

Fullerene Nanotube Can Be Bound to Surfaces

A team of researchers at Rice University, led by chemist Richard Smalley, have converted fullerene nanotubes into short, open-ended pipes, and attached molecules to their open ends, providing the ability to bind nanotubes to a variety of other chemical groups or surfaces. This form enables scientists to manipulate the nanotubes chemically. The research team demonstrated this ability by tethering the

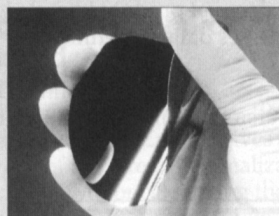
pipes to gold particles 10 nm in diameter. To create the fullerene pipes, Smalley and the researchers first purify the raw nanorope material in large batches using nitric acid, followed by a filtration technique similar to dialysis, yielding about 10–20% pure nanoropes by weight. Then the nearly endless ropes, which are made of several nanotubes nestled parallel to one another, are separated and cut into individual open-ended pipes ranging in length from 100 to 300 nm.

According to the researchers' article published in the May 22 issue of *Science*, the cutting method involves sonic bombardment with high-energy sound waves in combination with concentrated sulfuric and nitric acids. The walls of the nanotubes are attacked by the sonication, creating a hole, and the oxidizing acids etch around the remainder of the tube. The cutting takes place over a period of one to three hours. The acid treatment leaves the open edge of the pipe with carboxylic acid groups hanging onto the end, which can easily be converted to the acid chloride.

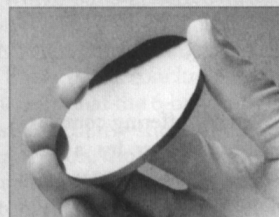
When mixed with water and soap-like molecules to keep them separated, the cut pieces, which are molecularly perfect and chemically clean, are in a form which allows them to be sorted by length using a type of chromatography. They can then be manipulated by exposing the ends to additional chemicals, such as alkane thiol chains attached by amide links.

"But still try—for who knows what is possible?"

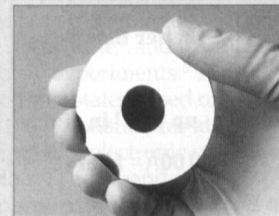
— Michael Faraday



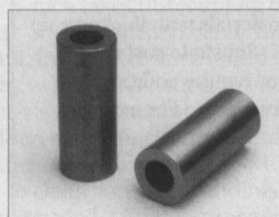
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Ultrashort X-Ray Pulses Shed Light on Dense Plasmas

In work fundamental to the nature of atoms, scientists at the University of Michigan have measured how matter changes under extreme pressure. Using a high-resolution femtosecond laser, Don Umstadter, a professor in the Department of Nuclear Engineering and Radiological Science and in the Department of Electrical Engineering and Computer Science, and his colleagues in the Center for Ultrafast Optical Sciences were able to watch how and when electrons and atoms organize themselves in the super-dense environments similar to those found in fusion reactors.

The work, which appears in the May 18 issue of *Physical Review Letters* and the May 29 issue of *Science*, confirms earlier predictions about the behavior of atoms in these conditions. In most phases of matter, electrons lead an orderly existence, confined to particular orbits around atomic nuclei depending on how much energy they have. Previously unverified mathematical and computer models, however, suggested that electrons in a super-dense plasma exist mostly in a free-for-all state, a particle sea unassociated with any given nuclei.

In their experiment, the researchers heated solid carbon with 100-fs pulses, converting it into a dense soup of ions. As the matter expanded and cooled, the electrons joined available nuclei at random. When they chose an atom, the electrons lost energy in the form of easily detectable x-rays. The x-ray emission is so regular that it can be read as a signature to deter-

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mine precisely when the electrons have settled into their new state, or phase. By measuring the x-ray emission signatures, the researchers were able to identify when and under what conditions of temperature and density the settling sea separated and the matter changed phase.

In addition to illuminating the timing of phase changes, the experiment also sheds light on the conditions under which electrons are bound to atoms, and thus, on the conductivity of matter. Whereas the electron sea freely conducts electricity, the bound state is nonconductive because there are no free electrons to carry current.

Umstadter said that femtosecond lasers allow for the first "clean measurements in a system that's usually never clean."

Although it has long been possible to generate x-rays with lasers, the efficiency of this table-top laser created at the Center is dramatically increased, making them useful for applications such as image, according to Margaret Murnane, professor of electrical engineering. According to the researchers, whereas traditional lasers emit visible and near-infrared light (with wavelengths in the 500–1000-nm range), those from this device are about 20 nm, with the possibility of being as short as 2 nm. The shorter the wavelength, the higher the spatial resolution of the beam.

The laser consists of a hollow glass tube filled with gas, sandwiched between a laser source and a detector. When the intense light passes through the gas, electrons are pulled away from the atoms, then slammed back when the field reverses direction. The electron then can give off its energy in the form of an x-ray photon. This x-ray light can be amplified by 100–1000 times by propagating through the hollow fiber at the same speed as the laser.

A major hurdle for earlier attempts at such a setup was that the x-rays tended to cancel each other out. The canceled waves produce very dim, diffuse x-ray patterns on the detector. But by adjusting the pressure of the gas in the hollow fiber to fine-tune the laser's speed, the scientists managed to keep the x-ray peaks properly matched, so that the beam amplified itself and remained "in phase" with the laser as it passed through the tube.

Viruses Act as Tools for Materials Science and Drug Delivery

Temple University chemistry professor Trevor Douglas and Montana State University plant pathology professor Mark Young have utilized a "gating mechanism" in the protein coats of some

simple viruses which allows them to admit and expel organic and inorganic material. Through these "reversible structural transitions" technicians will be able to remove the genetic component of a virus (the DNA or RNA that allows the virus to reproduce) and use the remaining protein coat as a container and delivery system for other substances.

Douglas and Young said that "In their native state, viruses are protein assemblies which act as host containers for nucleic

acid storage and transport. We have subverted this natural function."

According to their article published in the May 14 issue of *Nature*, the simple protein coats that Douglas and Young work with can also be "easily and routinely modified by design," meaning that the loaded virus can be altered to target certain types of cells (like cancer cells) and holds the promise of being able to deliver drugs to very specific sites.

Young said that the viruses they work

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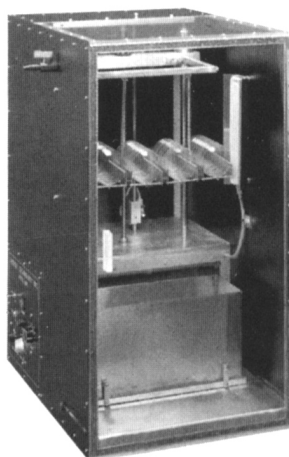
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with are relatively simple plant viruses which (1) "are incredibly host-specific" and do not use animals as hosts; (2) are routinely and safely eaten by people as part of vegetable material; and (3) have had their genetic material removed, leaving only a coat which can be used as a container. As such they pose no health threat to people.

Douglas and Young have used the protein coats of viruses as "size and shape constrained" chambers in which minerals crystallize in very specific and precise dimensions. The possibility of creating an unlimited number of homogeneously sized crystals would have a profound impact, for example, on the production of miniaturized semiconductors and other

patterned materials.

Medical advances may come from the fact that the two researchers were able to load an organic substance similar to heparin—which is routinely used to treat coronary thrombosis—into cowpea chlorotic mottle virus. Because this phenomenon of gating is possible for a large number of viruses of different shapes and sizes, options for drug delivery are expected to not be limited to any particular class of medicines.

Deformed Microdisk Laser Exhibits Increased Output Power and Directionality

Researchers from the Max Planck Institute for the Physics of Complex Systems in Dresden, Germany, Yale University in New Haven, Connecticut, and Lucent Technologies/Bell Laboratories in Murray Hill, New Jersey, have demonstrated semiconductor microlasers with more than 1,000 times the power of conventional, disk-shaped microlasers. The lasers, which are 0.05 mm in diameter, rely on the presence of chaotic light rays inside a deformed resonator.

According to the researchers' article published in the June 5 issue of *Science*, experiments on semiconductor microlasers with a quadrupolar deformation revealed that, within a critical deformation range, the light pulses will travel in a bow-tie pattern, which suffers less internal reflection and emits light in four narrow, controllable beams. Yale applied physicist A. Douglas Stone said that this change is accompanied by a large increase in power output. Each beam has an output of 10 mW of power, which increases the laser's total output to 40 mW.

This class of microlasers has been pioneered by researchers at Bell Labs in the early 1990s. The key difference between conventional devices and the microlaser lies in the shape: To create the perfectly synchronized photons, a resonator has to be formed by trapping the light. The early pioneering microresonators consisted of perfectly round disks that can store light in wavefronts circulating around the rim, that is, so-called "whispering-gallery" mode.

The scientists at first thought the light in their deformed microlasers operated in this "whispering-gallery" mode instead of a bow-tie pattern, Stone said. "In a circular whispering gallery, such as the dome in St. Paul's Cathedral in London, sound flows along the walls. A whisper can be heard by someone standing against the opposite wall, but not by someone standing in the center of the room," he said.

Harry P. Gregor, a Columbia University chemist and leading authority on ion exchange and membrane separation technology, died May 3 in Manhattan. He was 81 years old.

He was Professor Emeritus of Chemical Engineering and Applied Chemistry at Columbia's Fu Foundation School of Engineering and Applied Science, where he taught for 23 years, and Distinguished Research Professor at Polytechnic University, where he was conducting research for the U.S. navy.

Gregor was the first to recognize that certain ionic polymers attract small ions and reject larger ions. The technology is used to promote a number of chemical reactions that involve ions, such as removing calcium to soften water or removing radioactivity to purify water.

"He had a keen insight into how physical processes take place in aqueous solutions," said Carl C. Gryte, professor of chemical engineering and applied chemistry at Columbia and a student of Gregor's.

Gregor's interest in membranes germinated in the laboratory of Karl Sollner, his dissertation advisor, who had done extensive work on ion-selective membranes. He held a postdoctoral appointment at the University of Minnesota from 1944 to 1945, which he used to study the problem of synthetic rubber since the United States had lost its principal supply of rubber, the Philippines, in the war. He was made a captain in the navy and was sent to Germany on several occasions after the war to evaluate the country's chemical industry.

He joined Brooklyn Polytechnic Institute as assistant professor of physical chemistry in 1946, and was eventually promoted to full professor. In 1967, he was named professor of chemical engineering and applied chemistry at Columbia and was named Professor Emeritus in 1987. He rejoined Polytechnic University in 1990 and was named Distinguished Research Professor.

In his early work at Polytechnic, Gregor conducted basic research on ion exchange resins, gels that have a fixed negative charge. When exposed to solutions, the gels reach an equilibrium with the solution in which they attract cations. But the gels prefer cations of small hydrated volumes, which Gregor realized could be the basis for a purification technology. He developed several applications for the gels, among them removing radioactive materials from water and, during the strontium scare of the early 1960s, strontium-90 from milk. The navy asked him to develop ion exchange resins to purify drinking water in case of a nuclear confrontation.

At Columbia in the late 1960s, he returned to membrane research and also conducted important work in polyelectrolytes, ion exchangers, and polymer catalysis. He did pioneering research on stabilized enzymes in which enzymes that speed a reaction are bound to polymeric hollow fibers, allowing reactions to take place as a liquid flows through a fiber wall and eliminating the need to recover the enzyme from the final product.

He developed a bipolar membrane that acts as a synthetic enzyme to split salt solutions back into their constituent acids and bases. A synthetic enzyme process he developed is also used in the production of high-quality plastics, said Fred Liu, a former student who is now president of Union Resources and Technology Co., Inc., a technology firm in Pittstown, NJ.

Gregor was also interested in biomass conversion, the fermentation of natural starches such as that found in corn to produce ethanol, which could be used to replace petroleum fuels in automobiles. He was an advisor to the Department of Energy on its Alcohol Fuels Program and a member of the Columbia University Seminar on Biomaterials.

Researchers had previously shown that a light ray trapped inside a perfectly round optic resonator would bounce along the perimeter in just such a fashion. Round microlasers have two drawbacks, however. First, because of excessive internal reflection, they emit only a few microwatts of optical power. Second, the direction of the emitted light is not well defined.

Federico Capasso, head of the Semiconductor Physics Research Department at Bell Labs, and Jerome Faist, formerly of Bell Labs and now with the Universite de Neuchatel in Switzerland, had earlier invented the quantum-cascade laser, which operates like an electronic waterfall. Multiple layers of gallium-indium-arsenide and aluminum-indium-arsenide are stacked on top of each other with atomic precision in such a way that mid-infrared light is generated when an electric current passes through this semiconductor sandwich. The recent breakthrough came when this technology was combined with the notion of asymmetric resonant cavities introduced by Stone and physicist Jens Nöckel, formerly a student of Stone and now with the Max Planck Institute.

Symmetric microlasers had been unable to deliver emission in the form of focused beams. Several years ago Stone, Nöckel, and Yale professor Richard K. Chang discovered that a suitably deformed oval resonator shows highly focused laser emission in certain directions, whereas round resonators radiate equally all around their perimeter. The oval shape allows trapped light rays to move on chaotic paths after several reflections at the surface. Why chaos should bring about focused emission can be understood by analyzing the hidden structure underlying the ray chaos, which is not random, but deterministic. Essentially, the "whispering-gallery" theory remains valid to a certain extent, except for some very unpleasant wobbling that causes a slingshot effect in specific directions. Initial experimental and theoretical results confirming the idea were published in 1996 and 1997 in *Optics Letters*, *Physical Review Letters*, and *Nature*.

According to the researchers, asymmetric microlasers have left the stage of trial and error in which their development threatened to become stalled while no appropriate theory existed. Because of their low power consumption and small size, they can be packed onto chips for optical computing, and offer promise for the growing fiber-optic telecommunication industry. The mid-infrared lasers with which the chaotic-resonator has been demonstrated and characterized are themselves potentially useful in pollution monitoring and breath analysis in medicine. However, the same concepts can be applied to a broad range of solid-state laser materials emitting in the visible part of the spectrum or at shorter infrared wavelengths relevant for fiber communications.

Si Pore Formation Process Begins at Crystalline Defects

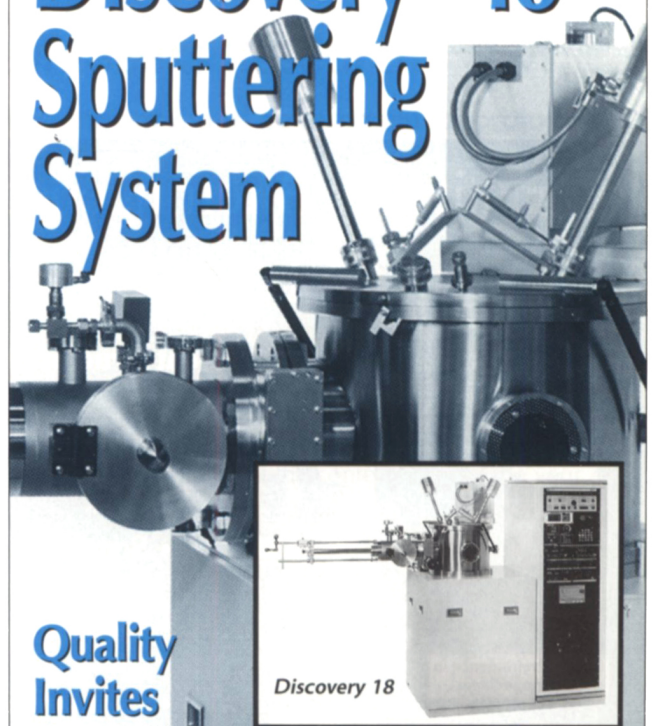
In their experiments with porous silicon, researchers at the National Research Council of Canada in Ottawa (NRC) and the Swiss Federal Institute of Technology (ETH) have determined that the pore formation process begins at crystalline defects, not at impurities as other studies have suggested. The researchers have also used a direct writing process to create microscopic light-emitting patterns of various shapes—squares, lines, letters, dots—in silicon.

As reported in the May 4 issue of *Physical Review Letters*, the researchers used a focused ion beam to implant, at room temperature, 200 keV Si^{++} ions in *n*-type Si (100) wafers doped with $5 \times 10^{15} \text{ cm}^{-3}$ As. Each pattern was formed with implant fluences of 3×10^{13} , 10^{14} , 3×10^{14} , 10^{15} , 3×10^{15} , 10^{16} , and 3×10^{16} ions cm^{-2} . Analysis using the TRIM code indicates that vacancies formed immediately below the surface, peaking at 270 nm below. The scientists then electrochemically treated the samples in 20% HF solution, step-

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Decades of R&D In Thin Film Manufacturing

ping the voltage potential from -0.5 V to different anodic potentials with a rate of 10 mV every 5 s. On unimplanted samples, a porous surface forms at a potential of ~3.8 V, thus this potential is called the pore formation potential (PFP). On a sample implanted at 3×10^{14} ions cm^{-2} , the PFP was measured as +0.25 V. The experiments showed that the photoluminescence (PL) spectrum of the implanted and electrochemically treated samples peaks at 655 nm in the orange-red region of the spectrum, typical of the PL response of porous Si. PL spectra of unimplanted areas as well as implanted areas prior to electrochemical treatment showed the spectra of clean silicon surfaces, indicating that neither lattice defects nor amorphization are responsible for the observed PL.

According to the report, "in the potential range between the PFP of the implanted area (+0.25 V) and the PFP of the untreated area (+3.8 V), pores can be selectively formed at ion beam treated parts of the sample." In earlier electrochemical treatment studies, porous Si formation was induced in *n*-type material under illumination. The carrier generation induced by the illumination yielded the holes nec-

essary for Si^0 oxidation and dissolution. In the current study, by P. Schmuki (ETH) and L.E. Erickson and D.J. Lockwood (NRC), *n*-type material is used in the dark, "which makes Schottky barrier breakdown the determining criterion for h^+ [vacancies] availability at the sample surface and thus for dissolution." Researchers have found a correlation between PL intensity and porosity in that the highest amount of holes in the nanoscopic range were found for the highest PL sample. Due to the size of the writing ion beam, light-emitting patterns range from 50 to 200 nm. The researchers said, "We clearly demonstrate that a creation of surface defects followed by an electrochemical treatment, tailored to trigger dissolution at defects, can be used to form visible light emitting porous Si selectively."

Unexpected Three-Stage Melting Transition Observed in Two-Dimensional Liquid Crystal System

Researchers at Princeton University, Applied Materials, Roswell Park Cancer Institute, the University of Minnesota, and

the State University of New York at Buffalo, have observed that when a two-dimensional (2D) liquid crystal melts, it undergoes two intermediate phase changes, which contradicts what had been previously predicted. An unusual liquid phase is observed that has some local characteristics of a solid, but is still technically a liquid. This liquid phase is preceded by the hexatic phase, the existence of which now confirms earlier theoretical predictions.

In this research, published in the May 29 issue of *Science*, the scientists used two-dimensional *n*-pentyl-4-*n*-pentanoyloxybiphenyl-4-carbonate films suspended in air because use of a substrate can affect the experiment. To understand the phase transitions as the temperature rose, the scientists studied the molecular structure of the films using an electron-diffraction microscope that was adapted to sustain the liquid-crystal films. In the hexatic phase, the molecules have no positional order just as in the liquid phase, but the molecular arrangement is not completely random, either, and there is some orientational (directional) order as in the solid phase. Other data reported includes the results of high resolution heat capacity and optical reflectivity measurements.

John Ho, Distinguished Service Professor in the Department of Physics and associate dean of the Faculty of Natural Sciences and Mathematics at Buffalo, said, "Our two-dimensional, liquid-crystal film provides the first unequivocal proof of the hexatic phase because its properties were revealed in the absence of a substrate. Now between this hexatic phase and the liquid phase, we have found another one, which has a slightly more ordered molecular environment than a liquid."

In this new phase, which has not yet been named, the immediate "neighborhood" of each molecule is more regular and, therefore, closer to a solid.

"The molecular arrangement is more regular locally, but it does not continue or propagate throughout the material and, therefore, it is still a liquid," said Ho.

In the experiments, the material repeatedly demonstrated distinct changes in its temperature and density as a result of the application of heat, strongly suggesting a distinct phase change. These findings are expected to help subsequent efforts to understand and model the melting process in 2D systems.

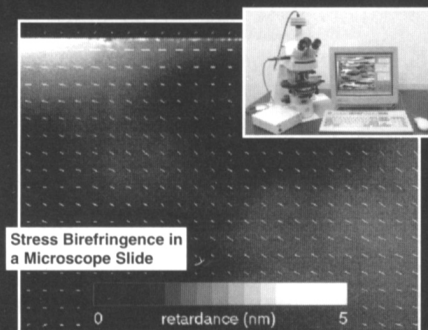
Hodgson Named Head of SLAC's Synchrotron Division

Keith Hodgson, deputy director of the Synchrotron Division of the Stanford

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Linear Accelerator Center (SLAC) and professor of chemistry and professor at the Stanford Synchrotron Radiation Laboratory (SSRL), has been named division director on July 1.

The announcement was made by SLAC Director Burton Richter who said, "Keith brings with him a strong scientific background, excellent management skills and an awareness of the larger context in which national science laboratories must function. His extensive role in national advisory activities position him well for leading SSRL into the next phase of its development."

Hodgson has been acting director of SSRL since last September, when the former director, Arthur Bienenstock, was appointed to a senior position in Washington, DC with the Office of Science and Technology Policy. Hodgson will be the first director of a major synchrotron facility who has a background and research interest in chemistry and biology rather than materials science or physics.

Hodgson's major research interests are inorganic and structural chemistry, using high-intensity synchrotron radiation for x-ray absorption, diffraction, and scattering studies. He joined Stanford's chemistry department in 1973 and became full professor of chemistry in 1984. In 1992, he became a joint professor of chemistry and at SSRL. In 1980, he originated focused efforts to develop a synchrotron-based research and user program in structural molecular biology.

His work has been recognized by awards and honors that include the Sidhu Award of the American Crystallographic Association in 1976, an Alfred P. Sloan Fellowship in 1976–1978, a Robert Welch Foundation Lectureship in 1981, and a World Bank Lectureship in Chemistry in 1984. He has co-authored more than 200 scientific publications.

In 1983, the Department of Energy (DOE) asked Hodgson to serve on a critical committee that defined the national need for new synchrotron facilities in the United States. He currently chairs the DOE Biological and Environmental Research Advisory Committee. He is also a member of the National Center for Research Resources Advisory Council in the National Institutes of Health.

Hodgson recently co-chaired a committee to evaluate and recommend future opportunities in the area of structural molecular biology, particularly a plan to build a new facility at SSRL jointly between Stanford and the Scripps Research Institute for state-of-the-art tools for protein crystallography research.

Society for Biomaterials Names Award Recipients

The Society for Biomaterials has named six award recipients at the 24th Annual Meeting and Exhibition last spring in San Diego. **Rena Bizios**, Rensselaer Polytechnic Institute, received the Clemson Award for Contributions to the Literature in recognition of Caldwell's contributions to the literature of biomaterials science; **Karin D. Caldwell**, University of Utah, received the Clemson Award for Basic Research in recognition of contributions to the basic knowledge and understanding of the interaction of materials with tissue; **Eugene P. Goldberg**, University of Florida and a member of the Materials Research Society, received the Clemson Award for Applied Research in honor of his utilization of knowledge in science to accomplish a significant goal in the biomaterials area; and **Larry L. Hench**, Imperial College of Science, Technology,

and Medicine in England, received the Founders Award which honors those members of the Society who have given much of themselves to research in biomaterials and in establishing, developing, maintaining, and promoting the Society. The award is determined from manuscripts which contain substantially new information and have the characteristics of a landmark contribution to the field. Hench is a past president of the Society. **Samuel F. Hulbert**, Rose-Hulman Institute of Technology in the United States, received the C. William Hall Award which honors individuals who have an outstanding record in establishing, developing, maintaining, and promoting the objectives and goals of the Society. Hulbert is a past president of the Society. Students **Susan J. Peter** and **Christopher R. Jenney**, both of Case Western Reserve University, were also recognized for their outstanding research. □

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