Electron Crystallography Provides Accurate Structural Determination of $Ti_{11}Se_4$

In an article published in the July 11 issue of *Nature*, researchers reported the successful use of electron crystallography to determine with 0.02 Å accuracy the structure for the compound $Ti_{11}Se_4$. With single crystals too small to be determined by x-ray crystallography and difficulties using powder diffraction, the compound was analyzed by electron crystallography which, in the past, suffered from complicated quantitative analysis due to dynamical effects. However, a research team from Max-Planck-Institut für Festköperforschung, Stockholm University, and Lund University took a sample size of 10,000 unit cells for electron diffraction and concluded a structure of $Ti_{11}Se_4$ in which the Ti atoms are part of condensed metal octahedra and the Se atoms are surrounded by Ti atoms forming trigonal prisms. The researchers report that every second layer consists of alternating strings of two and four condensed octahedra chains. They further report the crystallographic R-value of 14.7% which represents only a minor influence of dynamical effects on the diffraction amplitudes.

Portable Soil Analyzer Speeds Up Search for Contaminants

Researchers at Los Alamos National Laboratory have developed a method for locating contaminated soils using a laser-based "backpack" system that electronically analyzes contaminants in the soils. The portable unit, co-designed by David Cremers and Monty Ferris of Advanced Chemical Diagnostic and Instrumentation Group, focuses infrared laser pulses on a soil sample and creates a series of sparks that cause the soil to heat rapidly and emit light. The unit then analyzes the spectrum of the emitted light to determine whether the soil is contaminated.

When the laser pulse hits the soil, a spark is produced, vaporizing a small amount of soil and creating a plasma. The unit then collects light from the plasma and passes it through a prism, constructing a spectral signature from which elements can be determined. The spectral results are analyzed electronically. The entire process, lasting 0.1 ns, is called laser-induced breakdown spectroscopy, or LIBS. The procedure repeats in three-second intervals.

"The light is emitted in a specific pattern for each atom and these patterns are analyzed to identify contaminants," said Cremers.

The device, about the size of a cosmetic case which can be worn on a person's back, was developed to rapidly screen soils for a few selected contaminants, such as lead, beryllium, and barium.

Ferris said that the treatment of contaminated soil is expensive and time-consuming, but workers in the field can screen an area for contamination rapidly with the backpack unit and map areas of high contamination. With LIBS, workers can perform measurements in the field and not lose time waiting for laboratory analysis. Volumes of waste are reduced because workers know where to dig rather than having to guess. Also, using the backpack unit, soils can be analyzed in locations that are not accessible with other analytical techniques.

Chinese Academy of Science Inducts Ching Wu Chu

Director of the Texas Center for Superconductivity, Ching Wu (Paul) Chu, a pioneer in high-temperature superconductivity, was inducted into the Chinese Academy of Science (CAS) during the organization's recent conference in Beijing. According to CAS, Chu has made substantial contributions to science in China and throughout world.

"It is indeed a special honor for me to join this small group of distinguished foreign scientists recognized by the most populous country on earth. It is apparent that friendship between two countries can be advanced through scientific cooperation and exchange," Chu said.

In 1987, Chu discovered a material that superconducts at 95.9 K. At this temperature, the superconductor can be cooled inexpensively with liquid nitrogen. His discovery made this technology practical and brought it into the mainstream of science.

"I have no doubt that superconductivity will touch every aspect of our lives that is touched by electricity," Chu said. "I'd like to see commercial projects come out of the center."

CAS began inducting foreign members in 1994. To date, 24 have been selected.

Chu is also a member of the U. S. National Academy of Sciences and the Third World Academy of Sciences.

Stress-Strain Microprobe to Assess Aging of Nuclear Power Plants

Nuclear power plants worldwide are aging, and with age comes deterioration. Current and planned nuclear power plant aging management practices are designed to identify and address this deterioration before it becomes a threat to plant safety. However, many of the practices are elaborate and expensive.

K. Linga Murty, professor of nuclear engineering at North Carolina State University, is developing a method that quickly and efficiently assesses the condition and life expectancy of various metallic components in the power plant. He is adapting a recently developed stress-strain microprobe to test nuclear power plant components. The microprobe can be used to determine the degree of damage that has occurred without causing harm to the structure.

Murty said that, while pressure vessels are made of low alloy steels and start with good ductility and toughness, exposure to radiation causes brittleness. Steel, the material used to make the reactor vessel, becomes more prone to fracture when exposed to radiation. Also, the steel parts are welded together, and the weld zone of the vessel is relatively more sensitive to radiation damage.

The stress-strain microprobe system is based on the automated ball indentation (ABI) technique, which involves lowering a small spherical indentation device into the reactor and gathering data with no harm to the vessel. The device measures force and depth of penetration and feeds this information to a computer, which calculates the amount of stress and strain at the site. With this information, utilities and other reactor operators can determine how much the components—such as pipes, pressure vessels, and turbine blades—have deteriorated and how long they will remain usable.

When the plants reach their design life, officials must decide whether to shut them down, thermally anneal them, which will restore some of the toughness, or replace the vessels.

"The Department of Energy and utility companies are planning thermal annealing of some power plants," Murty said. "Therefore, we will need to know if we have recovered the key mechanical properties."

According to Murty, no methods exist now to do this well. A second potential use, then, for the ABI technique is to measure the mechanical properties of the vessel materials before and after annealing. He anticipates that his method will quantify both the improvement in ductility and the recovery of mechanical properties.

Murty will compare his results with data from conventional methods to confirm that ABI is as good as destructive tests on surveillance specimens but more easily applied to power plants in operation.

13 Scientists Receive National Medal of Science and National Medal of Technology

President Clinton announced the 1996 recipients of the United States' highest science and technology honors, the National Medal of Science and the National Medal of Technology.

In the field of materials research, **C. Kumar N. Patel**, vice chancellor for research, University of California—Los Angeles, received the 1996 National Medal of Science for his invention of the carbon dioxide laser, a major scientific and technological breakthrough which continues to be an important tool in manufacturing, medical treatment, scientific investigations, and materials processing. His carbon dioxide laser also led to the creation of new generations of lasers and laser systems. The other recipients are **Wallace S. Broecker** (Newberry Professor of Geology, Lamont-Doherty Earth Observatory at Columbia University, Palisades, New York), **Norman Davidson** (Norman W. Chandler Professor Emeritus and executive officer of the Division of Biology at the California Institute of Technology in Pasadena, California), **James L. Flanagan** (Director of the Center for Computer Aids for Industrial Productivity and Vice President for Research at Rutgers University in Piscataway, New Jersey), **Richard M. Karp** (professor, Department of Computer Science and Engineering at the University of Washington, Seattle, Washington), **Ruth Patrick** (Francis Boyer Chair of Limnology, Academy of Natural Sciences, Philadelphia, Pennsylvania), **Paul A. Samuelson** (economist and institute professor emeritus, Massachusetts Institute of Technology, Cambridge, Massachusetts), and **Stephen Smale** (mathematician and professor emeritus, University of California—Berkeley.

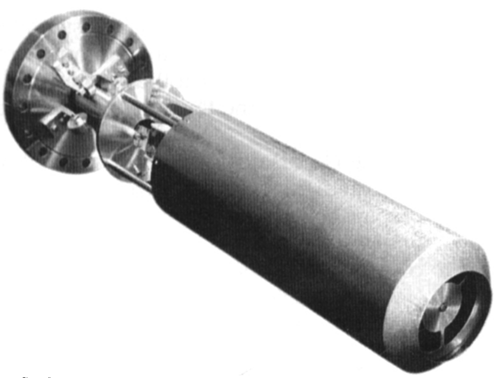
The 1996 National Medal of Technology recipients are **Charles H. Kaman**, president, chair, and CEO, Kaman Corporation, Bloomfield, Connecticut, for his pioneering work in helicopter technology and for making present-day helicopters more stable and easier to fly and for many other innovations including artificial intelligence in medicine and electromagnetic motors to run cleaner public transit buses; **Stephanie Louise Kwolek**, consultant and former research associate, DuPont Company, Wilmington, Delaware, for her contributions in the discovery and development of high-performance aramid fibers which are used in products such as lightweight bullet-proof vests and fiber optic cables; **James C. Morgan**, chair and CEO, Applied Materials, Inc., Santa Clara, California, for his vision and leadership in the development of the U.S. semiconductor manufacturing equipment industry; **Peter H. Rose**, president, Krytek Corporation, Danvers, Massachusetts, for his leadership in the development and commercialization of ion implantation products, which are necessary for the production of modern semiconductors; and **Johnson & Johnson**, New Brunswick, New Jersey, for a century of innovation in the research, development, and commercialization of products, including the first disposable contact lens.

R&D Magazine Announces 100 Awards

The trade journal *R&D Magazine* announced its 1996 awards for the top 100 technological achievements. Brookhaven National Laboratory was named for its plasma window, a device useful for electron-beam welding, recovering scrap metal through electron-beam melting, using industrial processes for making new materials or modifying existing materials, and transmitting beams of radiation for experiments in advanced synchrotron light sources. Developed by Ady Hershcovitch, the plasma window is an ionized gas that separates atmospheric pressure from a vacuum. At 12,000°C, the plasma window is about 300 times as hot as the air at room temperature. This intense heat makes the ionized atoms and molecules move around faster and collide more often with air molecules, thus stopping most of them when they try to pass through the plasma window. Since the plasma window matches atmospheric pressure with only one-fortieth its density, less air pressure can escape from it into the vacuum.

Researchers at Sandia National Laboratories were recognized for developing glasses out of phosphate rather than silicate to hermetically seal aluminum electronic components, useful for automobile and aerospace companies. The molecular structure of phosphate-based glass permits it to flow at much lower temperatures than silicate-based glass so that it is complimentary to the use of aluminum. Researchers have modified the molecular structure of phosphate glass through the incorporation of aluminum oxide, maintaining the chemical durability of the glass.

Several awards went to the development of new materials. Sandy Krezmien-Funk (Owens-Corning) developed glass



fibers to replace synthetic fibers. Fabricated by fusing two forms of glass into a single filament, the MIRAFLEX fiber acts as a fire-retardant material and can be used as insulation for speakers and electrical products, among other applications.

Researchers at Gel Sciences and the Massachusetts Institute of Technology developed smart hydrogels and smart lyogels, a family of gel materials designed with smart solvophobic and solvophilic forces that alter their properties predictably due to environmental influences. Among other applications, smart hydrogels can be used for drug delivery within the body.

In response to a need to remove heat from circuits in electronic packaging, researchers at Applied Sciences developed a substrate of materials called BlackIce, demonstrating thermal-expansion properties that perform better than polymers, ceramics, various metals, and some emerging composites.

Awards went to American Superconductor Corp. for its high-temperature superconductor fabrication technology that uses a composite that has filaments of a ceramic superconductor embedded in a metal matrix; Pacific Northwest for an electrochemical process that operates at low temperature and ambient pressure and uses a recyclable electrocatalyst for the economical treatment of hazardous solid and liquid wastes; Oak Ridge National Laboratory for thin-film rechargeable lithium batteries less than 10 μm thick consisting of an electrolyte made of the ceramic material lithium phosphorus oxynitride rather than of an organic liquid; Matsushita Electrical Industrial Co. (Japan) for a red semiconductor laser consisting of a saturable absorbing layer using metal oxide growth techniques; and to scientists at SRICO, Inc., Army Research Laboratory, and the University of Cincinnati for a photonic electric field sensor made of dielectric materials that reduce the sensor's susceptibility to electric noise, improving its accuracy over other devices.

Other awards include laboratory-on-a-chip developed by Oak Ridge National Laboratory (see *MRS Bulletin*, April 1995 for more details); the integration of micromachines with electronics in manufacturing processes (see *MRS Bulletin*, June 1996) and a low-temperature/pressure process to produce aerogels in bulk and thin-film form (see *MRS Bulletin*, August 1995), both by Sandia National Laboratories; and the magnetic flux imaging system by Argonne National Laboratory (see *MRS Bulletin*, September 1995).

For more details on all of the awards, see *R&D Magazine*, September 1996, Volume 38, No. 10. The 1997 entry forms will be available November 1 by fax 847-390-2618, via the World Wide Web at http://www.rdmag.com, and in upcoming issues of *R&D*.

Recently Announced CRADAs

Los Alamos National Laboratory (Los Alamos, New Mexico) and **Triton Thalassic Technologies Inc.** (T3I) (Ridgefield, Connecticut) signed a nine-month, cost-shared agreement of $103,000 to develop an ultraviolet fluid treatment system.

Appliance Recycling Centers of America, Inc. (ARCA) (Minneapois, Minnesota) and **Argonne National Laboratory** (Argonne, Illinois) signed an $800,000, 16-month agreement to study economical ways to separate and recover highly pure plastics, such as acrylonitrile-butadiene-styrene (ABS) and high-impact polystyrene (HIPS), from shredded household appliances.

Hecker to Receive Navy League Award

Siegfried Hecker, director of Los Alamos National Laboratory, will receive the Navy League New York Council's Roosevelts Gold Medal for Science Award.

Retired Navy Admiral S. Robert Foley, president of Raytheon, Japan, nominated Hecker for his leadership in laboratory programs such as the Trident program, Russian collaborations, new satellite developments, advanced computer technologies, AIDS research, genome studies, and the breakthroughs in neutrino research and superconductivity.

"Under Sig Hecker's leadership and direction, Los Alamos National Laboratory has made and will continue to make dynamic and momentous contributions not only in science and technology, but to our national defense," Foley said in the nomination letter.

The 10-year-old Roosevelts Gold Medal award honors an individual, corporation, or institution for extraordinary contributions through science to the security of America, according to the Navy League. The award was named for both Theodore and Franklin Delano Roosevelt, both former New York Navy League members, who contributed to the U.S. Navy fleet's modernization.

The Navy League was formed in 1902 with cooperation and funding from Theodore Roosevelt. It serves as a civilian organization that speaks for the Navy and serves to educate and motivate Americans to support maritime capabilities, services, and personnel.

Hecker will receive the medal at a formal dinner in New York on November 20.

Silver Grown Epitaxially on Gallium Arsenide

Researchers at the University of Texas—Austin have synthesized a flat epitaxial Ag film on a GaAs (110) surface. At a temperature of 135 K, nominally 15 Å of Ag was deposited on a GaAs substrate, forming a dense nanocluster film. When annealed at room temperature, "a perfectly flat, epitaxial, single-crystal film with uniform thickness was formed," according to the researchers. The films did have some pits, but for a film deposited at temperature, complete islanding occurs. Based on the pit depths, the film thickness was ~15 Å, with a close-packed (111) structure. In their article published in the July 12 issue of *Science*, the researchers said that the structure is "modulated by a 'silver mean' quasi-periodic sequence" along the [001] direction of the substrate, appearing as stripes with spacings of ~13 Å and ~17 Å (referred to as short segment and long segment) in correspondence to five and seven atomic row spacings in Ag crystals. The researchers reported that a scanning tunneling microscopic image shows that the sequence follows that of the silver mean except at one location that is identified as an extra long segment and at another location that represents a missing short segment. The researchers said, "These two locations can be considered as the positions where the silver mean sequence is broken." The average coherent length between broken sequences is ~100 Å. The researchers concluded that the flat film forms at a critical thickness of ~15 Å, but they cannot explain why. For films nominally thinner than 15 Å, the annealed film formed flat-topped islands of 11–15 Å thickness. For thicker films, excess Ag formed islands on top of a 15-Å-thick continuous layer.

News Briefs of Special Science Reports from The German Research Service

■ By bombarding atoms with a highly energized beam of charged atomic nuclei in a heavy-ion accelerator system, researchers at the Society for Heavy-Ion Research in Darmstadt have produced, for a brief moment, an element with atomic number 112. Peter Armbruster led an international team of scientists who beamed heavy zinc ions onto a foil of lead atoms as a target. The fusion of zinc, with atomic number 30, and lead, with atomic number 82, synthesized into the element with atomic number 112.

■ Udo Schwertmann led a team of scientists from the Faculty of Soil Science at the Technical University of Munich-Weihenstephan in the discovery of a natural source of iron hydroxysulfate in Pfitzcherjoch in South Tyrol. The International Minerological Association has officially named the mineral "Schwertmannite."

Hahn Selected for Medal of Excellence

H. Thomas Hahn, Hughes Aircraft Company Chair in Manufacturing Engineering at the University of California—Los Angeles, has been selected to receive the 1996 Medal of Excellence in Composite Materials, given annually by the University of Delaware Center for Composite Materials. According to Tsu-Wei Chou, Medal Award Committee Chair, Hahn "has been a leading contributor to the science and technology of composites for nearly a quarter of a century. His scholarly work in the mechanics and manufacturing science of composites will have lasting impact on our knowledge of the field."

One of Hahn's major accomplishments was founding the Integrated Manufacturing Engineering Program at UCLA. The Program's focus is on the integration of design, manufacturing, business, and management.

Hahn's areas of technical expertise include composites manufacturing and processing, composites design, fracture and fatigue, concurrent engineering, and rapid prototyping. He has published close to 175 papers and chapters. His current efforts are directed toward the integration of composites manufacturing with traditional manufacturing through rapid prototyping.

Among his past and current professional activities and accomplishments, Hahn is President of the American Society for Composites, an ASME Fellow, and has been a member of the NRC National Materials Advisory Board committees and the Technical Review Board of the Army Research Laboratory. He received his PhD degree at The Pennsylvania State University.

Edited from *Composites Update*, a Newsletter of the University of Delaware Center for Composite Materials

Two Advances Overcome Obstacles to EUV Lithography Use in Fabricating Computer Chips

Two advances made by researchers at Lawrence Livermore National Laboratory appear to overcome two obstacles in the development of using extreme ultraviolet light (EUV) lithography to make computer chips. EUV lithography would allow computer chip makers to work with light wavelengths 20 times shorter than today's technology, reducing line widths or feature sizes on chips from 0.35 μm to 0.1 μm and below.

The advances were made in two key areas:

■ a 300,000-fold reduction in the number of defects for the multilayer coated reflective masks used to transfer circuit patterns onto silicon wafers, or chips;

■ a critical 20- to 50-fold improvement in accuracy for measuring the surface shapes of optical components used in the lithography process.

The reduced mask defects spring from an ion beam sputter deposition system. Current methods to make EUV multilayer masks use a technique called magnetron sputtering that produces about 10,000 defects in a square centimeter. The ion beam sputter deposition system yields only three defects in 100 cm. For EUV lithography to be ready for commercial production, this defect ratio would need to be reduced further.

The second advance, by optical physicist Gary Sommargren, is an instrument that significantly improves the measurement accuracy of spherical and nonspherical optical surfaces. Don Kania, deputy AMP leader, said, "Without the ability to measure accurately these aspherical optics, it is impossible to build the optical systems required for EUV lithography." □