Silicon Cantilever Measures Attonewton Forces

During the American Physical Society meeting in Kansas City on March 17, Timothy Stowe, a graduate student at Stanford University, described an ultrasensitive cantilever that he and a team of researchers from Stanford and IBM's Almaden Research Center have developed to measure attonewton forces. The measurements were made using a microscopic cantilever being developed for a magnetic resonance force microscope (MRFM), which is still under development. The MRFM combines the scanning tunneling microscope's ability to image individual atoms with magnetic resonance imaging's capability of telling one kind of atom from another.

Like standard MRI, the MRFM technique uses a radio-frequency (RF) coil to excite magnetic resonance in a sample. Like conventional MRI, the MRFM can be tuned to interact only with specific types of atoms by varying the frequency of the radio waves. Rather than using the same coil to detect the magnetic signals coming from the atoms, however, the MRFM relies on a microscopic cantilever with a magnetized tip.

The force between the tip and sample oscillates as the RF coil periodically reverses the polarity of the atoms in the sample. This, in turn, causes the cantilever to vibrate. An optical fiber interferometer records the cantilever's movements. As the tip is scanned over a surface, it responds to magnetic signals coming from specific atoms within an annulus on the sample's surface. By recording variations in the amplitude of the cantilever's vibration at different positions, the scientists are able to make a three-dimensional map of the position of the resonant atoms.

So far, the MRFM has taken images at the micron scale. Dan Rugar, IBM Nanoscale Studies manager, said, "One key to extending MRFM capability to the atomic scale is the ability to detect forces at the attonewton level. This is the motivation for developing the new ultrasensitive cantilevers."

The cantilever measures 230 μm long, 5 μm wide and less than 600 Å thick. The team made these micro-devices by starting with silicon-on-insulator, which consists of an ultrathin layer of silicon bonded onto a layer of silicon oxide coating a silicon wafer. They etched cantilever shapes out of the ultrathin silicon layer and then chemically removed the underlying layers of oxide. They chose silicon in order to minimize the mechanical damping that occurs when the cantilever vibrates, and because it is compatible with semiconductor processing techniques.

The researchers found that they could not use normal drying techniques after the final fabrication step. The surface tension of the water is strong enough so that as it dries it bends the cantilevers into "U" shapes, causing them to reattach to the underlying material. The team circumvented the problem by employing the technique called critical point drying, which uses liquid carbon dioxide at high pressures.

Sculptured 3-D Thin Films Fabricated

Researchers at The Pennsylvania State University and the University of Alberta, Edmonton, have devised a deposition scheme for creating nanometer-scale three-dimensional sculptured thin films.

"We've built [sculptured thin films] from four elementary microstructures: matchsticks, zigzags, bent nematics, and coils," said Penn State researcher Akhlesh Lakhtakia.

Sculptured thin films are formed by tilting a substrate at a highly oblique angle to the incident flux of a deposition source and rotating the substrate perpendicular to its surface. Because of shielding effects, the films formed are relatively porous. They consist of material columns about 30–100 nm in width and spaced 100–500 nm apart, taking up about 10–30% of the film space. Films 10–1,000 nm thick can be formed.

Researchers at Penn State evaporated 99.9% pure MgF_2 in an e-beam source in a vacuum chamber evacuated to 2×10^{-7} Torr. Using a quartz crystal deposition rate controller, the researchers controlled the deposition rate at the substrate in the perpendicular position to the vapor direction at 2.0 nm/s. They set the source-to-substrate distance at 25 cm. They deposited the sculptured thin films onto flat substrates of silicon and glass. The samples measured by scanning electron microscopy (SEM) were coated with a very thin layer of gold to prevent sample charging. The thin films of MgF_2 deposited onto substrates at room temperature produced "columnar morphologies ranging from chevron structures to continuously varied structures resembling S-shapes and C-shapes," according to the researchers.

Films consisting of differing elementary microstructures and materials can be stacked to form composite structures, such as in optical circuits.

Applications envisioned for these materials include substrates for new catalyst systems, electroluminescence devices, and *in vivo* biological-tissue-growth devices.

Ultrasound Creates Metal Powders

Chemists at the University of Illinois at Urbana-Champaign have used sonochemistry to make extremely fine metal powders. The highly magnetic powders have potential uses in information storage, audio reproduction, and magnetic sealing.

"These small magnetic metal clusters have attracted great scientific interest because they behave as superparamagnets," Suslick said. "In superparamagnetic materials, the magnetic moments consist of clusters of atomic spins in which all the spins are aligned in the same direction. Each cluster behaves like a separate magnet, and magnetic moments of these clusters are more than 100 times higher than normal material. He said that in the presence of molecules that will coat these particles, stable iron colloids are formed in which the metal particles stay suspended indefinitely. As reported in the November 27 issue of *Journal of the American Chemical Society*, the clusters contain a few hundred atoms and are a few nanometers in diameter.

Colloidal suspensions of fine magnetic particles are commercially produced as ferrofluids and have a wide range of applications. "Commercial ferrofluids are generally produced by exhaustively grinding iron oxide particles for many weeks," Suslick said. "By comparison, our new sonochemical synthesis technique is both simple and quick."

Type-II Quantum Cascade Laser Developed

A team of researchers from the University of Houston Space Vacuum Epitaxy Center (SVEC) and Sandia National Laboratories has developed a type-II quantum cascade laser which has the potential to operate at a higher temperature and deliver more power at the 3–12 μm wavelength than previous diode laser technology.

As reported on February 12 at the Photonic West Conference at San Jose, the type-II quantum cascade laser combines the advantages of a cascade design and the interband transitions of the conventional diode laser.

The laser is based on the type-II heterostructure of which the conduction-band edge of the InAs layer can be lower in energy than the valence-band edge of the GaInSb/GaSb adjacent layer.

This energy-band alignment allows a staircase arrangement of the laser's active regions. Every time a carrier jumps down an energy step in the staircase as it crosses an active region, a photon is emitted.

Previous InGaAs/InAlAs quantum cascade lasers are based on relaxations between two electron energy states, intersubband transitions, which are less efficient than the type-II quantum cascade utilizing relaxations from an electron to a hole state, interband transitions.

The type-II quantum cascade laser emits mid-infrared light around 4 μm at temperatures up to -100°C under 100-ns current pulses at a repetition rate of 1,000/s. The average output power at the ambient temperature is 140 μW, which is much brighter than the commercially available LEDs at the same wavelength.

Cardanol Blends with Latex to Create Durable Rubber

Vu-Thi-Yen, a graduate student at the University of Cincinnati (UC), has developed rubber made from renewable sources that is more durable than oil-based rubber. According to Vu, rubber made from latex is easily degraded from sunlight. "Natural rubber needs an anti-oxidation agent to prevent it from degrading in sunlight and air," she said.

Vu heated cashew shells, producing a sticky resin known as cardanol. In her first set of experiments, Vu purified samples of cardanol she brought to UC from Vietnam and blended them with latex to see if the rubber would be more durable. The samples turned out to be stronger and longer-lasting than latex alone.

In addition to cardanol, Vu is also making blends using castor oil. She said that castor oil is very different chemically from cardanol, so she expects it to have a different effect on the properties of rubber.

Corn By-Product Functions as Biodegradable Packaging Material

A mix of zein—a corn by-product—and fatty acids, coated in flax oil, has created water-resistant, plasticlike food containers with the potential to be biodegradable. Zein is extracted from corn gluten meal left over after ethanol manufacture and from dry milling waste. Only a small portion of the zein currently available is used commercially in the formulation of specialty coatings for candies, rice, dried fruits, and pharmaceutical tablets. In a paper published in the January/February issue of *Cereal Chemistry*, researchers at the University of Illinois at Urbana-Champaign reported on how they turned zein into plasticized resins that can be molded into plates, sandwich containers, sealing material, and trays.

Scientist Graciela Wild Padua said, "Part of the problem in using zein before now has been its price. If you try to develop products out of commercial zein that is available, it wouldn't make financial sense. We need to be able to obtain zein in a greater quantity."

As part of the overall effort, the scientists have developed a process to manufacture biodegradable plastics from corn gluten meal. The process involves melting the zein with fatty acids to produce moldable resins. When disposed of, the plasticized zein is expected to degrade naturally, acting as a slow-release nitrogen source for soil, Padua said.

According to the report, the scientists turned the zein into thin plasticlike sheets by mixing it with palmitic or stearic acids at varying weight ratios. While the structure of the sheets changed according to the ratios, the sheets with the zein palmitic acid mixture showed a substantially stronger tensile strength.

Without the plasticizing acids, zein sheets were brittle and had higher water-absorption qualities. Zein sheets without the fatty acids lacked a definitive structure and showed cracking and voids under a microscope. The strongest, most watertight zein sheets were those containing a mixture of palmitic acid and fatty acids and coated with heated flax oil.

Microelectrode Measures Various Components of Marine Environment

With a tip 25 μm in diameter, a microelectrode developed at the University of Delaware measures key components of marine environments—including dissolved oxygen, iron, manganese, hydrogen sulfide, and iodide—simultaneously. According to the researchers' report on March 11 during the National Association of Corrosion Engineers (NACE) meeting, the microelectrode can be used to examine the corrosive ocean biofilms that damage boats, docks, and off-shore platforms.

Oceanographer professor George W. Luther, III invented the probe by inserting a tiny gold wire, plated with mercury, into the center of a very thin-walled glass tube 200 μm in diameter and about 4 cm long. Undesirable chemical species were then removed by applying electrical voltages across the surface of the electrode. Luther field-tested the microelectrode in Hawaii's Kaneohe Bay. "It's very much like a Delaware salt marsh," he said, "in that it cycles iron very rapidly."

After shrinking the sensor's tip to 25 μm, Stephen C. Dexter, professor of marine studies, and graduate student Kunming Xu began using the microelectrode to simultaneously measure dissolved oxygen, manganese, and iron, as well as pH levels, in seawater biofilms grown on platinum and stainless steel surfaces. The sensor generated accurate measurements at 1.5 μm intervals within the biofilms, they said.

Whenever metals corrode in seawater, Dexter said, they interact with microscopic organisms and metabolic by-products. These interactions trigger an elaborate series of reactions as microorganisms consume oxygen to produce various other chemical species. In this way, microbes may speed the corrosion of metal surfaces. Researchers will not be able to prevent biofilms from forming until they know more about the chemical reactions taking place inside these films, Dexter said.

According to Dexter, traditional devices, made of a sensitive membrane placed over an electrode, are limited mainly to measuring oxygen and hydrogen sulfide gases, requiring a separate electrode for each gas. In the future, Luther might subject the probe to more extreme tests: in Hawaiian volcanoes and hydrothermal vents that are loaded with gases such as hydrogen sulfide and methane, as well as iron and other metals.

Computer Model Provides Insight into Squeezing Out Porosity during Aluminum Casting

Researchers at Oak Ridge National Laboratory (ORNL) and Thompson Aluminum Casting Company (TAC) have developed a metal forming process that not only produces a consistent and sound aluminum part, but one that is resistant to cracking caused by service stress. Cast aluminum parts can have defects in the form of small cavities. To alleviate that problem, TAC sought to develop a process called metal compression forming, a variant of the squeeze casting process, that could produce parts with fewer cavities reliably and consistently.

The team headed by Srinath Viswanathan, a researcher in the Materials Processing Group of ORNL's Metals and Ceramics Division, used computer models of the casting process to find ways of pouring aluminum into the mold more smoothly and with fewer impurities than has been done.

"In this process, the metal flows slowly and the mold is not being filled at high pressure that often causes turbulence in the liquid metal, thus trapping gases and creating undesirable cavities in the final part," Viswanathan said. "In addition, the metal compression forming process allows for uniform pressure to be applied over the entire part, virtually squeezing out porosity that would normally be present."

Brady Receives Navy Meritorious Civilian Service Award for Development of Organic Surface Coatings

Robert F. Brady, Jr., head of the Coatings Section in the Materials Chemistry Branch in the Chemistry Division of the Naval Research Laboratory, has received the Navy Meritorious Civilian Service Award during a ceremony held at the Laboratory on January 31. According to the award citation, "Dr. Brady successfully developed numerous organic surface coatings now used on ships of the United States Navy. His work is characterized by a comprehensive view of the chemistry of materials, insight in recognizing opportunities for performance enhancements, and creativity in devising practical solutions for critical operational needs. His basic research generates fundamental knowledge of organic and polymer chemistry, which is relevant to naval requirements. Dr. Brady is commended for elevating the Navy's competence in materials chemistry and producing advanced coatings that extend the performance and endurance of the Fleet."

Brady directs and conducts basic research in polymer chemistry and coatings science, formulates and tests advanced marine and industrial coatings and devices, and validates test methods for coatings. His present interests include coatings based on silicone and fluorinated resins, high-solids epoxy and urethan coatings, reactions in super critical fluids, and nontoxic foulant release coatings. He received his PhD degree in chemistry from the University of Virginia, and has received numerous awards, including the NRL Technology Transfer Award, and the Gordon Award from the Chemical Society of Washington. He has published 225 papers and reports of chemical research, and holds six patents.

Demand-Control Filtration Increases Energy Efficiency of Cleanrooms

Investigators at the Ernest Orlando Lawrence Berkeley National Laboratory (Berkeley Lab) have conducted a pilot project to test methods of reducing energy use in cleanrooms. According to David Faulkner of Berkeley Lab's Energy and Environment Division, "Common industry practice is to keep the fans on even when the cleanroom is not being used because it is thought that if they're turned off, or if the fan speeds are changed, it will take hours to restore the room to the required particle specification."

To test this belief and determine whether an alternative airflow environment could maintain particle concentrations at nominal levels, Faulkner, William Fisk of the Indoor Environment Program, and John Walton of the Engineering Division developed a scheme called demand-control filtration (DCF).

The researchers ran the fan under two automatic control schemes. In the first scheme, the counter measured particle concentration each second. At any time, if the count exceeded the maximum allowable concentration, the fan speed increased 10%. If the count was below the allowable limit, the fan speed decreased by 0.1%. In the second scheme, the fan speed increased in proportion to how many particles were in the air if the particle count was above the allowable threshold. Thus, if the counter detected substantially more than 100 particles per cubic foot, the control system increased fan speed by up to 70%; if the counter measured a slight increase in particles, the fan speed increased, but only up to about 10% of its original.

The results of this research, published in the November/December 1996 issue of the *Journal of the Institute of Environmental Sciences*, show that both methods saved energy—60% of the fan energy used in the pre-existing control method, in which a variable-speed drive reduced fan speed at night.

According to the researchers, DCF may not work for cleanrooms that require the lowest possible level of airborne particles (Class 1), or production cleanrooms where operators want to maintain as clean an environment as possible. Further research may find the thresholds of acceptable cleanliness to which DCF could be optimized for a broader range of applications. □

SBIR Update

MRi (Lansdale, Pennsylvania) has been awarded two Phase II SBIRs with a total of $1.2 million for a two-year development project to commercialize NASA's WideGap™ and SuperBraze™ brazing technology for the joining of composites (carbon:carbon and carbon:silicon carbide) and ceramics to metals.