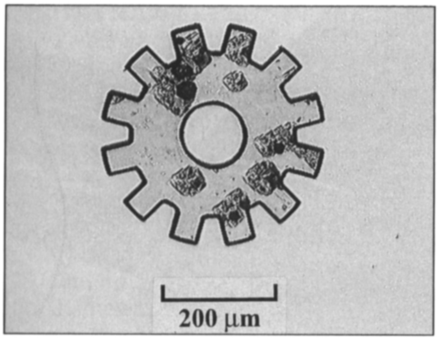
Microgear Produced from Single-Crystal Diamond

John D. Hunn of Oak Ridge National Laboratory and Paul Christensen, president of Potomac Photonics, collaborated to produce freestanding single-crystal diamond microstructures by combining a technique for the "lift-off" of thin diamond films with a technique for engraving diamonds with a focused eximer laser. They have made a twelve-tooth diamond gear that is ~400 microns in diameter and ~13 microns thick (see Figure), as well as other shapes.



Freestanding 12-μm-thick gear made from a nearly single-crystal homoepitaxial CVD diamond film.

Their simple method for producing miniature parts makes diamond a reasonable alternative to the more commonly used silicon in devices for microrobotic and micromechanical applications. Because of diamond's exceptional physical properties, diamond microstructures can operate at higher temperatures, perform in harsher environments, and last much longer than similar devices of silicon.

The lift-off technique, developed by Hunn and fellow researchers at Oak Ridge, uses ion implantation to create a buried damaged layer in a polished bulk diamond crystal, then removes that damaged layer by selective etching, thus lifting a thin sheet of diamond from the surface. The method for laser machining of diamonds, developed by Christensen at Potomac, involves using a pulsed ultraviolet laser emission shaped and collimated by beam-delivery optics and focused onto a work surface with a uv-transmitting microscope objective. The workpiece is mounted on an *xy*-motion stage whose velocity is controlled by CAD/CAM software. An article in the September 24 issue of *Science* describes how Christensen had succeeded in carving a relief prototype of a microgear into the flat surface of a diamond, but had been unable to remove the structure from the diamond. After reading this, Hunn called Christensen and suggested the collaboration.

Their freestanding diamond microgear was fabricated from a single-crystal homoepitaxial diamond film. At Oak Ridge, the substrate was implanted at -196°C with 5 MeV oxygen ions to a dose of 10^{16} ions/cm^2 to produce a buried damage layer ~2 μm below the surface. A film of homoepitaxial diamond, ~11 μm thick, was grown on the implanted substrate by hot filiment CVD from a methane-hydrogen gas mixture.

The sample was then sent to Potomac, where a 400-μm-diameter gear with one dozen 50 μm square teeth was then patterned in the CVD film, using a wave-

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guide ArF laser producing 50 μJ pulses of 80 ns duration at pulse repetition rates extending to 2 kHz. The *xy* motion stage was programmed to step out a pattern with a 15-μm-diameter focused laser spot, each laser pulse removing approximately one vertical micron of diamond from the irradiated area. After 20 passes, an ~20-μm-deep trench outlining the pattern was formed. After patterning, the sample was returned to Oak Ridge National Laboratory, where it was heated at 565°C under flowing oxygen for 90 minutes. This resulted in selective etching of the buried damage layer created by the initial implantation step, thereby freeing the gear from the substrate.

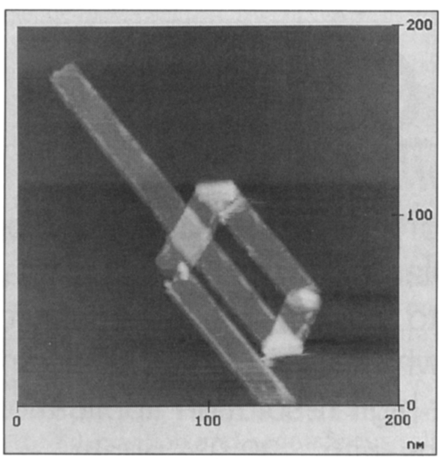
This method has been used to produce other 13-μm-thick diamond shapes, including a five-pointed star measuring 280 μm, tip to tip, a small circle with a 90 μm diameter, and a capital-H-shaped cantilever structure measuring 122 μm wide by 155 μm high, with 20-μm-wide legs. CAD/CAM software and the direct-write approach to laser machining allows almost any shape to be generated in a diamond surface.

In Pursuit of "Graphite Origami"

At NEC's Fundamental Research Laboratories located in Tsukuba, Japan, researchers have been studying fullerenes, carbon nanotubes, and the manipulation of carbon sheets. As a part of this work, T. Ebbesen, H. Hiura, and other members of their team–after discovering the mass production of nanotubes in 1992–realized that it should be possible to tailor the properties of graphite sheets by controlling their geometry. Work along this line led to an article in the January 1994 *Nature*, titled "Role of sp^3 Defect Structures in Graphite and Carbon Nanotubes." Subsequently, the team has come up with the concept of "carbon origami," achieved by passing the tip of an atomic force microscope (AFM) over a carbon sheet in a specific way. When carbon sheets fold along defect structures, they do so in a way that strongly reminds one of the Japanese paper-folding technique known as origami.

As reported in the previously mentioned paper, the NEC researchers found that the folding and tearing of graphite sheets follow well-defined patterns which seem to be governed by the formation of sp^3-like line defects in the sp^2 graphite network. Studies with the AFM and a scanning tunneling microscope revealed that these folds and tears occur preferentially along the symmetry axis of graphite, and that ripples are observed in the curved portions of the folds. Ripples were also seen in deformed carbon nanotubes. They lie along the directions for which sp^3-like line defects can form most easily to relieve strain. *Ab initio* molecular-orbital calculations indicated that the ripples stabilize the π-electronic energy in the bent structures, with the total energy of balance being determined by the amount of nuclear repulsion. These results provided insight into the geometries that graphite structures will preferentially accommodate, and the properties that might emerge.

In the same paper, the researchers speculated that it might be possible to fabricate idealized wires made of a carbon sheet with conductive zones separated by ripples or bends. These sp^3-like line defects would act as nonconducting barriers separating structures in the overall carbon network; further theoretical and experimental studies should reveal what to expect and what is possible in such cases. Ebbesen (on leave for one year at the NEC Research Institute, Princeton, New Jersey) and Hiura are now working toward this goal, hoping to make use of these inherent properties of graphite which allow for the formation of three-dimensional structures.



Graphite sheet 0.34 nm thick, 20 nm wide, and about 400 nm long, that folded due to the friction of an atomic force microscope tip.

The Figure illustrates the kinds of shapes that spontaneously form on the surface of highly ordered pyrolitic graphite due to the friction of an AFM tip (this effect was serendipitously found while studying carbon sheets and other structures with an AFM). The shapes are reminiscent of those produced by origami. The angles of the object shown in the photo are only those predicted by the rules established by Ebbesen and Hiura.

The researchers say that by changing the direction of folding, they can pile carbon sheets into structures with a tilt of 30, 60, 90, and 120 degrees. In this way, single-atom-thick sheets can be folded into triangular, square, and trapezoidal shapes. In the Figure, the object created by such folding was originally a single thin strip of graphite only 0.34 nm thick, 20 nm wide, and about 400 nm long. Ebbesen points out that the ribbon passes over itself, following the contour of the underlying ribbon very precisely, indicating again the out-of-plane flexibility of the graphite sheet. This has also been observed in other experiments. Although these structures were formed accidentally, it should be possible with the present level of technology to build them in planned and systematic ways, said Ebbesen. This and related work will be discussed in an upcoming paper and also presented at the 1994 MRS Fall Meeting, Symposium G.

F.S. MYERS

High-Brightness, 512 nm Green LED Announced

Sony Corporation has announced the development of a 512-nm-wavelength, green light-emitting diode (LED) offering a brightness of 4 candela (cd). The LED consists of a layer of ZnCdSe sandwiched between two cladding layers of ZnMgSSe in a double-heterostructure.

This LED emits (pure) green light (between blue-green and yellow-green), operating at a wavelength of 512 nm. The wavelength of an LED depends on the materials used to construct the semiconductor and on the crystal-growth technique. The LED's success resulted from the development of a cladding layer of ZnMgSSe and crystal-growth technology. The 512 nm wavelength lies almost at the center of the green bandwidth, resulting in "true green" light, which can be effectively mixed with primary-colored lights to generate a wide range of hues. At 4 cd, the LED's brightness is the highest yet recorded for green light.

The prototype LED consists of an emission layer of ZnCdSe sandwiched between two cladding layers of ZnCdSSe, which confine the holes and electrons, all on a GaAs base. The crystalline structures are grown using molecular beam epitaxy. The ZnMgSSe cladding layer yields a large energy bandgap which, in turn, allows for more efficient conversion of the current injected into the emission layer, into light. From a 20-mA current, light output of 2.1 milliwatts can be obtained. The resulting external quantum efficiency (the number of photons emitted compared to the number of electrons injected) of 4.3% is roughly four times greater than that of the conventional green bandwidth LEDs; the voltage required to generate a current of 20 mA is 3.9 v. At 4 cd, the on-axis luminous intensity of the LED, with a 12-degree cone-viewing angle (the full width at half maximum angle of beam divergence), is higher than previously recorded.

The purity of the emission layer restricts the spectrum of the wavelength to 10 nm (+/− 512 nm), resulting in very pure green light. Pure colors are a key factor in the development of full-color displays, since they allow effective blending of primary-

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color lights to achieve a broader spectrum of hues. Conventional high-brightness LEDs, such as those used in the level meters of stereo systems, or to display text on store displays, emit light in a range of four different colors using different operating wavelengths: red (630–700 nm), orange (610 nm), yellow (570–590 nm), and yellow-green (655, 566 nm). When they are used in combination with other high-brightness, short-wavelength LEDs (which emit blue or green light), all primary colors can be generated. This would open the way for large-scale full-color flat panel displays based only on LEDs. Research into short-wavelength, high-brightness LEDs continues in companies and research centers around the world and recent announcements include the development by Nichia Kagaku of LEDs emitting blue light (450 nm wavelength) and blue-green light (510 nm wavelength).

Sony will now concentrate its research on developing an LED that offers greater brightness and a longer emission time. Yoshimumi Mori is in charge of the green LED research at Sony's Research Center, Tokyo.

F.S. Myers

SVG to Develop Polymer Coating Technology for Flat Panel Displays

The Silicon Valley Group Inc. (SVG), San Jose, California, has been awarded a contract to design and develop a completely automated large-area polymer coating system for manufacturing flat panel displays. FAS Technologies will be a partner with SVG in the project. The U.S. Display Consortium (USDC), a government-industry partnership created to develop U.S. flat panel displays, awarded the $2.5 million contract to SVG with a completion date of November 1995.

Polymer coatings are critical to the development of cost-effective flat panel displays and are used in photoresists for the lithography process, in polyimide alignment layers, and in color filter layers. As the size of glass substrates used in manufacturing flat panel displays enlarges to 500 mm and more, maintaining deposition uniformity and polymer coating quality becomes more difficult. This project aims to address these difficulties and develop a technology that can be brought to the marketplace quickly.

SVG will oversee and integrate the development process and will provide manufacturing equipment such as ovens, robotic handling equipment, and packaging. FAS Technologies will provide the coater modules used to apply polymers to flat panel displays. The goal of FAS is to fashion a system that, with the use of spin coating, produces defect-free coatings, operating at greater throughput rates and utilizing polymer materials more efficiently.

Macromolecular Precursors Used to Form Ordered Multilayered Thin Films

Multilayered three-dimensional molecular structures have been formed by stacking microscopic sheets of synthetic silicate clay alternately with a positively charged polymer. Gregory S. Ferguson, an assistant professor at Lehigh University, and PhD student Elaine R. Kleinfeld described their accomplishments in a July 15 *Science* article titled "Stepwise Formation of Multilayered Nanostructural Films from Macromolecular Precursors." The article detailed their method for the stepwise preparation of ordered multilayered films from ultrathin (~1 nm) layers of organic and inorganic molecules.

In their approach, polyelectrolyte polydiallyldimethylammonium chloride (PDDA) and exfoliated sheets of synthetic hectorite, a mica-type layered silicate, are sequentially stacked. The hectorite is dispersed in water, where the mineral exfoliates into crystalline, quasi-two-dimensional sheets that are 0.96 nm thick and about 25 to 35 nm in diameter. The sheets, which bear a negative charge are, in turn, treated with the positively charged cationic polymer (PDDA). The opposing electrical charges cause the two surfaces to adhere to each other. Alternating layers of clay and polymer are added to the structure; the adsorption cycle takes about one minute. The growth of the structure's thickness is a linear function of the number of adsorption cycles.

In the past, both Langmuir-Blodgett (LB) deposition and spontaneous self-assembly (SA) have been used to form multilayered systems. LB multilayers are mechanically unstable, however, because they are held together primarily by van der Waals forces; while in SA systems, adsorption of multilayers displaying structural order has proven difficult. In addition, problems may occur when defects create unreactive patches that eventually result in uneven multilayer growth and a decay in structural order.

The new layering technique employing hectorite sheets also exhibits some packing imperfections, such as open space at their boundaries. However, these imperfections are "healed" because of the large lateral extent of the sheets, which allows these areas to be covered where the underlying layer is incomplete.

The multilayering method developed by Ferguson and Kleinfeld offers alternative strategies to scientists interested in building ordered organic-inorganic thin films, with systematic control over both structure and thickness. The technique can also be adapted to other systems using substrates, or interfaces, including semiconductors, electrical insulators, and electrodes.

D. Seidman Receives Max Planck Research Prize

David N. Seidman, professor of materials science and engineering at Northwestern University, has been awarded a Max Planck Research Prize by the Alexander von Humboldt Foundation and the Max Planck Research Society. The prize consists of 100,000 German marks for research over a three-year period. Seidman was cited for outstanding lifetime achievement in materials science.

Jointly nominated with the late Prof. Peter Haasen (see August 1994 *MRS Bulletin*, p. 7) of the Institute for Metal Physics, University of Göttingen, Seidman is currently researching grain boundaries and heterophase interfaces on an atomic scale via atom probe field-ion microscope experiments and computer simulations.

Seidman has been a professor at Northwestern University since 1985. Before that, he was a professor of materials science at Cornell University. He has published extensively on point defects and radiation damage in metals, field-ion and atom-probe microscopies, and interfacial phenomena. He received a PhD degree from the University of Illinois at Champaign-Urbana.

The recipient of the Robert Lansing Hardy Gold Medal of TMS-AIME and two Guggenheim Memorial Foundation Fellowships, Seidman is also a Fellow of the American Physical Society and an Alexander von Humboldt Senior Fellow at the University of Göttingen. □

SBIR Update

PSI Technologies, Andover, Massachusetts, has received a Phase I SBIR grant from the U.S. Department of Energy to develop a low-cost process to produce ultrafine silicon carbide (SiC) powders, which could be used in manufacturing high-temperature tolerant, high-strength ceramic parts. PSI Technologies will use a high-temperature, continuous aerosol method and a customized reactor to produce nanoscale SiC powders with a 10 to 100 nanometer grain size, substantially smaller than the conventional 1,000-nm-size grains currently available commercially.

American Superconductor Corporation, Westborough, Massachusetts, will use a $600,000 Phase II SBIR award from the U.S. Department of Energy to enhance its processes for fabricating high-temperature superconductor wire for commercial applications.

Linus Pauling, the only person to win two Nobel Prizes by himself, died August 19, 1994. Few scientists contributed to as many fields as Pauling, whose interests ranged from chemistry to molecular biology, nuclear weapons, and medicine. During a plenary address at the 1989 MRS Spring Meeting, he explained his work on the chemical bonding in metals and also offered explanations related to quasicrystals, high-temperature superconductors, and cold fusion. He argued that the heat attributed to fusion in the experiments going on at the time might actually occur when bonds form between deuterium and the metal atoms in palladium—then break. Pauling is, of course, also well-known for his advocacy of vitamin C and his fight to ban nuclear testing in the atmosphere and to ban nuclear weapons. The British magazine *New Scientist* ranked him among the 20 most influential scientists in history.

Pauling didn't receive his high school diploma until after he had won the Nobel Prize for chemistry in 1954. He won the Nobel Peace Prize eight years later. Pauling left high school without his diploma when he wasn't allowed to take, in the same semester, two American history courses that he needed to graduate. Knowing he had enough credits to be admitted to Oregon State University, Pauling instead took college algebra and trigonometry. After receiving his bachelor's degree in chemical engineering, he went on to earn a doctorate in chemistry and mathematical physics from the California Institute of Technology. He then spent two years in Europe on a Guggenheim Fellowship and returned in 1927 to Caltech, where he stayed for the next 36 years. He finished his academic career at Stanford University, where he was a professor from 1969 to 1973. Pauling continued to work out of the Linus Pauling Institute of Science and Medicine in Palo Alto, California.

Pauling published more than 650 scientific papers, about 200 articles on social and political questions, and many books. *The Nature of the Chemical Bond* by Linus Pauling is one of the most cited scientific books of the 20th century.

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