THE CNST NEWS

SUMMER 2017

A BETTER WAY TO BUILD WITH DNA

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MOVIES OF A NANOCATALYST

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Cover: Illustration shows that the protein RecA (blue) wraps around and strengthen double-stranded DNA (red line), enabling researchers to build larger structures with the genetic material. Credit: NIST

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RESEARCHERS USE A SUBTLE TWIST TO MANIPULATE MAGNETISM

In a pioneering effort to control, measure and understand magnetism at the atomic level, researchers working at the CNST have discovered a new method for manipulating the nanoscale properties of magnetic materials.

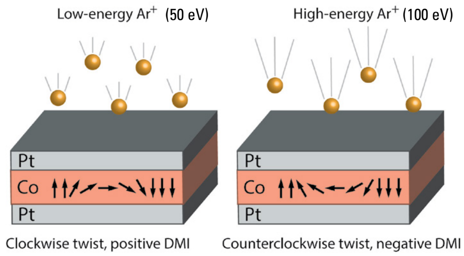
The ability to control these properties has potential applications in creating and improving the magnetic memory in consumer electronic devices, and developing a sensitive detector for magnetic nanoparticles.

The discovery focuses on a quantum-mechanical property known as spin, which endows electrons with a tiny magnetic field. Electron spin can point in either of two directions, "up" or "down," as does the accompanying magnetic field. Over the years, scientists have become adept at flipping the direction of spin, and therefore the direction of the magnetic field. But the new finding has a novel twist.

In materials such as cobalt, the spins of neighboring electrons interact, causing them to all point in the same direction. If some of the spins are forced away from that direction, they pull some of the nearby spins with them. This causes the spins to undergo a gradual twist—clockwise or counterclockwise. In some materials, the spins prefer to twist in only one direction.

A team led by CNST researcher Samuel Stavis and Andrew Balk, now at the Los Alamos National Laboratory, found a way to control the direction of this twist in a cobalt film just three atomic layers thick. Moreover, they could set this direction to be different at different locations on the same film of cobalt, and do so independently from other magnetic properties of the metal.

The team achieved this new capability by controlling an effect known as the Dzyaloshinskii-Moriya interaction (DMI), which imposes a preferred twist direction on spins. The DMI typically occurs at the boundary between a thin film of a magnetic metal and a nonmagnetic metal layer. The electron spins in the magnetic film interact with atoms in the nonmagnetic film, creating a preferential twist.



Schematic shows how different energies of argon atoms bombarding a thin film of cobalt can twist or rotate the spin of electrons in a particular direction. Credit: CNST.

Controlling the DMI can boost magnetic memory, which uses the orientation of spin to store information. A memory device needs two distinct states, representing either a one or a zero—in the case of a magnetic hard drive, electrons with spin pointing up or down. To write data, designers need a predictable way to flip from one spin orientation to the other. Controlling the direction and amount of twist could allow the spin flip to happen more efficiently and reliably than if the twist were random, Balk notes.

The DMI also plays a key role in another type of magnetic memory. If the DMI is strong enough, it will twist neighboring spins into a vortex pattern, and could potentially create exotic magnetic knots called skyrmions. These particle-like knots can store information, and their existence or absence in a magnetic thin film could act much like the ones and zeros of electronic logic circuits. By regulating the DMI, researchers can create skyrmions and guide the motion of these knots through a magnetic material. Skyrmions would require less power to operate than other types of magnetic memory.

The researchers describe their work in *Physical Review Letters*.

In their experiment, the researchers sandwiched a thin film of cobalt between two layers of platinum, a nonmagnetic metal. They then bombarded the trilayer with argon ions, which blasted away the top platinum film and roughened the top boundary between platinum and cobalt, depending on the ion energy.

The scientists discovered that when they used argon ions with an energy of 100 electron volts (eV), the DMI was negative, twisting the spins of the cobalt counterclockwise, and when they used argon ions with a lower energy of 50eV, the DMI was positive, and would twist the spins in the clockwise direction. When exposed to argon ions with an energy in between these values, the DMI was zero, making it equally likely that spins would twist clockwise or counterclockwise.

The researchers made their discovery while fine-tuning the magnetic properties of a cobalt film to develop an improved sensor for magnetic nanoparticles. In doing so, the team realized it had found a new way to manipulate the DMI.

The team discovered that the energy of the argon ions changed the DMI independently from another magnetic property, the strength of the magnetic field required to reverse the direction of the spins in the cobalt. This was unexpected, since both DMI and the reversal field are governed by similar interactions—the coupling of spins at the boundary between the cobalt and platinum layers.

Because argon ions with different energies could be aimed at specific regions within the cobalt, the researchers were able to fabricate cobalt films whose DMI varied across the surface of the material.

Finally, the scientists found that controlling the DMI did indeed make the film more sensitive to magnetic fields from single nanoparticles. Such measurement capability would benefit several types of research that rely on the CNST's facilities, including studies focused on finding the most effective ways to treat cancer using magnetic nanoparticles. At a later date, the team plans to publish work on applying the film as a nanoparticle sensor for facility users.

A. L. Balk, K-W. Kim, D. T. Pierce, M. D. Stiles, J. Unguris, and S. M. Stavis. Simultaneous control of the Dzyaloshinskii-Moriya interaction and magnetic anisotropy in nanomagnetic trilayers. *Phys. Rev. Lett.* **119**, 077205. Published 17 August 2017. DOI: *https://doi.org/10.1103/PhysRevLett.119.077205*

OPTICAL INTEGRATION REVOLUTIONIZES THE ATOMIC FORCE MICROSCOPE

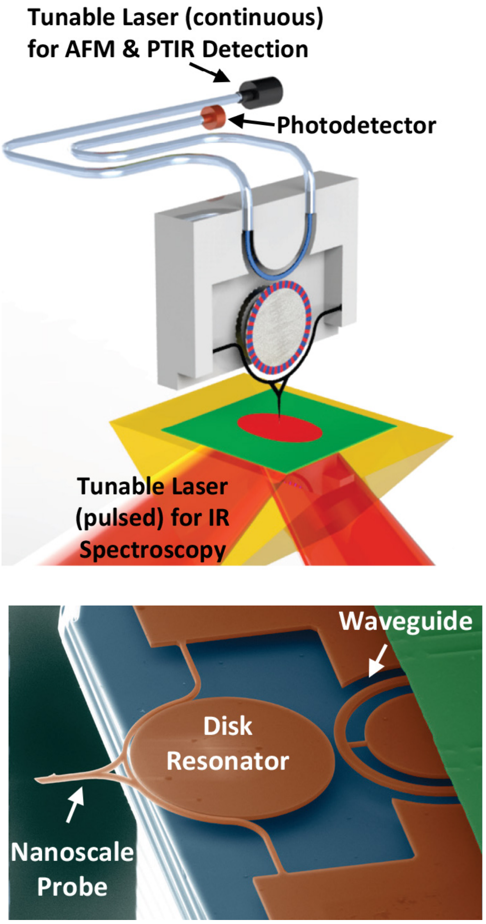
Most measuring instruments are limited by the tradeoff between how precisely and how rapidly a measurement is made: the more precise the measurement, the longer it takes. But because many phenomena occurring at the nanoscale are both rapid and tiny, their detection demands a measuring system that can record signals rapidly and with high precision.

Taking up that challenge, researchers at the CNST have redesigned the detection system at the heart of the atomic force microscope (AFM). A premiere tool of the nanoworld, the AFM uses a small probe, or tip, to map the minuscule hills and valleys that define the surface of materials, along with other properties. Although the AFM has already revolutionized the understanding of nanostructures, scientists are now eager to study nanoscale phenomena, such as protein folding or heat diffusion, which happen too quickly and generate changes too small to be accurately measured by existing versions of the microscope.

By fabricating an extremely lightweight AFM probe and coupling it to a nanoscale device that converts minuscule deflections of the probe into large changes of an optical signal inside a waveguide, the CNST researchers have made a groundbreaking advance: their AFM system measures rapid changes in structure with high precision.

The achievement takes the AFM into a new realm, enabling the instrument to measure time-varying nanoscale processes that may change on time scales as short as ten-billionths of a second. “This is truly a transformational advance,” says CNST scientist Andrea Centrone.

Centrone, Vladimir Aksyuk, and their colleagues employed the new AFM capabilities in experiments using photothermal induced resonance (PTIR), a technique that combines the acuity of an AFM with the ability to determine chemical composition provided by infrared spectroscopy.



Schematic (top) and micrograph (bottom) of the newly fabricated, extremely light-weight probe of an atomic force microscope integrated with a device that converts tiny motions of the probe into a light signal. Credit: NIST

With the new AFM-PTIR system, the scientists measured with high precision the rapid, but minute expansion of individual microcrystals heated by a light pulse. The microcrystals examined by the team belongs to a class of materials known as metal-organic frameworks (MOFs). These materials contain nanosized pores that act as miniature sponges, which can store gas and serve as drug delivery containers, among other applications.

Accurate knowledge of how well MOFs conduct heat is crucial for designing these materials for specific uses. However, most MOFs are microcrystals, which are too small for conventional instruments to measure their thermal conductivity. Instead, the team used the new AFM-PTIR system to record how long it took for the MOF crystals to cool down and return to their original size after they were heated by the light pulse and thermally expanded. The researchers then used that information to determine the thermal conductivity of individual MOF microcrystals, a feat that had never before been accomplished

The AFM system features two key elements. The team shrunk and slimmed down the AFM’s probe, a small cantilever that acts like a spring, deflecting and vibrating when a sample exerts a force on it. The new probe, fashioned at the CNST’s NanoFab weighs a mere trillionth of a gram. The minute mass enabled the probe to respond more quickly to a tiny force or displacement, such as the one induced by the thermal expansion of the MOF.

The researchers integrated the cantilever with a miniature disk-shaped cavity that acts like the optical version of a whispering gallery. Just as a whispering gallery allows certain frequencies of sound waves to travel freely around a dome, the cavity allows certain frequencies of light to resonate, circulating around the disk.

The AFM cantilever and the disk are separated by a mere 150 nanometers. That’s close enough that tiny motions of the cantilever change the resonant frequencies of the disk, in effect transforming the small mechanical motion of the AFM probe into a large change in an optical signal. The team’s system is the first to integrate this kind of optical devices in an AFM.

The researchers described the findings in a recent publication in *Nano Letters*.

Aksyuk and his collaborators painstakingly designed, fabricated and tested the system using an array of nanofabrication tools at the CNST. The new AFM-PTIR system can record a displacement as small as a trillionth of a meter that occurs over a timescale as short as ten billionths of a second. The team now plans to increase the speed of the PTIR technique and make measurements in water, a more suitable environment for examining biological samples.

Jungseok Chae, Sangmin An, Georg Ramer, Vitalie Stavila, Glenn Holland, Yohan Yoon, A. Alec Talin, Mark Allendorf, Vladimir A. Aksyuk, and Andrea Centrone. Nanophotonic Atomic Force Microscope Transducers Enable Chemical Composition and Thermal Conductivity Measurements at the Nanoscale. *Nano Lett.* Article ASAP DOI: 10.1021/acs.anolett.7b02404.

TO MAKE LARGER STRUCTURES WITH DNA, CNST SCIENTISTS FORTIFY IT WITH A PROTEIN

The DNA molecule is the stuff of life, but it is also the stuff of nanotechnology. Because strands of DNA with complementary chemical structure recognize and bind to one another, they can fit together like LEGOs to make nanoscale objects of complex shape and structure.

But researchers need to work with much larger assemblages of DNA in order to realize a key goal: using the genetic material to build durable, miniature devices such as biosensors and drug delivery containers. That's been difficult because long chains of DNA are floppy and the standard method of assembling them becomes increasingly error prone as structures become larger.

Now, using a DNA-binding protein called RecA as a kind of nanoscale rebar to reinforce the floppy DNA scaffolding, researchers at the CNST have constructed several of the largest tetragonal, rectangular and linear shapes ever assembled from strands of the DNA molecule. The structures can be two to three times larger than those attained using standard DNA self-assembly techniques.

In addition, because the new DNA-building method requires fewer chemically-distinct pieces to build organized structures than the standard technique, known as DNA origami, it is likely to reduce the number of errors in constructing the shapes. That's a big plus for the effort to produce reliable DNA-based devices in large quantities, says CNST researcher J. Alexander Liddle.

Although RecA's ability to bind to double-stranded DNA has been known for years, the NIST team is the first to integrate filaments of this protein into the assembly of DNA structures. The addition of RecA offers a particular advantage: once one unit of the protein binds to a small segment of double-stranded DNA, it automatically attracts other units that line up along side it, in the same way that bar magnets will join end-to-end.

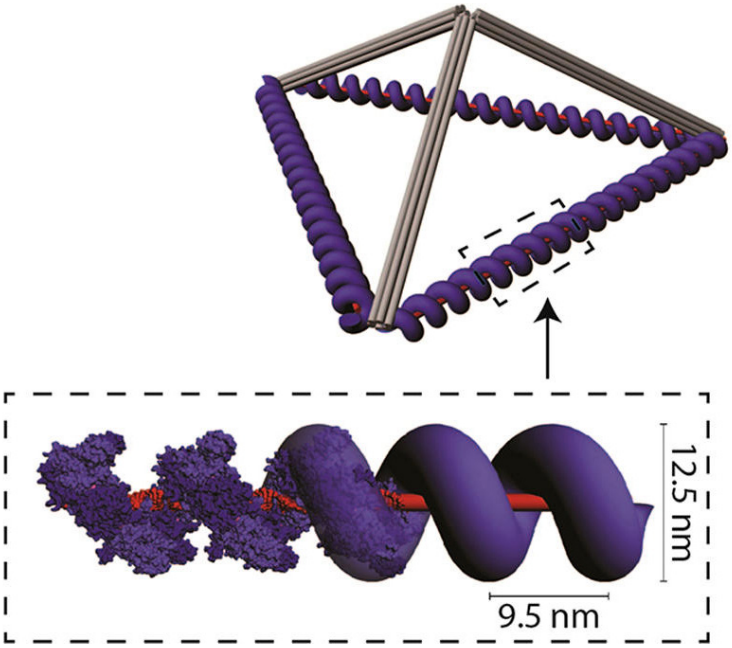


Illustration shows how the RecA protein (blue) wraps around, strengthens, and stretches double-stranded DNA (red line). Credit: CNST

Like bricks filling out a foundation, RecA lines the entire length of the DNA strand, stretching, widening and strengthening it. A floppy, 2 nanometer-wide strand of DNA can transform into a rigid structure more than four times as wide.

"The RecA method greatly extends the ability of DNA self-assembly methods to build larger and more sophisticated structures," says Daniel Schiffels of the CNST.

Schiffels, Liddle and their colleague Veronika Szalai describe their work in a recent article in *ACS Nano*.

The new method incorporates but goes beyond the DNA origami technique, according to Liddle. In DNA origami, short strands of DNA that have a particular chemical signature—a specific sequence of its four base pairs—are used as staples to tie together, or fold up, a long string of DNA, binding to sections that have the complementary sequence of base pairs. To make the skinny DNA skeleton stronger and thicker, the strand may have to loop back on itself many times, quickly using up the long string.

If DNA origami is all about the folding, Liddle likened his team's new method to building a room, starting with a floor plan. The location of the short, single-stranded pieces of DNA that act as staples mark the corners of the room. Between the corners lies a long, skinny piece of single-stranded DNA. The enzyme DNA polymerase transforms a section of the long piece of single-stranded DNA into the double-stranded version of the molecule, a necessary step because RecA binds best to double-stranded DNA. Then RecA assembles all along the double strand, reinforcing the DNA structure and limiting the need for extra staples to maintain its shape.

With fewer staples required, the RecA method is likely to build organized structures with fewer errors than DNA origami, Liddle says.

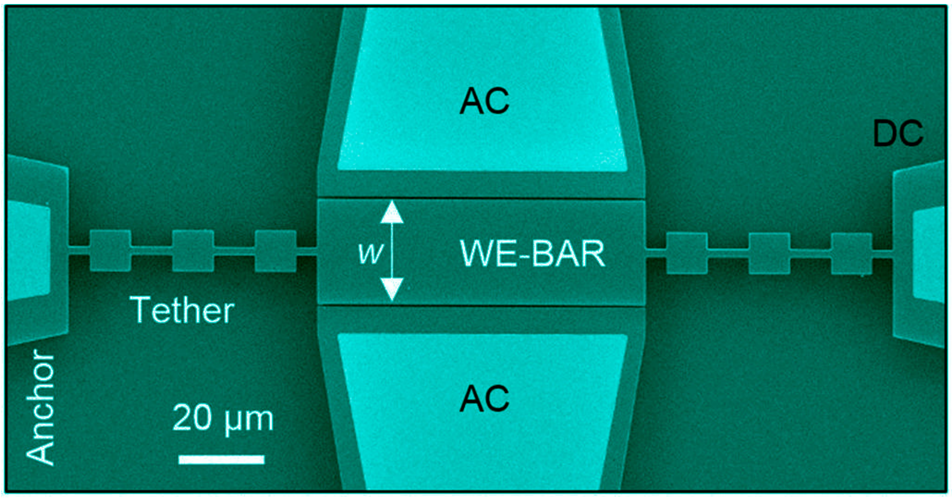
D. Schiffels, V. Szalai and J. A. Liddle. Molecular Precision at Micrometer Length Scales: Hierarchical Assembly of DNA-Protein Nanostructures. *ACS NANO*: 2017 Jul 25; **11** (7):6623-6629.
DOI: 10.1021/acsnano.7b00320

NEW DESIGN FABRICATED AT CNST MAKES MICRO-CLOCK RESONATORS RING LIKE A BELL

You can't hear most of them, but the world is running on different kinds of mechanical oscillations. For example, inside the average electronic wristwatch is a sealed canister containing a three-millimeter-long quartz crystal. In response to electrical feedback, the crystal continuously vibrates at about 33,000 times per second. The remarkable stability of that resonance frequency, which provides the clock's "tick" rate, keeps you on time.

"But in today's rush to make smaller, lighter devices, space on the printed circuit board is very valuable, and quartz crystals are large, expensive, and fragile," says Jason Gorman of NIST's Physical Measurement Laboratory. "So, over the past 10 years there has been a push to make microscale clocks, with a focus on silicon resonators. The goal is to develop microscale clocks that surpass quartz clocks in performance while also being 1/100th the size and using a fraction of the power."

In pursuit of that goal, Gorman and colleague Vikrant Gokhale—using structures fabricated at the CNST that are no larger than one-fifth the width of a human hair—have devised and tested a novel method that substantially improves silicon resonator performance, and may also benefit many different types of sensors. The scientists published their results recently in *Applied Physics Letters*.



A conventional design employing single straight beams. The sections labeled WE-BAR are the acoustic resonators. Credit: NIST

Clocks require some mechanism that oscillates (ticks) at almost exactly the same rate and strength over time, whether it is a swinging pendulum or atoms absorbing and releasing photons. A resonator's ability to do so precisely is directly related to its quality factor (*Q*). A high-*Q* resonator is one that stays close to a single frequency and bleeds off very little energy to its surroundings; its signal remains strong and stable over time.

In microscale devices—fabricated at dimensions measured in micrometers—a key factor for the achievable *Q* is the amount of vibrational energy absorbed by the tiny buttresses or "tethers" that suspend the resonator from the supporting substrate. Tethers are designed to reflect as much of the vibrational energy as possible back to the resonator, minimizing dissipation. The standard configuration for a tether is just a straight beam of solid silicon.

Recently, other researchers have employed tethers with more complex structure based on repeating geometry. Depending on the optimization of this geometry, these tethers can allow only certain frequencies of quantized vibrations called phonons to pass through while reflecting others back. Thus, an ideal "phononic crystal" (PnC) tether would reflect the resonator's resonance frequency, while transmitting others. "Since more vibrational energy is confined within the resonator due to reflections off of the phononic crystal, the quality factor is expected to improve compared to straight-beam tethers," says Gokhale.

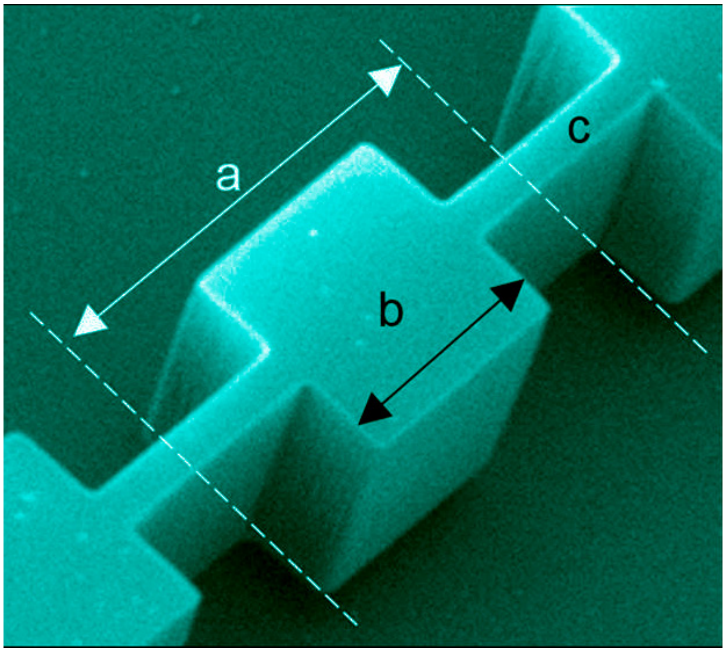
Early experiments with PnCs in different tether configurations by others showed that the quality factor could be improved by as much as a factor three. However, other energy dissipation mechanisms, such as stress at the interfaces between multiple materials and thermoelastic dissipation in metal electrodes, dominated the quality factor in the piezoelectric* resonators used in these tests.

"We decided to take that further," Gorman says. "We knew that by developing a resonator made of a single material, silicon in this case, we could get rid of most of the other dissipation mechanisms that limit the quality factor." Doing so reduced the dissipation to just a few effects that are unavoidable and small compared to the energy dissipation typically resulting from tethers.

At the CNST, Gokhale used a plasma-etching tool to make silicon tether arrays in rows containing one, three, or five PnC "cells." The resonator sidewalls had to be straight and smooth to minimize energy dissipation .In addition, the gap between the resonator and the electrodes that drive the resonator must be as small as 500 nanometers to generate an adequate electrostatic force. To meet these requirements, the researchers etched in small steps (approximately 80 nm), resulting in resonators fabricated with nanoscale accuracy and sidewall roughness smaller than 15 nm.

Gorman and Gokhale determined that larger numbers of the tethers increased the reflectance, and thus improved the *Q*. The results not only far surpassed the performance of conventional tether bars, but approached the fundamental limit of intrinsic dissipation for the material, achieving higher *Q* than ever before recorded for silicon at a resonance frequency above 100 MHz.

In addition to micromechanical clocks, this work may impact a number of sensor approaches based on resonators. "Resonant sensors are commonly used for sensitive measurements of acceleration, rotation, force, and mass changes, and the sensitivity is proportional to the achievable *Q*," says Gorman.



Tether configuration consisting of three cells in NIST's phononic crystal design. Credit: NIST

As an example, resonant chemical sensors rely on the fact that a resonator's center frequency depends on its mass. If a molecule of some kind—such as a pollutant—hits the resonator and sticks there, it changes the resonance frequency. The amount of change depends on the mass of the molecule, allowing users to determine the chemical species. "High *Q* matters in sensors because it improves the sensitivity to changes in the resonance frequency when a stimulus is applied to the resonator," Gokhale says. New sensor technologies based on the resonator with phononic crystal tethers are now being pursued.

* *Certain materials—including the quartz crystal in an electronic clock—respond to mechanical stress by generating electric voltage. Conversely, they will vibrate when an oscillating voltage is applied to them. Because piezoelectric resonators typically have metal components, there are more possible sources of energy dissipation than a resonator of monocrystalline silicon.*

Vikrant J. Gokhale and Jason J. Gorman. Approaching the intrinsic quality factor limit for micromechanical bulk acoustic resonators using phononic crystal tethers. *Appl. Phys. Lett.* **111**, 013501 (2017);
DOI: http://dx.doi.org/10.1063/1.4990960

QUANTUM THERMOMETER OR OPTICAL REFRIGERATOR?

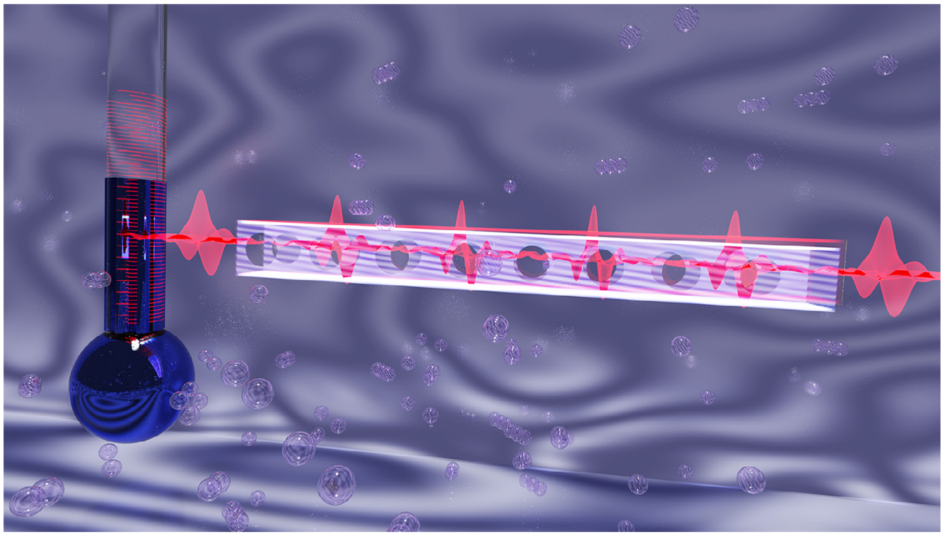
In an arranged marriage of optics and mechanics, physicists have created microscopic structural beams that have a variety of powerful uses when light strikes them. Able to operate in ordinary, room-temperature environments, yet exploiting some of the deepest principles of quantum physics, these optomechanical systems can act as inherently accurate thermometers, or conversely, as a type of optical shield that diverts heat. The research was performed by a team led by the Joint Quantum Institute (JQI) , a research collaboration of the National Institute of Standards and Technology (NIST) and the University of Maryland.

Described in a pair of new papers in Science and *Physical Review Letters*, the potential applications include chip-based temperature sensors for electronics and biology that would never need to be adjusted since they rely on fundamental constants of nature; tiny refrigerators that can cool state-of-the-art microscope components for higher-quality images; and improved "metamaterials" that could allow researchers to manipulate light and sound in new ways.

Made of silicon nitride, a widely used material in the electronics and photonics industries, the beams are about 20 microns (20 millionths of a meter) in length. They are transparent, with a row of holes drilled through them to enhance their optical and mechanical properties.

"You can send light down this beam because it's a transparent material. You can also send sound waves down the beam," explained Tom Purdy, a NIST physicist who is an author on both papers. The researchers believe the beams could lead to better thermometers, which are now ubiquitous in our devices, including cell phones.

"Essentially we're carrying a bunch of thermometers around with us all the time," said JQI Fellow Jake Taylor, senior author of the new papers. "Some provide temperature readings, and others let you know if your chip is too hot or your battery is too cold. Thermometers also play a crucial role in transportation systems—airplanes, cars—and tell you if your engine oil is overheating."



Artist's rendition of a quantum thermometer, a micron-scale mechanical device that can provide highly accurate temperature. Credit: Emily Edwards/Joint Quantum Institute

But the problem is that these thermometers are not accurate off the shelf. They need to be calibrated, or adjusted, to some standard. The design of the silicon nitride beam avoids this situation by relying on fundamental physics. To use the beam as a thermometer, researchers must be able to measure the tiniest possible vibrations in the beam. The amount that the beam vibrates is proportional to the temperature of its surroundings.

The vibrations can come from two kinds of sources. The first are ordinary "thermal" sources such as gas molecules buffeting the beam or sound waves passing through it. The second source of vibration comes purely from the world of quantum mechanics, the theory that governs behavior of matter at the atomic scale. The quantum behavior occurs when the researchers send particles of light, or photons, down the beam.

Struck by light, the mechanical beam reflects the photons, and recoils in the process, creating small vibrations in the beam. Sometimes these quantum-based effects are described using the Heisenberg uncertainty relationship: The photon bounce leads to information about the beam's position, but because it imparts vibrations to the beam, it adds uncertainty to the beam's velocity.

"The quantum mechanical fluctuations give us a reference point because essentially, you can't make the system move less than that," Taylor said. By plugging in values of Boltzmann's constant and Planck's constant, the researchers can calculate the temperature. And given that reference point, when the researchers measure more motion in the beam, such as from thermal sources, they can accurately extrapolate the temperature of the environment.

However, the quantum fluctuations are a million times fainter than the thermal vibrations; detecting them is like hearing a pin drop while taking a shower.

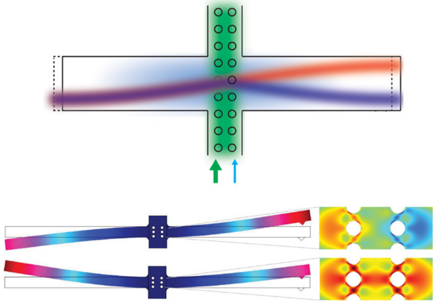
In their experiments, the researchers used a state-of-the-art silicon nitride beam built by Karen Grutter and Kartik Srinivasan at the CNST. By shining high-quality photons at the beam and analyzing photons emitted from the beam shortly thereafter, "we see a little bit of the quantum vibrational motion picked up in the output of light," Purdy explained.

Their measurement approach is sensitive enough to see these quantum effects all the way up to room temperature for the first time. He and his colleagues reported their work in a recent issue of *Science*.

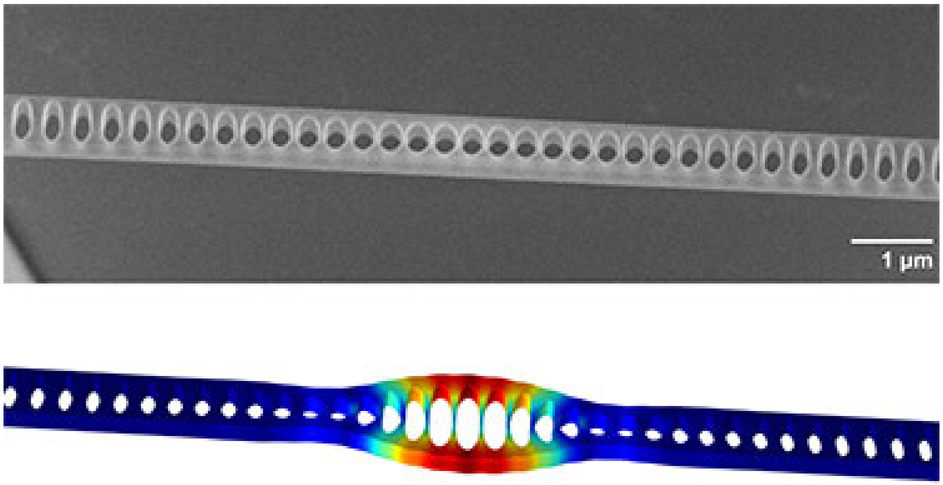
Although the experimental thermometers are in a proof-of-concept phase, the researchers envision they could be particularly valuable in electronic devices, as on-chip thermometers that never need calibration, and in biology.

"Biological processes, in general, are very sensitive to temperature, as anyone who has a sick child knows. The difference between 37 and 39 degrees Celsius is pretty large," Taylor said. He foresees applications in biotechnology when you want to measure temperature changes in "as small an amount of product as possible," he said.

The researchers go in the opposite direction in a second proposed application for the beams, described in a theoretical paper published in *Physical Review Letters*.



Schematic (top) and simulation (bottom) of an optomechanical device that takes advantage of optical cooling. Light travels through the series of holes with a chance to reflect back towards its source that depends upon the motion of the two 'arms' of the mechanical system. Laser cooling damps the motion in which the arms move in opposite directions, which improves the performance of the symmetrical moving mode (arms in synchrony). This symmetrical mode is, in turn, modified by the interaction of the small tip with a nearby surface, enabling optical detection of the surface properties in a standard atomic force microscope setup. Credit: Xu et al./ NIST and University of Maryland



At top is an electron micrograph of a silicon nitride beam, which can act as a highly accurate "quantum thermometer" through measurements of its tiniest vibrations. The bottom shows how the beam deforms as it vibrates (length scale greatly exaggerated) with the red regions showing the most deformation, and the blue regions not moving at all. Credit: Purdy et al., NIST/JQI

Instead of letting heat hit the beam and allow it to serve as a temperature probe, the researchers propose using the beam to divert the heat from, for example, a sensitive part of an electromechanical device.

In their proposed setup, the researchers enclose the beam in a cavity, a pair of mirrors that bounce light back and forth. They use light to control the vibrations of the beam so that the beam cannot re-radiate incoming heat in its usual direction, toward a colder object.

For this application, Taylor likens the behavior of the beam to a tuning fork. When you hold a tuning fork and strike it, it radiates pure sound tones instead of allowing that motion to turn into heat, which travels down the fork and into your hand.

"A tuning fork rings for a long time, even in air," he said. The two prongs of the fork vibrate in opposite directions, he explained, and cancel out a way for energy to leave the bottom of the fork through your hand.

The researchers even imagine using an optically controlled silicon nitride beam as the tip of an atomic force microscope (AFM), which detects forces on surfaces to build up atom-scale images. An optically controlled AFM tip would stay cool—and perform better. "You're removing thermal motion, which makes it easier to see signals," Taylor explained.

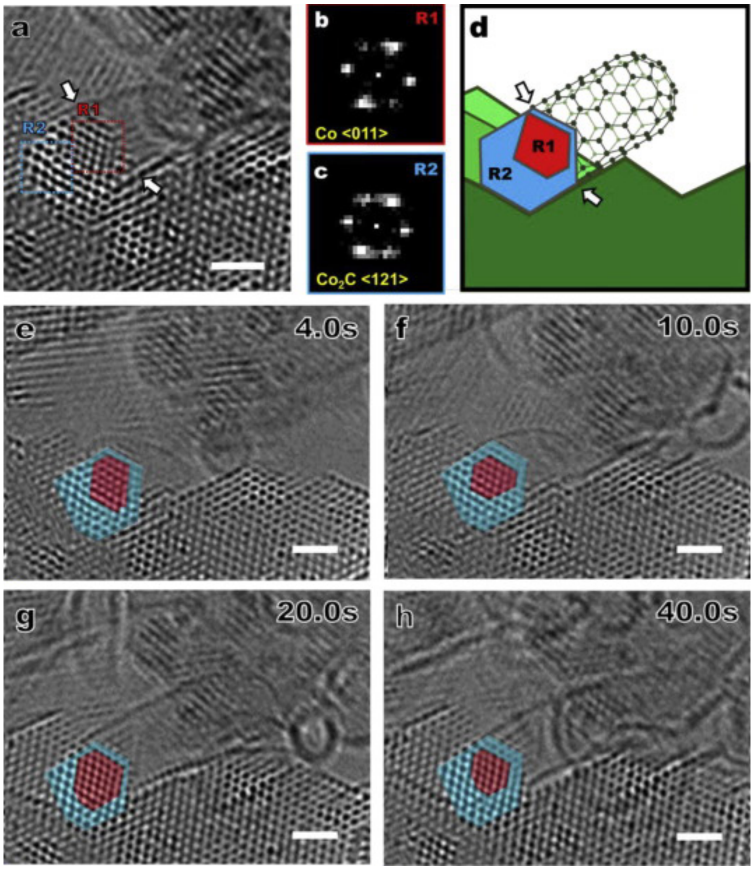
This technique also could be put to use to make better metamaterials, complex composite objects that manipulate light or sound in new ways and could be used to make better lenses or even so-called "invisibility cloaks" that cause certain wavelengths of light to pass around an object rather than bouncing from it.

"Metamaterials are our answer to the question, 'How do we make materials that capture the best properties for light and sound, or for heat and motion?'" Taylor said. "It's a technique that has been widely used in engineering, but combining the light and sound together remains still a bit open on how far we can go with it, and this provides a new tool for exploring that space."

T.P. Purdy, K.E. Grutter, K. Srinivasan and J.M. Taylor. Quantum correlations from a room temperature optomechanical cavity. *Science*. Published June 23, 2017. DOI: 10.1126/science.aag1407

X. Xu, T. Purdy and J.M. Taylor. Cooling a harmonic oscillator by optomechanical modification of its bath. *Physical Review Letters*. Published May 31, 2017. DOI: 10.1103/PhysRevLett.118.223602

VIDEO REVEALS ATOMIC-SCALE WORKINGS OF NANOTUBE CATALYST



Direct evidence of atomic-scale structural fluctuations in catalyst nanoparticles.
Credit: NIST

CNST researchers have made some of the first movies of the structural changes that tiny catalyst particles undergo to help build materials for miniature electronic circuits and other nanotech devices. The movies may suggest new ways to custom-design the catalyst nanoparticles, leading to more efficient production and improved control of the materials they help fabricate.

Catalysts are critical for many chemical reactions because they provide speedier, lower-energy pathways to break and make chemical bonds necessary for the reactions to proceed. Catalyst nanoparticles are a mainstay for such industrial processes as converting crude oil to gasoline and growing single-walled carbon nanotubes—superstrong arrangements of carbon atoms.

To build a better catalyst nanoparticle, researchers need a detailed understanding of the chemical pathways involved in the catalytic process, says Renu Sharma of the CNST. “If you want to make a good catalyst, you want to know why it works and why it doesn’t work, and it’s often changes in the atomic structure of the particle that hold the key to that understanding,” Sharma says.

To gain that knowledge, she and her colleagues relied on an innovative electron microscope and a high-resolution camera to track in real time the chemical evolution of cobalt nanoparticles as they acted as catalysts for growing single-walled carbon nanotubes. Although cobalt plays an important role in forming the nanotubes, “the question has always been how does this catalyst actually work,” says Sharma.

Sharma and her colleagues, including colleagues from the University of Maryland, and Texas A&M University, described their findings in a recent issue of the *Journal of Catalysis*.

Scientists have often assumed that a catalyst for making carbon nanotubes functions by attracting precursor gas molecules to its surface, where it’s easier to break and forge bonds. But Sharma’s team discovered that in the case of the cobalt nanoparticle catalyst, the entire particle is involved, not just its surface.

In the experiment, the team used acetylene (C_2H_2) as a source of carbon to build the nanotubes. The researchers found that some of the carbon, instead of directly making carbon nanotubes at the surface of the cobalt nanoparticle, diffused into the nanoparticle. Inside the nanoparticle, the diffused carbon temporarily formed cobalt carbide. This was confirmed by atomic-resolution images taken with an electron microscope, which showed that within a single catalyst nanoparticle, cobalt and cobalt carbide co-existed.

Eventually, the cobalt carbide decomposed back into cobalt and carbon. Only this carbon contributed to the formation of the nanotubes. The study revealed for the first time that the fluctuating amount of carbon within the catalyst nanoparticles mirrored the fluctuating rate at which the carbon nanotubes grew. Simulations of the molecular process corroborated the finding.

Now that the team has documented the atomic-scale dynamics of the cobalt catalyst nanoparticles, further studies will be needed to determine how to apply that knowledge to more efficiently form carbon nanotubes, Sharma says. In the meantime, the approach adopted by the researchers—combining real-time, atomic-resolution imaging with molecular dynamics simulations—can be applied to understanding and improving the function of a wide array of catalysts as well as fine-tuning the growth of the structures they help produce.

Pin Ann Lin, Jose L. Gomez-Ballesteros, Juan C. Burgos, Perla B. Balbuena, Bharath Natarajan, Renu Sharma. Direct evidence of atomic-scale structural fluctuations in catalyst nanoparticles. *Journal of Catalysis*, **Vol. 349**, May 2017, pages 149–155. DOI: 10.1016/j.jcat.2017.03.009

JOSEPH A. STROSCIO TO RECEIVE HEINRICH ROHRER GRAND MEDAL

The Surface Science Society of Japan has awarded CNST physicist and NIST fellow Joseph A. Stroscio the 2017 Heinrich Rohrer Medal-Grand Medal, named for the late Nobel Laureate physicist Heinrich Rohrer. In announcing the award, which recognizes researchers who have made top-level achievements in the fields of nanoscience and nanotechnology, the Society cited Stroscio "for his pioneering and ground-breaking achievements on the spectroscopic capability of scanning tunneling microscopy leading to deep insight into the physics of nanostructures and opening novel perspectives for revealing the quantum nature of the nanoworld."

Stroscio has been a leading scientist in the surface/nanoscience field for more than 30 years. His major contribution was one of the first measurements of scanning tunneling spectroscopy, which showed that the scanning tunneling microscope (STM) does not simply image atoms, but measures the energy-resolved electronic wavefunction probability distributions. He demonstrated this with elegant measurements of the Si(111)2x1 surface, showing that the atomic-like features undergo a 180° phase reversal when imaging the Si(111)2x1 π-bonded chain structure at energies below and above the Si(111)2x1 band gap. In this work, Stroscio introduced the concept of the normalized differential tunneling conductance as a measure of the energy-resolved local density of electronic states and showed its correspondence to the Si(111)2x1 surface state band structure.

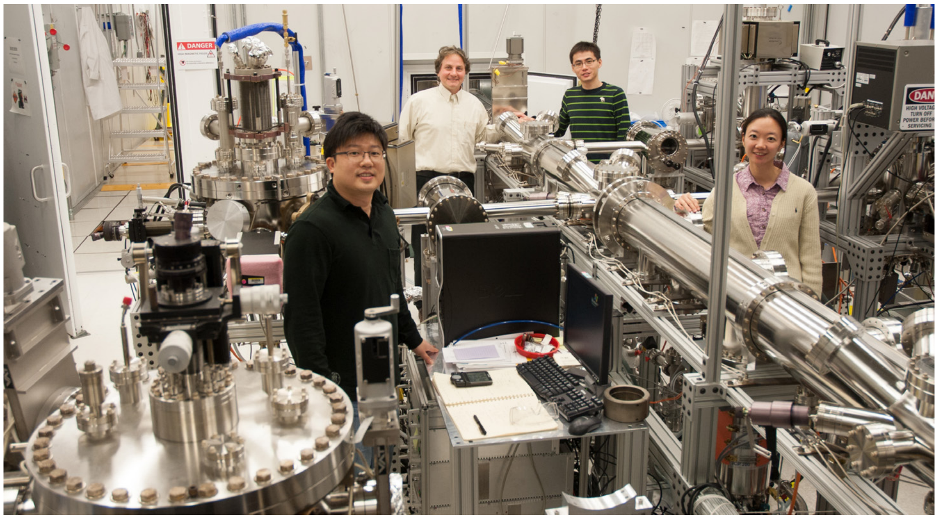
STM thus became an atomic-resolution tool with the combined power of the transmission electron microscope and photoemission. Taking advantage of the knowledge he gained, Stroscio performed the first tunneling measurements that demonstrated chemical selective imaging. He performed those measurements on the GaAs(110) surface, showing how to tune and image Ga versus As atom electronic states. In addition, he demonstrated how adsorbates on semiconductor surfaces effect band-bending and screening on an atomic scale.

Credit: Dill/CNST

Stroscio's other landmark contributions to surface/nanoscience include the discovery that individual atoms could be manipulated with the STM. He explained how "sounds" in the tunneling current during single atom manipulation result from telegraph noise in the tunneling current during atom manipulation. In this work, he showed how tunneling noise spectroscopy could be used to probe the potential energy surfaces for adatoms. This finding ushered in an unparalleled era of discovery about the nanoworld. His passion for developing atomic scale instrumentation led to the construction of tunneling microscopes operating at milli-Kelvin temperatures, dramatically improving the energy resolution down to tens of μeV. By developing these more advanced instruments. Stroscio resolved both spin and valley degeneracies in tunneling spectroscopy of graphene at mK temperatures. In his most recent work with the graphene system, he created *pn* junction resonators, which led to Stroscio's discovery of the *whispering gallery modes* of Dirac fermions resulting from Klein scattering. He also discovered a new quantum *Berry phase switch* of resonator states in graphene, using advanced spectroscopic measurements he developed in his laboratory.

His great contributions in scanning tunneling spectroscopy, atom manipulation and ultra-high energy resolution scanning tunneling microscopy have made scanning tunneling microscopy and atomic force microscopy the most important structural and spectroscopic tools with atomic resolution and μeV energy resolution, leading to many discoveries in surface science and nanotechnology.

The award, established in 2013 by the Surface Science Society of Japan in collaboration with IBM Research - Zurich, the *Swiss Embassy in Japan* and Ms. Rohrer, will be presented this October at the 8th International Symposium on Surface Science in Ibaraki, Japan.



View of Stroscio's millikelvin STM Lab at the CNST. Credit: Dill/CNST

CNST Center for Nanoscale Science & Technology

CENTER FOR NANOSCALE SCIENCE AND TECHNOLOGY

The CNST is a national user facility purposely designed to accelerate innovation in nanotechnology-based commerce. Its mission is to operate a national, shared resource for nanoscale fabrication and measurement and develop innovative nanoscale measurement and fabrication capabilities to support researchers from industry, academia, NIST and other government agencies in advancing nanoscale technology from discovery to production. The Center, located in the Advanced Measurement Laboratory Complex on NIST's Gaithersburg, MD campus, disseminates new nanoscale measurement methods by incorporating them into facility operations, collaborating and partnering with others and providing international leadership in nanotechnology.

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SUPPORTING THE DEVELOPMENT OF NANOTECHNOLOGY FROM DISCOVERY TO PRODUCTION

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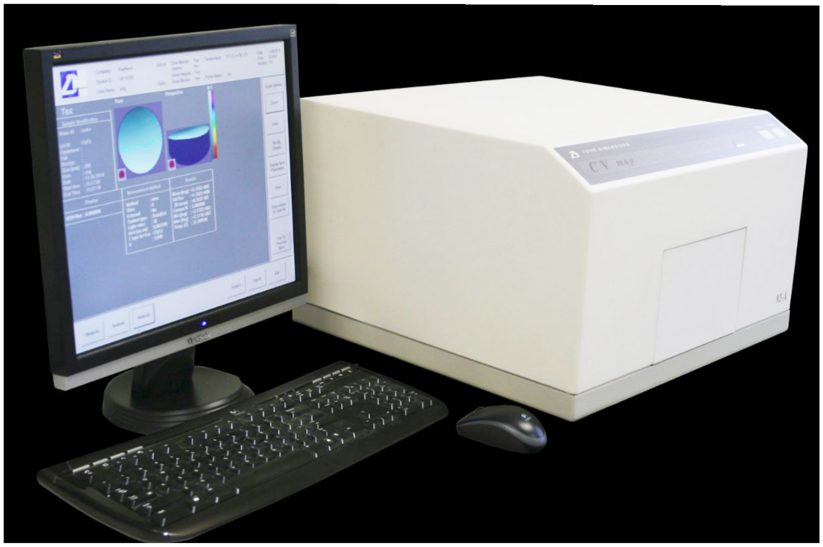
NEW IN THE NANOFAB

Mercury Probe Mapping System

By October, NanoFab users will have access to a mercury probe, a tool to make noninvasive and rapid measurements of the electrical properties of thin dielectric films. The measurements enable researchers to derive the resistivity, breakdown voltage and thickness of the dielectric films. The system uses a drop of mercury that acts as an electrical contact for a small, well-defined area of the film. The mercury contact enables researchers to make electrical measurements without having to coat the film with a thin layer of metal and fabricate capacitors, as more traditional measurement systems require.



The chemical mechanical polisher/post polisher system. Credit: Axus Technology

Polisher and Post-Polisher

Next February, the NanoFab will introduce two related tools—a chemical mechanical polisher (CMP) and a wafer cleaner for use after the polishing process. The automated CMP, which can treat wafers from 75 to 200 millimeters in diameter, ensures that wafers have a flat surface by polishing off any surface features. The CMP features a two-step process, with a primary platen to remove material and a secondary platen to perform the final polishing and minimize surface roughness. The polisher produces uniform, planar surfaces with deviations from flatness that are smaller than 3 percent.

The post-CMP wafer cleaner uses a mild ammonium hydroxide solution, brushing action and megasonic agitation to gently scrub away slurry particles remaining after polishing.