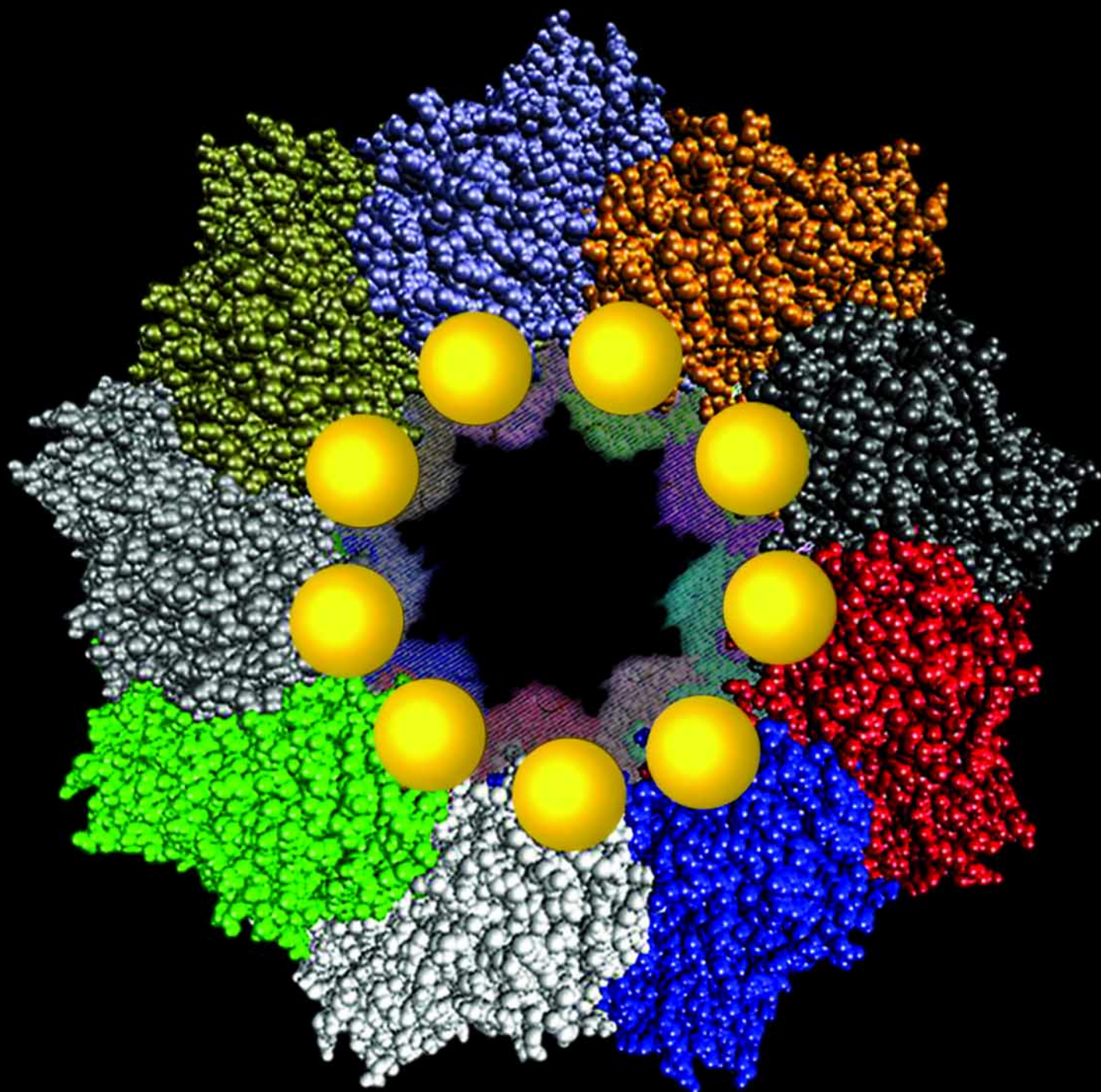
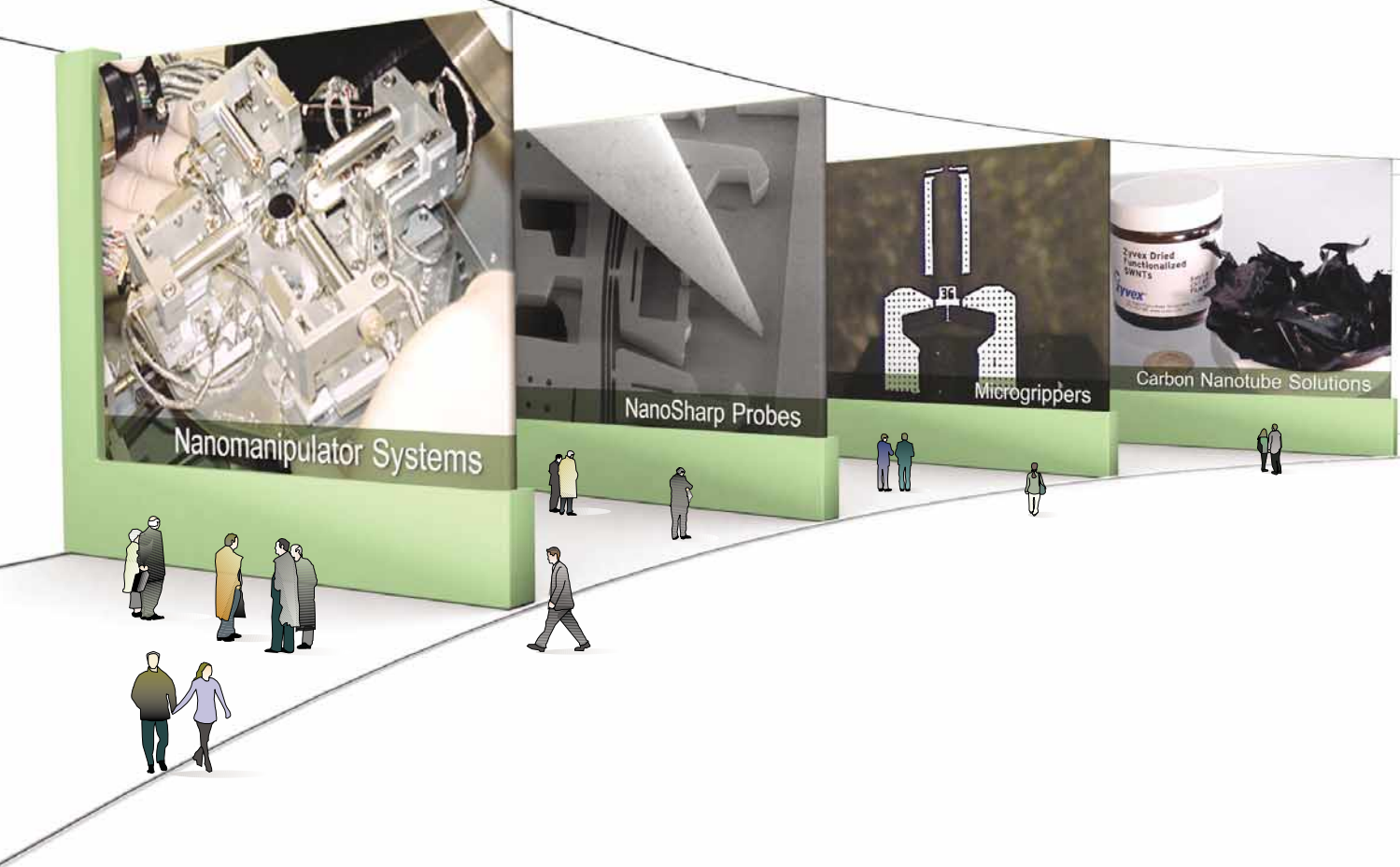


Nanotech Briefs



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Putting the "Tech" in Nanotech

Almost 45 years ago, physicist Richard Feynman saw a connection between biology and small-scale manufacturing. In 1959, Feynman explained his theory.

"A biological system can be exceedingly small. Many of the cells are very tiny, but they are active — they manufacture substances, they walk around, they wiggle, and they do all kind of marvelous things, all on a very small scale. Consider the possibility that we, too, can make a thing very small, which does what we want — that we can manufacture an object that maneuvers at that level."

Feynman was describing nanotechnology, long before the word even existed. Today, that once bizarre theory of manipulating atoms and molecules is one of the fastest-growing technology areas ever developed.

And technology is why ABPI — the publishers of *NASA Tech Briefs* — is pleased to welcome you to this premier issue of *Nanotech Briefs*, the first small-tech publication that focuses on the technology of nanotechnology — the best of government and industry nanotech innovations with real-world applications, developed for the commercial market. Our publication was conceived as a conduit for these innovations to reach design engineers and engineering managers who are developing tomorrow's small-tech products today.

In each issue of *Nanotech Briefs* — which begins as a bimonthly publication in January — you'll also find nanotech government and industry news in "Small Talk," the most exciting real-world uses of nanotechnology in our feature story, a profile of one of the country's cutting-edge nano research facilities in our "Inside" feature, and an "In Person" interview with a leading nanotech pioneer. In this issue, we're pleased to feature a discussion with Dr. Mihail (Mike) Roco, the key architect of the U.S. National Nanotechnology Initiative.

We'd like you to use *Nanotech Briefs* — not just read it. Use it as your central source of leading-edge government and industry nanotech innovations. Use it to spawn new ideas and solve problems in your design process. And use it to learn more about the staggering potential of nanotechnology in our daily lives.

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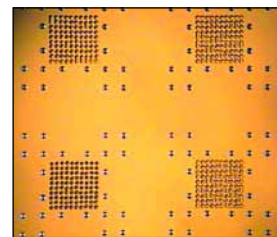
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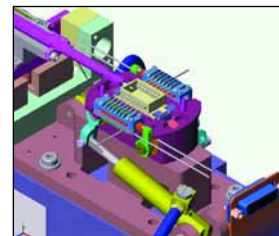
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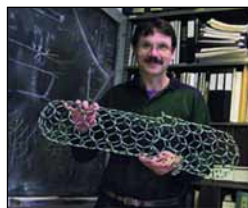
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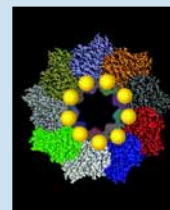
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ON THE COVER

Researchers at NASA's Ames Research Center in California formed this planar array of gold nanoparticles on a carbon film using chaperonin polypeptides. This and other periodic and otherwise ordered nanostructures can be fabricated less expensively than using conventional lithographic methods. See the tech brief on page 18 for details on the technology. (Image courtesy of NASA Ames)

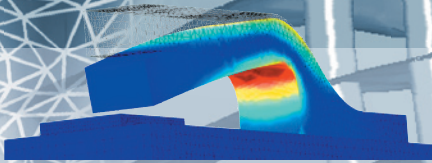


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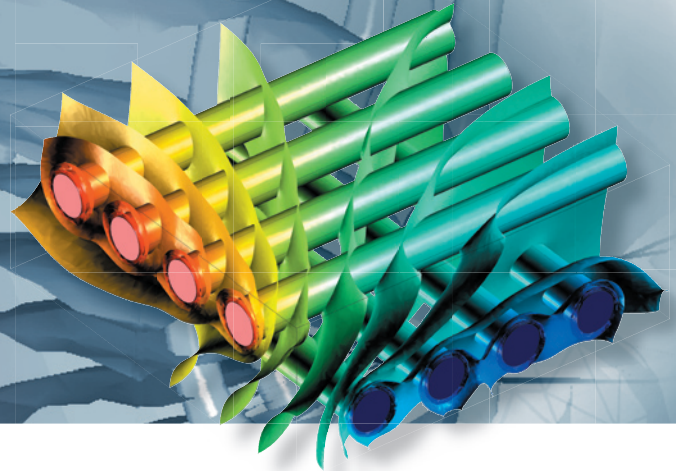
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Keithley and Zyvex Join Forces for Nano Solutions

Keithley Instruments (Cleveland, OH), a world leader in instrumentation for low-level electrical measurements, and Zyvex Corp. (Richardson, TX), a molecular assembly company, have joined forces to develop new solutions for the nanotech marketplace. The agreement calls for the companies to share marketing, sales, and applications engineering resources, and develop new solutions that combine Zyvex's nanomanipulator expertise and Keithley's ultra-low-level measurement technology.

Keithley's line of precision electrometers, picoammeters, and nanovoltmeters can measure electrical signals generated by nanodevices. Much of the company's technology used for characterizing semiconductor devices and test structures at the wafer level is applicable to nanotechnology.

Zyvex's family of nanomanipulation systems consists of modular R&D tools that are compatible with a variety of microscopes and probe stations. Keithley's Model 4200-SCS semiconductor characterization system (pictured), when coupled with Zyvex's S100 nanomanipulator, for example, will help engineers developing nanostructures, nanomaterials, and MEMS.

Visit Keithley at www.keithley.com. Visit Zyvex Corp. at www.zyvex.com.



NSF Grant to Study Nano's Societal Impact

The National Science Foundation (Arlington, VA) has announced two new grants of more than \$1 million each to study the societal implications of nanotechnology. Often referred to as a "transformative" technology — one that could change the way we live and work as profoundly as the automobile — nanotech has "the potential for unintended consequences," according to NSF Director Rita Colwell. Said Colwell, that's precisely why "we can't allow the societal implications to be an afterthought. The program has to build in a concern for those implications from the start."

According to David Baird of the University of South Carolina, which received one of the grants, technologies that don't do that have a way of coming to grief later on. "So, how can we go down a better path with nanotechnology?" Baird asked. The NSF grant will allow Baird and his team to tackle that question by setting up an ongoing dialog with as many points of view as possible. Just as researchers need to consider societal implications from the start, scholars need to understand what's possible in the lab.

The second grant, awarded to the University of California, Los Angeles (UCLA), will help Lynne Zucker and her team study how newly acquired knowledge about nanotechnology makes its way from the lab to the marketplace. Zucker explained that this is not something that happens automatically, and many startup companies fail because it's not done well. One of the products of the UCLA study will be an extensive database on small startup firms in the nanotech arena, and what factors influenced how well ideas succeed in the marketplace. "It will help us understand how the knowledge is transmitted, what facilitates that transfer, what blocks it, and what works well," Zucker said.

Visit www.nsf.gov/od/lpa/news/03/pr0389.htm.

Olympus Forms MEMS Division

Olympus Partnership Development Group (PDG) of San Jose, CA, has announced that Olympus Optical has formed a new MEMS Technology Division that will unify all of the company's previous MEMS activities, including R&D and MEMS foundry services. The present MEMS foundry will be expanded and have the capability to handle both existing and developing MEMS technologies on both 4" and 6" wafers, with future capability to handle 8" wafers. The MEMS center will produce and develop its own products, as well as provide foundry, R&D, and assembly services to other companies.

Visit www.olympuspartnership.com.



Nanobattery Patent Awarded

The University of Tulsa announced that chemistry professor Dale Teeters and two former students, Nina Korzhova and Lane Fisher, have been awarded a patent for a method of making nanobatteries for use in tiny machines. The team has made batteries that are so small, that more than 40 of them could be stacked across the width of a human hair. They are continuing to make even smaller batteries.

The patented invention is a manufacturing process that can build, charge, and test the nanobatteries. The method includes use of a porous membrane, filling the pores with an electrolyte, and capping the pores with electrodes. Conventional batteries have two electrodes that deliver the charge and an electrolyte through which charged ions move. Each battery packs as much as 3.5 volts.

Funding for Teeters' research was sponsored by the Department of the Navy's Office of Naval Research and the Oklahoma State Regents for Higher Education. Electronics companies Sanyo and Panasonic are interested in the battery-making process. The batteries can be used to power computers, medical devices, robots, and MEMS.

View the complete patent text at www.uspto.gov. Refer to patent #6,586,133.



Dr. Mihail C. Roco

Founder and Key Architect, National Nanotechnology Initiative (NNI)

Mike Roco is the Senior Advisor for Nanotechnology at the National Science Foundation, and serves as Chair of the Subcommittee on Nanoscale Science, Engineering and Technology (NSET) for the U.S. National Science and Technology Council. Roco is most notably the founder and key architect of the National Nanotechnology Initiative (NNI).

Nanotech Briefs: What is the National Nanotechnology Initiative and what are its origins?

Dr. Mihail C. Roco: The National Nanotechnology Initiative (NNI) is a visionary research and development program that coordinates 16 departments and independent agencies, with a total investment of about \$770 million in fiscal year 2003. The program started formally in fiscal year 2001 (October 2000), and was the result of a bottom-up activity proposing the idea of developing nanoscience and engineering that started in 1996.

At that time, I organized a small group of researchers and people from government, and we started to do our homework in preparing a vision for nanotechnology. In March 1999, on behalf of this interagency group, I proposed at the White House the National Nanotechnology Initiative with a budget of half a billion dollars. At that time, nanotechnology was not known to anyone in Congress or the Administration. We involved six major agencies that would place nanotechnology as a top research priority — the National Science Foundation (NSF), the Department of Defense (DOD), the Department of Energy (DOE), National Aeronautics and Space Administration (NASA), National Institutes of Health (NIH), and the National Institute of Standards and Technology (NIST).

In January 2000, President Clinton announced the Initiative. It was the first time we had a fundamental-science-driven initiative that was announced by

a president. In the first year, the six agencies of the NNI spent about \$470 million. Since then, NNI has increased significantly, and we now have 16 departments and agencies.

Nanoscale science and engineering are the wave of scientific discovery and have relevance in almost all disciplines and areas of the economy. Initially, the topics were driven by science, but since last year, industry has become a strong supporter and is expected to surpass the expenditures made by government next year in research and development. Also, states realize that nanotech has economic potential, and in 2002 made a commitment for nanotechnology that is more than half the NNI budget in 2002.

NB: How does the NNI work with industry to promote and develop nanotechnology?

Dr. Roco: The role of NNI is to create the foundation to do things that industry cannot do well, and to promote the transfer of information to industry and society at large. NNI will develop visionary projects that only government can do because it's cross-cutting, addresses common issues, and is long-term in its impact on society.

We help industry in many ways. We are looking to create a workforce for tomorrow for nanotechnology. Second, we want to develop the knowledge base that can be applied for different areas of development. Third, we want to create an infrastructure. This is an important issue, because the infrastructure has to be flexible, applicable in different domains, and geographically distributed. The National Science Foundation (NSF) has more than 20 centers funding nanotechnology, and two networks. One is the National Nanofabrication User Network for experiments, and the other is the Network for Computation of Nanotechnology. Next year, we'll expand the

National Nanofabrication User Network to more than double its size.

NB: What are the NSF's main focus areas in nanotechnology?

Dr. Roco: NSF in 2002 spent more than \$10 million in Small Business Innovation Research/Small Business Technology Transfer (SBIR/STTR) grants for nanotechnology. In 2002 we spent \$22 million for nanomanufacturing, mostly focused on fundamentally new concepts. Just last month, we announced two new centers for nanomanufacturing and a new center on instrumentation at the nano scale.

This is a very broad program. In all of NNI, we have only about 2,000 active projects. At NSF, we have about 1,500 active projects. We try to cover almost all the disciplines and areas of activity in a balanced way. We have involvement with industry, the states, foreign governments, and industrial partners abroad.

NB: How does the United States compare with other countries in terms of nanotechnology development?

Dr. Roco: Nanoscale engineering is a very broad field, and no country has supremacy in all of the fields. There are several areas in which the U.S. is more advanced, such as nano/bio, or in materials and simulations. We have more organized activity and we have a slightly better infrastructure in our physics, chemistry, and genetics departments. Overall, the US probably has a slight advantage, but it's difficult to quantify in each field.

Nanotechnology is slightly different from other technologies. First of all, we started earlier and felt an external pressure. For instance, when the space program started, it was because of competition with Russia. In nanotechnology, it was driven by the vision of scientific opportunity. The U.S. did not have a dominant advantage. In nanotechnology, the

U.S. does not have the overwhelming advantage we have in other technologies. We have to compete harder.

NB: How do you respond to concerns about the potential societal and environmental dangers of nanomaterials?

Dr. Roco: Like any emerging technology, there are questions about societal implications. And this was addressed from the beginning. In October 2000, we organized the first major workshop on societal implications. Societal implications are quite broad — it implies increasing productivity, changing healthcare, and expanding the limits of sustainable development of the Earth. With existing technologies, we have a limit to the number of people who can live on Earth in a sustainable way. If we improve that technology, moving to processes with no waste, or with a small amount of materials, this limit will be extended. Also, like any new technology, there are social effects like potential negative impacts if by accident, materials are released into the environment. All of this has to be taken into account.

We are moving ahead because all the indications so far show that the advantages of nanotechnology by far outweigh the potential unexpected consequences. For example, nanoparticles can eventually target cancer cells and visualize illnesses before they happen. At the same time, nanoparticles may have effects that are not yet fully determined. The positive aspect is that already nanoparticles exist all over the Earth. With nanotechnology, we can address current issues that cannot be solved otherwise.

At the same time, one has to develop nanotechnology in a responsible way. We have to pay sufficient attention to environmental and health issues, as well as to ethical and economic issues. It's interesting that NNI, because it was developed as a science project, planned for this from the beginning. In the first announcement the NSF made in June 2000, we had six major themes and one was on environmental issues and one was societal and educational issues.

NB: Is the development of nanotechnology proceeding at an expected pace, or is practical application still years away?

Dr. Roco: It's been said that nanotechnology is as complex as the space program. The main advantage to the space program was developing knowledge about the universe. Moving to nanotechnology is not only exciting, but will change the way we live on Earth. Both bring a lot of excitement. But one thing that's different is that in moving to the nano world, you have significant implications for living conditions on Earth and on health that are probably broader than any other technology developed so far. It is a risk sometimes to focus too much on day-to-day activities and not to have a guiding, long-term vision that helps to develop the programs, find the best investments, and find the best ways to prepare the people.

"Nanotechnology is not science fiction or hype. The development is real, and is penetrating into many fields."

We are just developing the science base of nanotechnology now. So far, we only have things that have been developed, I'll say, by accident. People working in other fields move to the smaller scale and discover things. They don't exactly know how they work or why they work. So we are still in an empirical phase of nanotechnology. There will be several phases. The first phase, which we're already in, is dealing with nanostructures and starting to have systematic control of this. The next phase, in three or four years, will be when we start to have active nanostructures such as a transistor, an actuator, an active drug-delivery device. It will be another several years before we have nano systems.

We don't want to be too pessimistic or too optimistic. We're moving in this direction — the only question is how far. While the space program had a lot of knowledge development, nanotechnology will have much more, besides an equal interest in knowledge and understanding once we develop the tools, and that will mark a major change in the economy.

One cannot predict exactly the time involved — many developments will be made by breakthroughs. We cannot make an exact timeline for each event. One has

to keep in mind that basically, we are still at the beginning of the road. Most of the activity in nanotechnology is empirically based — it's experimental, stemming from another field prior to nano.


NB: What is the long-term vision for nanotechnology?

Dr. Roco: The vision has three or four components. It's not important what's happening on the "small" end. What's important are the new phenomena, the new processes, the new functions that happen at the intermediate-length scale. It is not important to work in one field — what's important is the exchange of tools, methods, architectures, and applications among different disciplines. And it's

important that you have the ability to transform with a small amount of energy or materials, and to do things that are not possible at any other scale, and to do them more economically.

This is robust development — it's not science fiction or hype. The development is real, and is penetrating into many fields. What I see for this year and next year is expanding into four new areas: food and agricultural systems, energy conversion and storage, regenerative medicine and nano/bio medicine, and environmental research and education.

One has to be very careful when reading comments from those who are not working in the field. You find people who say, "There's nothing new in nanotechnology. We've been doing this for the past 20 years." These people don't even know that 20 years ago, you had microscopes 1,000 times larger, but not to the nanoscale, and you could not manipulate to see the shape of the molecule, to manipulate them one by one. We've developed a vision collectively.

Contact Dr. Roco at the National Science Foundation at mroco@nsf.gov. For more information on the NNI, visit www.nano.gov. 

Sending Nanotechnology into Space

Since 1993, the Hubble Space Telescope (HST) has been delivering some of the best images of space ever seen. The HST has allowed NASA to observe massive black holes, the birth and death of stars, the origins of gamma rays, and much more. As evidenced by the spectacular images taken August 27th when Mars made its closest approach to Earth in almost 60,000 years, HST is without a doubt an important tool for improving our understanding of the universe.

On its upcoming fourth servicing mission — expected to launch when Space Shuttle flights resume — HST will receive new and high-power instruments in the telescope's aft shroud section. However, the Space Telescope Imaging Spectrograph (STIS) is also located in this section, and the heat generated by these new instruments would wreak havoc with STIS's ability to deliver high-quality space images. To address this issue, the fourth servicing mission also will include adding a capillary-pump loop (CPL) technology to draw the waste heat away from STIS and toward the aft shroud's exterior, where it will be rejected into the cold darkness of space. The challenge with this cooling solution is how to attach the CPL system to the STIS radiator.

The STIS Thermal Interface Kit (STIK) will provide the means to attach the CPL to STIS (see Figure 1). However, STIK's interface material must be a good thermal conductor that:

- can withstand the harsh vacuum of space,

- provides electrical isolation,
- has a low contact pressure (so as not to damage the STIS instrument),
- does not contaminate the spacecraft or its instruments, and
- is abrasion tolerant (astronauts will have to install STIK around a blind corner).

Compounding this situation is the fact that the STIS radiator was never envisioned to be an on-orbit interface, so no one knows the condition of its surface. Although it is safe to say that STIS's surface is likely to be very uneven, measurements are impossible to obtain as HST travels above the Earth at 18,000 miles per hour.

Finding an effective interface material is essential. "The problem is that the compliant polymer materials providing the flexibility we need are not good thermal conductors or would contaminate HST," explained Dan Powell, a researcher at NASA's Goddard Space Flight Center in Greenbelt, MD. "And the materials that are good conductors aren't going to be compliant enough to mold to STIS's rough surface, creating a good thermal contact. What we need is a material that is as flexible as a polymer but as conductive as, or better than, copper, and that can be attached with a low contact pressure."

The Nanotechnology Solution

That's where nanotechnology — specifically, the carbon nanotube (CNT) — comes in. Although carbon nanotubes (CNTs) were discovered a decade ago, their practical applications are just beginning to be explored. Measuring one-bil-

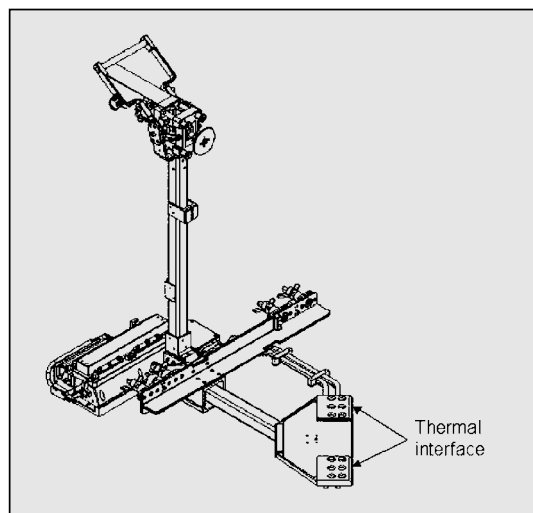


Figure 1: STIS Thermal Interface Kit (Source: NASA Goddard Space Flight Center)

lionth of a meter in diameter, CNTs are carbon molecules that have been covalently bonded in a seamless, hexagonal network architecture (see Figure 2).

CNTs offer highly useful material characteristics such as:

- **Flexible:** CNTs offer a linear elasticity of up to 10%. They also can be manufactured with several concentric tubes that can expand like the sections of a pocket telescope.

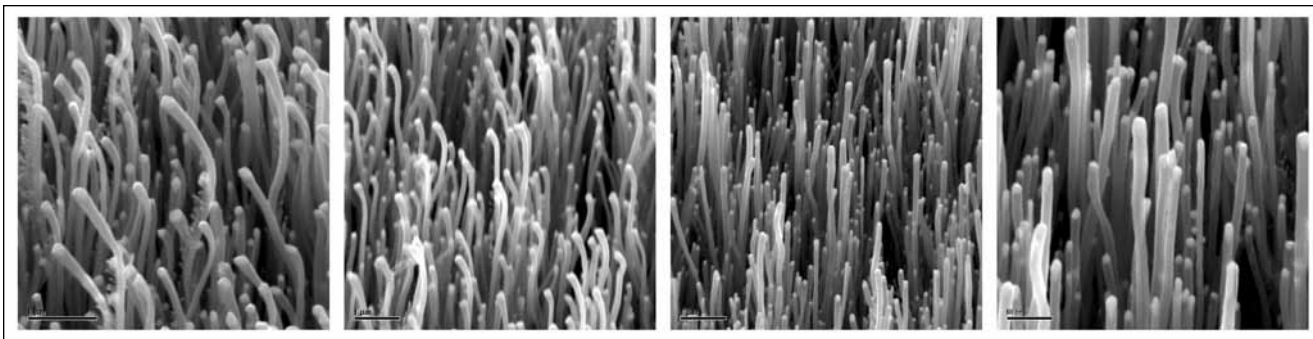


Figure 2: Scanning electron microscope views of 30µm-long vertical carbon nanotube arrays (Source: NASA Ames Research Center)

- **Strong:** With a Young's modulus of more than 1 terapascal, CNTs offer a strength-to-weight ratio 500 times better than aluminum, steel, and titanium as well as an order of magnitude improvement over graphite/epoxy.
- **Electrically conductive:** CNTs, particularly multiwalled CNTs, are robust ohmic conductors with extremely high current-carrying capability. They remain stable at current densities as high as 10^9 A/cm², more than three orders of magnitude higher than copper wires. Yet in situations like with HST, electrical conductivity can be eliminat-

ed so as not to pose any shorting or contamination problems.

- **Thermally conductive:** Although testing is still being done, the theoretical conductivity of CNTs in the axial direction is 6,000 W/m-K, whereas copper's conductivity is only about 400 W/m-K.

Seeking an appropriate interface material for the STIK, NASA decided to try to leverage these characteristics and, more importantly, the fact that a CNT interface would have a low contact pressure. Powell and his colleagues are now working out the engineering details to put the CNTs into practical application.

Because the timeframe for developing the CNT interface is very short, NASA asked its Ames Research Center in Moffett Field, CA, and the Applied Physics Laboratory (APL) at Johns Hopkins University to create a usable nanoconductor. "Having two labs working simultaneously helps reduce the risk of failure," said Powell. "It increases the chances that we'll get what we need for the HST servicing mission on time." These collaborative efforts are just one example of some of the benefits yielded by the Nanotech Alliance (see the sidebar on page 9).

NASA's Nano Research

Since Goddard is a space flight center, it is continually working to take the scientific breakthroughs discovered at NASA's research centers and get them ready for launch. "It's like a jigsaw puzzle," explained Goddard's Dan Powell. "Ames Research Center provides us with the pieces, and we figure out how to put them together to make a picture. Maintaining ongoing communication between the scientists and the engineers helps ensure that the pieces will fit in the picture NASA is trying to create."

In addition to the research related to the Hubble servicing mission, NASA also is exploring other space flight applications for its nanotechnology research. Much of this work is being performed jointly with members of the Nanotech Alliance:

- Goddard researcher Jeannette Benavides has developed a new noncatalytic method for manufacturing carbon nanotubes, which might have by-the-ton yields, cost of less than \$10 per gram to produce, and could greatly enhance the feasibility of using CNTs in space as well as other applications.
- Using oriented nanocomposite extrusion to produce "designer" materials — tailoring their properties to the specific application without changing their constituents — could provide a more reliable alternative to graphite-epoxy composites.

- Filling CNTs with lithium-methane and/or lithium-hydride could protect space, terrestrial, and even biological systems from energetic neutrons/protons and gamma radiation.
- A software program for finite element analysis with atomic-scale resolution might be used to study a wide range of nano systems — from theoretical material design to structural analysis to designing energetic materials (e.g., rocket propellant, car fuel).
- Nanoelectronic, cosmochemistry, and nanoelectrode array biochemical sensors could serve as low-power, high-sensitivity electronic sensors for NASA's space science, Earth science, and biological and physical research missions as well as DNA, RNA, and protein sensors for "bioexplorers."
- A CNT X-ray source has been incorporated into an instrument for analyzing the chemistry and mineralogy of solar system objects and later could be applied to microelectronics for surface analysis for <1kg mass spectrometers, spacecraft sterilization, or in vivo cancer treatment.


Other possibilities for nanotechnology at NASA include ultralightweight electrically conductive CNT-based satellite tethers, selectively rechargeable quantum chemical sensing assays, self-actuating radiometer hinges, and long-range self-deploying phased array antennae.

Ames and APL are aligning arrays of CNTs, and optimizing their characteristics for the needs of the HST project. "Our first CNT product will be a 6-square-inch, copper-backed thermal conductor," explained Dr. Robert Osiander, who is leading APL's research effort. "On it will be approximately 4 billion nanotubes sticking up about 40-millionths of a meter from the copper surface. Think of a laundry brush with stiff but somewhat flexible bristles rising from its wooden base."

Ames is working on arrays with similar length and tube density specifications, which were selected to ensure good contact between STIK and STIS. Ames researchers have established a plasma-enhanced chemical vapor deposition method that can grow freestanding CNT arrays on various substrates with densities from 10^7 to 10^9 CNTs per square centimeter, which fits the requirements of this application.

HST researchers are testing the CNT arrays for STIK. By mounting the nanoconductor and a compliant pad on a thin copper foil at the end of a mechanical arm, NASA researchers hope to establish billions of points of contact with STIS.

Several technical issues must be addressed before the CNT interface will be ready for the HST servicing mission. These issues include determining exactly what the thermal conductivity of the CNT interface is and exactly how an astronaut will attach STIK's interface to STIS. "We still have a lot of work to do," said Ted Swanson, Goddard's Assistant Chief for Technology for the Mechanical Engineering Division. "We began testing in June, and so far it's looking promising. The Hubble Development Project Office is very supportive and is as excited about this technology application as we are."

Contact Dan Powell at Dan.Powell@nasa.gov. 

The Nanotech Alliance

Much of the nanotechnology work that is proceeding for the space program has come out of the Nanotech Alliance (NTA). Currently composed of nearly 20 government and academic labs — including NASA, the Applied Physics Laboratory (APL) at Johns Hopkins University, the University of Maryland at College Park, the Naval and Army Research Labs, the National Institute of Standards and Technology, and many others — the NTA is dedicated to advancing nanotechnology research. But rather than focusing on delivering a tangible product, the NTA members focus on building their relationships to allow the benefits of collaboration to be realized.

The NTA began simply as informal discussions between researchers at NASA Goddard Space Flight Center and APL. "I had been working with APL folks on other research," recalled Goddard's Dan Powell, who founded the NTA in September 2002. "When we realized we had a mutual interest in nanotechnology, it made sense to leverage our current relationship into that new area."

Over time, Powell made similar contacts within other organizations, building the NTA's membership with two representatives each from eight labs. "We always have one scientist and one engineer from each organization for balance," said Powell. "The scientists consider what's possible, and the engineers focus on what's practical."

As the NTA members continued to meet monthly, they found specific

research areas where their interests and goals overlapped. In those cases, NTA members would team up to conduct research jointly. A key benefit of this joint research has been the efficient use of resources — most of which are provided by the taxpayers. "By knowing who the key players are, what they're looking at, and where things can be combined, you can direct your efforts where they bring the most value," Powell said. "There's not enough money for all of us to be working alone, so we'd better work together."

And such collaboration has paid off. "We have done a lot to advance NASA's research in nanotechnology for the space program, and much of that progress can be attributed to the NTA," emphasized Powell. "On average, research efforts take 20 years to get from concept to flight. For the Hubble repair we're testing now, which was a cooperative effort between NASA and APL, it took two and a half years."

Now that the NTA has grown to nearly 20 organizations, the group is planning its first conference for November 25, 2003, in Laurel, MD. Attendees, who will be invited from the broader nanotech community, will look at the latest advances in nanotechnology, taking an applications focus. The emphasis will be on system development and the integration of nanotechnology into those systems.

For more information about the Nanotech Alliance, visit www.nanotech-alliance.org or contact Dan Powell at Dan.Powell@nasa.gov.

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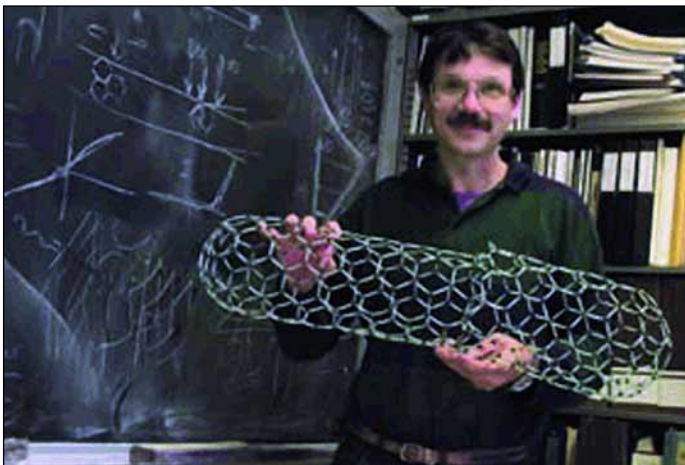
Nanotube Heterojunctions

These carbon nanotubes can be used to form electronic devices.

Ernest Orlando Lawrence Berkeley National Laboratory, U.S. Department of Energy, Berkeley, CA

Carbon nanotube structures have been designed that enable the creation of nanometer-scale diodes, transistors, and sensors. These carbon nanotubes, containing metal/semiconductor or semiconductor/semiconductor junctions, may be used to form electronic devices that are 1 to 2 nanometers in each dimension. Nanoscale devices envisioned include Schottky barriers, quantum wells, and transistors 10,000 times smaller in area than present commercial silicon devices.

A novel topological solution that matches tubes with different electronic structures enables the creation of these junctions. Carbon nanotubes are synthesized to contain pentagon-heptagon




Researcher Alex Zettl, displaying a model of a **Carbon Nanotube**, has created mechanical switches far too small to be seen without the aid of an electron microscope.

pair defects in their normal hexagonal structure. The defects change the helicity of the nanotube and alter its electronic structure. In addition to forming all carbon heterojunctions, the tubes can

be doped with boron or nitrogen to add charge carriers to the semiconducting region.

Applications for the technology include microswitches, micromachines, microsensors, computation elements, high-strength fibers, smart materials, and other devices that require high thermal conductivity, high mechanical strength, high temperature stability, and high-potential device density.

This research was performed by Alex Zettl of Lawrence Berkeley National

Lab. For further information, contact Pam Seidenman, Technology Transfer Dept., 510-486-6461, e-mail: ttd@lbl.gov, or visit www.lbl.gov/tt. Refer to Technology #IB-1181. 



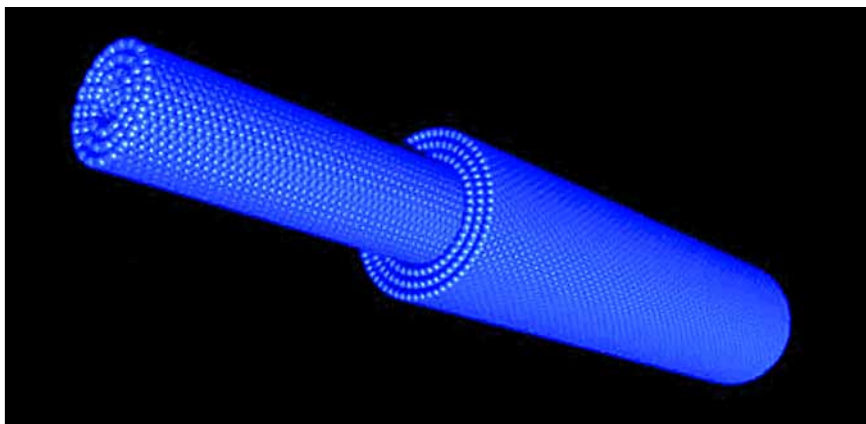
Nanotube Bearing and Spring

Multiwall nanotubes can perform as nanoscale linear bearings and nanosprings.

Ernest Orlando Lawrence Berkeley National Laboratory, U.S. Department of Energy, Berkeley, CA

Through controlled and reversible telescopic extension, multiwall nanotubes have been shown to perform as extremely low-friction nanoscale linear bearings and constant-force nanosprings. Measurements of individual custom-engineered nanotubes — performed *in situ* inside a high-resolution transmission electron microscope — have explicitly demonstrated the anticipated van der Waals energy-based retraction force.

These measurements have also placed quantitative limits on the static and dynamic nanotube/nanotube interwall friction forces, and have shown that the



A telescopically extended **Multiwall Nanotube**.

nanotubes behave as constant-force springs that do not follow Hooke's law.

On the atomic scale, no wear and fatigue were observed after noting repeated extension and retraction of telescoping nanotube segments.

This indicates that the new multiwall nanotubes may constitute wear-free surfaces.

This research was performed by John Cummings, Alex Zettl, Steven Louie, and Marvin Cohen of Lawrence Berkeley

National Lab. For further information, contact Pam Seidenman, Technology Transfer Dept., 510-486-6461, e-mail: ttd@lbl.gov, or visit www.lbl.gov/tt. Refer to Technology #IB-1649.



MATERIALS

Organic/Inorganic Hybrid Polymer/Clay Nanocomposites

The exfoliation and dispersion of clay particles are improved.

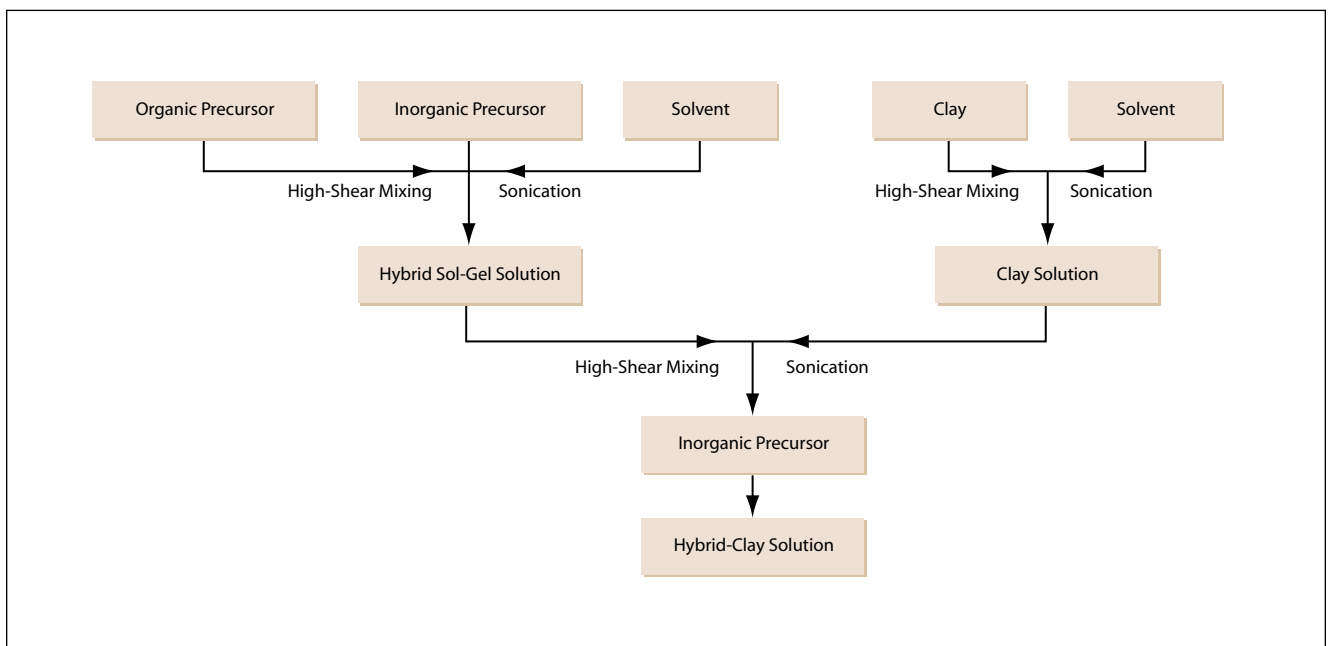
NASA's Langley Research Center, Hampton, Virginia

A novel class of polymer/clay nanocomposites has been invented in an attempt to develop transparent, lightweight, durable materials for a variety of aerospace applications. As their name suggests, polymer/clay nanocomposites comprise organic/inorganic hybrid polymer matrices containing platelet-shaped clay particles that have sizes of the order of a few nanometers thick and several hundred nanometers long. Partly because of their high aspect ratios and high surface areas, the clay particles, if properly dispersed in the polymer matrix at a loading

level of 1 to 5 weight percent, impart unique combinations of physical and chemical properties that make these nanocomposites attractive for making films and coatings for a variety of industrial applications. Relative to the unmodified polymer, the polymer/clay nanocomposites may exhibit improvements in strength, modulus, and toughness; tear, radiation, and fire resistance; and lower thermal expansion and permeability to gases while retaining a high degree of optical transparency.

The clay particles of interest occur naturally as layered silicates. In order

to fully realize the benefits of a polymer/clay nanocomposite, it is necessary that the clay particles become fully exfoliated (delaminated) and uniformly dispersed in the polymer matrix. Concomitantly, it is necessary to maintain the exfoliation, counteracting a tendency, observed in prior formulations of polymer/clay nanocomposites, for dispersions of exfoliated clay particles to collapse back into stacked layers upon thermal treatment. One reason for the difficulty in achieving and maintaining exfoliation and uniform dispersion is the



This **Flow Diagram** shows the major steps of a process for making a film of an organic/inorganic hybrid polymer/clay nanocomposite.

incompatibility between the silicate particle surfaces (which are hydrophilic) and the polymer matrix (which is hydrophobic). The present invention addresses these issues.


The figure depicts a process for making a polymer/clay nanocomposite film according to the invention. In one of two branches of the first step, a hybrid organic/inorganic matrix resin in a sol-gel form is prepared. The organic precursor of the hybrid is a compound or oligomer that contains both a cross-linkable functional group (e.g., phenylethynyl) and an alkoxy silane group. The inorganic precursor of the hybrid is also an alkoxy silane. Both precursors are mixed with a solvent to form the sol-gel matrix resin. In the other branch of the first step, a clay solution is prepared by initially dispersing layered clay particles in the same solvent as that used to form the sol-gel matrix resin. To achieve intercalation of

the solvent into the stacked layers, the mixture is subjected to high-shear mixing and to ultrasound. Suitable clays to provide compatibility to the organic polymer include chemically modified organophilic cation-exchanged smectite type clays and synthetic clays having hydroxyl functional groups on the edges and/or elsewhere on the surfaces of the particles.

In the second step of the process, the clay solution is added to the hybrid sol-gel solution and the resulting mixture subjected to high-shear mixing and ultrasound. The hydroxyl groups of the organic/inorganic hybrid react with hydroxyl groups on the surfaces and the edges of the exfoliated clay particles, forming covalent and/or hydrogen bonds that enhance exfoliation in the presence of high shear.

The third step involves a film casting process. For example, to make an unoriented film, one begins by simply casting

the solution onto a glass plate or other suitable clean, dry surface. The solution is allowed to dry to a tack-free film in ambient, desiccated air, then further dried and cured in flowing heated air. During this thermal treatment, the remaining silanol groups of the hybrid undergo condensation reactions, forming a molecular network that prevents the reunion of the exfoliated particles into stacked layers. In addition, the organic matrix can be consolidated with further crosslinking among the cross-linkable functional groups during thermal cure. It may be possible to prepare oriented films and fibers by using shear, drawing, and fiber spinning processes.

This work was done by Cheol Park (NRC), John W. Connell, and Joseph G. Smith, Jr., of NASA's Langley Research Center. For more information, contact the Langley Commercial Technology Office at (757) 864-3936. Refer to LAR-16216 

Low-Temperature Plasma Functionalization of Carbon Nanotubes

This process is dry, clean, and relatively simple.

NASA's Ames Research Center, Moffett Field, California

A low-temperature plasma process has been devised for attaching specified molecular groups to carbon nanotubes in order to impart desired chemical and/or physical properties to the nanotubes for specific applications. Unlike carbon-nanotube-functionalization processes reported heretofore, this process does not involve the use of wet chemicals, does not involve exposure of the nanotubes to high temperatures, and generates very little chemical residue. In addition, this process can be carried out in a relatively simple apparatus and can readily be scaled up to mass production.

The apparatus used in this process includes two vacuum chambers, denoted the target chamber and the precursor chamber. A plasma of the chemical precursor of the molecular groups to be deposited is generated in the precursor chamber. The plasma flows from

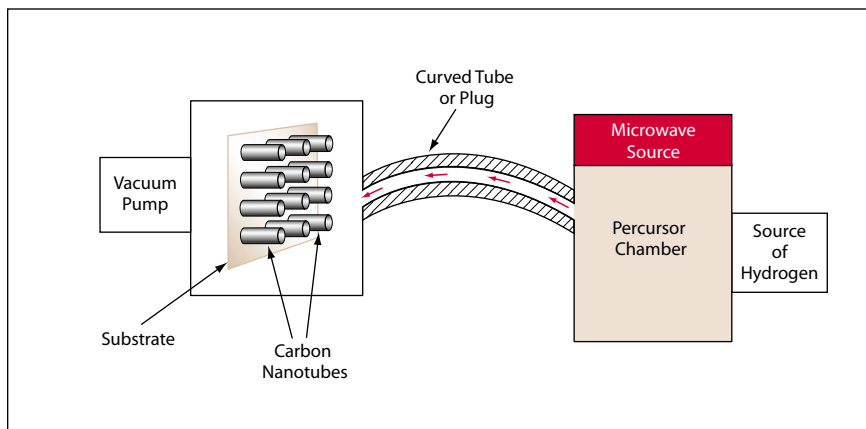
the precursor chamber to the target chamber, wherein the carbon nanotubes to be functionalized are mounted on a substrate.

The process is best described by use of an example of functionalizing carbon nanotubes with hydrogen atoms. The precursor chamber is backfilled with high-purity (99.9999 percent or greater) H₂ gas, optionally mixed with an inert carrier gas (N₂, Ne, or Ar), to a total gas pressure between 0.1 and 1 mm of Hg (between about 13 and about 130 Pa). The gas is irradiated with microwaves, thereby generating free electrons and a partially ionized gas that includes free radicals (in particular, monatomic hydrogen). Instead of a microwave source, a DC, non-microwave-radio-frequency, inductive-discharge, or electron-cyclotron-resonance source can also be used to generate the plasma. The temperature of the free electrons is typi-

cally of the order of a few electron volts (1 eV = 11,604 K). The temperature of the partially ionized gas typically lies in the approximate range from 350 to 1,000 K.

The two chambers are connected by a curved tube or plug made of polytetrafluoroethylene or other suitable material. Typically, the tube or plug has an inner diameter of about 1 mm, an outer diameter of between 5 and 25 mm, and a length between 5 and 25 mm. The substrate holding the carbon nanotubes is positioned to face directly into the plasma flowing into the target chamber through the hole in the tube or plug.

The tube or plug is curved to eliminate a direct line of sight between the interiors of the chambers in order to prevent ultraviolet light originating in the precursor chamber from reaching the carbon nanotubes. This is necessary for the following reasons: Some ultraviolet radi-



A Hydrogen Plasma is generated in the precursor chamber and flows into the target chamber, where hydrogen atoms become chemically bonded to the carbon nanotubes.


ation is generated in the undesired but unavoidable recombination of some of the monatomic hydrogen into H_2 molecules. This radiation is capable of breaking the C-H bonds in hydrogenated materials, including hydrogenated car-

bon nanotubes. Other ultraviolet radiation generated in the precursor chamber may be capable of breaking C-C bonds in the nanotubes.

There is no need to maintain the temperature in the target chamber at any par-

ticular value in order to achieve functionalization. Experiments have shown that carbon nanotubes can be functionalized with hydrogen to the point of saturation in a process time of about 30 seconds, at or below room temperature. It is also possible to functionalize carbon nanotubes with molecular groups other than hydrogen. For example, by choosing a suitable precursor, one could attach halogen or alkali metal atoms or low-molecular weight hydrocarbons.

This work was done by Bishun Khare of SETI Institute and M. Meyyappan of NASA's Ames Research Center. For further information, please contact M. Meyyappan at (650) 604-2616 or E-mail: meyya@orbit.arc.nasa.gov.

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-14661-1 

Nanoparticulate Electrodes Enable Improved Rechargeable Li-ion Batteries

NEI Corporation, Piscataway, New Jersey

The unique structural features of nanostructured powders lead to enhanced diffusion of Li-ions, thereby delivering high power density, along with a long cycle life and high charge/discharge rate capability. The surface and internal structures of nanomaterials are often very different from that of conventional coarse particles. This has several ramifications, including the fact that the primary particle or grain sizes are at least an order of magnitude smaller than those used in conventional electrode materials. This implies that a battery can be fully charged or discharged at a much faster rate, without compromising the capacity.

Nanostructured electrode materials for rechargeable Li-ion batteries, branded as Nanomyte™, may reduce the wait associated with recharging laptops and camcorders from hours to tens of minutes. Intrinsically low-cost materials, when used in the nanostructured form,

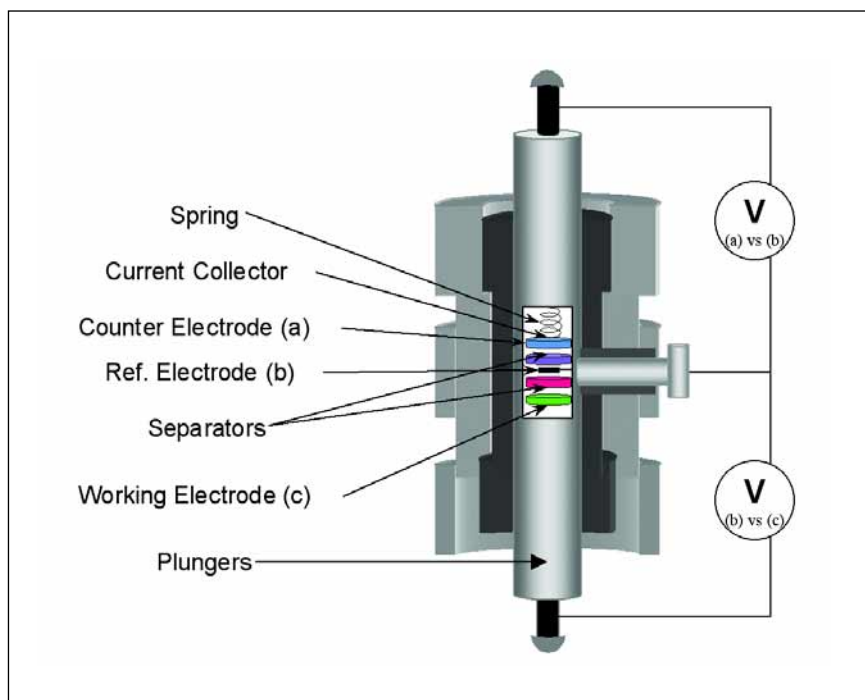


Figure 1. Schematic of an **Electrochemical Test Assembly**.

lead to higher capacity compared to their coarse-grained counterparts.

Quick charge/discharge capabilities are needed in applications such as uninterruptible power supplies, power tools, and hybrid electric vehicle batteries. Nanoparticles of certain compositions and structures can potentially increase the energy density of Li-ion batteries; this is because of the additional sites available on the surface of the nanoparticles in addition to the intercalating sites — only the latter being available in micron-sized particles. Also, the volume distortion associated with intercalation is relatively small in nanomaterials. As a result, the reversible intercalation reaction can occur several times without any “damage” to the electrode material. This results in a long cycle life; i.e. the initial capacity can be maintained for a large number of cycles.

Another unique feature of nanoparticles is the ability to use alternative inter-


calating ions — such as magnesium — instead of lithium. The advantage is that magnesium is significantly less expensive than lithium, and the resultant rechargeable device has the capability of delivering still higher power densities, along with good energy densities.

Over the past few years, NEI has been working closely with a number of groups in academia and industry to develop nanostructured materials for use in a variety of rechargeable “rocking-chair” devices, the rechargeable Li-ion battery being one of them. For example, under a NASA-sponsored program, NEI is developing a nanostructured and layered lithium manganese oxide-based material, LiMnO_2 . To date, an initial discharge capacity of ~ 250 mA/g has been achieved. The capacity drops somewhat at higher discharge rates, but there is practically no capacity loss over 100 cycles. This material has the potential to

surpass the energy density achieved in spinel LiMn_2O_4 as well as in LiCoO_2 , which is the standard cathode material.

On the anode side, NEI has developed nanostructured lithium titanate ($\text{Li}_4\text{Ti}_5\text{O}_{12}$) using a novel low-cost method. The ultrafine material displays near-theoretical capacity at moderate charge rates (1 hour charge), and $>90\%$ capacity at exceptionally high charge rates (20-minute charge). Other cathode compositions, based on phosphates, are also in advanced stages of development.

In summary, nanomaterials present unprecedented opportunities for achieving the performance goals of several applications at competitive cost.

This article was written by Amit Singhal, Chief Scientist, and Ganesh Skandan, CEO, at NEI Corporation. For more information, visit www.neicorporation.com or call (732) 868-1906. 

Fundamentals of Magnetic Nanomaterials

Magnetic interactions between nanomaterials can be characterized using this method.

Materials Science Department, Brookhaven National Laboratory, U.S. Department of Energy

Local magnetic interactions between ferromagnetic and antiferromagnetic nanocrystallites strongly depend upon the chemical and topographical nature of the interface, and the exchange-bias effect is a sensitive measure of these interactions. Additionally, the geometry of the interfacial region may also affect the magnetism. To examine the effects of the geometry of the ferromagnetic/antiferromagnetic interfacial region upon the intrinsic and extrinsic magnetic properties of an exchange-biased system, a 13-nm-thick film of interspersed Ni and NiO nanocrystallites was produced using reactive dual ion-beam deposition.

X-ray diffraction, Rutherford backscattering spectroscopy, and SQUID magnetization and susceptibility measurements were used to characterize the sample. Results indicate that the film has an overall composition of 75 at. % Ni and 25 at. % O with average grain sizes of 6 nm (Ni) and 7 nm (NiO). A measured $T_N =$

510 K for the NiO nanocrystallites is slightly reduced from the bulk value (525 K); however $T_C = 580$ K for the Ni is severely depressed compared to the bulk value (627 K), possibly due to exchange effects or chemical inhomogeneities.

A strong temperature and measuring field dependence for both the coercivity and exchange field strength is present. The coercivity rapidly decreases with increasing temperature in large measuring fields (5 T) and remains essentially constant with small measuring fields (0.05 T). Most notably, the exchange field varies from positive to negative with rising temperature in a strong measuring field but remains positive with rising temperature in smaller measuring fields.

In addition to their technological attributes, magnetic nanocomposite materials exhibit interesting, non-intuitive physical properties such as an apparent increased Curie temperature of the lower- T_C constituent and anomalous high-tem-

perature coercivities. To investigate the origin of these phenomena quantification of the internal strain state of the individual phases in melt-quenched $\text{Nd}_2\text{Fe}_{14}\text{B} + \alpha\text{-Fe}$ nanocomposites was undertaken. Synchrotron x-ray diffraction studies were performed at the National Synchrotron Light Source, and the data was analyzed within the framework of a modified Williamson-Hall method. It was determined that the constituent $\text{Nd}_2\text{Fe}_{14}\text{B}$ grains are largely strain-free, but the $\alpha\text{-Fe}$ component exhibited significant stress (~ 1 GPa) with an associated strain of approximately 0.1%.

The results of this study helped to confirm that the enhanced T_C of $\text{Nd}_2\text{Fe}_{14}\text{B}$ was likely an exchange- and not stress-induced effect as well as provide a basis for the marginal remanence enhancement and anomalous elevated-temperature coercivities found in these nanocomposites.

Fe layers of 100 nm and 200 nm thickness evaporated onto slices of $\text{Gd}_5\text{Si}_{1.5}\text{Ge}_{2.5}$

are found to increase the dc initial susceptibility and shift the metamagnetic transition to low field. These effects lead to an enhancement of the magnetocaloric effect around the entropy peak temperature. At the current time, the mechanism underlying the observed

phenomena is not clear; candidate mechanisms are a magnetostatic effect, an exchange interaction between the ferromagnetic Fe layer and the first few nanometers of the $Gd_5Si_{1.5}Ge_{2.5}$ and a mechanical “clamping” effect from the layer.

This work was done by Dr. Laura J. Henderson Lewis of the Center for Functional Nanomaterials at Brookhaven National Laboratory. Visit www.cfn.bnl.gov/research/nanoassemblies/ for more information. Contact Dr. Lewis at lhlewis@bnl.gov.

Elastomer Filled With Single-Wall Carbon Nanotubes

Strength and stiffness increase with SWNT content.

NASA's Lyndon B. Johnson Space Center, Houston, Texas

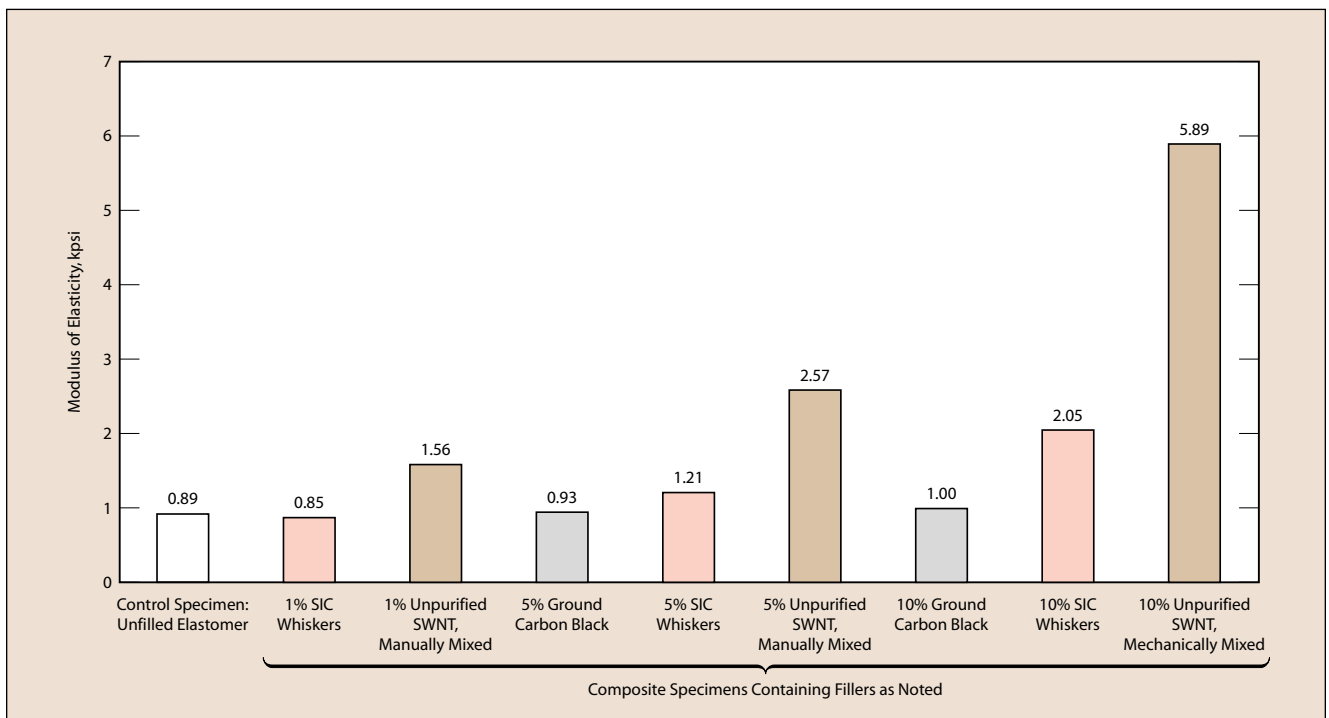
Experiments have shown that composites of a silicone elastomer with single-wall carbon nanotubes (SWNTs) are significantly stronger and stiffer than is the unfilled elastomer. The large strengthening and stiffening effect observed in these experiments stands in contrast to the much smaller strengthening effect observed in related prior efforts to reinforce epoxies with SWNTs and to reinforce a variety of polymers with multiple-wall carbon nanotubes (MWNTs). The relative largeness of the effect in the case

of the silicone-elastomer/SWNT composites appears to be attributable to (1) a better match between the ductility of the fibers and the elasticity of the matrix and (2) the greater tensile strengths of SWNTs, relative to MWNTs.

For the experiments, several composites were formulated by mixing various proportions of SWNTs and other filling materials into uncured RTV-560, which is a silicone adhesive commonly used in aerospace applications. Specimens of a standard “dog-bone” size and shape for

tensile testing were made by casting the uncured elastomer/filler mixtures into molds, curing the elastomer, then pressing the specimens from a “cookie-cutter” die.


The results of tensile tests of the specimens showed that small percentages of SWNT filler led to large increases in stiffness and tensile strength, and that these increases were greater than those afforded by other fillers. For example, the incorporation of SWNTs in a proportion of 1 percent increased the tensile strength by



The **Stiffness of a Silicone Elastomer** filled with several different kinds and proportions of reinforcing materials was measured in standard tensile tests.

44 percent and the modulus of elasticity (see figure) by 75 percent. However, the relative magnitudes of the increases decreased with increasing nanotube percentages because more nanotubes made the elastomer/nanotube composites

more brittle. At an SWNT content of 10 percent, the tensile strength and modulus of elasticity were 125 percent and 562 percent, respectively, greater than the corresponding values for the unfilled elastomer.

This work was done by Bradley S. Files and Craig R. Forest of NASA's Johnson Space Center. For further information, access the Technical Support Package (TSP) free online at www.techbriefs.com/tsp under the Materials category. Refer to MSC-23301 

Modifying Silicates for Better Dispersion in Nanocomposites

Processability and final material properties are improved.

NASA's John H. Glenn Research Center, Cleveland, Ohio

An improved chemical modification has been developed to enhance the dispersion of layered silicate particles in the formulation of a polymer/silicate nanocomposite material. The modification involves, among other things, the co-exchange of an alkyl ammonium ion and a monoprotonated diamine with interlayer cations of the silicate. The net overall effects of the improved chemical modification are to improve processability of the nanocomposite and maximize the benefits of dispersing the silicate particles into the polymer.

Some background discussion is necessary to give meaning to a description of this development. Polymer/silicate nanocomposites are also denoted polymer/clay composites because the silicate particles in them are typically derived from

clay particles. Particles of clay comprise layers of silicate platelets separated by gaps called "galleries." The platelet thickness is 1 nm. The length varies from 30 nm to 1 μ m, depending on the silicate. In order to fully realize the benefits of polymer/silicate nanocomposites, it is necessary to ensure that the platelets become dispersed in the polymer matrices. Proper dispersion can impart physical and chemical properties that make nanocomposites attractive for a variety of applications.

In order to achieve nanometer-level dispersion of a layered silicate into a polymer matrix, it is typically necessary to modify the interlayer silicate surfaces by attaching organic functional groups. This modification can be achieved easily by ion exchange between the interlayer metal cations found naturally in the sili-

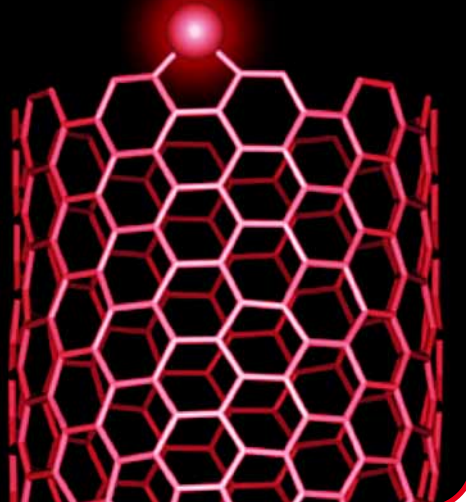
cate and protonated organic cations — typically protonated amines. Long-chain alkyl ammonium ions are commonly chosen as the ion-exchange materials because they effectively lower the surface energies of the silicates and ease the incorporation of organic monomers or polymers into the silicate galleries. This completes the background discussion.

In the present improved modification of the interlayer silicate surfaces, the co-ion exchange strengthens the polymer/silicate interface and ensures irreversible separation of the silicate layers. One way in which it does this is to essentially tether one amine of each diamine molecule to a silicate surface, leaving the second amine free for reaction with monomers during the synthesis of a polymer. In addition, the incorporation of alkyl ammonium ions

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into the galleries at low concentration helps to keep low the melt viscosity of the oligomer formed during synthesis of the polymer and associated processing — a consideration that is particularly important in the case of a highly cross-linked, thermosetting polymer. Because of the chemical bonding between the surface-modifying amines and the monomers,

even when the alkyl ammonium ions become degraded at high processing temperature, the silicate layers do not aggregate and, hence, nanometer-level dispersion is maintained.

This work was done by Sandi Campbell of NASA's Glenn Research Center. For further information, access the Technical Support Package (TSP) free on-line at

www.techbriefs.com/tsp under the Materials category.

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Commercial Technology Office, Attn: Steve Fedor, Mail Stop 4-8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17339. 



SENSORS

Sensor for Monitoring Nanodevice-Fabrication Plasmas

Temperature and trace amounts of chemical species could be measured *in situ*.

NASA's Ames Research Center, Moffett Field, California

The term "plasma process diagnostics" (PPD) refers to a spectroscopic technique and sensing hardware that have been proposed for monitoring plasma processes used to fabricate electronic devices that feature sizes as small as several nanometers. Nanometer dimensions are characteristic of the quantum level of miniaturization, where single impurity atoms or molecules can drastically change the local properties of the nanostructures. Such changes may be purposely used in nanoscale design but may also be extremely damaging or cause improper operation of the fabricated devices. Determination of temperature and densities of reactants near the developing features is important, since the structural synthesis is affected by characteristics of the local microenvironment. Consequently, sensors capable of nonintrusive monitoring with high sensitivity and high resolution are essential for real-time atomistic control of reaction kinetics and minimizing trace contamination in plasma processes used to fabricate electronic nanodevices. Such process-monitoring sensors are required to be compact, multiparametric, and immune to the harsh environments of processing plasmas. PPD is intended to satisfy these requirements.

The specific technique used to implement plasma diagnostics with a PPD sensor would be an advanced version of

continuous-wave cavity-ringdown spectroscopy (CW-CRDS) capable of profiling spectral line broadenings in order to derive both Doppler and Stark components. CRDS is based on measurements of the rate of absorption of laser light in an optical resonator. The ultimate sensitivity results from a very long absorption path length within the cavity and immunity to variations in incident laser intensity. The proposed version of this technique would involve the use of multiplexing tunable laser diodes and an actively modulated high-reflectivity optical resonator, thus offering a synergistic combination of simplicity, compactness, high sensitivity, and high resolution.


The multiplexing capabilities of diode lasers could be utilized to make the PPD sensor a single, simple, compact, and inexpensive tool for the acquisition of multiparametric data. A PPD sensor would be capable of continuous measurement of such physical parameters as gas temperature, gas velocity, electron number density, and absolute densities of reacting chemical species. A laser beam can be easily adjusted to analyze the immediate vicinity of the growing nanostructures (or features etched down) in real time. The absorption enhancement in an optical cavity would afford the sensitivity needed for measurement of the temperature and densities of species at concentrations sig-

nificantly lower than measurable by other nonintrusive techniques.

It is anticipated that fully developed PPD sensors would enable simultaneous measurement of local temperature and determination of plasma species responsible for the synthesis and functionalization of nanodevices. These sensors would also enable tracking the pathways and origins of damaging contaminants, thereby providing feedback for adjustment of processes to optimize them and reduce contamination. The PPD sensors should also be useful for optimization of conventional microelectronics manufacturing plasma processes.

Going beyond plasma processes for fabrication of electronic devices, PPD sensors could be used for monitoring of atoms, molecules, ions, radicals, clusters, and particles in a variety of other settings, including outer space. Because of their high sensitivity, such sensors could also prove useful for detecting traces of illegal drugs and explosives.

This work was done by Alexander Bol'shakov while he held a National Research Council associateship award at NASA's Ames Research Center.

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-15084-1. 



Ordered Nanostructures Made Using Chaperonin Polypeptides

This method exploits the ability of chaperonins to assemble into complex structures.

NASA's Ames Research Center, Moffett Field, California

A recently invented method of fabricating periodic or otherwise ordered nanostructures involves the use of chaperonin polypeptides. The method is intended to serve as a potentially superior and less expensive alternative to conventional lithographic methods for use in the patterning steps of the fabrication of diverse objects characterized by features of the order of nanometers. Typical examples of such objects include arrays of quantum dots that would serve as the functional building blocks of future advanced electronic and photonic devices.

A chaperonin is a double-ring protein structure having a molecular weight of about 60 ± 5 kilodaltons. In nature, chaperonins are ubiquitous, essential, subcellular structures. Each natural chaperonin molecule comprises 14, 16, or 18 protein subunits, arranged as two stacked rings approximately 16 to 18 nm tall by approximately 15 to 17 nm wide, the exact dimensions depending on the biological species in which it originates. The natural role of chaperonins is unknown, but they are believed to aid in the correct folding of other proteins, by enclosing unfolded proteins and pre-

venting nonspecific aggregation during assembly.


What makes chaperonins useful for the purpose of the present method is that under the proper conditions, chaperonin rings assemble themselves into higher-order structures. This method exploits such higher-order structures to define nanoscale devices. The higher-order structures are tailored partly by choice of chemical and physical conditions for assembly and partly by using chaperonins that have been mutated. The mutations are made by established biochemical techniques.

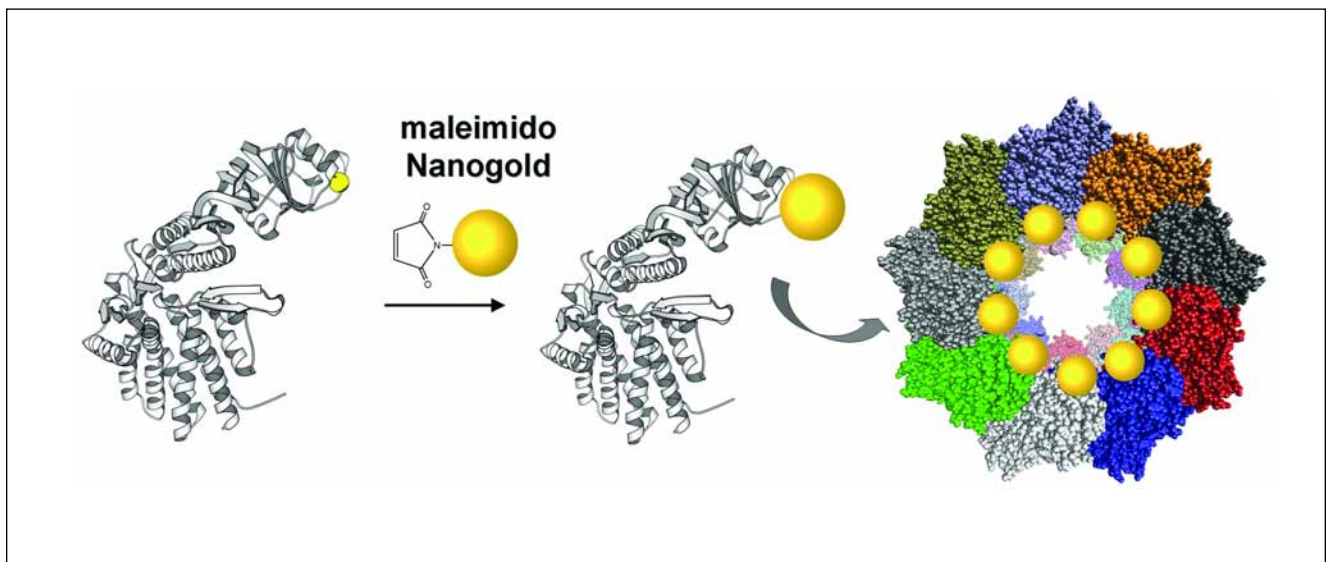
The assembly of chaperonin polypeptides into such structures as rings, tubes, filaments, and sheets (two-dimensional crystals) can be regulated chemically. Rings, tubes, and filaments of some chaperonin polypeptides can, for example, function as nanovessels if they are able to absorb, retain, protect, and release gases or chemical reagents, including reagents of medical or pharmaceutical interest. Chemical reagents can be bound in, or released from, such structures under suitable controlled conditions.

In an example of a contemplated application, a two-dimensional crystal of chap-

eronin polypeptides would be formed on a surface of an inorganic substrate and used to form a planar array of nanoparticles or quantum dots. Through genetic engineering of the organisms used to manufacture the chaperonins, specific sites on the chaperonin molecules and, thus, on the two-dimensional crystals can be chemically modified to react in a specific manner so as to favor the deposition of the material of the desired nanoparticles or quantum dots. A mutation that introduces a cysteine residue at the desired sites on a chaperonin of *Sulfolobus shibatae* was used to form planar arrays of gold nanoparticles (see figure).

This work was done Jonathan Trent, Robert McMillan, and Chad Paavola of NASA's Ames Research Center; Rakesh Mogul of University of California, Santa Cruz; and Hiromi Kagawa of SETI Institute. For further information, access <http://www.ipt.arc.nasa.gov/trent.html> and <http://amesnews.arc.nasa.gov/audio/bionanosound/bionano9.html>.

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-14744. 



This Planar Array of Gold Nanoparticles was formed on a carbon film by the method summarized in the text.

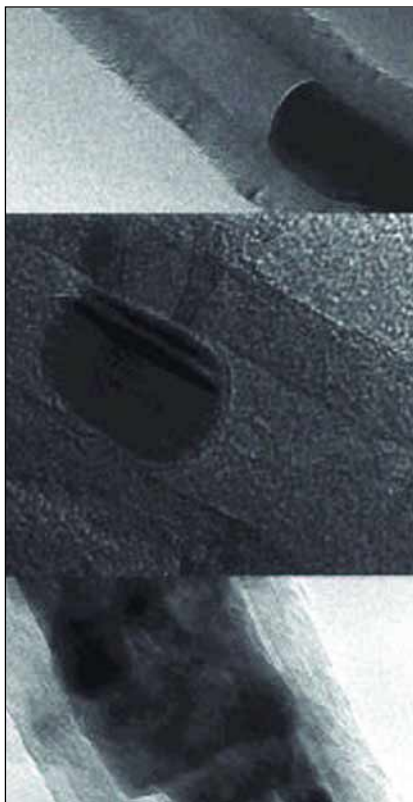
Simple Fabrication of Very Long Carbon Nanotubes

This inexpensive process yields very long carbon nanotubes with narrow-diameter distribution.

Ernest Orlando Lawrence Berkeley Laboratory, U.S. Department of Energy, Berkeley, CA

A simple, inexpensive, high-yield process requiring no special apparatus has been devised for fabrication of high-quality, very long carbon nanotubes with narrow-diameter distribution. This method improves upon existing arc discharge, laser ablation, and chemical vapor deposition (CVD) methods, all of which have various problems, including lack of control of catalyst/precursor ratios in starting materials and products; the need for fabrication of specialized apparatus; limited area coverage of nanotube mats (which translates into a low yield); and the need for overly high temperatures for fabrication.

The new process successfully fabricates large-area, freestanding films of very long carbon nanotubes by heating a transition metal complexed alkyne with aryl and/or alkyl substituents. The heating of the transition metal complex can be completed in 0.5 to 3.0 hours at 550 to 700°C in a sealed vessel under partial pressure of argon or helium (in contrast to typical chemical vapor deposition, which involves a flow of organic compound over a transition metal catalyst).




TEM (200 keV, JEOL 200 CX microscope) images of **Carbon Nanotubes**.

The heating results in high yields of carbon nanotubes as freestanding films or nanotube mats, which can be simply lifted off the inside of the vessel. Coverage of areas more than 4 cm² has been achieved from about 30 mg of the starting complex. Compared to other processes, the yields are essentially quantitative.

The removal of the transition metal from the films is a simple process, achieved by immersing the film in aqueous hydrochloric acid and filtering out the dissolved metal. This allows for greater purity in the final carbon nanotube yield.

Applications for the technology include field emitters, high-strength fibers, strong radiation shields, energy-absorbing materials, nanoscale catalytic beds, nanocircuits, nanoscale transistors, frictionless nanobearings, and electro-mechanical and optoelectronic devices.

This research was performed by K.P. Vollhardt and V.S. Iyer of Lawrence Berkeley National Lab. For further information, contact Pam Seidenman, Technology Transfer Dept., 510-486-6461, e-mail: ttd@lbl.gov, or visit www.lbl.gov/tt. Refer to Technology #IB-1719. 

Conductive Adhesives Enable Assembly of MEMS Chips

Flip chip mounting enables fragile MEMS devices to be assembled without damaging process steps.

Polymer Assembly Technology, Inc., Billerica, Massachusetts

Some MEMS (MicroElectroMechanical System) chip designs are simply too fragile for traditional solder flip chip assembly. Examples would include MOEM (MicroOptoElectroMechanical) mirror arrays with delicate balance beams, and fluidic and DNA analysis chips that cannot tolerate exposure to either high temperatures or aggressive cleaning procedures

to remove flux residues. For sensitive components such as these, electrically conductive, silver-filled adhesives may be the only available option for first-level chip assembly.

Conductive epoxies typically are cured at temperatures ranging between only 70-150°C compared with 235-265°C for reflow-soldering and require no post-

cleaning procedure. They are also surprisingly good electrical conductors when compared to solder. A typical flip chip connection made with conductive epoxy ranges from 6-10 milliohms, compared to an equivalently sized solder connection with 3-5 milliohms resistance.

Automated dispensing equipment is often employed when depositing non-

conductive “underfill” epoxies underneath flip chip mounted devices to increase chip interconnect reliability, but is less effective when applying conductive epoxy bumps. Unlike their non-conductive counterparts, the silver-filled epoxies are more viscous and prone to particle agglomeration, which can clog smaller dispensing needles. Dot size uniformity can therefore vary considerably and is presently limited to an approximately 125 μm diameter. A more practical approach, especially for high pin-count applications when pitch dimensions between flip chip bumps are small and when processing whole wafers, is the stencil printing process.

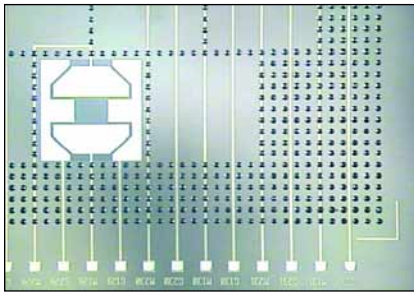


Figure 1: **MOEM Test Substrate** illustrating conductive epoxy spreading pattern.

An array of stencil-printed epoxy bumps can be seen in Figure 1, which was taken from a section of a 3D MOEM mirror array substrate. This unit required accurate placement of the mirror cells in three axes. The Z-height needed to be controlled by a soft placement tech-

nique onto a tightly packed pattern of conductive epoxy bumps that would merge into a contiguous conductive thin film. The MEMS mounting surface included regions with very thin, fragile walls separating adjacent mirror cells. In these sections, only a single row of bumps was deposited to prevent any epoxy from spreading into the cell's interior region and potentially interfering with the mirror's movement.

In this application, over 40,000 bumps were required per doublet-cell. The stencil-printing process required only a few seconds to perform this process step, yielding a near-perfect array of uniform conductive bumps. Attempting to deposit this pattern using an automatic dispenser would have consumed far too much time, and the epoxy would have begun to set up before the pattern could be deposited and transferred into the placement machine. Many low-temperature-curing (<100°C) conductive epoxies have a short “pot-life” and begin to harden within a few hours, placing a restriction on the process cycle time.

The stencil-printing process also can be adapted for multiple prints using different epoxy materials on the same surface. Another application, for example, required a MEMS device be attached to an Au-coated silicon substrate atop an integral 5- μm -high insulator. First, a pattern of 5- μm -high dielectric epoxy bumps was stencil-printed and cured, followed by a separate print depositing an outer

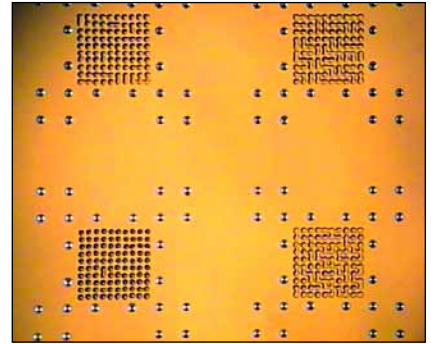


Figure 2: **MEMS Device Mounting Substrate** with stencil-printed, conductive, and dielectric epoxy patterns.

pattern of conductive epoxy bumps to electrically and mechanically secure the MEMS device to the substrate surface (Figure 2). The required 5- μm -high insulator bumps were achieved by determining the right combination of stencil aperture and printing parameters for the particular viscosity of the dielectric epoxy used. The conductive epoxy in this case was a b-staged, solvent-based, thermoplastic material to which the MEMS device was subsequently thermo-compression bonded.

As wafer scale packaging techniques are considered for MEMS devices, the advantages of using stencil-printed polymer materials should not be overlooked.

This article was written by James Clayton, President of Polymer Assembly Technology, Inc. For further information, visit www.polymerassemblytech.com, or call 1-978-667-0071.

Growing Aligned Carbon Nanotubes for Interconnections in ICs

Carbon nanotubes are embedded in silica with their tips exposed as contacts.

NASA's Ames Research Center, Moffett Field, California

A process for growing multiwalled carbon nanotubes anchored at specified locations and aligned along specified directions has been invented. Typically, one would grow a number of the nanotubes oriented perpendicularly to a silicon integrated-circuit (IC) substrate, starting from (and anchored on) patterned cat-

alytic spots on the substrate. Such arrays of perpendicular carbon nanotubes could be used as electrical interconnections between levels of multilevel ICs.

The process (see Figure 1) begins with the formation of a layer, a few hundred nanometers thick, of a compatible electrically insulating material (e.g., SiO_x or Si_3N_2)

on the silicon substrate. A patterned film of a suitable electrical conductor (Al, Mo, Cr, Ti, Ta, Pt, Ir, or doped Si), having a thickness between 1 nm and 2 μm , is deposited on the insulating layer to form the IC conductor pattern. Next, a catalytic material (usually, Ni, Fe, or Co) is deposited to a thickness between 1 and 30 nm on the

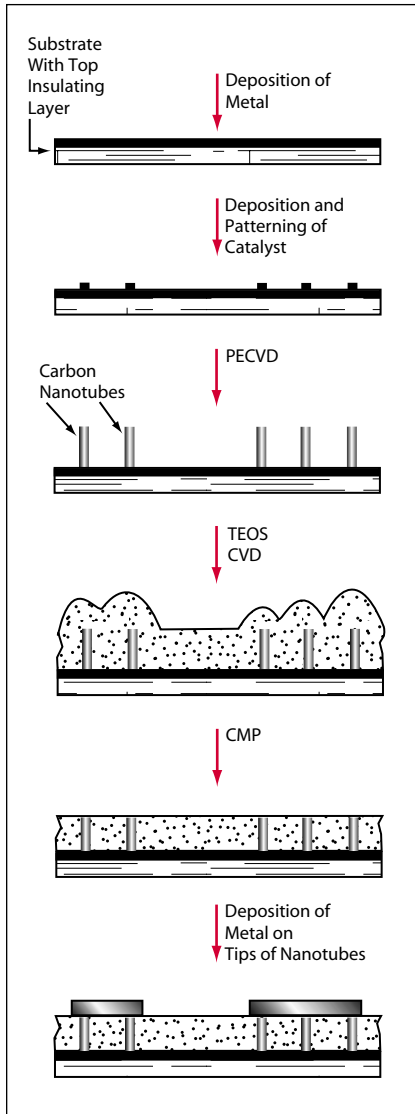


Figure 1. A **Bottom-Up Approach** is followed in growing carbon nanotubes, embedding them in a silica matrix, exposing their tips, and using the exposed tips as electrical contacts.

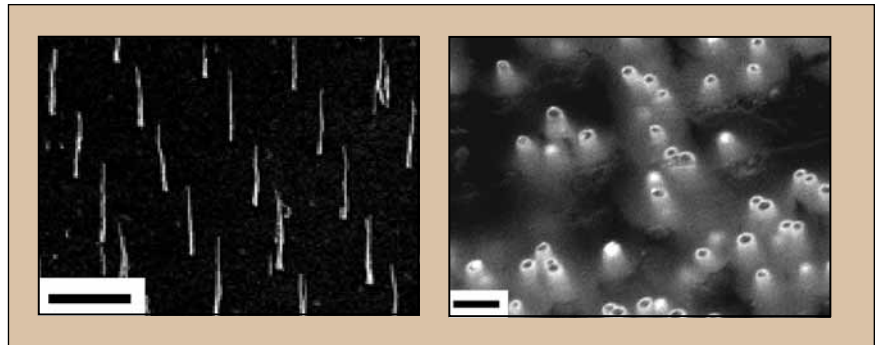


Figure 2. These **Electron Micrographs**, both taken at an angle of 45°, show specimens of arrays of carbon nanotubes at two different stages of processing. (The scale bar on the left is 5 μm, the one on the right is 0.2 mm.)


spots from which it is desired to grow carbon nanotubes.

The carbon nanotubes are grown by plasma-enhanced chemical vapor deposition (PECVD). Unlike the matted and tangled carbon nanotubes grown by thermal CVD, the carbon nanotubes grown by PECVD are perpendicular and freestanding because an electric field perpendicular to the substrate is used in PECVD. Next, the free space between the carbon nanotubes is filled with SiO₂ by means of CVD from tetraethylorthosilicate (TEOS), thereby forming an array of carbon nanotubes embedded in SiO₂. Chemical mechanical polishing (CMP) is then performed to remove excess SiO₂ and form a flat-top surface in which the outer ends of the carbon nanotubes are exposed. Optionally, depending on the application, metal lines to connect selected ends of carbon nanotubes may be deposited on the top surface.

The left part of Figure 2 is a scanning electron micrograph (SEM) of carbon nan-

otubes grown, as described above, on catalytic spots of about 100 nm diameter patterned by electron-beam lithography. These and other nanotubes were found to have lengths ranging from 2 to 10 μm and diameters ranging from 30 to 200 nm, the exact values of length depending on growth times and conditions and the exact values of diameter depending on the diameters and thicknesses of the catalytic spots. The right part of Figure 2 is an SEM of an embedded array of carbon nanotubes after CMP.

This work was done by Jun Li, Qi Ye, Alan Cassell, Hou Tee Ng, Ramsey Stevens, Jie Han, and M. Meyyappan of NASA's Ames Research Center. For further information, access the Technical Support Package (TSP) free on-line at www.techbriefs.com/tsp under the Manufacturing category.

Inquiries concerning rights for the commercial use of this invention should be addressed to the Patent Counsel, Ames Research Center, (650) 604-5104. Refer to ARC-15042-1. 

BIOTECHNOLOGY

Quantum-Dot-Based Cell Motility, Invasion, and Metastasis Assays

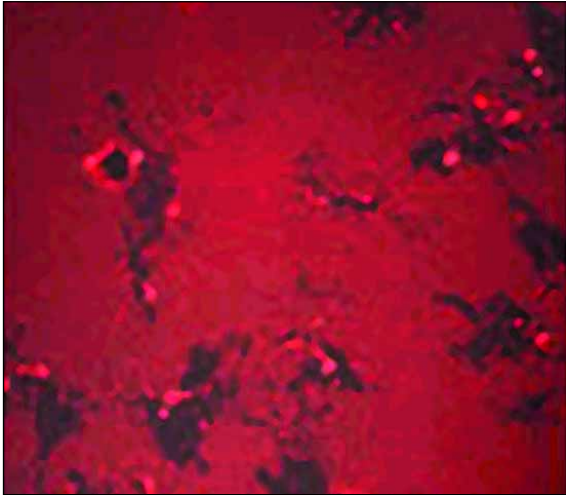
A new technique enables cell motility and migration studies.

Ernest Orlando Lawrence Berkeley National Laboratory, U.S. Department of Energy, Berkeley, CA

Researchers have discovered a powerful tool for studying cell mobility and migration — behaviors that are

responsible for metastases of primary cancers. The researchers compared the motions of cancerous and healthy

human breast cells, as well as several other cell types, as they migrated across a layer of colloidal semiconductor



Engulfment of Nanocrystals reveals migratory paths of breast tumor cells. A wide variety of mammalian cells engulf quantum dots when migrating. This migration is correlated to the cell's metastatic potential. After 24 hours, dark clearings in the nanocrystal layer are observed around motile tumor cells.

nanocrystals. As they move, they engulf the quantum dots and leave behind a phagokinetic track that yields information about the health of the cells.

Migration of cells and their metastatic potential are well known to be correlated.


Colloidal quantum dots are robust and efficient light emitters that have been used for static biological labeling. They are superior to organic dyes, which fade quickly and perturb the assayed cells. These nanocrystals are spontaneously ingested by a wide variety of cells while remaining fully luminescent, enabling researchers to examine live cells over extended time periods, and to quantify changes in response to various molecular manipulations.

This new technique is less labor-intensive than the Boyden Chamber invasion assay method. It does not require killing the cells, and it enables

real-time variation of external conditions and analyses of cellular responses. It also overcomes the marker problems encountered in the original phagokinetic tracking methods.

This method can be coupled to information about chemical signals and allows a wide variety of tissue culture substrates to be used, including growth on all extracellular matrix substances.

The improved cell motility studies and rapid assaying of metastatic potential enabled by this quantum dot method will provide improved diagnostics and enhanced information for cancer drug development.

This research was performed by Carolyn Larabell, Paul Alivisatos, W.J. Parak, R. Boudreau, M. LeGros, D. Gerion, D. Zanchet, C.M. Micheel, and S.C. Williams of Lawrence Berkeley National Lab. For further information, contact Pam Seidenman, Technology Transfer Dept., 510-486-6461, e-mail: ttd@lbl.gov, or visit www.lbl.gov/tt. Refer to Technology #IB-1755. 



SOFTWARE

Speeding Up the Internet With MEMS Technology

Simulation software is used to design a MEMS optical switch.

ALGOR, Inc., Pittsburgh, Pennsylvania and SiWave, Arcadia, California

As Internet traffic continues to grow, so does consumer demand for increased bandwidth to support data-intensive multimedia applications. In response, telecommunications companies are expanding and upgrading their fiber-optic networks. A key to improving performance is the use of new optical switches, which can redirect signals among thousands of different ports. Optical switch systems offer improvements over alternate methods in speed, data capacity, data management, and cost.

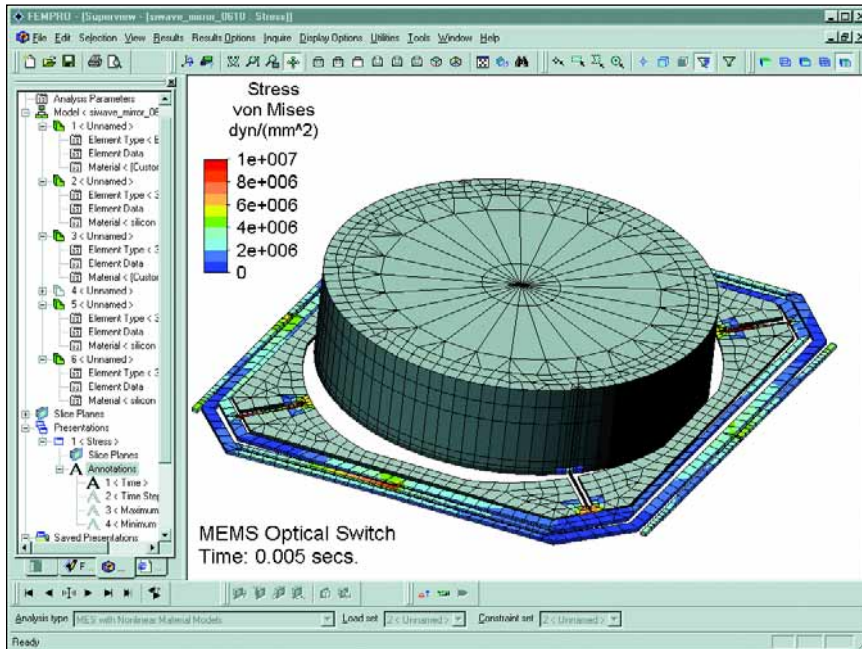
SiWave, a supplier of optical switching components and subsystems, is developing an optical switch using microelectromechanical systems (MEMS) technology.

MEMS are micromachines the size of a grain of salt or the eye of a needle that integrate mechanical elements, sensors, actuators, and electronics on a common silicon substrate. MEMS devices are fabricated using photolithographic-based semiconductor processes that selectively etch away parts of the silicon wafer or add new layers to form the various devices. The unique characteristics of MEMS devices allow the manufacture of extremely small optical switching products that provide new levels of capability, reliability, and complexity at a relatively low cost.

The telecommunications industry has stringent shock specifications for all

components — even if they can fit on the head of a pin. According to Telcordia standards, all components must be able to survive a 500-g, 1-millisecond, 1/2-sine pulse. Equivalent to dropping a rigid object six feet onto concrete, this standard ensures that components will withstand shocks they might see in the field; for example, during installation or when a technician thrusts a card into an adjacent rack. For geometrically complex devices such as a MEMS optical switch, the specification becomes an important design consideration.

SiWave used ALGOR finite element analysis-based simulation software in the development of this product to predict



ALGOR Mechanical Event Simulation (MES) software was used to calculate **Stress and Displacement Results** over time for an applied shock load on the MEMS optical switch. The image shows a display of von Mises stress results.

the performance and durability of the device. The 1-mm optical switch mirror was modeled in SolidWorks and InCAD technology was used to seamlessly capture the model for use in ALGOR. After creating an automatic mesh, it was refined in Superdraw, ALGOR's finite element modeling tool.

Anticipating that the optical switch might be susceptible to shock, a built-in 100-Hz frequency isolation system within

the MEMS device's packaging was planned that would provide about 40% damping. The optical switch within the isolation system would actually experience a peak acceleration of 163 g's. It was this attenuated shock value that was input into the simulation.

Because critical resonance frequencies were high compared to the duration of the shock, an inertial acceleration method was considered as a good way to approx-

imate the behavior of the mirror under shock conditions. This method required no special constraints, only a standard gravity input and a time history curve. Although the Telcordia standards call for a 1/2-sine pulse, the isolation system affects the shape as well as the magnitude of the shock load — resulting in a sine-shaped load — so that the optical switch actually will get shaken in two directions within just a few milliseconds.

In addition to the shock load, published, orthotropic material properties for the silicon and silicon compounds that comprise the optical switch were input. Also, the surfaces of the part that were most likely to experience possible contact pairs were examined, and restricted contact pairs were defined.

Both von Mises stress and displacement results were examined. One concern for this component is the orientation into which it displaces. If the part were to contact the packaging, it could get stuck. The mirror's motion could not result in interference with surrounding components. Another concern was that the device might drum itself against its base, so it was important to know whether contact occurred and the stresses that resulted from it.

This work was done by Robert Calvet of SiWave, Inc. To read this complete story, visit <http://SiWavetechnology.ALGOR.com>. For more information on ALGOR, visit www.ALGOR.com.

Using Mainstream CAD Software in MEMS and Micro-Mechanical Design

Solid modeling tools can simplify the complexity of the MEMS design process.

SolidWorks Corporation, Concord, Massachusetts

Until recently, microelectromechanical systems (MEMS) and micro-mechanical design and production were more often research-oriented activities that took place in university labs, than commercially viable manufacturing enterprises. The design and manufacturing tools used for MEMS and micro-mechanical design were highly special-

ized, and very few engineers knew how to accomplish micro-design without developing manufacturing techniques and enabling technologies as part of the design process. Much of this work was done using 2D layouts to represent configurations of the separate layers of silicon that are deposited sequentially to create a conventional MEMS component.

One development that is helping to facilitate the widespread use of MEMS and micro-design techniques is the inclusion of MEMS-specific functionality in mainstream 3D computer-aided design (CAD) applications. The ability to use a familiar tool and design environment for designs ranging from MEMS and micro devices to larger assemblies

and components eliminates the time, effort, and cost involved with learning specialized tools.

The typical sequence for designing a MEMS device is to begin with a model of the component created out of multiple semiconductor layers. Designers then produce photo masks — 2D layouts for each layer that match each specific cross-section configuration to drive manufacturing. A significant challenge in MEMS design arises in working with photo masks for several cross-sections of a solid model at the micron and sub-micron level for a device that will be packaged in a much larger assembly. Without MEMS-specific CAD functionality, designers must move back and forth in their CAD systems between different dimensional scales, from microns to millimeters to meters; are unable to truly visualize the complete assembly; and cannot take advantage of basic solid modeling features such as parametric associative design.

Solid modeling systems simplify the complexity of the process by providing capabilities that specifically address MEMS design functions. For example, SolidWorks® 3D CAD software enables a broad geometric range, which allows designers to work on the same assembly at the micron level all the way up to many

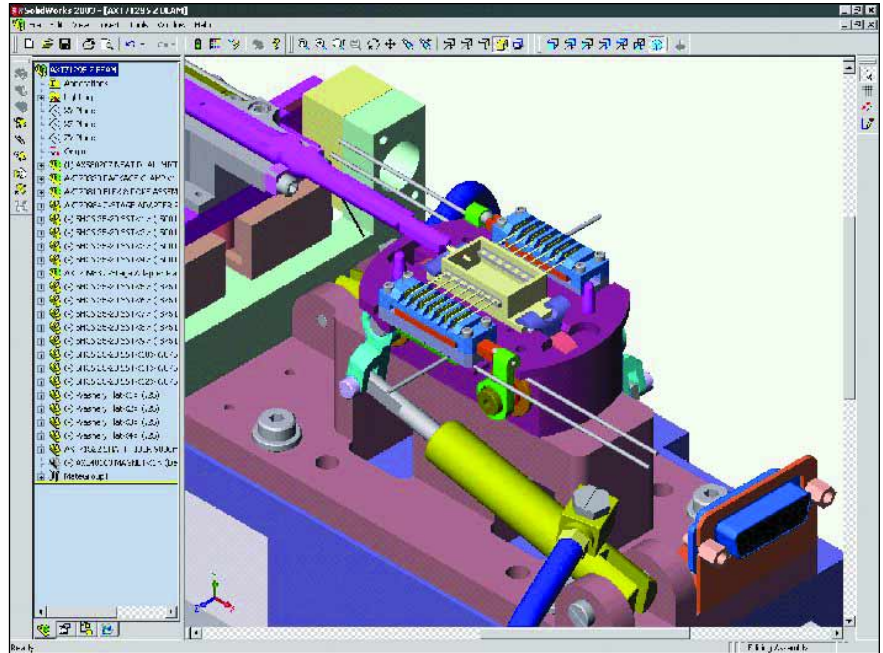



Figure 1. **This Automated Packaging Cell** was designed with SolidWorks software, which enables engineers to design the MEMS device, output the photo mask, design the packaging, and put it together in a single software package.

meters. Engineers can zoom into the MEMS detail and zoom out to the larger assembly, providing full 3D visualization of both the MEMS component and its packaging. The software also automatically cross-sections the MEMS component and creates fully associative photo masks for each layer. As the design is modified

and refined, changes propagate to all associated design documents, including components, assemblies, detail, and photo-mask drawings. Sub-micron feature definition, collision/interference detection of components, and the creation of feature patterns and patterns of patterns are capabilities that are also useful for MEMS design.

Another important benefit in using mainstream CAD for MEMS design is the ability to conduct structural, thermal, electromagnetic, and fluid flow analyses directly on the solid model. Finite element analysis (FEA) is extremely important for predicting the behavior and performance of a MEMS component or micro device before producing costly prototypes.

The introduction of MEMS-specific capabilities in mainstream design tools is helping to pave the way to greater use of micro technology. These tools will help companies facing miniaturization challenges to break new ground; to innovate products, devices, and micro-manufacturing technologies; and to broaden the use of MEMS systems for a variety of applications.

This article was written by Ilya Mirman, Vice President of Marketing, SolidWorks Corp. Visit www.solidworks.com for more information. 

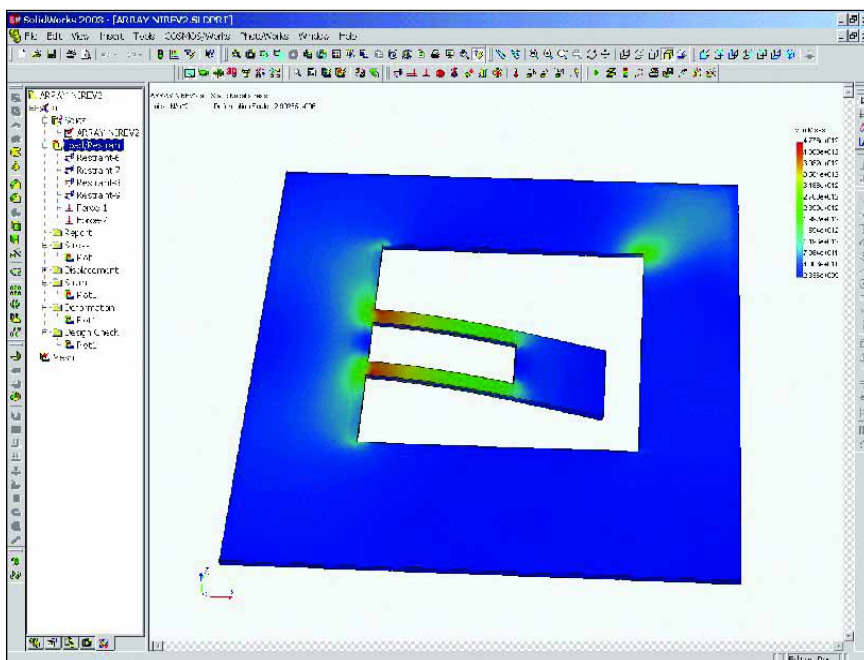


Figure 2. **Finite Element Analysis**, such as the stress analysis shown here, is important for predicting the behavior and performance of a MEMS component before producing costly prototypes.



Nanofluidic Size-Exclusion Chromatograph

This device would perform the functions of a much larger instrument.

NASA's Jet Propulsion Laboratory, Pasadena, California

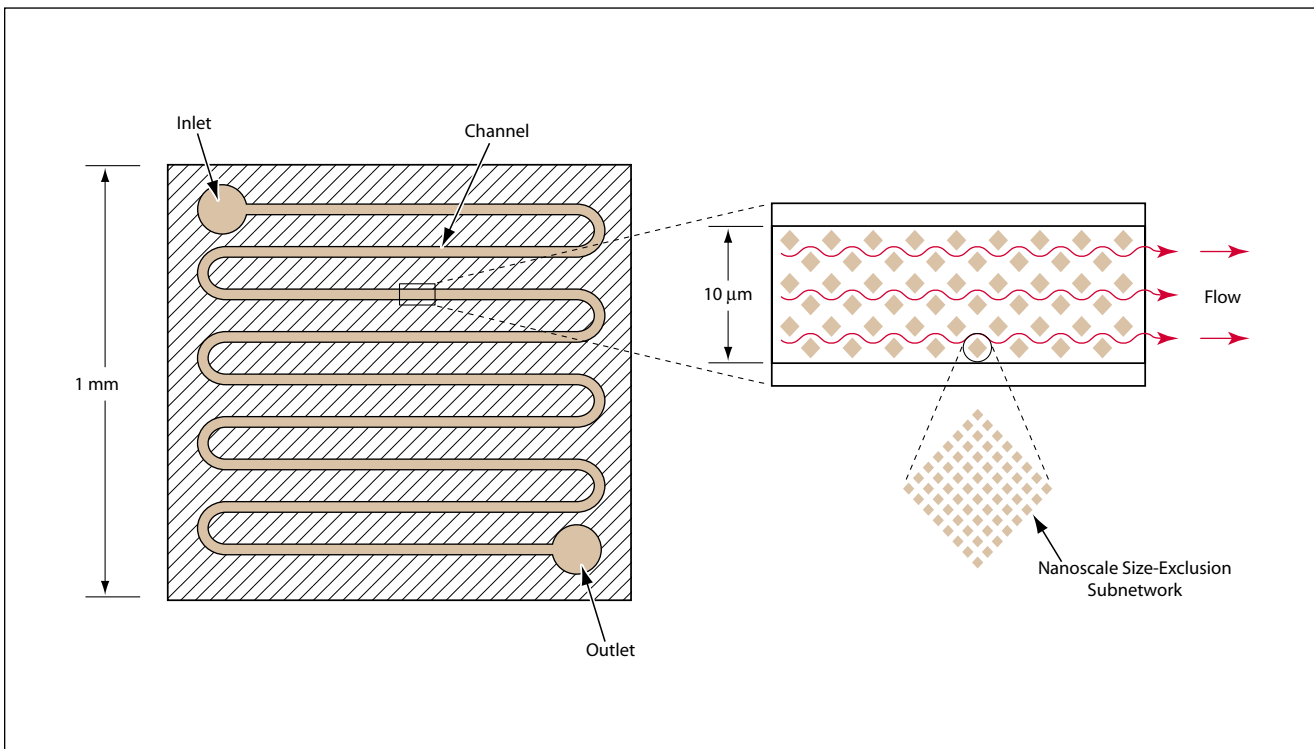
Efforts are under way to develop a nanofluidic size-exclusion chromatograph (SEC), which would be a compact, robust, lightweight instrument for separating molecules of interest according to their sizes and measuring their relative abundances in small samples. About as large as a deck of playing cards, the nanofluidic SEC would serve, in effect, as a "laboratory on a chip" that would perform the functions of a much larger, conventional, bench-top SEC and ancillary equipment, while consuming much less power and much smaller quantities of reagent and sample materials. Its compactness and low power demand would render it attractive for field applications in which, typically, it would be used to identify and quantitate a broad range of polar and nonpolar organic compounds in soil, ice, and water samples.

Size-exclusion chromatography is a special case of high-performance liquid chromatography. In a conventional SEC,

a sample plug is driven by pressure along a column packed with silica or polymer beads that contain uniform nanopores. The interstices between, and the pores in, the beads collectively constitute a size-exclusion network. Molecules follow different paths through the size-exclusion network, such that characteristic elution times can be related to sizes of molecules: basically, smaller molecules reach the downstream end of the column after the larger ones do because the smaller ones enter minor pores and stay there for a while, whereas the larger ones do not enter the pores. The volume accessible to molecules gradually diminishes as their size increases. All molecules bigger than a pore size elute together. For most substances, the elution times and sizes of molecules can be correlated directly with molecular weights. Hence, by measuring the flux of molecules arriving at the downstream end as a function of time, one can obtain a liquid mass

spectrum for the molecules present in a sample over a broad range of molecular weights.

The developmental nanofluidic SEC is based on the same size-separation principle as that of a conventional SEC. However, instead of a packed macroscopic column containing porous beads, the nanofluidic SEC would contain a size-exclusion network in a miniature column in the form of a microscopic channel containing nanometer-scale features (see figure). More specifically, the nanometer-scale features in the channel would be sized, shaped, and positioned to define a matrix of micron-width subchannels topped with a gap of varying thickness of the order of tens of nanometers. The miniature column would be fabricated by established techniques now used to produce integrated circuits (ICs) and microelectromechanical systems (MEMS). One or more device(s) to detect molecules could be integrated onto the column



A Size-Separation Network in a Miniature Channel on a chip would contain nanoscale features. The channel and features would be formed as one piece in a chip by use of IC and MEMS fabrication techniques.

chip at the downstream end. These devices could be based, for example, on electrochemical (in particular, amperometric) and laser-induced-fluorescence detection techniques.

This work was done by Sabrina Feldman, Danielle Svehla, Frank Grunthaler, Jason Feldman, and P. Shakkottai of Caltech for NASA's Jet Propulsion Laboratory.

For further information, access the Technical Support Package (TSP) free on-line at www.techbriefs.com/tsp under the Physical Sciences category.

In accordance with Public Law 96-517, the contractor has elected to retain title to this invention. Inquiries concerning rights for its commercial use should be addressed to

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(818) 354-2240*

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Refer to NPO-30499.*

Scanning Nanomechanics

A hybrid nanoindentation instrument is capable of measuring dynamic materials properties.

Naval Research Laboratory, Chemistry Division, U.S. Department of Defense, Washington, DC

The invention of the scanning tunneling microscope (STM) and atomic force microscope (AFM) in the 1980s has led to rapid commercialization of microscopes that image surfaces with atomic resolution. As a result, materials analyses capabilities at the nano scale have expanded enormously and now include magnetic, dielectric, tribological, electrochemical, thermal, and mechanical properties.

However, despite the many advances in commercially available instrumentation, dynamic mechanical analysis at this scale has been an elusive goal. To resolve this problem, we have developed a hybrid nanoindentation system capable of measuring quantitatively dynamic materials properties. The output of this instrument also can be in the form of an image or map of mechanical response or property (e.g., stiffness, modulus).

High-resolution mapping of mechanical properties is possible through the use of a hybrid nanoindenter that combines depth-sensing nanoindentation with an AFM. This combination enables quantitative nanomechanical properties analyses with nanometer-scale positioning and topographical mapping. These scanning and positioning capabilities allow investigations of materials and structures (e.g., composites, nanostructured materials, lithographic patterns, or microelectromechanical systems (MEMS)) that have features below the optical limit.

In an indentation experiment, mechanical properties are evaluated using a rigid probe of well-defined shape (e.g., a pyramid or sphere) to elastically or plastically deform a sample while moni-

ing the load and displacement response. We have improved the sensitivity of the hybrid nanoindenter by introducing a small sinusoidal component to the indentation force and detecting the displacement signal with a lock-in amplifier. The result is a dynam-

ic measurement of contact stiffness that is related to both the contact size and elastic properties of the contacting materials. Two significant capabilities arise from these dynamic stiffness measurements. First, by scanning the sample under the oscillating probe at low loads (elastically), a two-dimensional map of the dynamic stiffness of the sample can be obtained. Second, by varying the frequency of the oscillations, we can investigate dynamic mechanical properties inherent in many polymers and biomaterials. Figure 1 is a schematic of the instrument.

Figure 2(a) shows a 10mm x 10mm image mapping the elastic (storage) modulus of a carbon fiber-epoxy composite. Contrast in the image, as well as the rendered height, correspond directly to modulus with lighter regions having higher modulus. Figure 2(b) shows a cross section through the center of the image of the elastic modulus. The image shows that the center of the carbon fiber has a lower modulus than the periphery, while the epoxy has a substantially lower modulus than the fiber.

The elastic modulus values obtained during the modulus mapping experiment were consistent with measured values from standard indentation experiments. More importantly, the low loads used during modulus mapping minimized the contact area between the probe and sample, thereby increasing the lateral resolution of the technique.

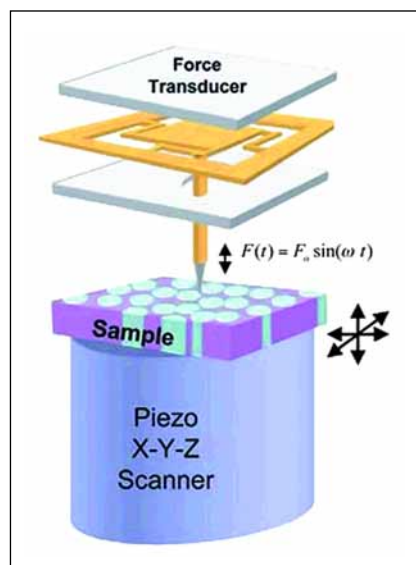


Figure 1. The Hybrid Scanning Nanoindenter. The load-displacement response of a probe attached to a movable plate is actuated electrostatically to apply force; the displacement response is monitored by capacitive techniques. Sample x-y positioning and scanning are accomplished through a piezo-tube scanner.

toring the load and displacement response. We have improved the sensitivity of the hybrid nanoindenter by introducing a small sinusoidal component to the indentation force and detecting the displacement signal with a lock-in amplifier. The result is a dynam-

Stiffness images can be acquired over a range of frequencies up to ~250 Hz, allowing investigation of dynamic mechanical properties of viscoelastic materials. Figure 3(a) and (b) shows two contact stiffness images of a cross section of a layered polystyrene (PS) sample, with alternating low (PS1) and high (PS2) molecular weights. The lighter regions in the images correspond to higher contact stiffness. The two images were taken from the same region of the sample while oscillating the probe at 105 and 200 Hz, respectively. At 200 Hz, the image contrast is reversed, indicating a strong frequency-dependent response of the two PS materials within this frequency range.

The image contrast reversal reflects a change in contact stiffness caused by the frequency-dependent dissipative properties of the two polymers. Figure

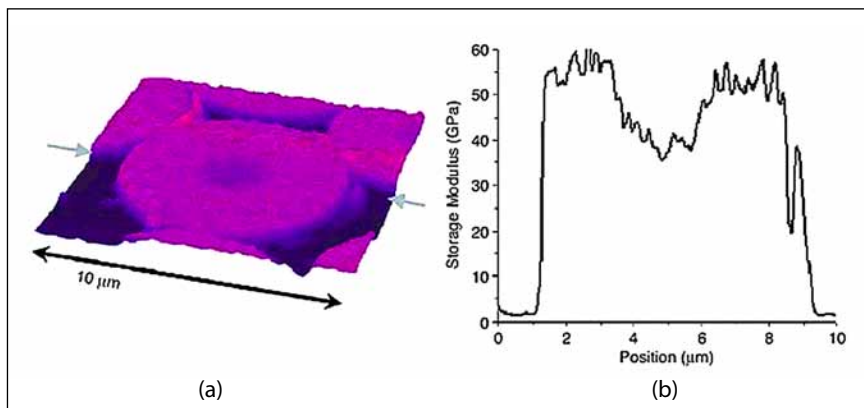


Figure 2. A two-dimensional Map of the **Elastic Modulus** (a) of a carbon fiber-epoxy composite material. Brighter regions correspond to higher modulus. (b) Cross-section line scan through the center of the image shows the storage modulus in the epoxy and the modulus gradient at the center of the fiber.

3(c) plots the dynamic compliance (1/stiffness) of the nanoindenter probe in contact with the two polymers as a function of frequency. This plot shows

that the stiffness of PS1 should be lower than PS2 at 105 Hz, but higher than PS2 at 200 Hz. Because the compliance maxima (and equivalent stiffness minima) of the polymers occurs near 100 Hz for PS1 and near 200 Hz for PS2, dynamic imaging at these two frequencies results in a stiffness contrast reversal due to the change in relative compliance between the polymers.

This new scanning nanomechanics technique is capable of mapping elastic and visco-elastic response, and is an ideal tool for multiphase materials, composites, polymers, and nanostructures.

This work was done by S.A. Syed Asif of Hysitron, Inc. and R.J. Colton and K.J. Wahl of the Chemistry Division of the Naval Research Laboratory. Visit www.nrl.navy.mil/content.php?P=02REVIEW155.

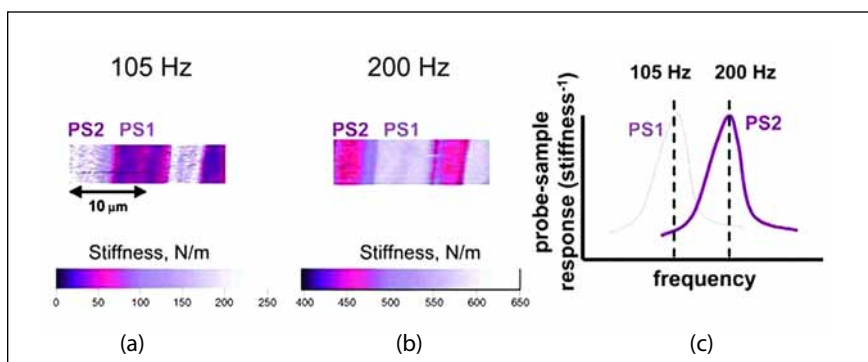


Figure 3. **Stiffness Images** of alternating layers of polystyrene (PS) of two molecular weights at (a) 105 Hz and (b) 200 Hz. The contrast is due to frequency-dependent mechanical response of the polymers. The probe-sample response as a function of frequency shown in (c) is consistent with the stiffness image.



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Stanford Nanofabrication Facility

The Stanford Nanofabrication Facility (SNF) at Stanford University in California is directed by Professor Yoshio Nishi, and supports research in areas such as optics, MEMS, biology, chemistry, and process characterization and fabrication of electronics devices. SNF also uses micro and nanofabrication technologies in non-traditional research applications.

Hands-on access to SNF is provided to lab members; remote use also is available for well-defined projects that are performed by the lab's staff. Remote users are members who do not come to SNF to do their own nanofabrication processing. Rather, SNF staff performs the processing. A variety of Web-based tools are available that allow remotely located researchers to view and inspect their nanofabrication process via Webcams, microscope cams, and automated downloads for still microscope images.

SNF also participates in the MEMS Exchange program, administered by the Corporation for National Research Initiatives. The MEMS Exchange is a network of fabrication facilities that provides MEMS process and design capabilities. SNF users include researchers from industrial companies and non-Stanford universities, as well as individual users.

The SNF was spawned from the Integrated Circuit Fabrication Facility, which began on the Stanford campus in the mid-1960s. The design and fabrication of MEMS devices has been a major component of the facility since its beginning, with much of its early support stemming from the medical systems area. This resulted in work in the areas of image sensors and image processing, accelerometers, and implantable electronics. Work in device and process modeling began in the mid-1970s, and resulted in the development of an array of device and process simulators. The facility was upgraded in the early 1980s to support larger chips, and then upgraded again in 1986.

Research Areas

Recent research at SNF covers a wide variety of nanotech and MEMS applications, from integrated bio-fluorescence



SNF is housed in the Paul Allen Center for Integrated Systems (CIS) building on the Stanford campus.

sensors and micromachined sensors for biological measurements, to designing and fabricating piezoresistive pressure sensors using carbon nanotubes, and integrated circuit thermal management.

• Integrated Bio-Fluorescence Sensor

— The need for small, portable biosensors becomes more important as biological analysis systems shrink. This project integrates vertical cavity surface emitting lasers (VCSELs), PIN photodetectors, and optical emission filters to be used as a fluorescence sensor. Integrating these components will drastically reduce the size and cost of fluorescence detection systems, and parallel sensing architectures of more than 100 channels will be possible. *(Work performed by SNF researchers Evan Thrush, Ofer Levi, James S. Harris Jr., and Stephen J. Smith.)*

• Microfluidic Channels With Integrated Ultrasonic Transducers

— Recent developments in the field of ultrasonic MEMS devices are being used to integrate ultrasonic sensors and actuators in biofluidic channels. The integration of ultrasonic transducers in small channels enables applications that previously were in the domain of larger-scale

sensors and actuators. Silicon micromachined ultrasonic capacitor transducers (CMUTs) and zinc oxide-based piezoelectric transducers are being studied for use in flow measurement, fluid pump, pressure, density, viscosity, and other mechanical properties. *(Work performed by SNF researchers Goksen G. Yaralioglu and Hemanth Jagannathan, with B.T. Pierre Khuri-Yakub of Stanford's Dept. of Electrical Engineering.)*

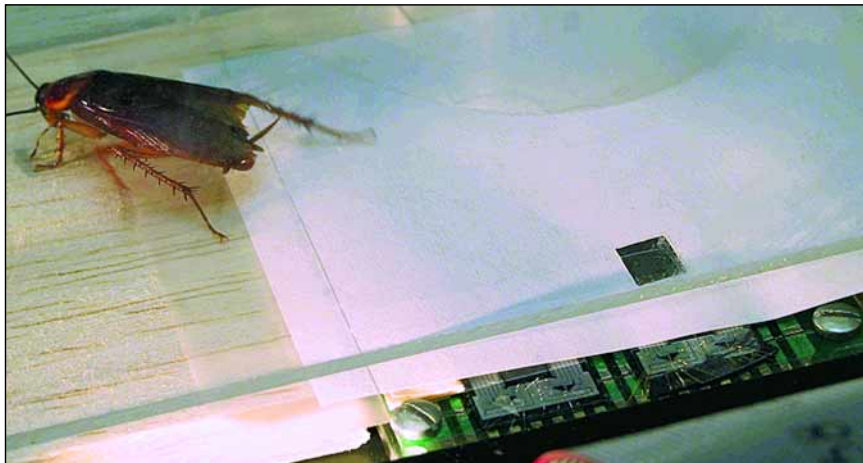
• Piezoresistive Pressure Sensors Using Carbon Nanotubes

— Traditional silicon piezoresistive pressure sensors are highly temperature-dependent. One alternative material that has been shown to exhibit the piezoresistive effect is the single-walled carbon nanotube (SWNT), a single graphene sheet rolled into a cylinder. SWNTs were grown on suspended polysilicon membranes, and air pressure was applied uniformly to press down on the membranes, causing the tightly attached SWNTs to deform. The current passing through the SWNTs was measured with respect to continuous time. The team's designs can be used in further investigation on the potential of SWNTs as piezoresistive pressure sensors. *(Work performed by Dr.*

Hongjie Dai and Randy Grow of Stanford, and researcher John Liu of the University of California Irvine.)

• **Micromachined Sensors for Biological Measurements** — A collaboration between SNF and members of the University of California at Berkeley is seeking to create a micromachined multi-axis force sensor to study biomechanical forces produced by insects such as cockroaches. The device enables three-axis measurement of ground reaction foot forces produced by running insects. The sensors, while originally designed to study insect biomechanics, also enabled the first detailed measurements ever made of biomechanical forces produced by ants as small as 4 mg. (*Work performed by SNF researcher Michael S. Bartsch, with Thomas W. Kenny of Stanford's Dept. of Mechanical Engineering.*)

• **Silicon Micropumps for IC Thermal Management** — New approaches to thermal management are necessary due to increases in high-end chip performance. An SNF team is developing thermal management systems based on microfluidic networks. Electro-osmotic pumping is a key enabling technology for such systems. These pumps operate well at the micro scale, require little power, and since they have no moving structural elements, they are inherently reliable. The electro-osmotic pumps leverage electrochemical interactions at a liquid-solid interface to induce flow through



A micromachined force sensor measures foot forces produced by running insects such as this 1g cockroach.


the application of an electric field. (*Work performed by SNF researcher Daniel J. Laser, with Kenneth E. Goodson, Juan G. Santiago, and Thomas W. Kenny of Stanford's Dept. of Mechanical Engineering.*)

• **Probe System for Electrochemical Analysis in Nanometer Dimension** — A pencil-shaped electrochemical transducer system for analysis or surface modification in the nanometer dimension has been developed. High-aspect-ratio tip structures are shaped by combining isotropic and anisotropic deep reactive etch processes to form the body of the transducer. Due to the high aspect ratio topography, the probe is well suited for Scanning Electrochemical Microscope (SEM) methodologies. The technology also shows production

possibility for both probe-arrays and probes on cantilevers. (*Work performed by SNF researchers R. Fasching, Y. Tao, and K. Hammerick, with F. Prinz of Stanford's Depts. of Mechanical Engineering and Materials Science.*)

Working With SNF

Lab membership at SNF is open to anyone from any academic, government, private, or industrial organization. SNF was designed to serve as a sophisticated "sandbox" where lab members can work hands-on with a broad range of state-of-the-art tools generally unavailable to researchers in academia or in small companies.

For information on SNF's facilities, including becoming a user, visit <http://snf.stanford.edu>. 

A Unique Network

SNF is one of five founding members of the National Nanofabrication Users' Network (NNUN), which was established in 1993 by the National Science Foundation to expand access to nanofabrication resources across the country. The combined staffs of the NNUN — which includes, in addition to SNF, the Cornell NanoScale Facility, the Penn State Nanofabrication Facility, the Materials Science Research Center at Howard University, and the Nanofabrication Facility at the University of California Santa Barbara — have experience in all phases of nanofabrication and its use in fields from nanophysics to biology to electronics.

Recognizing the need to foster growth in many integrated fields, the NNUN has chosen to focus on the interface between biology and engineering. Clinical diagnostics and high-throughput screening of genes and drug candidates are prime areas of rapid growth in this field. NNUN Biology Domain Experts serve as technical liaisons between the biological and engineering research communities, coordinating resources and maintaining information on biotechnology research.

For more information on the NNUN, and how to become a user, visit www.nnun.org.

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