Mercury-Based Oxide Under High Pressure Superconducts at 153 K

Several research groups have reported obtaining superconductivity at temperatures above 153 K in a mercury-based oxide compound under elevated pressure. Pressurizing the mercury compound, which has a layered structure, is believed to reduce the distances between the layers, enabling electrons to flow more freely.

The history of this latest series of discoveries goes back to April 1993, when a team of scientists from Moscow and Grenoble reported superconductivity at 94 K in an oxide compound of mercury, barium, and copper. In May a Swiss group at the Eidgenössische Technische Hochschule in Zurich introduced calcium into this mercury-containing compound and reported detecting superconductivity up to 133 K in an impure sample.

Researchers at the Texas Center for Superconductivity at the University of Houston (TCSUH) reproduced this observation and also isolated and characterized the phases. They found that $HgBa_2Ca_2Cu_3O_{8+\partial}$ (Hg-1223) exhibited superconductivity at 135 K at ambient pressure. Subjecting the compound to 150 kbar, the team observed superconductivity at 153 K, with a main transition temperature of 147 K. This work was published in the September 23, 1993, issue of *Nature*.

Paul C.W. Chu, leader of the TCSUH team, speculates that the effect is due to charge transfer plus some other yet unidentified effect. According to Chu, the main layer of mercury atoms tends to have an oxygen deficiency. "Not all the sites are filled with oxygen. So when we apply pressure, there's a greater charge-transfer effect," said Chu.

Chu thinks that T_c above 153 K may be possible in the Hg-1223 compound through physical or chemical doping, and that the high-pressure element could be eliminated.

Immediately after the TCSUH report, Manuel Nunez-Regueiro, a physical chemist, and colleagues at France's National Center for Scientific Research in Grenoble described similar methods for causing superconductivity in Hg-1223. In the October 1, 1993, issue of *Science*, they report a transition temperature of 157 K at 238 kbar.

Nunez-Regueiro hopes to raise the transition temperature of the materials at normal pressure, first by improving the doping process by adding other trace elements to improve conductivity. He also wants to chemically mimic the effects of pressure by incorporating smaller atoms into the material. "This change compresses the material's atomic latttice," he said.

In September, Chu told *Science News* that his group had just attained a transition temperature of 161 K in their compound at 233 kbar and that they would be publishing the results soon.

Sulfide Ceramics Can Form High-Strength Bonds

Sulfide ceramic bonding could replace welding for joining materials that are difficult to weld like graphite, molybdenum, and tungsten, says Tom Kaun of Argonne National Laboratory. Welds in such materials are often weak, and the area around the weld, called the "heat-affected zone," becomes brittle. Because sulfide ceramics bond materials at lower temperatures than those required for welding, sulfide ceramics produce a less brittle joint.

"Sulfide ceramic bonds are up to 20 times stronger than those of other bonding agents in joining metals to ceramics," Kaun said. Besides forming high-strength bonds between ceramic and nonceramic materials, they are stable in corrosive environments like high-temperature molten salts.

The increased bond strength, he said, may make practical a high-temperature molten-salt battery being developed by the U.S. auto makers. The battery is based on lithium and iron sulfide and operates at 400°C. The new ceramics, Kaun said, can bond the battery's stacked, disk-shaped cells for operation in the corrosive, high-temperature environment. Sulfide ceramics have also been used to increase the diameter of advanced battery cells to a size practical for a prototype electric car.

Sulfide ceramics can serve as a structural foundation for voltage sensors in metals production and other corrosive environments, added Kaun. For example, the typical method of producing light metals like aluminum, lithium, and titanium—called "electrolytic recovery"—involves recovery of the metal from a high-temperature molten salt. In this process, the molten metal is collected on an electrode. Sulfide ceramics can withstand this molten salt environment that is destructive to many other materials. Kaun said sulfide ceramics could also facilitate production of a space-based solar-energy converter that uses high-temperature liquid and vapor sodium.

Kaun said sulfide ceramics also can bond materials with different coefficients of thermal expansion. In the past, it has been difficult to bond materials with coefficients that differ as much as 10%. "Using sulfide ceramics, we've been able to bond materials with coefficients that differ by as much as 200%," Kaun said.

Porous Metals from Ukraine To Be Investigated

In cooperation with the U.S. Specialty Metals Processing Consortium Inc. (SMPC), researchers at Sandia National Laboratories are investigating a group of porous, lightweight metals relatively unknown in the United States but with potential as high-strength, high-performance materials. Under terms of a contract signed with the Dnepropetrovsk Metallurgical Institute in Ukraine, its U.S. representative DMK TEK Inc., and the Department of Energy, Sandia will fabricate and test the properties of the porous metals. A furnace for producing the porous metals will eventually be constructed at Sandia's Albuquerque facilities.

The metals, called GASAR materials by Ukrainian metallurgists, are created with a casting process under high pressures. GASARs are reported to possess a high degree of structural integrity, giving them high strength and rigidity when compared to traditional porous materials. Mike Maguire, lead researcher for the project at Sandia, said, "These metals have been the cornerstone of the Soviet's rocket program."

Ukrainian researchers have fabricated GASAR materials from a wide variety of metals and from some ceramics and glasses. The pore size, pore orientation, and pore shape can be closely controlled by manipulation of pressures and cooling processes, Maguire said. Pore sizes can vary from 5 μm to 10 mm. Pore volume can be as low as 5% or as high as 75%. Pore orientation can be radial, longitudinal, or spherical. Unlike fragile conventional porous materials, GASARs retain properties that make metal forming, cutting, welding, bending, and machining possible. In former Soviet Union countries, the materials are being used commercially for chemical filters, bearings, ceramic supports in rocket and jet engines, lightweight panels for space usage, and oxygenators for water purification.

The agreement calls for research at the Dnepropetrovsk Institute on three different alloys: PH13-8Mo stainless steel, Inconel 625, and Haynes 25. This information will be presented to the consortium and Sandia in the form of phase diagrams which describe the pressures and mixtures of gas needed to achieve desired structures. "Gas pressures and compositions have to be maintained in a very narrow range for success," Maguire said. Dnepropetrovsk scientists will also provide plans for a furnace to create the materials. The plans will be compared with plans now under development at Sandia.

Bridgman Award Goes to Ruoff for Contributions to High-Pressure Science

Arthur L. Ruoff, Cornell University professor of materials science and engineering, has received the Bridgman Award of the International Association for the Advancement of High Pressure Science and Technology, given for outstanding contributions to high-pressure science. Ruoff and his research group have achieved 560 GPa static pressure in pressure vessels using diamond tips squeezed together. In 1990, he was the first to reach a static pressure of 416 GPa, greater than that at the center of the Earth. Ruoff also helped pioneer the use of x-ray diffraction to measure such ultrahigh pressures.

Using the diamond anvil technique, Ruoff has found that oxygen becomes an extremely reflective metal at 95 GPa. Sulfur changes, too, he said. With increasing pressure it first becomes amorphous, then transforms to a new crystal structure, and finally to a different crystal structure that is metallic. "As a result, it's possible both oxygen and sulfur could be excellent superconductors," he said.

Ultrahigh pressure also provides a test of bonding theories, Ruoff said. Recently, it was calculated that aluminum nitride would transform to a new crystal structure at high pressure. The group made this structure and found that it remained after the pressure was removed. "The new material is stiffer than the starting material, as predicted by theory," Ruoff said. He hopes to explore the frontiers of ultrahigh pressures, eventually achieving 1,000 GPa.

M. Weber is 1993 Luminescence Award Recipient

Marvin Weber, a Lawrence Livermore National Laboratory physicist, is the recipient of the 1993 International Conference on Luminescence Prize. The prize, established in 1984 to recognize individuals who have made outstanding contributions in luminescence research, carries with it a plaque and a stipend of $1,000. Awarded every three years at the International Conference on Luminescence, the prize was given to Weber for his "fundamental studies of dynamical process in solids which affect luminescence efficiency and the application of that knowledge to laser and scintillator materials."

While he was in the Laser Program at Lawrence Livermore National Laboratory, Weber was the recipient of the George W. Morey Award and an R&D 100 Award for his research on fluorescence and stimulated-emission in laser glass and for the insights that research has provided into glass structure. In the area of scintillator materials, Weber is the discoverer of scintillation in bismuth germanate, a crystalline material widely used in detecting high-energy particles and radiation.

He has been a consultant to Pacific

Northwest Laboratories on scintillator materials for radiation detection for several years and is currently collaborating with researchers at Lawrence Berkeley Laboratory on scintillator materials for medical imaging applications. Weber is also a member of the steering committee of the Crystal Clear Collaboration based at the Centre European pour la Recherche Nucléaire in Geneva, an international group interested in research and development of scintillator materials for high-energy physics detectors.

CO_2 Gas Removed Before Processing High T_c Materials

A way to remove CO_2 in the production of high-temperature superconductors has been developed by researchers at Argonne National Laboratory. They say the method is also 10 times faster and 60% cheaper than conventional processes and allows the resulting material to carry twice as much electricity.

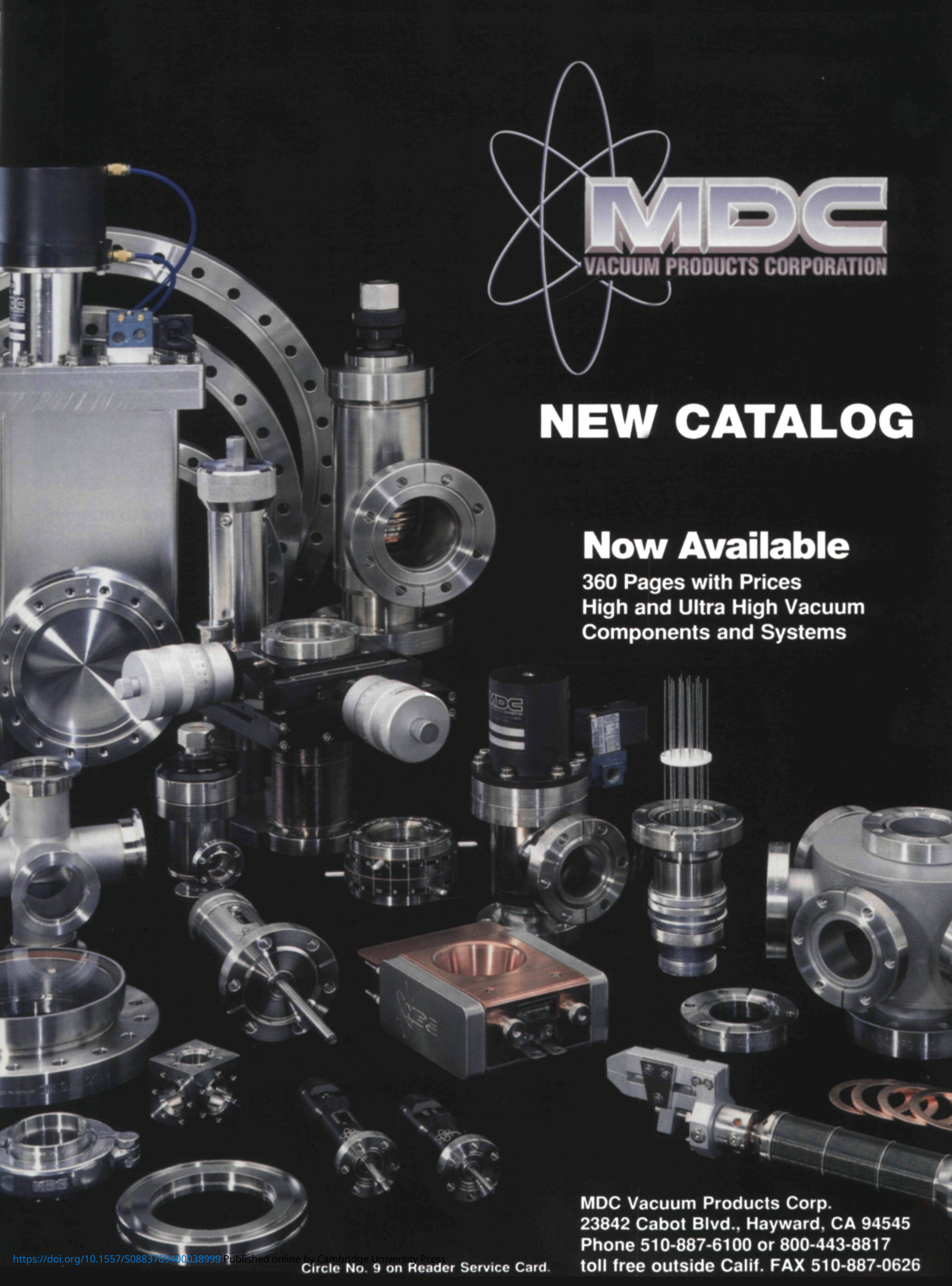
In the conventional process for manufacturing high-temperature superconductors, mixed salts are heated to 950°C for more than 100 hours causing the salts to give off CO_2 gas. The gas reacts with the salts to form nonsuperconducting impurities that lower the current carrying ability of the final product. The new technique removes the CO_2 before it reacts with the salts and needs heating to only 850°C for four hours.

Utham Balachandran, one of the inventors, said, "When you use conventional processing methods, you also have to periodically grind the salt crystals. Inevitably, microscopic particles from the grinding tool get into the mix as impurities. We avoid this because we've eliminated the need for grinding."

Alfred University Creates X-Ray Lab with United Technologies Equipment

A gift from the United Technologies Research Center in New Hartford, Connecticut gives the New York State College of Ceramics at Alfred University the largest academic x-ray laboratory in the world, said Robert L. Snyder, professor of ceramic science at Alfred. Snyder, also director of the Institute for Superconductivity, said the equipment donated by United Technologies will allow College researchers to "implement the latest advances in x-ray characterization of ceramic materials."

The equipment will allow Alfred researchers to bring to the academic laboratory setting high-cost materials characterization techniques previously possible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only at outside sources. Plans are to eventually focus parallel beam diffraction and reflection techniques on practical manufacturing problems and the ultimate transfer of them to the factory floor, said Snyder.

Even before the equipment was put in place, students were involved in analyzing 5,000 powder patterns a year with existing x-ray diffraction equipment, Snyder said, underscoring the importance of the equipment to the school's educational as well as research mission.

In the newly created Thin Film and High Temperature Analysis Facility, one unit will be used to characterize thin films by x-ray reflection, measuring the films' thickness, density, and surface roughness. A second unit will be used for analyzing elemental and phase composition of thin films. That unit is also equipped with a pole figure device which allows researchers to look at the orientation of crystallites within a ceramic body. Pole figure analysis, for example, tells researchers about a superconductor's current carrying capacity and is particularly useful in the effort to develop practical superconducting devices. The third unit is equipped with a Guinier diffractometer, used for high-resolution x-ray diffraction, and can also operate at high temperatures, making the unit particularly useful for the dynamic characterization of advanced ceramic materials, Snyder said.

Ceramic Membrane May Help Convert Methane to Syngas

Amoco Corporation and Argonne National Laboratory recently announced a three-year, $2.5 million CRADA to develop technology to improve conversion of methane, the main component of natural gas, to synthesis gas (syngas).

Current catalytic processes that can carry out the necessary reactions are not economical because the catalysts need a steady, inexpensive supply of oxygen. A potential solution may lie in ceramic membranes that selectively filter oxygen from air.

Reptation Theory of Molecular Mixing Gains Support

The question of whether molecules mix at their middles or in a snakelike head-first fashion has been answered by researchers at the University of Illinois. In the September 16 issue of *Nature*, Richard P. Wool, professor of materials science and engineering, and graduate student G. Agrawal show that the snakelike model is essentially correct. "The question is one of the most critical in polymer science," Wool said. The knowledge should lead to a better understanding of welding, glue stickiness, paint adhesion, photographic processes and high-tech composites, and could result in more efficient manufacturing methods in the plastics industry.

The reptation (for reptile) theory was developed in 1971 by French physicist Pierre-Gilles de Gennes, who speculated that molecular chains moved like snakes. The theory was criticized by scientists who believed molecular motion was Brownian, or random.

In their experiment, Wool and Agrawal used two groups of polystyrene molecules. One group had deuterium atoms at the ends of its molecular chain, and ordinary hydrogen in the middle. In the other group, the researchers reversed the order, inserting hydrogen at the chain's ends and deuterium in the middle. The scientists reasoned that a neutron beam would refract at the point at which the plastics were contiguous. First, with the two sets of molecules adjacent but unmixed, they measured no refraction by a neutron beam at the wavelength of deuterium. Then they heated the molecules so that the plastics began to mix. Had the molecules surged forward from any point along their chains, random mixing would have occurred and the angle of beam refraction should have been unchanged.

Instead, a definite change in refraction occurred at first—a change accounted for if the molecules had led with their ends, moving the leading deuterium from one group to the other while leaving the deuterium atoms in the middle of the second group of molecules initially unmixed. As heating continued, the refraction returned to its original amount as full mixing took place.

The neutron beam experiment was conducted at Argonne National Laboratory. The experiment was repeated using ion beams at the University of Illinois Materials Research Laboratory and at IBM's Almaden Research Center.

High-Flux Solar Furnace Offers Research Opportunities

This past July, the Department of Energy designated the National Renewable Energy Laboratory's (NREL) solar furnace research facility a National User Facility, opening it nationwide to industry, university, and government scientists interested in using the power of up to 50,000 suns for technology development.

The furnace, located on South Table Mountain in Golden, Colorado, was built in 1989 to explore the use of highly concentrated sunlight for various materials and chemical processes, ranging from advanced materials processing to detoxification of contaminated wastes. Its configuration of large mirrors concentrates sunlight (potentially to an intensity equal to that of 50,000 suns) onto a small target at temperatures as high as 3000°C.

NREL will continue to use the facility for DOE research, but it is now available for outside research one or two weeks every month. "We ultimately expect to accommodate up to two dozen users a year," said Allan Lewandowski, NREL project leader for advanced processes.

For information contact: A. Lewandowski, Thermal Systems Branch, National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401-3393. Phone (303) 231-1972; fax (303) 231-1331.

Joint Project Seeks to Improve Lasers for Manufacturing

Under a CRADA, Argonne National Laboratories and Spawr Industries, Lake Havasu City, Arizona, will work on improving lenses and mirrors to shape laser beams for industrial uses. The project could also improve "laser thermal simulation," a way to test how machine parts withstand heat.

Components of high-temperature devices such as jet engines or particle accelerators are normally tested under operating conditions, but laser thermal simulation, performed in the laboratory, could streamline heat and stress testing, saving money and reducing research and development costs.

The Spawr-Argonne work focuses on improving a radiometric power meter, made by Spawr, which measures the power of high-energy lasers in about one second by sampling a known fraction of the laser light. The conventional method absorbs the total laser beam energy and takes about 10 seconds to measure.

A shorter measuring time gives the Spawr instrument a wider range of applications. "Argonne and Spawr will work on improved coatings for Spawr's laser dump to handle laser beams with high power density," Keng Leong of Argonne said. The laser dump absorbs laser energy while the beam is being tuned for specific applications. □

Correction: On p. 30 of the September 1993 *MRS Bulletin*, James G. Fagan's photo was identified as being that of Alexander R. Lodding, and vice versa.