

Large Negative Magnetoresistance Observed in Purified AlGaAs/GaAs Layers

Physicists Moty Heiblum, V. Umansky, and their colleagues at the Weizmann Institute in Israel have used an ultraclean vacuum system and highly purified materials, devising a semiconductor consisting chiefly of gallium arsenide (GaAs) and aluminum gallium arsenide (AlGaAs) layers. Cooling the material to 0.1 K and subjecting it to a small electric field, electrons at the GaAs/AlGaAs interface drifted in the direction of the field at speeds 40% greater than those in the best previous GaAs-based materials. The electrons journeyed an average of 120 μm before colliding. In general, electrons tend to scatter less in GaAs than in silicon; moreover, this material outperformed its GaAs-based cousins because it had significantly fewer impurities. This is not the case at room temperature, however, where thermal vibrations of the semiconductor crystal diminish the importance of purity. As reported in the August 4 issue of *Applied Physics Letters*, the researchers detected possible signs of large negative magne-

toresistance in which magnetizing the cold sample appeared to decrease its electrical resistance substantially—something not seen before in similar AlGaAs/GaAs materials.

NSOM Used to Image Photonic Crystals

Near-field scanning optical microscopy (NSOM) is an emerging technique that combines the nondestructive advantages of optical microscopy with nanometer-scale resolution near that of atomic force or electron microscopes. NSOM works by channeling laser light through a fiber-optic probe, scanning it about 10 nm above a sample surface, and then collecting it on the other side. An opening at the tip of the probe is only about 50 nm wide, smaller than a wavelength of visible light but large enough for a small portion of the light energy, or photons, to escape. Physicists at the National Institute of Standards and Technology (NIST), working in collaboration with researchers at the University of Virginia and the Naval Research Laboratory, recently created an image of a "photonic crystal," a test material made by embedding an array of tiny glass cylinders

in a matrix glass. To the eye, these two clear glasses are indistinguishable. However, they have slightly different indices of refraction. Consequently, the NSOM image shows that, rather than traveling straight through the sample, light is "guided" through the crystal by the cylinders.

The NSOM technique has been developed by numerous laboratories worldwide in order to image and characterize nanometer-scale features on biological membranes, semiconducting devices and

Review Articles

Ph. Colomban of CNRS and J. Tomkinson of Rutherford Appleton Laboratory have published a review article in *Solid State Ionics* 97 (1997) entitled, "Novel Forms of Hydrogen in Solids; The 'Ionic' Proton and the 'Quasi-Free' Proton." The article reviews recent incoherent inelastic neutron scattering studies of proton conductors, emphasizing un-solvated or poorly solvated proton systems which remain stable in the temperature range of 100–500°C.

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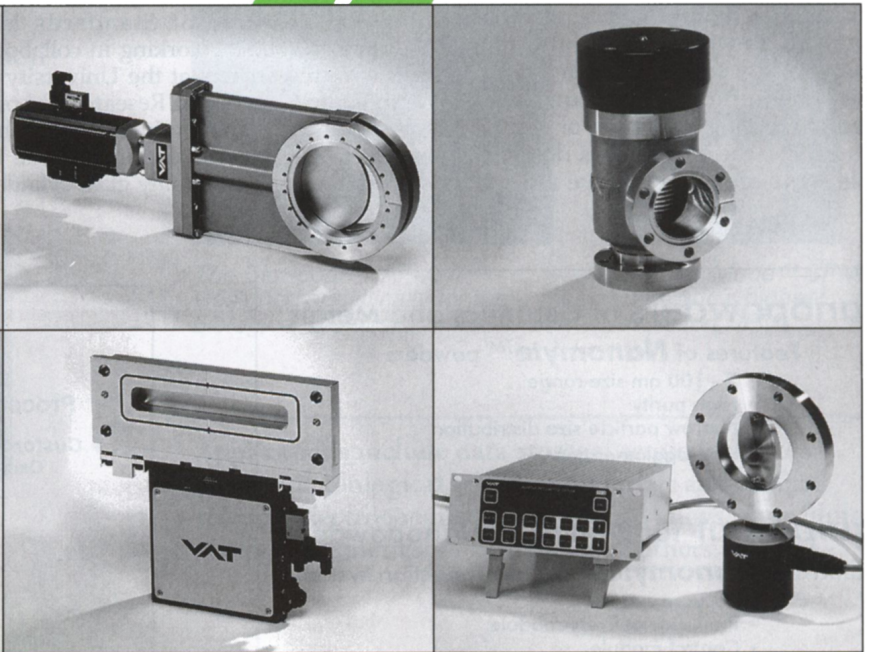
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substrates, fiber-optic communications components, and many other materials. A major goal of the NIST program is to further refine NSOM measurements and modeling so that NSOM can provide truly quantitative measurements of the optical properties of these structures. For example, accurate measurements of the size of the glass cylinders and their index of refraction should result from the researchers' collaborative research on photonic crystals. In addition, other research groups at NIST are working on developing applications for NSOM, including nanometer-scale chemical composition analysis.

NIST Scientists Develop Highly Stable Optical Retardation Plates

Optical retarders, known as waveplates, are critical components for polarization control in the optics industry. They play an important role in optical sensor, data storage, medical and military systems as well as a variety of test and measurement instruments. Waveplate manufacturers typically measure retardance using laboratory benchtop systems with varying uncertainty. Customers also require different levels of retarder uncertainty—ranging from greater than 10% for multiple-order waveplates to less than 0.1% for critical devices. Scientists in the Optoelectronics Division of the National Institute of Standards and Technology developed and demonstrated a stable linear retarder for use as a calibration reference. The device is a nominally quarter-wave retarder at 1.3 μm ; this wavelength was chosen to meet fiber optic telecommunication needs. The retarder is stable to within 0.1 degree over a wavelength range greater than 50 nm, an input angle range greater than 1 degree, and a variation in room temperature greater than 10°C. The devices are packaged in a protective housing that ensures retardance stability in the presence of humidity and contamination. Researchers estimate that the retardance will remain stable within 0.01 degree for 10 years when the device is stored properly in typical laboratory conditions. Each device will be individually measured and a certified retardance value with an expanded uncertainty less than 0.08 degree reported.

General Technique Devised for Engineering Nanocomposites

Douglas Gin of the Materials Sciences Division at the Ernest Orlando Lawrence Berkeley National Laboratory and assistant professor of chemistry at the University of California—Berkeley, has devised a general technique for engineer-

ing nanocomposites that begins with the self-assembly of synthetic starting materials. Polymerizable liquid crystals form the skeleton of Gin's composites, matrices containing stacks of hexagonally packed tubes whose diameter and spacing is measured in nanometers. These ordered tubes contain a chemical precursor in solution, which can be converted to solid filler material after the architecture of the liquid-crystal matrix has been locked into place by polymerization.

Unlike the sort of liquid crystals found in digital displays, which change in response to temperature or an electromagnetic field, Gin uses lyotropic liquid crystals; in addition to changes in temperature, these respond to additives and changes in the chemical solution in which they are immersed.

"The design of unique lyotropic liquid crystals is the key to everything that follows," said Gin. He works with polymerizable surfactants. "Like laundry soap, they're made of amphiphilic monomers"—molecules, each of which has a hydrophilic end and a hydrophobic end. By adding more and more monomers, spherical micelles can self-organize and lengthen into cylinders.

Instead of submerging his monomers in water, Gin reduces the amount of water in his system and designs monomers to form "inverse" cylindrical micelles with their water-loving heads inward. Meanwhile the water-fearing tails on the outside of the tubes seek each other's company, and the tubes pack themselves into hexagons. After the hexagonal architecture is locked in place, Gin said, "We can do ordinary synthetic-organic chemistry inside the channels."

Using two different kinds of monomers and two different filler precursors, Gin and his colleagues have demonstrated two novel self-organizing nanocomposites with unique properties. In one technique the liquid-crystal matrix has been formed in a solution containing a precursor to poly(para-phenylenevinylene) or PPV—a light-emitting, electrically conducting polymer—which fills the tubes. When Gin turns up the heat, the precursor converts to PPV inside the tubes to form a bundle of long, discrete, exceedingly fine wires. His group has made uniformly oriented films of this material up to 8 cm wide, yet only 30–100 μm thick. Nanoscale materials often show markedly different properties from the same materials in bulk, and PPV is no exception: Gin's hexagonal matrix of PPV has over twice the fluorescence, per unit volume, of PPV in bulk.

In related work, Gin is studying an entirely different liquid-crystal system,

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The screenshot displays a grid of windows from the HSC Chemistry software. The top row includes a plot of Gibbs free energy vs. temperature and a data table. The middle row shows a phase diagram and another data table. The bottom row contains a plot of heat capacity vs. temperature and a final data table. The interface is clean and professional, typical of scientific software from the late 1990s.

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which uses a different monomer to build the hexagonal-tube framework and a different filler precursor, tetraethyl orthosilicate, in a solution of water and ethanol. The solution also includes a small amount of a chemical that generates an acid when illuminated. In the presence of the acid the precursor converts to silicate glass, even at room temperature.

Because of the hexagonal array of confining channels, the glassy composite has a fine, nanoscale structure unlike that of normal amorphous glass or plastic. Gin and his colleagues describe it as "a tough, pale-yellow, slightly opaque, glassy material . . . Completely insoluble in common organic solvents and water." It promises unusual properties, including hardness, now under investigation.

YBCO Breaks Both Gauge Symmetry and Time-Reversal Symmetry

Recent experiments on yttrium-barium-copper-oxide (YBCO) superconductors have clarified understanding of the peculiar behavior of this unconventional material. Most significant among the findings, by studying a characteristic called the zero-bias conductance peak, scientists at the University of Illinois Urbana-Champaign—working with scientists at

Northwestern University—have discovered the first example of a solid superconductor displaying broken time-reversal symmetry, as reported in the July 14 issue of *Physical Review Letters*.

Laura Greene, professor of physics who directed the research effort, said that the pertinent news in the field of high-temperature superconductors (HTS) is that the zero-bias conductance peak splits at low temperatures in the absence of an externally applied magnetic field. Greene said, "Not only does our experiment again prove that the dominant symmetry in YBCO superconductors is *d*-wave, it also shows that two different pairing mechanisms—or order parameters—can coexist in the same material, creating spontaneous currents that are a signature of broken time-reversal symmetry."

To perform the experiment, Greene and her colleagues grew thin films of YBCO by off-axis magnetron sputter deposition. The researchers then used planar tunneling spectroscopy to measure the tunneling conductance across different junctions as a function of crystallographic orientation, temperature, and externally applied magnetic field.

According to the experimental results, at 90 K (the critical temperature for YBCO) the superconductor has *d*-wave symmetry, Greene said. "When cooled to about 7 K,

however, a second superconducting channel opens up which has *s*-wave symmetry. Because the two symmetries coexist, the differences between their phases spontaneously generate a current. The current creates a magnetic field, and that is what splits the zero-bias conductance peak."

According to Greene, the spontaneously generated current is also what breaks the time-reversal symmetry. Greene said, "Because the current is flowing in a certain direction, you can tell whether it's going forward or backward with respect to time."

The result offers proof of a new state of matter. Greene said, "This is the first case of a solid superconductor breaking both gauge symmetry and time-reversal symmetry. The only other material proven to break both symmetries is the unconventional superfluid helium-3, the discovery of which was awarded the 1996 Nobel Prize in physics."

Acoustic Liner Material Uses Tiny Spheres to Absorb Noise and Withstand High Temperatures

Tiny, hollow spheres developed at the Georgia Institute of Technology nearly 10 years ago for high-temperature insulation offer competitive noise-absorption properties, recent studies show. Researchers are

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using the spheres to create an acoustic liner material they believe has several advantages over existing materials, including its ability to withstand temperatures of more than 2,000°F (1082°C). The material consists of hollow, ceramic spherical beads ranging from 1 to 5 mm in diameter. They have eggshell-thin walls and multiple needle-size holes on their surfaces.

Joe K. Cochran, a professor of materials engineering, developed the original spherical shells—which he calls aerospheres—as an alternative for industrial and home insulation. They are made from readily available ceramic powders like alumina and mullite. The initial design did not include the surface holes which are now added to improve the spheres' noise-absorption properties.

To test the noise-reduction ability of liners made with the spheres, researchers conducted a variety of acoustic tests with an impedance tube. They collected their data with a computer and a two-channel signal analyzer that sent a broadband sig-

nal toward a collection of ceramic spheres through an amplifier, then to a speaker in the impedance tube. By examining the amount of reflection of the incident sound, they determined the sound absorption properties of the spheres.

Researchers checked their results for accuracy by testing other materials in the same manner, then comparing these results with data gathered by other researchers with other impedance tubes. They also tested standard steel BBs and spheres without surface holes to confirm that the hollowness and the holes aid sound absorption.

The results show that the new liner material can absorb both low and high sound frequencies at levels comparable to traditional bulk-absorbing liners like fiberglass, Kevlar, and foam. Krishan K. Ahuja, Regents Researcher in the Georgia Tech Research Institute's Aerospace & Transportation Laboratory and a professor in the School of Aerospace Engineering, said that these materials are not as malleable,

however, and cannot withstand temperatures over 2,000°F (1082°C).

Other materials such as ceramic wool, mineral wool, and some metallic honeycomb structures—can withstand high temperatures, but are produced in preshaped forms. Ahuja said that the new, versatile spheres could be poured into existing structures (such as walls of homes and frameworks of aircraft) and could be encased in a quiltlike fabric to make portable curtains and blankets for use in noisy factories where permanent structures are not needed.

Researchers also tested the liner material to see how it would perform in aerospace applications, where it would have to stand up to high-velocity, heated air flow. The spheres were poured into a hollow shroud surrounding a noisy jet issuing from a nozzle. Far field noise data measured in an echo-free chamber confirmed that the spheres can reduce noise in a high-temperature flow environment.

Future tests will seek to pinpoint exact-

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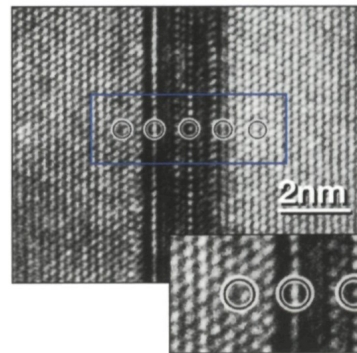
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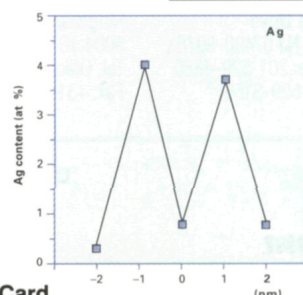
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Al-Cu-Mg (Ag) precipitate in aluminum showing silver segregation to interface.



Data courtesy of Dr. James M. Howe, Department of Materials Science & Engineering, University of Virginia, U.S.A.



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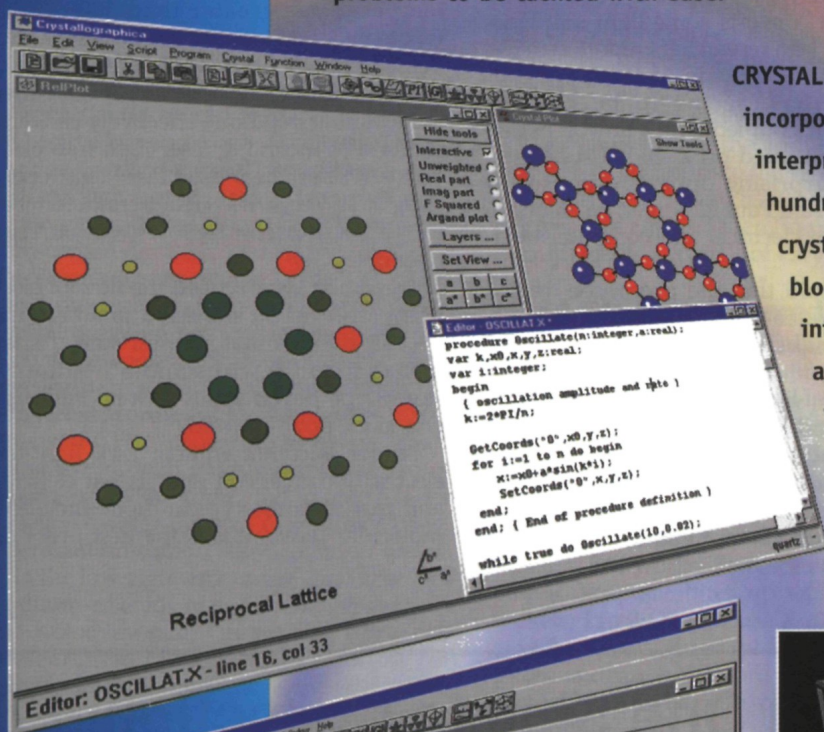


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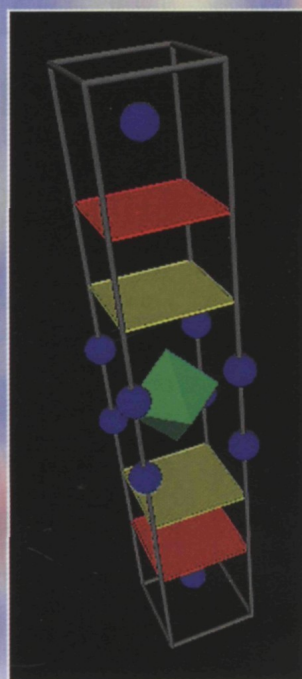
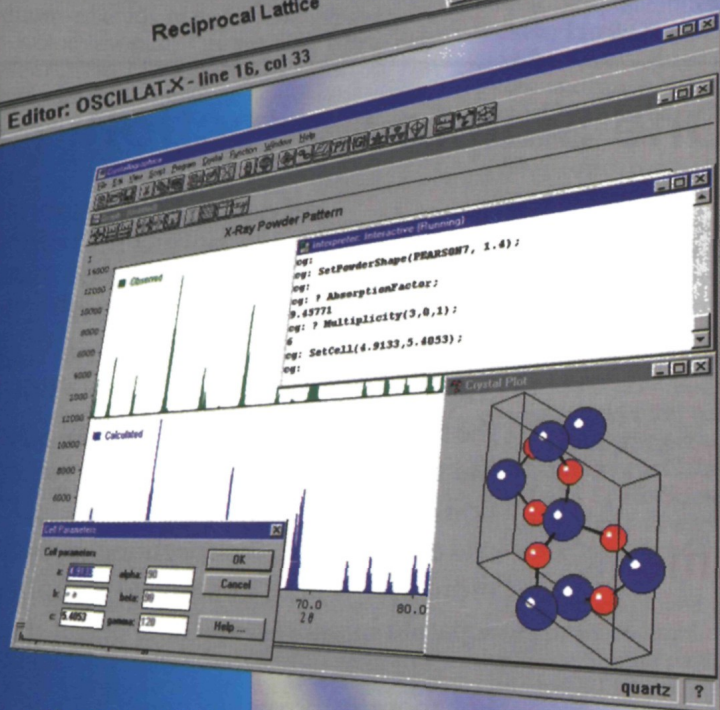


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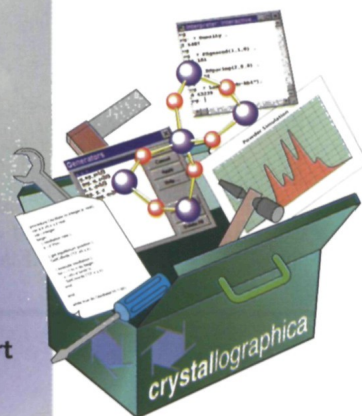
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ly how the spheres reduce noise. Researchers also want to conduct tests with thicker spheres, which they believe will be more durable, and ones made of lightweight plastic, which would be cheaper to produce.

Plastic spheres would be less brittle than ceramic ones and could be used in applications without heated air flow, such as in buildings, hand tools, and the inlet sections of aircraft engines. However, a manufacturing process to produce plastic spheres must be developed first.

Recently Announced CRADAs

The National Institute of Standards and Technology (NIST) (Gaithersburg, Md.) and Schott Glass Technologies Inc. (Duryea, Pa.) have signed an agreement to look together at different types of glass used for carrying laser light in photonic systems.

Carbon-Doped Germanium Layers on Silicon Respond to 1.3 μm Light

Researchers at the University of Delaware have developed a silicon-based device that converts some light into electricity. The key, researchers reported in the September 1997 issue of *IEEE Electron Device Letters*, seems to be carbon. Germanium, coupled with a tiny bit of carbon and mounted on a silicon substrate, exhibits surprising optical responses to laser light, said Paul R. Berger, an associate professor of electrical and computer engineering.

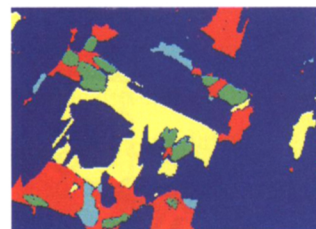
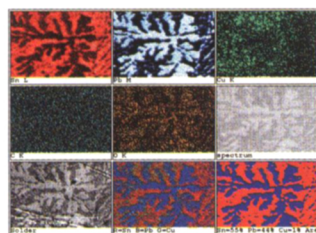
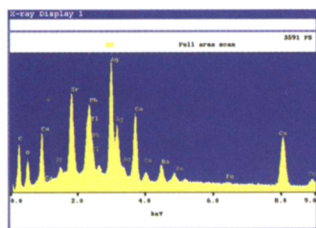
Berger said that the prototype device "demonstrates the feasibility of using a conventional silicon platform, enhanced by a germanium-carbon alloy, to exploit the power of light."

Berger and James Kolodzey, a professor of electrical engineering, teamed up with students Xiaoping Shao, Sean L. Rommel, and Bradley A. Orner to grow a single epilayer of germanium, infused with 0.2% of

carbon, on a silicon substrate. Using molecular beam epitaxy (MBE), the researchers "spray-painted" the alloy onto silicon. Germanium and carbon were loaded into separate effusion cells operating at high temperatures greater than 600–700°C where they emitted a vapor that stuck to a heated silicon substrate loaded inside a high-vacuum chamber. Berger said that the resulting *p-n* diode effectively converted 1.4% of incoming laser light into electricity. (Researchers used laser light operating at 1.3 μm , a wavelength undetectable to silicon, but compatible with current fiber optics.)

The diode also rectified the flow of electricity even when subjected to an abrupt 80-volt reverse bias, Berger said. "That means it could withstand a lightning-storm type power spike with no interruption in the flow," he said.

Berger said that while a 1.4% light-to-electricity conversion rate "is not stellar," and that the prototypical diode included molecular flaws, this is the first proof of



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this principle. He said, "It shows that, even when we cut corners to develop this crude, stripped down device, we still got pretty good light-conversion and rectification numbers—and we did it all on a silicon platform."

He said that, moreover, the device demonstrated a relatively efficient conversion rate, given that light was transformed through direct contact with an ultrathin (50 Å) active region of only 20 atomic layers, under "surface-normal" conditions—and not through a concentrating waveguide.

Berger said that silicon-based, germanium-carbon devices could "bridge the gap" between current silicon computer chips and next-generation microprocessors based on gallium-arsenide alloys. As silicon chips approach speeds of 2 GHz, their performance wanes. Gallium-arsenide devices promise faster speeds—up to "tens of GHz," according to Berger. But a single, two-inch gallium-arsenide wafer might cost \$100, compared to only \$10 for a silicon wafer the size of a dinner plate. Germanium-carbon alloys, offering speeds "in the low gigahertz range" cost less than gallium-arsenide materials, Berger said.

Kolodzey said, "In the near term, our research might lay a foundation for improved radio-frequency wireless communication devices such as cellular telephones, as well as solar energy conversion."

Nanocrystal Size Can be Controlled During Electrodeposition

Researchers at the Weizmann Institute have shown that by manipulating the mismatch between the spacing of atoms in two materials they can control the size of microscopic semiconductor crystals. As described in a series of papers, the most recent two published in *Advanced Materials*, using electrodeposition, the scientists laid down crystals of the semiconductor cadmium selenide, each measuring 4–5 nm, onto a gold substrate. The crystals were found to be oriented in a uniform manner. This configuration is highly beneficial for controlling the semiconductor properties and it occurs because the atoms of the crystals tend to align themselves with the atoms in the surface layer of the substrate. Such alignment is attributed to the good match between the interatomic spacings of cadmium selenide crystals and of gold.

Gary Hodes of the Materials and Interfaces Department said, "We now run into what might at first glance seem to be a paradox. On the one hand, attaining a close match is the important first step leading to crystal alignment. On the other hand, the match is not perfect—but this imperfection can also be made to work to

our advantage. In fact it is the small remaining mismatch that allows for precise control over crystal size."

The substrate stretches the growing crystal in an attempt to minimize the mismatch, creating a strain within the crystal that eventually causes it to stop growing altogether. Therefore, by fine-tuning the mismatch the scientists were able to determine when the crystal will stop growing.

They showed that the addition of small amounts of tellurium to the semiconductor reduced the mismatch to a predictable degree. This allowed them to produce uniformly oriented crystals of varying sizes.

NASA Steadies Tethered Satellite for Propulsion and Power

Scientists and engineers at NASA's Marshall Space Flight Center are developing a test model of a device, ProSEDS (Propulsive Small Expendable Deployer System), that will use Earth's magnetic field to make a rocket stage re-enter the atmosphere in a few days instead of months. If it works, then scientists will be able to keep satellites up without using rockets. ProSEDS will use a 20 km extension cord that plugs into the magnetosphere and turns the cord into an electric motor that slowly raises or lowers a satellite's orbit.

The concepts behind ProSEDS are derived from the Tethered Satellite System (TSS) flown on the Space Shuttle in 1995 and 1996. Although the tether broke as it reached its 19.6 km length on its 1996 flight, scientists gathered data about tether behavior during five hours of operation.

"There's a new model out there on how you collect electrical current in space," said Nobie Stone, project scientist for the TSS. Stone and Dennis Gallagher, both in NASA Marshall's Space Sciences Laboratory, are advising Marshall engineers on the electrodynamic aspects of the ProSEDS experiment.

The TSS employed a large deployment mechanism, resembling a deck winch, in the Space Shuttle payload bay. The winch unreeled 20 km of insulated, conducting tether with a spherical satellite at the end. As the Shuttle orbited the Earth, the electrical wire cut through the Earth's magnetic field, and the motion produced an electrical current. Electrons were collected by the satellite, through the tether, and flowed out the Shuttle by way of an electron gun that dumped the charge as it built up.

The scientists found that the tethered system produced more current than expected. Specifically, the models require that the voltage be 10 times greater to collect a cur-

rent than what was observed. Before the flight, the models predicted that the tether would produce 0.5 amp under ideal conditions. Instead, it produced more than 1 amp under less than ideal conditions.

The TSS carried on the Space Shuttle was a large, complex system. Discoveries from it show how smaller tethers can be used to keep satellites in orbit.

Stone said, "The models were a factor of two or three off because they don't include

Special Journal Issue Describes Research on Mid-IR Laser Materials

The August 18th issue of *Optics Express* (1, #4, 1997, <http://epubs.osa.org/opticsexpress/framestoc5.htm>), an online multimedia journal published by the Optical Society of America, is a special issue dealing with mid-infrared laser materials. The issue contains four articles and an introduction. Sean M. Kirkpatrick, L.B. Shaw, S.R. Bowman, S. Searles, and B.J. Feldman from the U.S. Naval Research Laboratory and Joseph Ganem from Loyola College of Maryland describe studies of the mid-infrared spectroscopy of erbium-doped chloride crystals. Growth techniques and analysis of potential 3.5 and 4.5 μm laser materials are described. L.B. Shaw, B.B. Harbison, B. Cole, J.S. Sanghera, and I.D. Aggarwal from the U.S. Naval Research Laboratory describe studies of the spectroscopy of Pr³⁺-doped BaInGaGeSe chalcogenide glasses for lasers, amplifiers, and high brightness sources for the mid-infrared. D. Zhang, R.Q. Yang, C.-H. Lin, and S.S. Peia from the University of Houston and E. Dupont, H.C. Liu, and M. Buchanan from the National Research Council of Canada describe work on the development and analysis of electroluminescence in the long-wavelength infrared (10–15 μm) spectrum region from Sb-based type-II interband cascade quantum-well structures. The final paper describes work by T. Schweizer, D. Brady, and B. Hewak from the University of Southampton, UK to develop and characterize gallium lanthanum sulphide-based glasses as high-quality hosts for rare-earth-doped, mid-infrared fiber lasers. These lasers would offer compact and rugged sources for gas sensing, atmospheric transmission, and medical applications.

the effects of orbital motion through the plasma of the ionosphere." While motion of a conductor through the magnetic field is crucial, motion through the electrons in space was thought to be a minuscule effect. The Shuttle moves at 7.7 km/s while the electrons move at 200 km/s. According to the scientists, the current carried by those electrons connected nicely with the tethered system and contributed significantly to the power generated.

J.R. Sanmartin of the Polytechnic University of Madrid, Spain, predicted that a tethered system did not need a large sphere at the end of the line to work. The motion of a satellite through space generates a plasma shield that stands off about 1 cm away from the spacecraft surface. On the 1.8 m diameter TSS, that 1 cm standoff adds only about 2% to the collecting area. On a wire, it increases the collecting area 400-fold or more, so that an 82-meter wire now has as much effective collecting area as the 1.8-meter sphere.

Gallagher said, "The applications of this

are, potentially, to produce power or thrust on the International Space Station." The tether could provide extra electricity to the station, or help maintain its altitude so it does not re-enter Earth's atmosphere. The tether will produce just a little force. The force on the Shuttle was 0.4 newton. But applied steadily, for hours or days, it makes a difference. Les Johnson of Marshall's Advanced Systems and Technologies Office predicts that a 10 km, 10 kilowatt tether system could boost a 1,000 kg satellite as much as 400 to 540 km in one day, depending on the orbit and other conditions.

Johnson and others in the Advanced Systems and Technologies Office are developing the ProSEDS concept to test this innovative idea.

Johnson said, "The big thing we're trying to do is demonstrate the propulsive utility of an electrodynamic tether. We view this as a precursor to a lot of different approaches that we've been studying."

ProSEDS is much smaller than the Shuttle's TSS. In operation on future satel-

lites, the tether, which will look more like dental floss than TSS's high-tech rope, will unreel from a bobbin in a can. The can is released from the satellite and the difference in Earth's gravity pulls the can down. Eventually, the tether is unwound to a distance of 25 km. About 5 km of tether near the spacecraft would be bare; the rest is nonconducting and provided to put enough distance on the tether so it stays taut. Stone said that if the new bare wire tether works as predicted, "it would allow us to collect considerably more current for a given length of tether." As a result, shorter tethers could be used for propulsion or to generate electrical power.

In the ProSEDS demonstration flight, the satellite will be the second stage of a Delta rocket. The tether bobbin will stay on the rocket and a weight will be unreeled upward on the tether. After the satellite is injected into orbit, the second stage normally would be slowly pulled back from 400 km to Earth by atmospheric drag. After 120 days, it re-enters and burns up.

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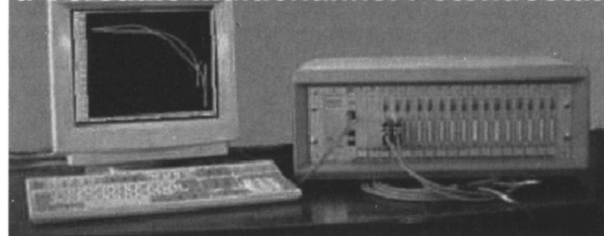
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ACS Meeting Held in Las Vegas in September

The American Chemical Society held its 214th National Meeting in Las Vegas from September 7 to 11. Different research teams from The Pennsylvania State University (Penn State) reported on fuel science research in which one team has developed jet fuel functioning simultaneously as a coolant and another group is working toward emissions control for coal-fired boilers at lower temperatures than is currently possible. Researchers from the Georgia Institute of Technology reported on a class of polymeric membranes that could expand gas separation uses through improved thermal and chemical resistance than is currently possible. Researchers from Duke University reported on a way to speed up quantum chemical calculations.

Research Points to Fuel Reformulation as Only Way to Avoid Fuel Degradation, Carbon Deposition When Jet Fuel Combined as Coolant

In preparation for the next generation of advanced supersonic aircraft, researchers at Penn State are experimenting with ways to prevent fuel degradation and carbon deposition when using jet fuel as a coolant before it is burned as a fuel.

"Today's aircraft are air cooled, but at the high speeds expected of advanced aircraft, air can't be used for cooling," said Semih Eser, associate professor of fuel science.

The coolant will cool the engine and also circulate through the wings and fuselage to reduce the high temperatures caused by air friction. Jet fuel can be used as a heat transfer liquid, but begins to break down rapidly at above 575°F (298°C). The degradation to smaller molecules will not affect the fuel's efficiency, but eventually carbon will deposit out of the fuel onto the fuel lines.

Eser, graduate student in fuel science Jun Li, and postdoctoral fellow in polymer science Maria Sobkowiak are studying the fuels to characterize the way carbon deposits on nickel, stainless steel, and copper. The research shows two main mechanisms of carbon deposition.

"Above a certain temperature, nickel and stainless steel begin to act as a catalyst," said Eser. "Filamentous carbon deposits on the surface and particles from the metal surface actually move into the carbon layer."

This erosion and pitting of the metal surface could eventually cause pinholes in the fuel lines, while the carbon deposition could clog the fuel lines.

On smooth nickel surfaces, carbon was deposited mainly in nonfibrous layers without interaction with the surface. This deposition takes place at higher temperatures than the catalytic reaction.

Eser said, "It appears that rough surfaces are more likely to act as a catalyst, while smooth surfaces simply build up layers of carbon."

Although copper lines would probably not be used in advanced aircraft, the researchers also tested copper. Copper grows fibrous, not filamentous carbon on rough surfaces. The carbon deposited on these copper surfaces resembles minute arrows, while those on stainless steel or nickel are uniform and the size of the metal particle on which they grew.

"If the metal is coated with silica, the catalytic type of deposition does not occur," said Eser. "It might also be possible in the future to modify the surface of fuel lines to passivate the catalytic sites so that filamentous carbon does not form. However, to eliminate carbon deposition completely, we need a reformulation of the fuel."

Low-Temperature NO_x Reduction Catalyst Tested for Future Use in Production Boiler Systems

Penn State researchers in another area of fuel science are studying emission control systems for boilers which generally consist of a baghouse that removes flyash and other particulate materials, a method for removing sulfur from the flue gas, and a method of converting nitric oxide produced during combustion to environmentally harmless forms of nitrogen. Typically, a sorbent is injected into the flue gas to remove sulfur before the gas enters the baghouse.

Ammonia is also injected to facilitate the selective catalytic conversion of NO_x. Frequently, NO_x conversion takes place after the gas leaves the baghouse, but researchers at Penn State want to place the conversion catalyst monoliths inside the baghouse.

Andre L. Boehman, assistant professor of fuel science, said, "Babcock & Wilcox have a high temperature process that provides combined sulfur dioxide, nitrogen oxide, and particulate emissions control for coal-fired boilers that they call the SO_xNO_x Rox Box. We are trying to modify this process to permit combined emissions control at the much lower temperatures common in industrial scale boilers."

Boehman's research group carried out bench scale tests using synthetic flue gas

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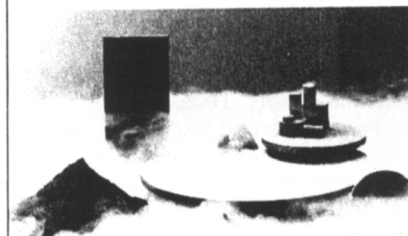
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containing sulfur. The first round of pilot-scale tests was run using a combustor fired by natural gas. The second round of pilot-scale tests will use a fuel containing sulfur. The eventual goal of this project is to test the system in a demonstration boiler.

Boehman said, "We need to ensure that sulfur removal occurs before the flue gas reaches the NO_x catalyst, so that the sulfur does not form ammonium sulfates and foul the catalyst. We are also investigating the variables that control the total proportions of nitric oxide converted and the proportions converted to nitrogen as opposed to nitrous oxide."

The conversion of nitric oxide to nitrogen is governed by temperature and the speed at which gas passes over the catalyst. The presence of sulfur and water also effects the outcome. The researchers can achieve NO_x conversion at 95%, but with 50% of the conversion product nitrogen and 45% nitrous oxides at the temperatures under study.

Boehman said, "We have designed this system to work between 350 to 400°F [175 to 202°C], which is in the range of temperature within the baghouse of our demonstration boiler. The nitric oxide conversion is comparable to that seen in high temperature systems. But this low-temperature catalyst has a tendency to form nitrous oxide instead of nitrogen. We are looking for operating conditions that minimize the formation of nitrous oxide."

Better Thermal and Chemical Resistance Achieved in Gas Separation Membranes Using Polyimide Materials with Crosslinkable Diacetylene Groups

Researchers from the Georgia Institute of Technology reported on improved thermal and chemical resistance in gas separation membranes produced by blending polyimide materials containing crosslinkable diacetylene groups. A solid-state crosslinking reaction initiated after formation of the membranes accounts for the improved properties. Because the reaction is believed to occur in the more ordered regions of the blend, it does not significantly increase sample density. Thus, improvements in mechanical properties and chemical and thermal resistance are realized without reducing the material's gas transport and separation abilities.

Mary E. Rezac, assistant professor in Georgia Tech's School of Chemical Engineering said, "We have jumped a very large hurdle in having a material

that is chemically and thermally resistant while retaining very attractive gas transport properties. There could be a very large commercial market, but there are a number of technical hurdles still ahead of us."

Conventional polymeric gas separation membranes lose mechanical strength at temperatures above 100°C and can be damaged by reactive components in gas streams, Rezac said. But tests show that the new polymers are stable at temperatures of more than 400°C, and are not significantly affected by the chemical contaminants.

Rezac and collaborators Haskell W. Beckham, Birgit Bayer, E. Todd Sorensen, and Njeri Karangu produced the new membranes by blending a non-reactive polyimide with a new diacetylene-functionalized polyimide. The blend was then dissolved in methylene chloride and formed into a film using conventional techniques. It was then heated to initiate a crosslinking reaction in the diacetylene-containing portion of the polyimide blend.

The researchers used polyimides based on hexafluoroisopropylidene diphthalic anhydride (6FDA) as the nonreactive component in the blend. The reactive portion contains both a hexafluoroisopropylidene bisphthalimide moiety and an aliphatic diacetylene moiety. Varying the proportions of the two materials produced blends with different degrees of thermal and chemical resistance.

Crosslinking reactions normally cause shrinkage of the material as the carbon chains link together. According to Beckham, assistant professor in Georgia Tech's School of Textile and Fiber Engineering, that has foiled previous attempts to produce stronger membranes because increasing the density of the membrane film decreases its gas permeability.

Beckham said, "We believe these new materials retain their gas transport properties because the crosslinkable groups can undergo a solid-state reaction without significantly increasing the density of the material. That is a special feature of this material."

Because of its cost, the material is unlikely to be used commercially. However, the researchers have produced a series of other diacetylene-containing polyimides to determine whether these special properties may be found in similar and less costly polyimide materials.

Beckham said, "Incorporating diacety-

lene groups into linear chain backbones to crosslink polymers is not itself novel, but nobody has done a systematic study of using these groups to crosslink polyimides. We want to see if the characteristics observed for the crosslinkable 6FDA-based polyimide blends are general to polyimides, or specific to this system."

Rezac believes the new membranes could make the recovery of hydrogen from petrochemical processes economically feasible. The temperature of refinery gas streams—containing hydrogen, propane, methane, and other hydrocarbons—exceeds the thermal operating range of current membranes, and cooling those streams can cost more than the recovered materials would be worth.

Rezac said, "If we can achieve these types of separations, we can reduce operating energy, waste production, and the pollutants going into the atmosphere. The dollar value of the hydrogen is significant, but there are external issues in terms of recycling and pollution control that are just as important."

In addition to evaluating other polyimide materials, Rezac and Beckham hope to confirm and understand how the crosslinking process can produce stronger materials without increasing density. They theorize that the linking takes place in regions of the polyimide that do not contribute to the gas permeability.

Beckham said, "By selectively crosslinking the more ordered regions of the polyimide that don't contribute to the gas transport anyway, the diacetylene groups allow us to tie up the structure without reducing the gas transport properties."

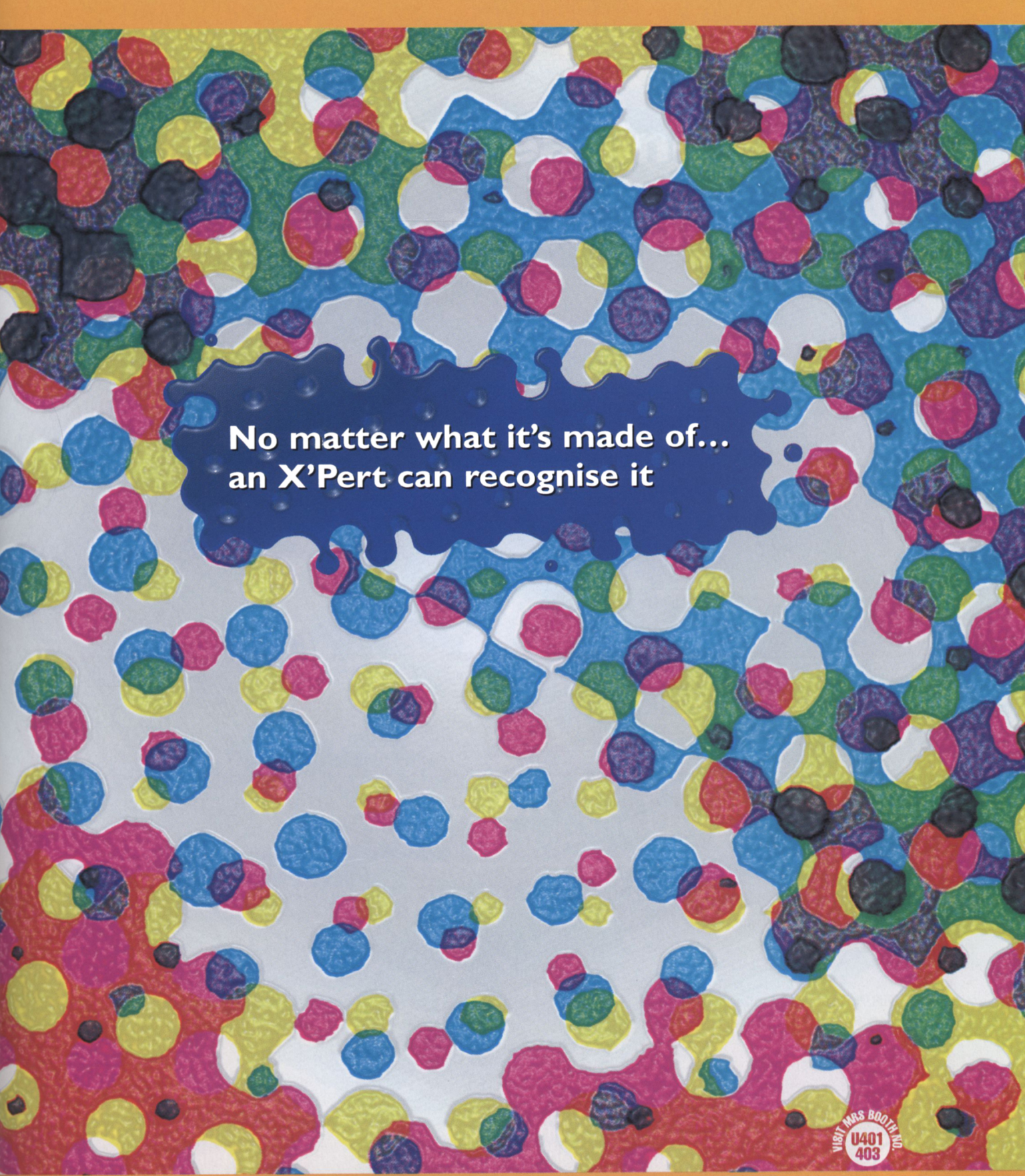
Computational Shortcut Speeds Quantum Chemical Calculations

Weitao Yang, an associate professor of chemistry at Duke University, has described the development and application of a "divide and conquer" method requiring far fewer calculations than currently used to model the electronic structure of large molecules.

"Electrons do not fit in our picture of the classical world," Yang said. "We need quantum mechanics to describe electrons. And when we do that, we are able to describe chemical reactions, the breaking and formation of chemical bonds, and many other interesting processes in chemistry and biology."

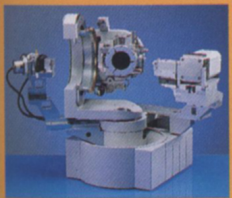
However, traditionally, chemists doing such quantum mechanical calcula-

Continued on page 24



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Continued from page 22

tions had to look at the big picture. "If they wanted to calculate one part of the molecule they had to calculate the entire molecule at the same time," Yang said.

Such calculations were impossible for molecules larger than a few hundred atoms, he said, because the required computations rose by a cube of the number of those atoms. "You soon run out of computer capability," he said.

Yang and his associates introduced a method that allows such large quantum mechanical calculations to be vastly speeded up by breaking up the problem.

"We call it the 'divide and conquer' method," he said. "The method enables us to calculate a molecule one piece at a time in a very sophisticated way. Such division is possible because we chemists know that the properties of a molecule are very localized. And these properties

depend on the local structure."

Calculations done by Yang's method rise linearly with molecule size, rather than rising by the cube of the molecule's size. Yang said, "If it takes an hour to do the calculation for 100 atoms, it would take two hours to do the calculations for 200. So it's much quicker. We are able to do calculations on our workstations that were impossible before."

Because Yang's method predicts electron properties so precisely, it can provide researchers a more refined picture of the behavior of big molecules, such as proteins. "We think of proteins as being made up of atoms. But those atoms have electrons, and electrons are the mediating forces between the atoms," he said.

In joint research with the University of North Carolina at Chapel Hill, Yang's method was recently used to reveal the

locations of some of the hydrogen atoms in an enzyme called cytidine deaminase. He said knowledge of the locations of two particular hydrogen atoms is very important for illuminating details of the enzyme's mechanism. Knowledge of how the enzyme promotes chemical reactions may have important therapeutic uses in antitumor drugs, Yang said. Hydrogen atoms, made up of a single electron and proton, are too light to be revealed by x-ray crystallography. That test reveals only the heavier atoms in the protein.

"So it is not possible to really tell where all the hydrogens were from experiments," Yang said. "One can only guess. But our calculations have nailed down hydrogen."

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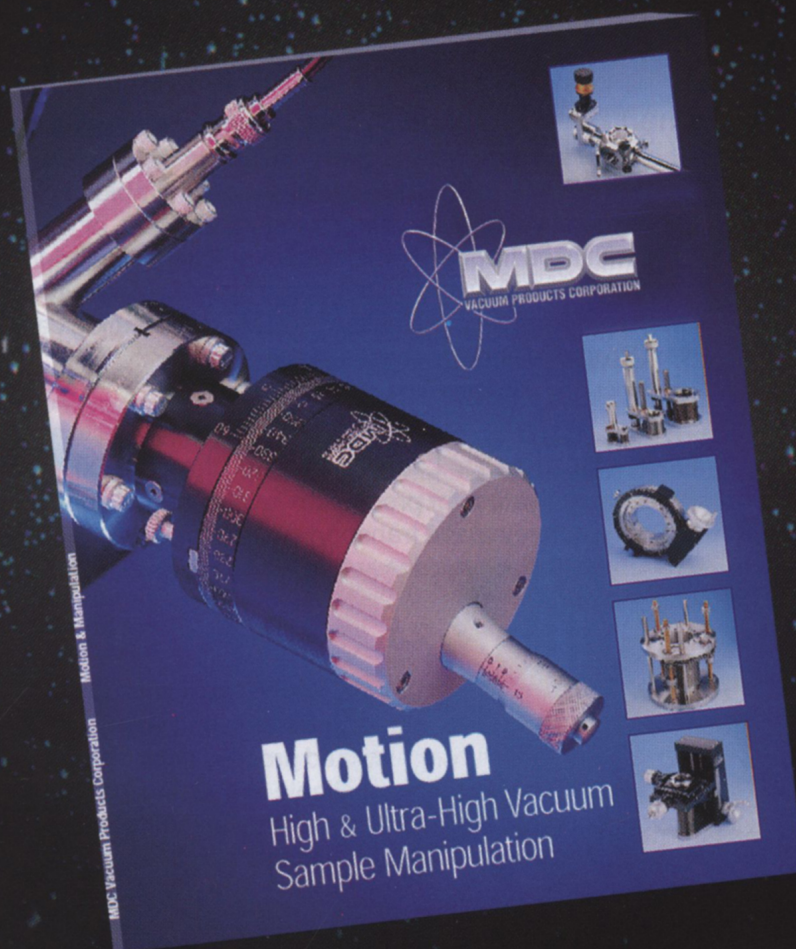


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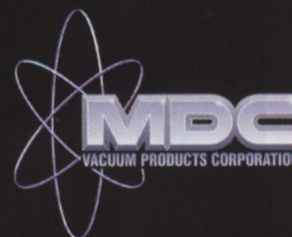
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