Atomic Imaging Identifies Surface Processes in Technologically Important Materials

Atomic imaging procedures used at Sandia National Laboratories have identified processes that take place at the surfaces of technologically important materials. The research, carried out by physicist Gary Kellogg in the Surface Science Division, has so far:

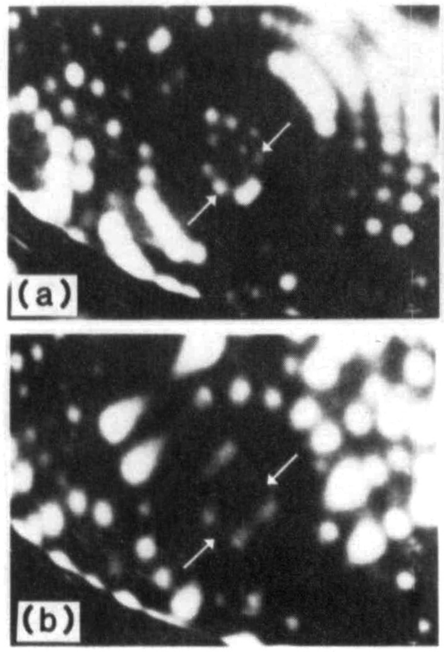
- Identified the process responsible for the decrease in catalytic activity of rhodium automotive exhaust catalysts during the carbon monoxide oxidation reaction. The drop in catalytic activity occurs when a stable oxide forms on the surface of the rhodium.
- Made the first direct observation of how a surface of platinum reconstructs itself from its expected arrangement of surface atoms. Images showed that the platinum atoms rearrange into alternate filled and empty rows. Studies of the motion of individual atoms also identified the fundamental forces that drive the restructuring of this single-crystal surface.
- Led to understanding the growth mechanism of a strained-metal overlayer system. These studies imaged both individual nickel atoms and clusters of nickel atoms as they attached to or moved about on tungsten substrates.

Field ion microscopy was used in the studies, and the study of catalytic reactions on rhodium combined field ion microscopy with imaging and pulsed-laser atom-probe mass spectroscopy techniques developed at Sandia.

The experiments with rhodium solved a puzzle about its catalytic action. With rhodium catalysts it is known that the reaction rate increases for a time with rising pressures of oxygen, but then suddenly goes down.

Kellogg ran the reaction under simulated real-world conditions in a catalytic converter, using field ion microscopy to examine the surface before the reaction started and using imaging atom-probe mass spectroscopy to study the surface composition after the reaction. With varying oxygen/carbon monoxide ratios up to 30:1, the atom-probe mass spectrometer saw no unusual surface species. Increasing the ratio to 40:1, however, resulted in signals indicating presence of a surface oxide. Analysis showed that a stable oxide of rhodium, Rh_2O_3, had formed.

The range of oxygen-concentration ratios where oxides were detected (30:1 to 40:1) correlates well with the ratio found for deactivation of the CO oxidation reaction, according to Kellogg. "That these conditions correlate with the deactivation of the reaction is confirming evidence that oxide formation is the surface process responsible for deactivation," he said.



Field ion microscope images show in atomic detail the reconstruction of atoms of platinum (a) from a bulk-terminated surface structure to (b) a missing-row structure. The reconstruction was produced by heating the sample from 77 K to 330 K for one minute. Arrows identify a row of atoms in (a) that is missing in (b).

Direct observations of the surface reconstruction of platinum helped settle a long-standing controversy over how platinum atoms rearrange as a new surface is formed. Observation before reconstruction showed rows of atoms adjacent to each other. After the surface reconstruction was stimulated by heating to temperatures between 300 and 450 K, the observations showed that every other row of atoms on the surface was missing, confirming the missing-row model (see photo).

Subsequent studies showed that the atoms rearrange into the missing-row structure because there is a strong attractive interaction between atoms in the same row or channel but a repulsive interaction between atoms in adjacent surface channels.

To achieve a better fundamental understanding of the properties of metal overlayers, Kellogg used atomic imaging to study what happens during growth of an overlayer of nickel deposited onto two different surfaces of tungsten. One goal was to learn whether an individual nickel atom is mobile or stationary on different tungsten surfaces. A single nickel atom became mobile well below room temperature on a relatively smooth surface of tungsten, but only above room temperature on a more "open" or rough surface. Pairs of nickel atoms exhibited much the same behavior.

Deposition at elevated temperatures on the smoother surface caused clusters of nickel atoms to form, their shape determined by the deposition temperature. Atoms deposited at 200 K formed linear one-dimensional chains aligned parallel to a particular crystal plane of the substrate. Atoms deposited at higher temperatures (230 K and above) formed two-dimensional clusters. At 300 K and below the clusters remained immobile; above 300 K they could be seen migrating as a unit. During intervals between addition of heat, smaller clusters could also be seen coalescing into larger clusters. Two clusters of 15–20 atoms, for example, came together and formed a larger cluster of 30–40 atoms.

At still higher temperatures (375 K), these islands of nickel atoms fell apart, and the clusters disappeared from the top of the tungsten plane. The growth of these islands was strictly one- or two-dimensional. Attempts to deposit nickel atoms on top of the nickel islands did not work.

Army Awards Grants for Diamond Films

The U.S. Army Strategic Defense Initiative Organization has awarded Ionic Atlanta (Georgia) Phase I of two Small Business Innovation Research (SBIR) grants to study the feasibility of producing diamond and diamond-like films using ion beam and plasma-enhancement techniques. Total funding is $131,487 for both programs, which are being headed by Dr. Keith Legg.

One program will concentrate on developing diamond and diamond-like coatings on space-based optical components. The coatings should not only be transparent from the infrared to the ultraviolet, but hard, chemically stable, and scratch-resistant as well. Since ion beam techniques will be used to ensure good adhesion at low temperatures, these films will be available for a variety of optical substrate materials.

The second SBIR program will develop ion beam techniques for doping of thin epitaxial diamond films grown via plasma-assisted techniques. Doped electronic diamond films would be ideal for space-based systems where new semiconductor materials are needed to be faster, less susceptible to radiation, and capable of higher-power densities and operating temperatures.

continued

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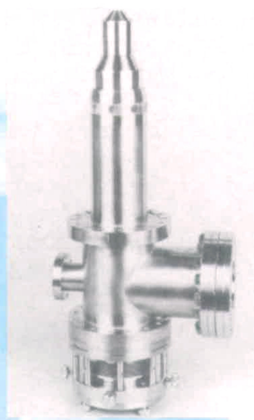
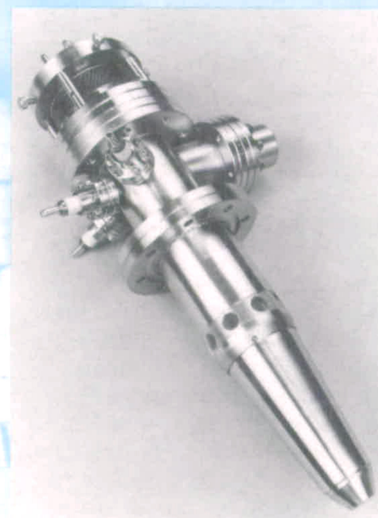
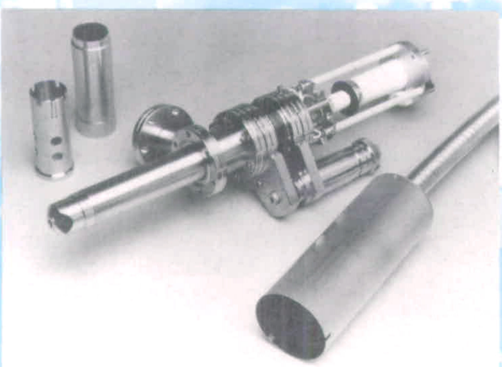
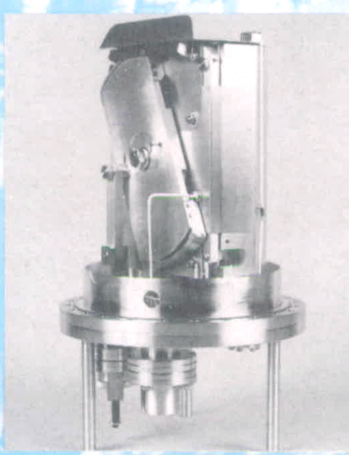


2000Å Electron Source



Twin Anode X-ray Source





CLAM 100 XPS/AES Analyzer

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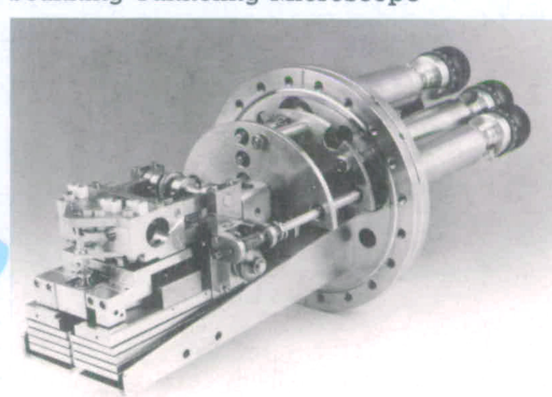

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NAE Report Says Engineering Ties to Foreign Technology are Critical to U.S. Economy

A report by a National Academy of Engineering committee suggests that academic, government, and industrial programs in the United States must be modified to respond to increasing engineering activity abroad. Many nations have developed centers of technological excellence, the committee noted, and newly industrialized nations such as Taiwan, Korea, and Brazil are attaining higher levels of competence for technical development as well as production. More than 60% of research publications in engineering and technology now originate outside the United States.

The report suggests that the United States would benefit more by ensuring that knowledge is created in American laboratories at a more rapid pace than it is transferred abroad and that American engineers and scientists have access to significantly more technological information from foreign research. "There is a growing appreciation that in today's world, a company or a nation must be cooperative to be competitive," wrote NAE president Robert M. White in a letter transmitting the report to its sponsor, the National Science Foundation.

The committee, chaired by H. Guyford Stever, NAE foreign secretary, offers recommendations not only to the NSF but also to other federal agencies, engineering schools, professional societies, and industry. It called on the NSF to increase its level of funding for participation in international collaborative engineering research and education and to modify its proposal formats to ensure that researchers are aware of results from engineering knowledge abroad.

Other committee recommendations:

• Engineering schools should instill in their students a greater appreciation for foreign achievements, and make available more opportunities for engineering students to study abroad. The committee called for establishing on the campus of every major engineering school an office responsible for presenting an international perspective in engineering and technology, and for providing information on international programs and developments. The committee particularly noted an "urgent need" for increased capability in Asian languages, especially Japanese, among American engineers.

• Professional engineering societies should increase their participation in international activities, and industry should adopt strategies "for connecting to engineering progress abroad." One way of doing this is by increased cooperation of U.S. engineers in international efforts, such as setting standards for commercial products. The committee urged federal agencies and American firms to become more involved in this area.

(See Material Matters in this issue in which MRS President K.C. Taylor looks at MRS's role in international cooperation.)

The report, *Strengthening U.S. Engineering Through International Cooperation: Some Recommendations for Action*, is available from: National Academy of Engineering, Office of Administration and Finance, 2101 Constitution Avenue NW, Washington, DC 20418.

Université de Sherbrooke Acquires SEM and X-Ray Analyzer

The Materials Characterization Laboratory at the Université de Sherbrooke (Quebec, Canada) recently installed a JEOL JSM 840 scanning electron microscope and a Link AN 10085 energy dispersive x-ray analyzer. The instruments were purchased using a grant of Can$263,408 awarded in April 1987 by the National Science and Engineering Research Council of Canada to Drs. P.-C. Aitcin and S.L. Sarkar of the Department of Civil Engineering and Drs. M. Boulos and E. Chornet of the Department of Chemical Engineering. Several private companies and the University provided additional funds for installing the two instruments which will be used for research on materials ranging from minerals, cement, concrete, refractories, grouts, ceramics, composites, metals and alloys to ultrafine particles.

U.S. Army Uses NMR Spectroscopy

The Materials Characterization Division of the U.S. Army Materials Technology Laboratory (Watertown, MA) will use a nuclear magnetic resonance (NMR) spectrometer to help select materials to incorporate into military systems. Research chemist Louis Carreiro and physicist Paul Sagalyn will perform analyses on a wide range of materials including polymers, ceramics, metallic alloys, and organic compounds. Eventually, NMR will become an integral part of the M1 tank track pad program, as different blends of rubber are analyzed for their composition. In addition to spectroscopy work, the Materials Technology Laboratory (MTL) will extend its efforts to include NMR imaging to detect flaws such as trapped water or leaked fuel which can degrade materials during their lifespan.

NSF Establishes Four Materials Research Groups

The National Science Foundation (NSF) has awarded $7.4 million to sponsor four teams of university scientists who will perform basic research on materials of potential technological interest. The three-year grants bring to 15 the number of university-based Materials Research Groups (MRGs) established by the NSF since the program's inception in 1985. The four new groups will emphasize distinct areas of research — the magnetic properties of thin-film materials, the toughening of various glassy polymers, the electronic properties of novel semiconductor compounds, and the factors that make materials susceptible to deformation and fracture.

The new MRGs are being established at Brown University, the University of Michigan, the University of California at San Diego, and Montana State University.

Brown University will receive $900,000 per year for three years to study how materials deform and fracture. Research will include experimental and theoretical modeling, and will focus on understanding the effects of temperature and stress rates, the relationship of microscopic mechanisms to materials behavior, localized deformation mechanisms, and the role of surfaces and interfaces.

The University of California at San Diego will be awarded $550,000 annually for three years to study the microscopic properties of magnetic materials in thin-film and particulate form, including materials used for storage in tape and disk recording.

Montana State University's three-year grant will total $1.68 million dollars, including $590,000 during the first year. Efforts will be directed toward better understanding basic properties of the semiconductor gallium arsenide and related compounds, including how these compounds "grow" during manufacturing processes. Scientists will use a range of experimental tools, including scanning electron microscopy, low-energy electron diffraction, and photoemission and energy loss studies. Theoretical research will also be conducted.

The University of Michigan will receive $343,000 the first year and an additional $1 million over the following two years to establish a group to study ways to toughen glassy polymers. Scientists from the field of ceramics, where toughening mechanisms are better understood, will work with experts in polymers, an area in which mechanisms still need to be tested and applied.

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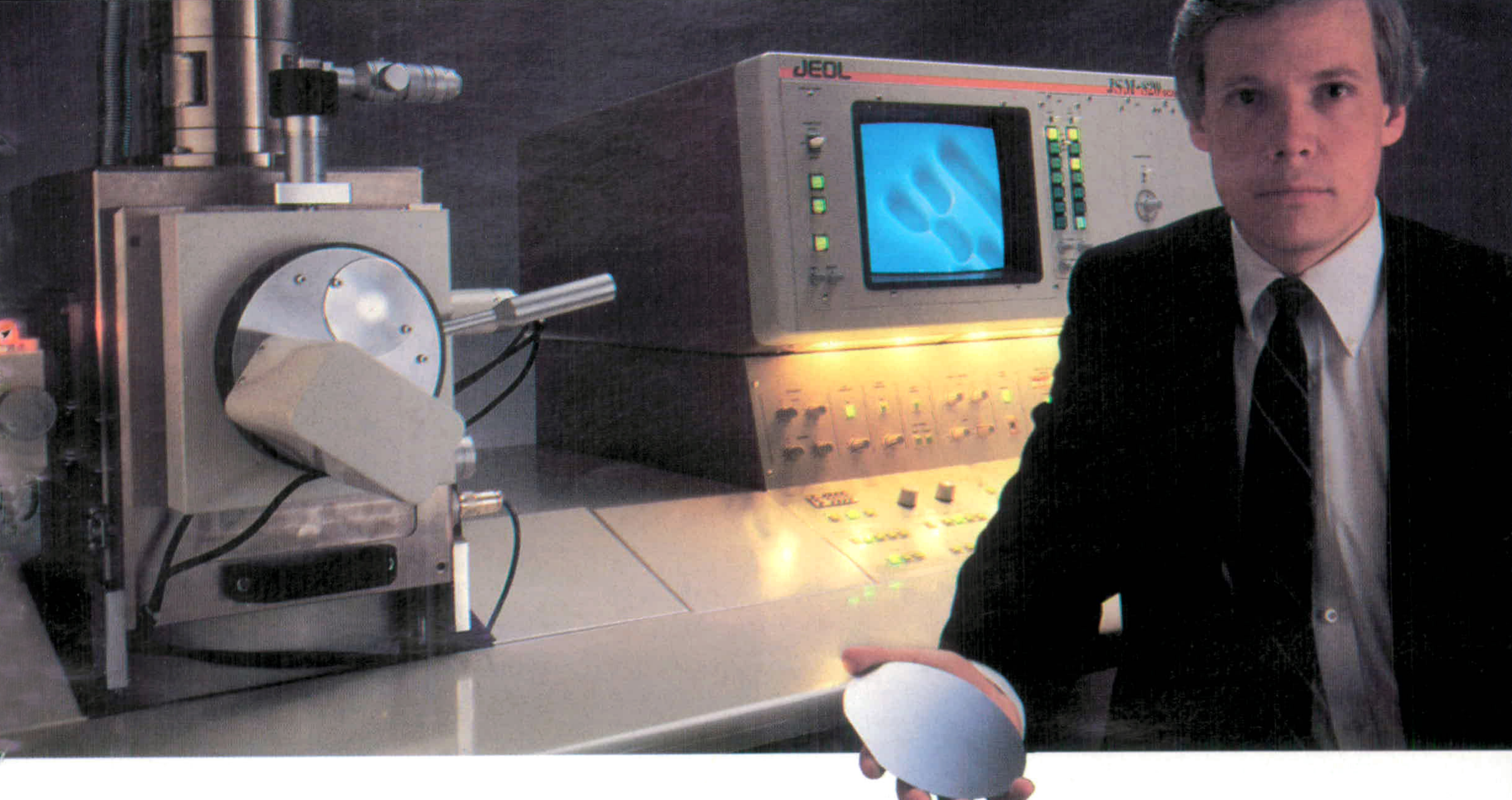

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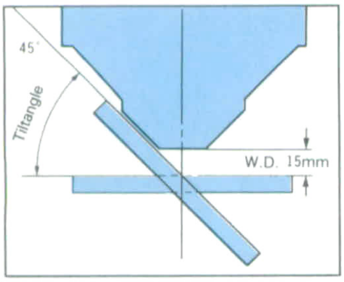

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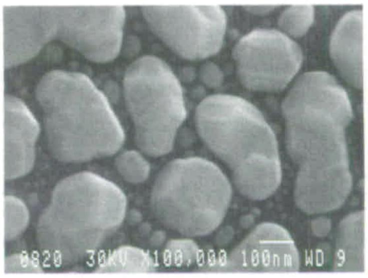

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Researchers Explore Use of Plastics in Development of Artificial Blood Vessels

Research at Los Alamos National Laboratory may lead to the development of artificial blood vessels that would be commercially available for human implantation. Except for large blood vessels, natural materials are now used for vascular implants in humans. The use of plastics to replace veins and arteries has been restricted by the tendency of blood to clot when it comes into contact with the artificial material. Chemist Debra Wrobleski and co-workers David Cash, David Duchane, Jerry London, and Bruce Lehnert have been studying ways to alter the surface of polymeric plastics to reduce the chance that blood will react adversely with the artificial material.

The researchers are using chemical infusion to modify the surface of polymeric plastics without changing fundamental properties, such as strength and flexibility, that make the materials biologically useful as synthetic blood vessels. In the chemical infusion process, for which Duchane earned a patent four years ago, the polymeric material is placed into a solution that softens the surface. The solution is gradually diluted, allowing the material to harden with new chemicals "infused" into the surface. A wide range of experimental conditions have been used in the research, including varying the type of plastic and changing the dilution times and concentrations of the infusant.

"An ultimate outcome of our efforts will be the establishment of an information base from which materials could be tailored to meet specific medical needs," said Wrobleski. The research may also have applications in other biomedical areas, including plastic surgery, the implanting of timed-release drugs in the body, and the use of plastic materials in insulin pumps and other artificial health aids such as bone implants.

Armada Research to Use Nickel Aluminide Alloy for Resistance Heating Elements

Armada Research Corporation (Detroit, MI) has acquired exclusive field of use rights to a high temperature nickel aluminide alloy for commercial use in electric resistance heating elements. The heat- and corrosion-resistant "intermetallic" material, developed at Oak Ridge National Laboratory, was also licensed to Cummins Engine Co. in 1985 for applications in large displacement (non-automotive) diesel engines.

Nickel aluminide increases in strength with higher temperatures—at 600°C, it has six times the strength of stainless steel. This characteristic, together with the availability of raw materials in the United States, makes the aluminide an attractive alternative to high temperature alloys that depend on foreign-supplied cobalt or chromium. Other potential applications include high performance jet engines, gas turbines, advanced heat engines, and heat exchangers in nuclear and coal-fired steam plants.

Ceramic Engines Promise Improved Performance

Ceramic engines may endow the car of the future with such attributes as rapid acceleration, durability, corrosion resistance, and fuel economy, according to a new National Research Council report. Ceramic parts are being widely used to replace metal in experimental engines, the report notes, and some foreign manufacturers have introduced them in a few commercial models. Ceramic materials offer certain advantages over metals, says the committee of automotive experts who prepared the report, but ceramics also present problems of brittleness, high cost, and difficulty in manufacturing large uniform quantities.

The committee visited automotive plants and engineering laboratories in the United States, Japan, and Europe. It reported that "aggressive and coordinated" programs to develop ceramic engines are under way in Japan, while several European countries are moving into ceramics at a more moderate pace. In contrast, "industry-government coordination of efforts to use ceramics commercially appears to be at a lower level in the United States than in either Europe or Japan," said the committee.

The study was undertaken in response to a Department of Energy (DOE) request for a review of research on advanced diesel engines, called "low heat rejection engines" (LHREs) in the report. Research in this area has been directed toward using ceramic insulation to prevent heat loss from the cylinder, on the theory that such an engine would use fuel more efficiently than traditional engines. The research generally has been applied to diesel engines because the higher temperatures cause unacceptable knock in a conventional gasoline engine, the report explains. Federally supported research on advanced engines has been funded by DOE and the U.S. Army; U.S. industries are also becoming increasingly involved in ceramics research and development.

LHREs may be especially important to the Army with its fleet of 25,000 tracked combat vehicles and 250,000 wheeled vehicles, according to the report. The committee estimated that LHREs in tanks could improve fuel economy 5–10% over conventional diesels, including the energy savings from operating a smaller cooling system. Adding in-cylinder insulation to existing turbocharged engines in commercial vehicles could improve fuel economy 3–4% when the vehicle is operated at full throttle; fuel savings may be even greater at part throttle, the committee estimated. For commercial trucks, Army tactical vehicles, and diesel automobiles, a smaller cooling system would permit redesign of the front end for reduction in aerodynamic drag, with a resulting improvement in fuel economy.

Despite the promises of ceramic insulation, the committee concludes that ceramic materials appear to be so versatile as replacements for metal engine parts that they will probably be developed more rapidly for this purpose. Many uncertainties in ceramic engine technologies remain, and research will be required to determine the best uses for the new materials. Unless costs are reduced considerably, ceramics will be unable to compete with metal and development will be slowed, the committee commented. But the prospects of future benefits are sufficient, it said, to recommend that the DOE and the U.S. Army continue their research support.

The report, *A Review of the State of the Art and Projected Technology of Low Heat Rejection Engines*, is available for $12.00 prepaid from the Energy Engineering Board, 2101 Constitution Avenue NW, Washington, DC 20418.

Glasstech Achieves Large-Scale Continuous Production of α-Si Coated Glass

Glasstech Solar, Inc. (Wheat Ridge, CO) has developed a multichamber, in-line system to coat glass substrates up to 120 cm × 180 cm with a uniform, amorphous silicon film on a continuous production basis. The coated panels are used in solar energy cells for the generation of electric power. Earlier this year, Glasstech received a contract from the government of India to provide a large-scale, fully automated turnkey plant to produce amorphous-silicon solar panels. GSI also has supplied smaller research systems to the Jet Propulsion Laboratory (NASA) and the Indian and Mexican governments.

Bednorz and Müller Awarded Nobel Prize for Physics

J. Georg Bednorz and K. Alex Müller were awarded the Nobel Prize for physics for their breakthrough discoveries in superconductivity. Bednorz and Müller, scientists at the IBM Zurich Research Laboratory, realized in 1983 that oxides can potentially be high temperature superconductors. The announcement of their results (*Z. Phys.* **B64**, 1986, p. 198) led to international skepticism that was overcome when K. Kitazawa and C.W. Chu confirmed "credible evidence" of superconductivity at 30-35 K in a lanthanum barium copper oxide at the 1986 Fall Meeting of the Materials Research Society in Boston.

Bednorz, a crystallographer, was born in Germany in 1950 and joined IBM's Zurich lab in January 1982. His current research interests include preparation, crystal growth and characterization of high refractive oxide materials (phase transitions and quantum ferroelectricity), oxides with metallic conductivity and superconductivity, and development of very high temperature superconductors.

Müller, a physicist, was born in Switzerland in 1927 and joined IBM's Zurich lab in 1963. His present interest is in fundamental questions leading toward understanding the nature of high temperature superconductivity.

"The Race for the Superconductor"

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