Copolymer Networks with Controllable Mesh Size Developed

Researchers at Purdue University have developed copolymer networks, which are "built" from intersecting chains of small molecules linked together to form a larger, meshlike structure. The two monomers used in the new materials are acrylic acid and a derivative of oligoethylene glycol. The properties of an individual material in the class can be varied depending on the relative amounts of the monomers used to prepare it.

Robert Scott, a PhD candidate at Purdue who helped develop the material, said, "Because these materials are copolymers, we can control their properties more precisely and over a wider range than we could if they were made of a single type of monomer. This level of versatility and control allows for a number of applications."

Scott presented information on the new materials at the annual meeting of the American Physical Society in Los Angeles in March. He said, "As we increase the acrylic acid content of the materials, the oligoethylene glycol chains that make up the network move farther apart, increasing the mesh size, which in turn determines what substances can pass through. By varying the acrylic acid content, as well as other parameters, we can precisely control the size of the molecules we allow through."

Another application Scott has investigated is the controlled release of substances. He said, "We've made systems that contain a model drug, and we're studying how the rate of diffusion of that drug out of this polymer varies as we vary the polymer structure. Using the material as a membrane for drug delivery is a particularly appealing application because we have very fine control over what drugs could be released through it and under what conditions."

In addition, he said, the acrylic acid in the materials makes them sensitive to acidity. The mesh size and diffusive properties vary depending on the pH of the environment, which is an important consideration for drug delivery applications because different parts of the body exhibit different pH levels. For example, a capsule incorporating this material and containing a particular drug might remain "closed" in the mouth and "open" in the stomach to release a drug.

One type of the material also can be made to have a very dense network of molecular chains, which would make it very resistant to liquids, according to Scott. He said, "In that case it might make an ideal coating for applications that require a very low permeability to moisture. We can also modify the properties so that it will absorb various amounts of liquid. We're looking very closely at how we can control its affinity for water."

Optical Tweezers Open Research on Magnetic Fields' Effect on Bose-Einstein Condensates

Physicists at the Massachusetts Institute of Technology have trapped condensates with light, and have "tuned" a condensate's behavior with magnetic fields, advancing new possibilities for the manipulation of ultracold atoms. The researchers first developed a trap to confine Bose-Einstein condensates that holds the atoms by purely optical forces exerted by an optical laser beam, which Wolfgang Ketterle, leader of the team who holds appointments in the Department of Physics and the Research Laboratory of Electronics, identifies as "optical tweezers" for condensates. He said, "We can now move a condensate around simply by steering a weak optical laser beam."

Key to the setup is an infrared laser beam that "sucks" the condensate into its focus, similar to how a toy magnet attracts a piece of iron. Ketterle said, "The laser field polarizes the atoms by separating the positive and negative charges a tiny bit, thus creating an electric dipole, which is trapped in the alternating electric field of the laser beam."

Physics graduate student Dan M. Stamper-Kurn said, "We expected the laser beam to heat up the ultracold atoms and destroy the condensate, but nothing happened. The condensate survived." As reported in the March 9 issue of *Physical Review Letters* and the March 12 issue of *Nature*, this was in contrast to earlier work on optical confinement of ultracold atoms which showed strong heating due to unavoidable laser beam jitter, laser power fluctuations, and spontaneous scattering of photons. However, the condensate created at MIT was so cold that extremely small laser powers of only a few milliwatts were sufficient to trap it, minimizing the heating.

The trap realizes "optical tweezers" for Bose-Einstein condensates, which opens many possibilities for future research, such as studying the interactions of condensates with surfaces, with electromagnetic radiation in cavities, or with other condensates.

The optical trap also opens the door for experimenting with magnetic fields and

their effects on condensates. Physics graduate student Michael R. Andrews said, "Trapping the atoms magnetically limited our exploration of magnetic fields. Now we can play with magnetic fields at will." The researchers were doing just that when they saw the first evidence for a Feshbach resonance on November 19, 1997. More specifically, they exposed a Bose-Einstein condensate of sodium atoms in the optical trap to a variable magnetic field.

When two atoms collide, they usually just "touch" each other and separate immediately. However, at a certain value of the magnetic field, the colliding atoms "stick" together, form a molecule for a while, and then separate again. This effect, the Feshbach resonance, was predicted for ultracold atoms by Boudewijn Verhaar and collaborators in the Netherlands.

According to Ketterle, atomic physicists have eagerly searched for this effect because it profoundly changes the properties of a Bose-Einstein condensate and can be used to design its properties. The interactions between atoms dramatically changed when the magnetic field was swept across the resonance. Physics graduate student Shin Inouye said, "The forces between the atoms below the resonance were ten times stronger than above."

Seismic Imaging of Heterogeneous Outer Core of Earth May Lead to Further Understanding of Earth's Magnetic Field

Using seismic wave data gathered from tens of thousands of earthquakes, researchers at Lawrence Berkeley National Laboratory (LBNL) have produced a three-dimensional image of the Earth's structure, from the crust to the inner core. In creating their model, Don Vasco and Lane Johnson of LBNL's Center for Computational Seismology found evidence that the outer core is not homogeneous as has been long hypothesized. This information could help scientists further understand the Earth's magnetic field, according to the researchers, who published their findings in the February 1998 issue of the Journal of Geophysical Research.

The researchers used seismic data collected during the 1960s through the 1980s and measured the time the waves took to travel from the epicenter of each earthquake to seismographic stations located around the world. By using computers to analyze travel times from some 40,000 earthquakes, Vasco and Johnson were able to characterize the seismic velocity of materials which make up the planet.

Until now, most researchers have focused on one region, such as the mantle, rather than the entire structure of Earth. Although they do not claim to have any definitive answers, Vasco said the work is another step in determining the Earth's makeup and its effects. The three-dimensional structure of the Earth's mantle has only been determined over the past 20 years and now scientists are digging deeper and studying the inner and outer cores. It is thought that the outer core, which starts about 3,000 km below the Earth's surface and is 2,300 km thick, is a liquid, with viscosity not much different from water. This led some to conclude that the outer core has no real structure.

Vasco said, "We found indications of heterogeneity at the bottom of the outer core." He described the material as an iron-nickel-sulfur compound. High pressures and temperatures could be causing nickel-rich iron to solidify and depleting the nickel at the base of the outer core, Vasco said, which could help explain the Earth's magnetic field. The depleted iron is less dense than the surrounding core, causing it to rise, leading to convection and a magnetic field.

Liquid Crystal Assay Prototype Detects Antibodies

Nicholas Abbott of the University of California—Davis and his colleagues have reported in the March 27 issue of *Science* their construction of a prototype liquid crystal assay in which they changed surface topography rather than apply an electrical field to orient the liquid crystals. The researchers' goal was to create a liquid crystal assay that would report whether an antibody was present in a test sample.

First the researchers spread an ultrathin layer of gold atop an inch-square glass plate, creating a microscopic landscape of parallel hills and valleys. Within that landscape they placed a field of molecular receptors with a particular affinity for the target antibodies. The sample solution was then poured over the assay. The molecular receptors took hold of the antibodies. When the solution ebbed away, the landscape was left studded with antibodies. A few drops of liquid crystal then were added across the landscape to settle.

Had there been no antibodies in the test solution, the liquid crystal molecules would have aligned in parallel along the hills and valleys. In that orientation, they would have transmitted no light. However, because there were antibodies, the liquid crystals were forced to bend around them, emitting light, creating patterns of yellow, orange, and red, or fluorescent pink and green.

Abbot said, "Right now, the assay can detect a threshold concentration—it can say whether the target material is there or not. But it can't yet tell us how much of the material is there."

Correction: In the article, "Jane-Lun Bredas Receives 1997 Francqui Prize," (*MRS Bulletin*, March 1998, page 19), the award recipient's name is **Jean-Luc Brédas** and his affiliation is Mons University, Belgium. He is an advisor at the Frontier Research program at RIKEN in Japan.



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Research News Briefs from ACS Meeting

The American Chemical Society held its 215th National Meeting and Exposition in Dallas from March 29 to April 2. Among the presentations is a report from Cornell University on fabricating smooth surfaces on silicon wafers and two from the University of Delaware in the areas of surface chemistry and microblock co-polymers.

Chemical Etching Produces Atomically Smooth Surface in Silicon (111)

Melissa Hines, an assistant professor of chemistry at Cornell University, has reported on the production of silicon surfaces that "are essentially totally flat." The possibility of surface perfection was discovered about five years ago when Bell Labs researchers sought a new method of removing dust from the silicon wafers used to produce integrated circuits. The old method, developed in the 1960s, involves washing the silicon wafers in basic peroxide baths. But much smaller circuitry develops atomic-scale roughness from the chemical, significantly reducing the transistor's performance. By changing the acidity and composition of the chemical solution the researchers discovered they were able to produce small areas on the silicon surface that were totally flat, even at the atomic level in which surface roughness was equal to only one protruding atom out of every 30,000 surface atoms.

However, this perfection is only reproducible on silicon (111), which is a different plane from the silicon (100) used for integrated circuits. Thus, said Hines, the goal of research is to find chemical solutions that will produce perfection on different surfaces. To do this, she said, it must first be understood how a basic hydrofluoric acid solution used in her research etches away protruding atoms.

The most perfect surface Hines and her colleagues have achieved to date appears through the electron tunneling microscope as a series of steps, with every step only a single atom high. The steps are the result of almost imperceptible errors in cutting the silicon wafer. Because of the chemical action, each step is evenly spaced and almost straight.

Another example of surface chemistry is the production of equilateral triangles. In this case, the chemicals appear to burrow into small defects on the silicon surface, each a few atoms across, and then open the defects out into triangles about 1,000 atoms across. The bottom of each triangle is flat. Hines said, "It turns out there is an atomic defect in the crystal that is very reactive. When etched, the atomic structure becomes triangular."

In both cases, the flat surfaces and triangles, the chemicals etch away surface atoms, one atom at a time, in a very precise order. Hines calls the process "unzipping" because neighboring atoms are etched in a sequential fashion in much the same way that teeth in a zipper are sequentially opened. It is this type of reaction that Hines is seeking to control in her quest for perfect surfaces.

Fluorescent Molecule Exposes Photomask Scratches

Mary J. Wirth, a professor of chemistry and biochemistry at the University of Delaware and recipient of a 1994 National Science Foundation Creativity Award, reported on fluorescent molecular markers that "measure imperfections on the surface of materials used as templates for integrated circuits." She said, "Our goal is to measure surface flatness optically, on the molecular scale, as fast as we can."

According to Wirth, as computer chips or integrated circuits become increasingly complex, with ever-smaller components, even molecular-scale flaws can create big problems. Each photomask must be polished to an atomically smooth finish. A scratch no larger than a thousandth of a micron could result in serious photomask limitations.

Wirth's collaborator, Daniel W. van der Weide, director of UD's Center for Nanomachined Surfaces and recipient of a 1998 Young Investigator Program Award from the U.S. Office of Naval Research, said that traditionally atomic force miscroscopy offered one way to spot flaws on photomasks, but this process is extremely timeconsuming.

Wirth said that her strategy is to use a molecule to measure something the size of a molecule. First, silica is washed with nitric acid and water to remove any contaminants on the surface. Next, a small amount of fluorescent dye with a high affinity for silanols groups of silicon, oxygen, and hydrogen that comprise scratches on silica—is placed on the sample. Doctoral candidate Derrick J. Swinton said, "Individual fluorescent molecules of an indocarbocyanine dye tightly stick to points along these shallow scratches because of an electrostatic attraction."

In this way, scratches of atomic dimensions turn into bright fluorescent lines that are visible through a high-quality optical microscope, which is positioned beneath the silica sample. Wirth and Swinton attach a camera to the microscope to capture realtime images of the dye fluorescence. Compared to existing tip-based techniques for probing surfaces, Wirth's approach "lets us quickly find small scratches over large areas," according to van der Weide; "The dye molecule amplifies the presence of these imperfections so that they can be detected quite easily with conventional microscopes."

Microblock Copolymers Exhibit Enhanced Properties

John F. Rabolt's research team at the University of Delaware reported on the properties of materials synthesized by strongly bonding together segments of different homopolymers. When the researchers combined various homopolymers to make microblock copolymers, they "expected a hybrid effect," Rabolt said, but discovered that "the resulting properties are much better than would be predicted from looking at the properties of the two constituents." Thus far, the research team has examined two combinations of microblock copolymers: one type (ethylene-tetrafluoroethylene) supplied by Anselm Griffin's research group at the University of Southern Mississippi, and another (ethyleneethylene oxide) provided by Greg Baker from Michigan State University.

Through chemical processing, the researchers have formed covalent bonds between the two polymers, and a phenomenon known as "conformational influence," bringing about changes in homopolymers that come into contact with the microblock copolymer. The enhanced properties that result from these novel combinations of polymers include higher tensile strength and temperature resistance as well as greater stability. Rabolt said, "For example, a polymer may be metastable as a homopolymer-that is, it can be extended when stretched from its coiled state but will spring back when the tension is removed. By making it a microblock copolymer component, we can change its properties so that it is extended and remains that way rather than coiling back up."

The researchers have found that in many cases a supramolecular helix results when microblock copolymers are formed. Rabolt has postulated that there is an attraction between like polymer segments, which means that when the material solidifies, it forms a coiled structure where like segments are lined up with each other. According to Rabolt, this effect is probably at least partially responsible for the unexpectedly enhanced properties seen in these materials. The segments usually fall into the 10- to 20-unit range, but can vary from three to 50 chemical units. The properties of two microblock copolymers will differ with different segment lengths, even if the same two polymer chemical architectures are combined, Rabolt said. Rabolt said, "What remains now is to determine which combinations of materials and segment lengths yield the properties that are desired for various applications."

Nanomachined Electrostatic Chuck Operates at High Temperatures in Microchip Production

Researchers at Sandia National Laboratories have patented a prototype of an electrostatic chuck that electrostatically clamps onto a silicon wafer, allowing the temperature to be regulated with confined helium gas. Because the chuck face is made of the same materials as computer chips—silicon, silicon dioxide, and silicon nitride—and is fabricated with wellunderstood silicon chip technology, manufacturing costs are expected to be far lower and batch contaminations far fewer than those for a conventional chuck's sprayed-on overcoat of diverse materials currently used by industry. The new chuck should be able to operate at higher temperatures more suitable to modern wafer processing.

The chuck, like more conventional

Studies on Ice Elicit Information on Adhesive/Separation Properties and on Nature of Hydrogen Bonds

Electric Charge Separates Ice from Metal

Thayer School of Engineering Professor Victor Petrenko of Dartmouth College has discovered that applying a small electric voltage across an ice-metal interface can break the bond between ice and metal surfaces. Several years ago, Petrenko became intrigued at similarities in the physical properties of semiconductors and ice. Technically, Petrenko said, ice is a semiconductor. This is important, he said, because of what happens when molecules of water become molecules with regard to their electrical charge. At the surface, however, molecules tend to line up in the same direction: primarily oriented with their protons facing out, or primarily with their protons facing inward, buried in the ice. The reason for this is unclear, but the effect is significant: a high density of electrical chargeseither positive or negative, depending on the material-on an ice surface.

As reported at the 215th National Meeting and Exposition of the American Chemical Society in Dallas on March 30, when an electrically charged surface comes into contact with any other surface, the charged surface induces an opposite charge in the facing surface and the two surfaces are drawn together. Petrenko said, "This simple attraction accounts for most of ice adhesion." Breaking the bond between ice and metal, he reasoned, might be as simple as neutralizing the surface charge with an equal amount of its opposite.

Petrenko tested the theory, using a sheet of ice, a globule of mercury, and a small battery with two wires attached. He touched one wire to the ice, the other to the mercury, and the mercury drew itself up and away from the ice: the current had loosened its grip. Petrenko repeated the experiments using steel and other solid metals. In each case, the electricity caused the ice to lose adhesion. The effect could also be reversed, causing a surface to stick more firmly to the ice.

Low-Energy He Scattering Measurement on Ice Provides Structural Information

An international team of physicists in the group of Peter Toennies at the Max-Planck-Institute for Fluid Dynamics in Göttingen have used the scattering of very low-energy He atoms for an analysis of the structural arrangement of water molecules on the ice surface and have also gained direct information on their vibrational motion. The results of these experiments, published in the March 23 issue of *Physical Review Letters*, indicate that the molecules are surprisingly mobile which explains many peculiarities in the interactions of ice with its environment.

The researchers have employed lowenergy helium atom scattering, a technique that has the advantage of being completely nondestructive and exclusively sensitive to the topmost layer of crystals. Since the (111) surface of platinum has nearly the same lattice spacing as ice, it was used as a template on which single crystal ice films of 10-100-nm thickness were grown. After cooling the surface to 30 K, the researchers were able to observe a sharp intense series of diffraction peaks. These not only provide information on the lattice spacing and arrangement of the first layer molecules but also indicate at least a partial alignment of the hydrogen atoms (ferroelectric ordering) at the surface.

A further advantage of the He atom scattering technique is that high-resolution time-of-flight energy loss and gain spectra can be measured with the same equipment. These spectra provide information on the frequencies and wave-lengths of the collective vibrations (phonons) at the surface. As the crystal was again cooled down to 30 K, a very intense inelastic peak emerged from a strong multiphonon background. This intense inelastic peak was simulated with a theoretical model which allowed its assignment to a special very large amplitude in-plane shearing motion of the surface molecules. At higher temperatures, this motion becomes increasingly enhanced leading to a high

density "phonon bath" and ultimately causing individual molecules to break away from their original sites. According to the researchers, this explains the liquidlike topmost layer.

The researchers said that this vibrational disorder at the ice surface also explains why two pieces of ice fuse when pressed together. The H₂O molecules at the ice crystal surface form hydrogen bonds with those of another ice surface when two crystals are brought in contact, thus increasing their coordination. This results in a stiffening of the soft surface vibrations, making the interface solid. In addition, the high rate of accommodation of molecules on the surface of ice particles in the stratosphere can also be understood in terms of the facile energy transfer of the molecules with the phonon bath available at the surface.

Ice at High Pressure Exhibits Proton Tunneling

Through studying the behavior of ice under pressure, a scientific team from the Max Planck Institute for Solid-State Research in Stuttgart and the University of Montpellier II report in the March 19 issue of Nature a study of the behavior of hydrogen bonds. By applying pressure, the water-water distance can continually vary, bringing the water molecules so close together that the proton can be shared between two molecules, leading to the so-called symmetric hydrogen bond. In these circumstances, the water molecule ceases to exist as a separate entity and the protons become transferred from one molecule to another.

When the molecules are subject to sufficient high pressure, the proton can jump from a molecule to a neighboring one, due to a typically quantum phenomenon called tunnelling, which allows light particles like protons to overcome energy barriers. At even higher pressures the proton sits on average at the middle of the bond (symmetric hydrogen bond), leading to the so-called ice X phase, which is a non-symmetric phase of ice. ones, confines a thin layer of helium gas that drains heat from a silicon wafer during plasma-etch fabrication steps. The relatively low pressure of this gas permits the use of electrostatic attraction to oppose it and seal the wafer to a bottom plate, thus trapping the helium.

Project leader Alan Hurd said, "An electrostatic chuck and the wafer held on it are essentially a big capacitor. The force generated within the capacitor tends to squish it together."

The electrostatic chuck face is, itself, an easily produced, patterned silicon wafer that consists of tiny, nonconductive silicon dioxide islands rising above the surface of the rest of the wafer. The intentionally fabricated islands physically support the wafer being etched. These islands are insulating, so a strong electric field can be applied between the chuck face and the clamped wafer without excessive currents being developed. This field can be turned on or off quickly, allowing the chuck to grab or release just as rapidly. The chuck face of the in-use design consists of a composite material, randomly sprayed on a base plate and then polished.

Bob Anderson, who holds the patent, said, "The coating on the in-use chuck needs some electrical conductivity so that high fields can be developed between the chuck face and the wafer intended to be held, but an excessive conductivity would short out the power supply that generates the clamping field." Therefore the conductivity must be kept fairly small to limit leakage, and results in appreciable time delays for applying or releasing the clamping force.

When the voltage is turned off, the wafer on a conventional chuck might remain "stuck" for a time. Anderson said, "Our design circumvents that problem by micromachining the chuck face to create nonconductive raised areas—the islands—while the bulk of the chuck facing remains electrically conductive."

According to Anderson, "The composite material on conventional chucks is complicated, difficult to make, and raises the possibility of contamination by foreign material." The useful temperature range of this composite is also restricted. The new design, instead, uses a chuck face that is tolerant of high temperatures.

Hurd said, "You can't rejuvenate the conventional base plate when it wears down over time—it's got to be thrown away." According to Hurd, the wellunderstood fabrication process for creating the new chuck face should significantly reduce the cost of chuck replacement.

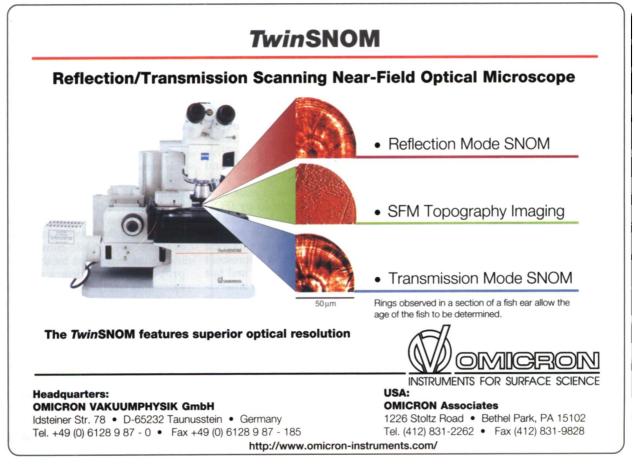
Self-Assembled Molecules 50-µm Long Produced

Researchers at the University of Rochester have fabricated self-assembled molecules that are 1,000 times larger than the previous largest synthetic self-assembled structures, as reported in the March 20 issue of *Science*. Each is made up of millions of molecules that organized themselves together. Graduate student X. Linda Chen said, "In the world of self-assembly, these structures are giant. We've created microstructures through self-assembly." Previous self-assembled structures have been made at the nanolevel. The largest of the team's structures are 50- μ m long.

Chen and Samson Jenekhe, a professor of chemical engineering, chemistry, and materials science at Rochester, used poly(phenylquinoline)block-polystyrene, a type of block co-polymer, which features both a rigid half and a flexible half. The researchers incorporated hydrogen bonds into polymer structures, giving them the same source of stability that helps DNA and self-assembled proteins in nature arrange themselves into functioning objects.

Chen said, "We first realized last summer that this polymer had special potential for self-assembly."

In one experiment the team used its self-assembled hollow spheres as tiny containers to carry billions of buckyballs. Some studies have shown that buckyballs can shield certain cells from many different types of damage, but their usefulness is limited because they are not very soluble. Shuttling billions of them via a selfassembled shell might prove a convenient means of drug delivery.



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Use of XTM and AFM Provides Insight into Tooth Dentin

Researchers at the University of California—San Francisco School of Dentistry (a team headed by Sally J. Marshall) are using an x-ray tomographic microscope (XTM) and atomic force microscopy (AFM) to observe structures in the dentin—the porous material that lies under the hard enamel of teeth—as small as 2 μ m. The researchers' goal is to understand the structure and properties of dentin in order to find methods and materials that will create a tighter, more permanent bond between the tooth and the plastic-based fillings now used to repair most cavities.

In the first stage of the study, as reported at the annual meeting of the American Association for Dental Research, the researchers examined the way dentin is structured and how a bond forms between the natural material of the tooth and the plastic filling. Strong bonds form easily between restorative materials and the surface enamel of teeth because about 97% of the hard surface material is mineral. But because tooth decay eats through the enamel and into the underlying layer of the tooth, a bond must be formed with the dentin, which is only 50% mineral.

Because dentin contains a great deal of moisture and organic tissue, bonds are more difficult to form. When dentists prepare a tooth for bonding a filling, they use an acid to demineralize the surface of the cavity. This process removes the mineral

Review Articles

S. Backus et al. of the University of Michigan have published a review article in *Review of Scientific Instruments* **69** (3) (1998) entitled, "High Power Ultrafast Lasers." The article reviews the development of high peak-power ultrafast lasers (from terawatt to over a petawatt) and the design issues which determine the performance of these systems.

A. Othonos of the University of Cypress has published a review article in *Journal of Applied Physics* 83 (4) (1998) entitled, "Probing Ultrafast Carrier and Phonon Dynamics in Semiconductors." While reviewing the status of ultrafast carrier and phonon dynamics in semiconductors, this article discusses experimental techniques such as excite-andprobe transmission, time-resolved up-conversion luminescence, and pump-probe Raman scattering. material from the dentin, leaving a framework of collagen tissue and tiny tubules that run from the center of the tooth toward the surface. When a plastic-based filling is placed in the cavity it is in a liquid form, allowing the material to penetrate the exposed framework before hardening. The researchers studied how quickly and efficiently different acids work in the demineralization process and how various materials used to form bonds withstand the constant stress placed on teeth. By using the microscopes to observe the bonding process, the researchers are better able to explain why bonds succeed and fail.

Using the AFM, which provides a high resolution image in any environment, including wet or acid solutions, the researchers were able to see that in the bonding process the liquid filling penetrates moist dentin more easily than drier dentin.

While metal alloys create a more durable restoration than newer plasticbased and ceramic materials used for filling tooth cavities, the ability to match the color of these materials to the natural color of a tooth makes the newer materials much more popular choices.

Sally J. Marshall, professor of restorative dentistry, said, "We want to make the polymer and ceramic materials as strong and long-lasting as metal."

Dual-Color Cross-Correlation Spectroscopy Enables Real-Time Analyses of Enzyme Kinetics

A research team at the Max Planck Institute for Biophysical Chemistry in Göttingen, Germany, headed by the Nobel laureate Manfred Eigen, published two articles in PNAS (Proceedings of the National Academy of Sciences) introducing dual-color fluorescence cross-correlation spectroscopy as a new method for an ultra-sensitive characterization of enzyme activity and demonstrating its potential for high throughput screening. In recent years, fluorescence correlation spectroscopy (FCS) has become an attractive analytical tool for the investigation of biomolecular processes. Due to the interplay of modern confocal optics, new dyes as efficient fluorescent probes, sensitive photodetectors, and fast data processing, FCS allows the observation of the dynamics of single molecules in real time while they pass an open volume element of about one femtoliter. This method was invented and worked out by groups at Cornell University, Ithaca, at the Karolinska Institute, Stockholm, and at the Max Planck Institute, Göttingen. It has now found its way into several laboratories and

companies worldwide as a tool for basic research as well as for industrial applications such as drug screening.

Dual-color fluorescence cross-correlation spectroscopy (dual-color FCS) was proposed by Rudolf Rigler, Stockholm and Manfred Eigen, Göttingen, in the early 1990s, and has recently been implemented by Eigen's group in Göttingen. In contrast to conventional single-color FCS, the new method uses two perfectly superimposed laser foci, and detects correlated fluctuations that arise from single molecules carrying two spectrally distinguishable fluorescent labels. It allows precise and highly specific detection of molecules on a large unspecific fluorescent background; moreover, the method is fully compatible with biological environments. Two important applications were addressed by the scientists: real-time measurements of enzyme kinetics were successfully performed, and the suitability of dual-color FCS for high throughput screening has been demonstrated.

Kinetics of biomolecular interactions like nucleic acid hybridization or protein aggregation were previously investigated by Eigen and co-workers applying dualcolor FCS. Now, it has been extended to real-time analyses of enzyme kinetics. As a first application, the kinetic parameters of the cleavage reaction of a double-helix DNA catalyzed by a specific endonucleolytic enzyme were determined. Furthermore, catalytic activity down to enzyme concentrations of one picomole per liter was detected with high reproducibility. This is at least one order of magnitude more sensitive than other homogeneous endonucleolytic assays. According to the scientists, this technique can, in principle, be used for any reaction in which a (covalent or noncovalent) linkage between two different fluorophores is either broken or formed. This includes other hydrolytic enzymes like proteases, esterases, or glycolyases, as well as ligating enzymes.

High-throughput screening with dualcolor FCS was demonstrated in the second article; this combination is termed RAPID FCS (rapid assay processing by integration of dual-color fluorescence cross-correlation spectroscopy). While conventional FCS identifies molecules by their diffusion properties, requiring a considerable amount of analysis time, dual-color FCS simply counts doubly labeled molecules; therefore, analysis times for simple yes-orno decisions are much shorter and data evaluation is faster. As reported by the authors, analysis times of one second per sample and less were achieved with endonucleolytic assays; sample volumes could even be reduced to submicroliters without decreasing the signal strength. Therefore, according to the scientists, this technology is an ideal tool for ultrahigh throughput screening when combined with nanotechnology; and it will gain access to progressive selection strategies in evolutionary biotechnology, in which rare and specific binding or catalytic properties have to be screened in large numbers of samples.

Compaction of Granular Materials Can Produce Irregular Regions of Highly Concentrated Stress

The grains of granular materials can abruptly concentrate stress by assembling themselves into brief but dramatic chains shaped much like jagged lightning bolts as they randomly push against each other within tightly compacted spaces, according to Robert Behringer, a Duke University physicist who has devised ways to observe this behavior in the laboratory. Behringer reported at the American Physical Society's Meeting in Los Angeles in March that this unexpected way granular particles build up and then dissipate collective tension may help explain why passive containers such as coal-filled hoppers sometimes selfdestruct for no apparent reason. These stress fluctuations may also contribute to the behavior of other natural phenomena.

Behringer said that when the particles are pressed as compactly as they would be in a confined space, "the grains suddenly wake up and they stop slipping. They begin to roll, as if they are clever enough to rotate as well as move." While grains would dissipate energy if they just slid among themselves, these rotating particles store up the energy of motion instead, he said. A network of such tense grains can form jagged stress chains that stand out among the larger sea of particles. As the particles continue to interact, these chains then dissipate suddenly, only to be replaced by other chains in other locations.

"These stress chains act to carry force from one place to another place, and they can come and go with very great speed," he said. "Therefore, they have the potential to do damage, because they are localized and because they come and go without a lot of warning."

The device Behringer used in his experiment consists of a wheel in the center of a shallow circular container, which is filled with small, flexible, translucent cylinders. These experimental "granular materials" are made of a compressible plastic that bends light as it undergoes tension. The tiny cylinders also were marked with a line on one end, which allowed scientists to establish that some rotated instead of simply sliding.

When the device is turned on, the wheel slowly moves and transmits its energy to the surrounding "grains," which respond by bumping into each other and compacting. Their optical properties then change whenever and wherever they build up stresses.

Experimenters can see the resulting flashing stress chain patterns by illuminating the apparatus from underneath and observing it through cross-polarizing filters. Those filters screen out any light whose optical properties have not been stress-distorted. Using a calibration device, investigators also can use it to painstakingly measure the forces on individual grains, as well as following how the grains move and orient themselves within their essentially two-dimensional world.

Behringer acknowledged that most granular particles exist in three-dimensional space, and that most are made of hard substances rather than squeezable ones. But he said separate 3-D experiments with hard glass beads have produced some analogous results. Some of the 3-D work was described in the October 7, 1996, issue of *Physical Review Letters*.

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60-Atom Cluster of He-Four Isotope Found to be a Superfluid

The basic understanding of superfluidity has challenged theoretical physicists ever since its discovery in helium 60 years ago. One fundamental question has been the minimum number of atoms needed for superfluidity. The answer to this question was recently provided by spectroscopic experiments performed by physicist Andrej Vilesov and PhD student Slava Grebenev, both natives of Russia, working together with Peter Toennies, scientific member and director at the Max-Planck-Institute for Fluid Dynamics in Göttingen. They discovered, as reported in the March 27 issue of *Science*, that a cluster having just 60 atoms of a heliumfour isotope is already a superfluid.

The researchers reported on a new experiment which strongly suggests that the ability of molecules to rotate freely is a result of the superfluidity of the droplets. To demonstrate this they compared the spectrum in a superfluid with that in a normal fluid. For the latter they used droplets consisting of the rare isotope helium-three, which being a Fermion system only becomes a superflu-

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id at much lower temperatures of 3 millidegrees K and therefore at the helium-three droplet temperatures of 0.10 K behaves as an ordinary classical fluid. For these experiments they chose the simple linear molecule OCS (oxygen carbon sulfide). Inside helium-four droplets a very sharp rotational line spectrum was observed whereas in helium-three only a single broad peak was found indicating that, as expected for an ordinary fluid, the rotational motion is strongly impaired by collisions. This experiment provided the key evidence that the phenomenon of free molecular rotations in liquid helium is a new microscopic manifestation of superfluidity which they call "molecular superfluidity."

To determine the critical number of helium-four atoms for molecular superfluidity they then added to the pure heliumthree droplets well-defined numbers of helium-four atoms. From other experiments and theory they could precisely determine the actual number of atoms picked up. Moreover they had evidence that the helium-four atoms aggregate around the OCS molecule. On the addition of about 60 helium-four atoms the sharp spectral lines reappeared, indicating the return to free rotational motion. According to the researchers, 60 atoms are sufficient to build up a cage consisting of a double layer of helium-four atoms which surround the molecule.



Boatner Elected to Mexican Academy of Sciences

Lynn A. Boatner of Oak Ridge National Laboratory has been elected as a corresponding member of the Aca-

demic Mexicana de Ciencias (Mexican Academy of Sciences). As a corresponding member of the Academy, Boatner, a section head in the Solid State Division, will collaborate with Mexican scientists for the further development of science in Mexico.

Boatner, a Lockheed Martin corporate fellow, has a BS and MS degree in physics and mathematics from Texas Tech University and a PhD degree in physics and mathematics from Vanderbilt University. He is a fellow of the American Physical Society, the American Association for the Advancement of Science, and ASM International. He received a Federal Laboratory Consortium Award for Excellence in Technology Transfer for 1997 and was an R&D 100 Award recipient in 1982, 1985, and 1996. He is a member of the Materials Research Society, the National Institute of Ceramic Engineers, the American Ceramic Society, and the American Welding Society.