer logic.<sup>12</sup>

- *Coupled nano-oscillators for image recognition.* NIST completed a multiyear collaboration with industry and academia, with support from the Defense Advanced Research Projects Agency (DARPA), to develop nanoscale, high-frequency nano-oscillators for rapid, low-energy processing of images in computer hardware, a task traditionally performed in software. Applications are in advanced computing, neuromorphic computing, and national security. The spin-torque oscillators have the potential to act as nano-neurons when used in an interacting array. Their nanoscale size, high-frequency dynamics, and non-linear properties provide the qualities required to build fast and scalable devices for artificial neural networks. The oscillators can communicate among each other because of their ability to be mutually synchronized. As part of its contribution, NIST developed novel microwave circuits, device concepts, and measurement techniques. In particular, NIST measured the locking properties of a new geometry of spin-torque oscillators having three terminals instead of two. The addition of a third terminal affords advantages for applications, such as better durability, a larger output signal, and the possibility to independently optimize excitation and readout.<sup>13</sup>
- *Chiral spin order in magnetic nanostructures.* Thermal fluctuations are the main limitation to the scalability of nonvolatile magnetic memory. To prevent thermal erasure of a bit in a magnetic memory element, the magnetization must be in a uniform, single-domain state. However, a novel physical mechanism at play in certain types of ferromagnetic/nonmagnetic multilayers might compromise the uniformity of magnetization: the Dzyaloshinskii-Moriya interaction (DMI). The DMI favors chiral magnetization orientation rather than a uniform state, and a sufficiently large DMI would greatly reduce the thermal stability of magnetic memory. Another manifestation of DMI is the creation of skyrmions, which have potential application as nanomagnetic recording bits that can be moved along racetrack structures. NIST developed a quantitative measurement technique based on Brillouin light scattering to characterize the DMI at different ferromagnetic/nonmagnetic interfaces.<sup>14</sup>

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

- *Return on Investment Initiative.* In April 2019, NIST published the Return on Investment (ROI) Initiative “Final Green Paper” (NIST Special Publication 1234),<sup>15</sup> which includes 15 key findings to maximize U.S. innovation from government-funded research. Incorporating extensive feedback from industry, academic, and government stakeholders, the report describes options for enhancing how federally funded inventions move from the laboratory to the marketplace. The paper’s findings are informing deliberations, decision-making, and actions by relevant departments and agencies. The ROI initiative is an effort to advance the President’s Management Agenda in support of the Lab-to-Market Cross Agency Priority goal.
- *Device design.* NIST has continued to develop and disseminate the Nanolithography Toolbox, which is in use at a variety of universities and corporations. This design software enables scripting and

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<sup>12</sup> <https://www.nist.gov/news-events/news/2017/04/supercomputing-probing-future>

<sup>13</sup> <https://www.nist.gov/programs-projects/neuromorphic-computing>

<sup>14</sup> <https://www.nist.gov/programs-projects/spin-dynamics-and-magnetic-microscopy>

<sup>15</sup> <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.1234.pdf>

generating layouts for lithography patterns, in particular facilitating the design of devices with complex curves and aggressive critical dimensions. Using parameterized shapes as building blocks, the Nanolithography Toolbox allows rapid design and layout of nanoscale devices of arbitrary complexity through scripting and programming. The Toolbox includes structure libraries for microelectromechanical systems, nanoelectromechanical systems, and nanophotonic devices. Furthermore, the Toolbox allows precise definition of the number of vertices for each shape or the creation of vectorized shapes using Bezier curves. The Toolbox is applicable to a broad range of design tasks in the fabrication of small devices.

- *Ultra-clean wafers for nanoelectronics.* The NIST Boulder Microfabrication Facility has a cooperative research and development agreement with a company to improve process development for substrate cleaning and die bonding needed for nanoscale device fabrication.<sup>16</sup>
- *Magnetic resonance spectroscopy for nanomagnetic memory.* Recently, NIST developed a new method to quantify the spin-Hall effect, a mechanism by which the quantum-mechanical spin of the electron couples with the direction of electron transport in certain kinds of metals. The new method employs a conventional ferromagnetic resonance spectrometer based on a vector network analyzer to measure the interaction of a ferromagnetic sample with microwaves. With it, NIST discovered that the transport of the electron spin across interfaces of dissimilar materials, a crucial component of most spin-Hall effect measurements, is highly inefficient. The NIST research results point to the importance of interface engineering to improve spin transport between magnets and metals. The rapid measurement method should facilitate the discovery of new materials that exhibit a large spin-Hall effect.<sup>17</sup>
- *Coatings.* NIST developed a suite of fast, reliable laboratory methods for simulating scratch damage at the nano-, micro-, and macroscales on automobile clearcoat 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.<sup>18</sup>
- *Nanolubricants.* NIST completed the evaluation of the boiling heat transfer enhancing potential of a nanolubricant for the Naval Facilities Engineering & Expeditionary Warfare Center. NIST presented an analysis that showed that the nanoparticles were too large and too dense to promote a boiling enhancement in chillers that cool buildings, and the fullerene-like structure and the large size encouraged nanoparticle settling that could lead to additional degradations.
- *Nanoparticle counting.* NIST expanded its capabilities for determining critical characteristics of nanoparticles for use in biological environments. One example is the development of a uniform process to determine nanoparticle count in environmental or biological samples. This work, performed in conjunction with a large chemical and materials manufacturer and EMPA, the Swiss Federal Laboratory for Material Testing and Research, resulted in a simplified process used by pharmaceutical researchers when testing a nanoparticle-based therapeutic.
- *Through-focus scanning optical microscopy (TSOM).* NIST continued to develop TSOM and applied the method to perform rapid and accurate measurements of the equivalent-volume diameter of nanoparticles with irregular shapes. While the optical signal is incapable of resolving the subwavelength features of nanoparticles, perturbations in the through-focus optical field are directly proportional to the total quantity of material. Nanoparticle volume is critically important, because it fundamentally affects reactivity and suitability for different materials or biomedical applications.

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<sup>16</sup> <https://www.nist.gov/news-events/news/2017/09/nist-boulder-microfab-lab-goes-nano>

<sup>17</sup> <https://www.nist.gov/programs-projects/spin-transport>

<sup>18</sup> <https://www.nist.gov/news-events/news/2018/09/improve-auto-coatings-new-tests-do-more-scratch-surface>

- *Pharmaceutical manufacturing.* NIST published a study on the reference standards needed to provide real-time feedback on the presence of bacteria in high-purity water systems for pharmaceutical manufacturers; the presence of these bacteria can adversely impact the final industrial products. The study suggested that small fluorescent spheres less than approximately 1,000 nm in diameter could be used to calibrate and validate new online instruments used to identify bacteria in these systems.<sup>19</sup>
- *Voluntary consensus nanotechnology standards.* NIST leads and participates in a range of international standards development activities that result in new standards and guidance documents. For example, a new ASTM E56 *Test Method for Quantitative Measurement of the Chemoattractant Capacity of a Nanoparticulate Material In Vitro* was approved for publication.<sup>20</sup> This method was led by FDA with substantial NIST contributions. In ISO TC229, standard ISO 20814 *Nanotechnologies – Testing the photocatalytic activity of nanoparticles for NADH oxidation* was published; this effort was undertaken by NIST together with Korea.<sup>21</sup>

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

- *Instrumentation upgrade for NIST Electron Beam Ion Trap.* NIST completed the installation and commissioning of a superconductor transition-edge x-ray sensor array in the NIST Electron Beam Ion Trap spectrometer. The sensor array allows the instrument to make x-ray reference measurements, with a resolution 40 times better than previously possible, over a wide range of energies, enabling high-precision measurements of ionized gases in minutes instead of weeks. The data will be used to reveal the dynamics of astrophysical plasmas, test atomic theories, and provide diagnostic measurements of fusion energy reactors, among other applications. A transition-edge sensor (TES) is a thin film of superconducting material that is electrically biased in its sensitive superconducting-to-normal resistive phase transition. When the film absorbs an x-ray photon, the temperature, and consequently the resistance, increases, allowing an exquisitely sensitive measurement of the photon's energy. Although the TES was invented in the 1940s, it was not until the development of the low-noise superconducting quantum-interference device (SQUID) array amplifier at NIST in the 1990s that it became possible to take full advantage of the TES's extreme sensitivity.<sup>22</sup>
- *New beamlines.* NIST expanded its capabilities at the National Synchrotron Light Source II located at Brookhaven National Laboratory, supplementing the existing NIST beamline, based on “hard” x-rays, with two additional beamlines with “softer” x-rays at NSLS-II. These additional beamlines were commissioned in September 2019, with most instrumentation already in place. Once complete, 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 the atomic, molecular, and electronic structure of nanostructured materials. 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

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<sup>19</sup> <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC6826051/>

<sup>20</sup> <https://www.astm.org/Standards/E3238.htm>

<sup>21</sup> <https://www.iso.org/standard/69298.html>

<sup>22</sup> <https://www.nist.gov/news-events/news/2019/04/ebits-improved-x-ray-vision-promises-astronomical-insights>

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. These beamlines will be used to support transformative research of high-risk and potentially high-reward science in partnership with industry.

#### Goal 4. Support Responsible Development of Nanotechnology

- *Nanoplastic metrology.* NIST developed a measurement system that advances and integrates complex nanofluidic replicas, localization optical microscopy, and Bayesian statistical analysis. This system enables dimensional and optical metrology of single nanoparticles with record precision, accuracy, and efficiency. The work can inform the characterization of environmental nanoplastics. NIST also developed a method to characterize the concentration of metal, oxide, and plastic nanoparticles in water involving microdeposition of aqueous suspensions onto a microscopy substrate by inkjet printing. Optical microscopy measurements of microdroplet volume in flight and nanoparticle count yield suspension concentration. The volume measurement achieves a relative uncertainty of approximately 0.2 %, an order of magnitude improvement in comparison to the benchmark method of microdroplet gravimetry.
- *Nanosilver.* 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. This work was completed in fiscal year (FY) 2019, resulting in two archival papers.<sup>23</sup>
- *Floor coatings and paints.* NIST researchers completed a five-year collaboration with the Consumer Product Safety Commission (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 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.
- *Airborne nanoparticles.* NIST 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 were released in FY ‘19 and can be accessed as applications within a web browser on the NIST website.<sup>24</sup>

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<sup>23</sup> <https://www.nist.gov/news-events/news/2018/11/do-kitchen-items-shed-antimicrobial-nanoparticles-after-use>

<sup>24</sup> <https://pages.nist.gov/CONTAM-apps/>