Electron Microscopy Shows How Carbon Bonds in Diamond Films

Workers at Cornell University have combined the high spatial resolution of a scanning transmission electron microscope (STEM) with electron-energy-loss spectroscopy (EELS) to probe the atomic structure of carbon and build a two-dimensional chemical map.

Done principally by doctoral students in Cornell's Materials Science Center for their dissertations, the work shows how the interaction between a carbon atom and a silicon wafer determines whether the carbon will become diamond or graphite within a distance of one nanometer. Physics student David Muller said, "This technique tells us what the atom is, what the bonds are made of, and where it is."

The researchers combined STEM and EELS technologies to study the first atomic layer on a silicon wafer and its interface with diamond thin film. Muller; Yujiun Tzou, graduate student in materials science and engineering; Rishi Raj, professor of materials science and engineering; and John Silcox, professor of applied and engineering physics and director of the Materials Science Center, reported the work in the December 23, 1993 issue of *Nature.*

"We've had the technology for about seven years," Silcox said, "but we've had to make upgrades to achieve the spatial resolution we have. This is the only machine that is stable enough to form a fully two-dimensional bonding map at such a high spatial resolution. And since the data goes directly into a computer, you can look at it later."

Unlike a scanning tunneling microscope, the STEM/EELS technology looks at information beneath the surface. Raj said the technology could allow materials researchers to view how an atom decides what kind of bonds to form. For example, the first carbon atom to be deposited on the silicon surface has a choice. Raj said, "You don't know whether it will form graphite or diamond. What happens in that first interlayer determines whether or not the diamond film grows on a single crystal." If the carbon forms double bonds, it becomes graphite. If it forms single bonds, it becomes diamond.

Raj and Tzou recognized that visualization of the atomic arrangement of atoms at the silicon/diamond interface was essential in guiding the growth process. Previous experiments in high-resolution microscopy were done at the Max-Planck- Institut in Stuttgart in 1992, where Raj and Tzou spent a year sponsored by the Alexander von Humboldt Foundation. Their work continued in collaboration with Muller and Silcox.

Muller and Silcox are examining the grain boundaries of nickel aluminide, a high-temperature intermetallic material with potential for use in jet engine turbines. The grain boundaries of this material are brittle, breaking easily at the points where the crystals meet. But when even a small amount of boron is added, the grain boundaries behave much more like the bulk material—durable rather than brittle. Scientists don't know why boron has such a potent effect on the properties of the grain boundary, but they hope that the high-resolution of EELS can answer the question.

Water Packing Discovered at Electrode Surface

Researchers have been surprised to find that water molecules crowd together at up to twice their normal density at the surface of a metal electrode. Michael Toney, a physicist at IBM's Almaden Research Center, said, "Experts have long believed that water molecules form several distinct layers around the surface of immersed metal electrodes, and some had suggested that the first, innermost layer would have unusual properties. But no one would have predicted what we found there: a high-density packing of up to double water's liquid density as the voltage on the electrode is increased." Toney and colleagues reported their results in the March 31 issue of *Nature.*

Toney said the finding changes the atomistic picture of how surface reactions occur. "The current molecular models of water used in computer simulations and statistical calculations are clearly inadequate for predicting such properties of electrode/electrolyte interfaces as corrosion and reactions rates, viscosity, and acidity."

The arrangement of water near electrodes has been of theoretical interest since the 1940s, but experimental data has been sparse and often inconclusive or contradictory, partly due to the extreme difficulty of probing submerged interfaces with precision. In the new study, the scientists aimed x-ray beams at the surface of a silver crystal that was immersed in water within a specially designed cell, then analyzed the pattern of scattered x-rays, which detailed the orientations of the water molecules. The water molecules were observed to organize themselves into discrete layers near such metal surfaces. Moreover, the molecules rotated their orientation from oxygen-up to oxygen-down, as predicted, when the voltage applied to the metal was changed from negative to positive.

But not predicted was a crowding together of the molecules in the first layer (next to the electrode) to a maximum of about twice liquid density as the voltage was raised to +0.52 V. This behavior implies that the hydrogen-bonding network in the first layer of water molecules is disrupted and that the properties of this surface water are likely to be very different from those in the bulk. While this density is surprisingly high, it is not physically unrealistic, since it is comparable to the densities of water molecules surrounding dissolved (solvated) ions, which also create local electric fields of comparable strength.

Co-worker Joseph G. Gordon said, "This suggests that the electric field plays an important role in the water molecules' increasing density at the electrode surface." Toney added, "We expect that these findings will also apply to other water-metal interfaces where there is no specific chemical interaction between the water and metal."

D. Wolf Receives Max Planck Research Award

Dieter Wolf, a physicist with Argonne National Laboratory, has received the Max Planck Research Award, bestowed by the Max Planck Society and the Alexander von Humboldt Foundation. The award, consisting of $65,000 over three years, is named for the German physicist who received a Nobel Prize in 1918 for discoveries in quantum theory.

Wolf shares the award with collaborator H. Gleiter of the University of Saarbrücken. The two study the physical structure of nanophase materials, which can be used to create ceramics and metals with new properties, including high strength.

Wolf, leader of the interface materials group at Argonne's Materials Science Division, was also honored in 1990 with the University of Chicago Distinguished Performance Award for important contributions to materials research. He has written more than 130 technical publications, and has been author or co-author of two books and several book chapters. Wolf received his BS degree in physics from the University of Stuttgart and his PhD degree in physics from the Max-Planck-Institut.

Low-Cost, High-Temperature Glue Developed

University of Illinois researchers say their new glue is resistant to high temperatures, cheaper and faster to make, and uses recyclable ingredients. The glue, a cross-linked copolymer, or thermoset, is stable in air to 350°C, said James Economy, Materials Science and Engineering Department head. "We surmise the adhesive will last many thousands of hours under stress at 200°C."

Cross-linked copolymer molecules form chains that connect anywhere along their lengths, not just at their ends. The material was previously discounted as a glue because acetic acid would be created during its production, weakening the glue. Economy, working with graduate student Dan Frich and postdoctoral researcher Konstantin Goranov, found that the glue could be used as a film to impregnate materials such as carbon or glass fabric, letting acetic acid molecules escape without creating detrimental pores when each layer of impregnated fibers was heated.

Economy said that fiber-and-resin compounds had "plenty of avenues for the vapors to escape even when layers were cooked together in a single baking. Thus, while high-temperature composites take 30 to 40 hours to bake, we could make ours in four or five hours."

The glue is fairly easy to make through the reaction of relatively inexpensive aromatic carboxylic acids with aromatic acetates. Unlike the ingredients of glues such as epoxies or phenolics, the materials used in the new adhesive should be recyclable, according to preliminary test results. While moisture absorption is a major factor in the long-term deterioration of epoxies, the thermoset glue absorbs 100 times less moisture than conventional commercial adhesives.

Frank L. Galeener, a professor of physics at Colorado State University, died last June of esophageal cancer. Galeener earned his SB and SM degrees in physics at Massachusetts Institute of Technoloyg (1958 and 1962, respectively) and was a staff member at the MIT Lincoln Laboratories (1959–61) and at the National Magnet Laboratory (1961–64). After receiving his PhD in physics from Purdue University in 1970, he became a founding member of the research staff at the Xerox Palo Alto Research Center. In 1977 he became a principal scientist at Xerox PARC. During and just after his tenure there, he held visiting positions at the University of Oxford, the University of Cambridge and the University of Paris VI. He moved to Colorado State University in 1987. Although his PhD studies were in solid-state theory, Frank became a consummate experimentalist, developing a state-of-the-art Raman spectroscopy laboratory at Xerox PARC that later formed the nucleus of a diversified glass physics laboratory at Colorado State University. Early in his career, his interests were centered on the atomic and vibrational structures of amorphous solids and thin films. Among his most important achievements were his demonstration of the splitting of transverse and longitudinal optical modes in glasses, the determination of selection rules for Raman and infrared absorption in glasses, the study of intermediate-range structural order in glasses and the identification of planar three-ring structures in glassy SiO_2. In recent years he shifted into the field of point defects and radiation-damage mechanisms in silica glass. He and his collaborators discovered that the defect production efficiency had unanticipated dependence on both the fictive temperature of the glass and the energy of the defect-inducing x-ray photons. Galeener was guest editor of the 1987 June/August *MRS Bulletin* on glasses.

Roy J. Plunkett, discoverer of DuPont Teflon–polytetrafluoroethylene (PTFE) resin–died May 12 after a brief illness. He was 83. Plunkett was a young research chemist at the DuPont Jackson Laboratory in Deepwater, NJ, when he made his discovery. On April 6, 1938, he was investigating the results of a failed experiment involving refrigeration gases when he discovered a white, waxy substance. The material proved inert to virtually all chemicals and the most slippery material known. In addition to his work with Teflon, Plunkett is named as the inventor or co-inventor of a dozen products patented by DuPont in the United States, and is responsible for many foreign patent filings on DuPont materials. His career with DuPont spanned 39 years. When he retired in 1975, he was production manager in the former Organic Chemicals Department. His contributions have been recognized by numerous scientific, academic and civic communities. He was inducted into the Plastics Hall of Fame, Inventors' Hall of Fame, and the Engineering and Science Hall of Fame. Dr. Plunkett received his BA degree in chemistry from Manchester College. He was awarded his Master's degree and his PhD from Ohio State University. DuPont Fluoropolymers conducts two awards programs named for him. The "Plunkett Awards for Innovation with DuPont Teflon," a worldwide competition conducted among manufacturers and end users, recognizes the most innovative commercial applications using DuPont fluoropolymer resins. The "DuPont Plunkett Student Awards for Innovation with Teflon," aimed at U.S. college and university students, encourages new developments in fluoropolymers and carries prizes and scholarships worth over $40,000.

A. Pines Receives Tech Transfer Award

Lawrence Berkeley Laboratory (LBL) researcher Alex Pines of the Materials Science Division received the 1994 Federal Laboratory Consortium (FLC) Award for Excellence in Technology Transfer.

Pines was recognized for the conception, transfer to industry, and implementation-in-place of double rotation solid-state nuclear magnetic resonance (DOR) probe technology for high-resolution instrumentation. The probe allows researchers working with solids to obtain nuclear magnetic resonance spectra of much higher resolution than previously were possible. The technology is used to study the structure and function of critically important catalysts in the petrochemical, pharmaceutical, and microelectronics industries. Chemagnetics, Doty Scientific, Inc., and Bruker Instruments have licensed and now market the DOR probe. Shell Oil Company has also adopted use of the technology into their catalysis research program.

Schwartz Receives TMS Leadership Award

Lyle H. Schwartz, director of the Materials Science and Engineering Laboratory at the National Institute of Standards and Technology received the 1994 Leadership Award of The Minerals, Metals & Materials Society during the TMS Annual Meeting in San Francisco. This award is presented to the representative of an industrial, academic, governmental, or technical organization who demonstrates outstanding individual leadership in metallurgy and materials.

Schwartz received his BS in science engineering and PhD in materials science from Northwestern University. He has published more than 90 papers on phase transitions in iron alloys, applications of Mossbauer spectroscopy, x-ray and neutron diffraction, characterization of catalysts, and policy issues related to materials science and engineering. He is co-author of two textbooks on x-ray diffraction.

Schwartz chaired the panel on international cooperation and competition in materials science and engineering of the National Research Council's MS&E study, Materials Science and Engineering for the 1990s, published in 1989. In 1990, he was appointed chairman of the intergovernmental Committee on Materials Science and Technology.

Cottrell Awards Fund Innovative Research and Teaching by Young Scientists

Some 17 young faculty members are the first to receive $50,000 Cottrell Scholars awards. Named for the founder of Research Corporation, the awards for young faculty scientists are intended to combat the split in academic science between "teachers" and "researchers" by funding independently proposed programs that help recipients develop their skills in both areas. The awards are fully peer-reviewed by the scientific community, support a variety of academic activities and can be flexibly spent by recipients.

The 1994 recipients are all within three years of a first faculty appointment leading to tenure. They include ten chemists, six physicists and one astronomer, some of them in interdisciplinary research or departments, at 16 different universities in 14 states.

One of the awards went to Heinrich M. Jaeger, assistant professor, Physics Department, University of Chicago, to investigate how granular materials behave when vibrated. (See "What is Shaking in the Sandbox?" by H.M. Jaeger, J.B. Knight, C-h. Liu, and S. R. Nagel in the May 1994 *MRS Bulletin*, p. 25.) Jaeger also teches introductory physics for undergraduates, including many not planning to major in science.

There is no fixed limit on the number of Cottrell Scholars awards. Applications for 1995 are invited from qualified faculty members who accepted tenure-track positions in 1992. The deadline for applications is **October 1, 1994**. Information and application forms are available from: Research Corporation, 101 N. Wilmot Road, Suite 250, Tucson, AZ 85711-3332. Phone (602) 571-1111; fax (602) 571-1119.

NAS/NAE Elect New Members

The National Academy of Sciences and the National Academy of Engineering have announced the election of new members.

Among those elected to NAS membership are the following members of the Materials Research Society: Leroy L. Chang, Hong Kong University of Science and Technology; John P. Hirth, Washington State University, Pullman; and David R. Nelson, Harvard University.

MRS members among those elected to NAE membership are as follows: M. George Craford, Hewlett-Packard;

Stephen H. Davis, Northwestern University; Lambert Ben Freund, Brown University; Doris Kuhlmann-Wilsdorf, University of Virginia; James D. Livingston, Massachusetts Institute of Technology; James E. McGrath, Virginia Polytechnic Institute and State University; Lyle H. Schwartz, National Institute of Standards and Technology.

Albert Sauveur Award Goes to Apelian

Diran Apelian, provost and Howmet Professor of Engineering at Worcester Polytechnic Institute, received the 1994 Albert Sauveur Achievement Award presented by the Philadelphia chapter of ASM International. Established in 1934, the award is presented annually in recognition of dedicated service to the society in pioneering materials science and materials engineering achievements and knowledge.

Apelian is known for his pioneering work in solidification processing, molten metal processing, infiltration of metals, aluminum foundry engineering, plasma disposition, and, most recently, spray casting/forming. He has received several honors for his work and has more than 200 publications to his credit and is co-editor of four books.

Apelian received his BS degree in metallurgical engineering from Drexel University and his doctorate in materials science and engineering from the Massachusetts Institute of Technology. After graduating, he worked at Bethlehem Steel's Homer Research Laboratories before joing Drexel in 1976.

D. Michel Receives Burgess Award

David J. Michel, associate superintendent of the Naval Research Laboratory's (NRL) Material Science and Technology Division, has received ASM International's 1993–94 George Kimball Burgess Memorial Award for "his outstanding contributions to research in elevated temperature deformation mechanisms and for his leadership qualities demonstrated, from 1988 to 1993."

Michel joined NRL in 1972 as a metallurgist. From 1973 to 1987, he headed the High-Temperature Materials Section of the Materials Science and Technology Division and, in 1992, served as acting head of the division's Physical Metallurgy Branch. He was appointed to his current position the same year.

Michel is the author of more than 100 refereed publications and co-editor of three books. He has made 96 technical presentations in the United States and Europe and also holds two patents. He received his BS degree from the University of Missouri and his MS and PhD degrees from the University of Pennsylvania. He has received numerous NRL awards, was honored as Outstanding Young Member of ASM's Washington Chapter in 1977, and was elected a fellow of ASM International in 1989.

Sunlight Concentrated 50,000 Times

Researchers at the National Renewable Energy Laboratory (NREL) have increased terrestrial sunlight 50,000 times with the High-Flux Solar Furnace, which uses an array of mirrors to concentrate the light into an intense, focused beam.

Previously using a reflective secondary concentrator to achieve 21,000 suns, the researchers used a lens-like refractive secondary concentrator for the 50,000-sun level. The refractor was designed by the University of Chicago. Both secondary concentrators are based on non-imaging optics principles developed by Roland Winston and colleagues at the University of Chicago.

Potential applications for such light include production of fullerenes and solar-pumped lasers. These lasers, more energy efficient than traditional lasers, are proposed for space communication systems, space power systems, terrestrial materials processing, and photochemistry. □