Process Opens Heat-Resistant Polymers to Manufacturing

The potential for some strong, heat-resistant polymers has been little or only partly realized because the very properties that make them useful also make them essentially impossible to manufacture.

Now University of Rochester scientists have developed a simple and inexpensive process that opens up manufacturing possibilities for several classes of polymers, including aromatic polyazomethines. An aromatic polyazomethine is so resistant to heat, it decomposes before it flows, and it will not dissolve in solvents except in highly corrosive acids which destroy it.

An easily reversible, harmless technique, complexation-mediated solubilization and processing, makes the polymer flow. A solid such as gallium chloride or ferric chloride is added to the polymer. The chemical then forms a complex, temporarily breaking the forces between molecules and making the polymer dissolve in common solvents so it can flow. After useful devices have been formed, the material is immersed in water or methanol and the process is reversed. The polymer resumes its strength, stiffness, and chemical resistance, but in a useful form.

Several other polymers (PBO, PTMHT, PBZT, BBB, BBL, and Nylon 66) benefit from the new technique, which reduces processing temperatures for each one, sometimes by several hundred degrees to near room temperature.

Because of their durability under high temperatures, aromatic polyazomethines might be used in applications that range from fire-resistant clothing or carpeting to lightweight car and airplane parts. Because they also react to laser light, switching the index of refraction in just one picosecond, they could also be used in optoelectronic materials for computers, communications equipment, and electronics.

SERI Receives National Laboratory Status, New Name

President Bush recently designated the Solar Energy Research Institute (SERI), located in Golden, Colorado, as the National Renewable Energy Laboratory (NREL), symbolizing a U.S. "commitment to renewable energy as an essential part of the National Energy Strategy."

Much progress in deriving electricity from solar thermal, photovoltaics, and wind took place at SERI, Deputy Energy Secretary W. Henson Moore said. "Elevating its status to the elite group of National Laboratories is both a recognition of these accomplishments and a commitment to continue this renewable energy progress in the future," he said.

SERI, authorized by the Solar Energy Research, Development, and Demonstration Act of 1974, is the nation's chief institution for solar and renewable energy research and development, with an annual operating budget of approximately $95 million and a staff of over 500.

Since SERI began operation in 1977, the laboratory's mission has grown to include all aspects of solar and renewable energy research, including photovoltaics, solar thermal, biomass, wind, building systems, resource assessment, analytical studies, and energy storage. The laboratory's cooperative programs and technology transfer initiatives with private industry have helped bring renewable energy technologies into practical use.

SERI is currently housed primarily in leased office space in Golden, but a field test laboratory building and related outdoor test installations were completed in 1985. Congress has approved $4.5 million to begin construction of a new, $20 million, 100,000 square foot laboratory this year.

Ultrasound Plus Tiny Bubbles Equals New Materials

University of Illinois scientists have determined that the collapse of tiny gas bubbles during exposure to ultrasound produces temperatures almost as high as the surface of the sun, and that this process can be used to create new materials.

The searing heat, about 4900°C, occurs when the bubbles implode, creating microscopic hot spots, said Kenneth S. Suslick, who directed the studies.

The chemical effects of ultrasound, pitched above 16,000 cycles per second, come from the creation, expansion, and collapse of small gas bubbles in an irradiated liquid. The process, called cavitation, creates the intense heat. Suslick and colleague Edward B. Flint examined the light emitted from these hot spots to measure the temperature that cavitation produces, as presented in the September 20 issue of *Science*.

"There is tremendous local heating and emission of light. It's a microscopic flame in a very cold liquid," Suslick said. The heating, however, lasts a very short time, and hot spots are cooled in much less than a millionth of a second.

In the October 3 issue of *Nature*, Suslick and colleagues Seok-Burn Choe, Andrzei A. Cichowlas, and Mark W. Grinstaff describe how they used this rapid cooling to produce a new amorphous metal. "By producing metal atoms and then cooling them at a rate of more than a billion degrees per second, the metal solidifies before it can crystallize," Suslick said. "This forms a metallic glass." They used this process to produce amorphous iron powders.

The metals formed by this process have important industrial applications. Since they have unusual magnetic properties, they could be useful for audio tape and videotape heads and computer disks. Also, they can act as catalysts to convert one chemical to another, which may be useful for the manufacture of pharmaceuticals, fine chemicals, and petroleum products.

Suslick and Grinstaff described a medical application of ultrasound in the September *Proceeding of the National Academy of Sciences.* By exposing proteins to ultrasound, one can make long-lived microscopic spheres, which can pass through the bloodstream. These microspheres produce sonographic images of the heart and other organs, and may be used in the future as artificial red blood cells and for use as magnetic resonance imaging agents.

Suslick and Grinstaff found that ultrasound disperses small droplets of air into water, and then it makes the microspheres permanent by causing chemical bonds to form between protein molecules. These bonds are created from reactions with superoxide, a highly reactive ion produced in the cavitation hot spot during ultrasonic irradiation of aerated water. Understanding the mechanism "allows us to know what proteins will and will not work," Suslick said. "With this knowledge, we are able to make better microspheres and of different proteins."

Optical Fiber Stores 50 Retrievable Holograms

Until recently, the idea of retrieving a whole page of information instantly from a data base using laser holography was hampered by a problem with the large crystals used to store the holograms. When data is retrieved, scattered light mixes information from more than one page at a time. Now, Lambertus Hesselink, a Stanford University professor of electrical engineering and aeronautics/astronautics, has come up with a solution that allows laser optics to store up to 50 holograms in a single crystal optical fiber. The strontium barium niobate (SBN) rod is too small for "crosstalk" between images.

Each hologram contains 100,000 to 1 million bits of data, stored in an optical fiber using two beams of laser light. An object beam is reflected off a recordable image, such as a spreadsheet, into the fiber. Then a reference beam crosses the object beam and records the image in the fiber as a pattern of electrons. Each reference beam is encoded so that, bounced into the fiber again, it retrieves only the pattern for the particular page that it stored. So 50 spreadsheet pages can share the same tiny space, and data can be retrieved from any one at the flash of a laser beam.

Hesselink and colleague Steve Redfield of Microelectronics and Computer Technology Corporation have patented their optical storage method. They are now working on a five-year, $22.5 million grant administered by the National Institute for

Standards and Technology to develop the architecture of input/output devices to take advantage of the new technology. A few thousand SBN rods packed into a plate the size of a 5 1/4-inch floppy disk could hold as much information as the large multiple storage disks of mainframe computers.

Though several groups in the United States and Japan are working to develop the technology, Hesselink said it will probably be five to 10 years before products reach the market. Some of the first applications are likely to be for large data base management systems, such as payrolls, and for image-based systems, such as high-definition television.

$23 Million Approved for MIT Magnet Laboratory

The National Science Board (NSB) approved a National Science Foundation (NSF) recommendation to provide up to $23 million to the Massachusetts Institute of Technology to support the Francis Bitter National Magnet Laboratory until September 1995, when a new National High Magnetic Field Laboratory (NHMFL), now under construction in Tallahassee, Florida, is expected to become fully operational. The NHMFL is being established with support from NSF and the state of Florida by Florida State University, the University of Florida, and Los Alamos National Laboratory in New Mexico.

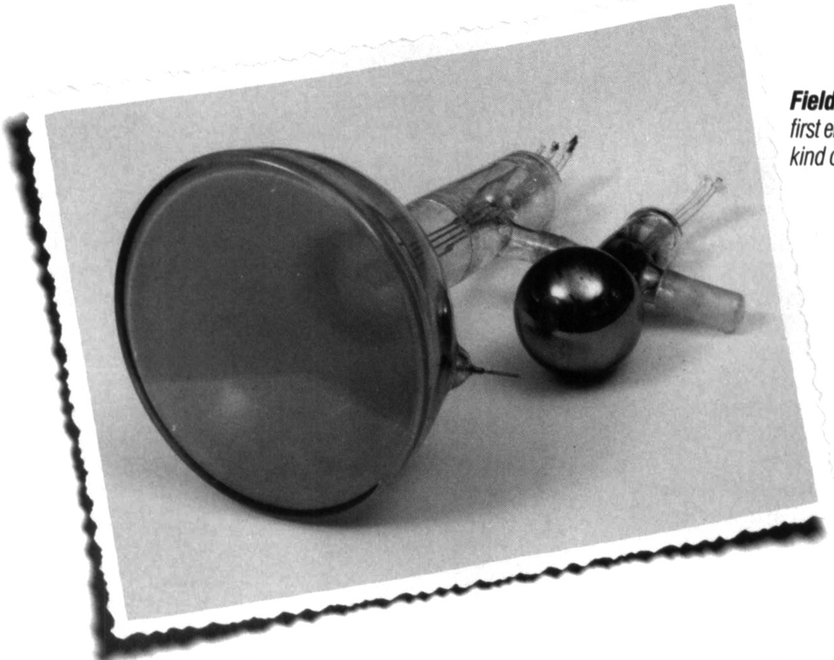
In addition to providing continuing services to the user community, part of the funding to MIT is earmarked for the design, development, and construction of a 45 Tesla class hybrid magnet. The magnet combines superconducting magnets and water-cooled magnets, and achieves a magnetic field 900,000 times the strength of the Earth's magnetic field. The hybrid magnet project is a joint venture between MIT's Bitter Laboratory and the National Laboratory in Florida. The magnet will be completed by 1995 and will be located in Tallahassee.

Once completed, the hybrid magnet will provide the highest steady-state magnetic fields available, enabling scientists to explore and develop high technology fields such as high-temperature superconductors, semiconductor multilayer structures used for high performance computers, liquid crystals, novel magnetic materials, and biomolecular materials.

Model Shows Surface Atoms Move by Exchange/Displacement

Recent theoretical and experimental evidence has revealed a new model for the movement of surface atoms based on an atom exchange/displacement mechanism. The long-standing view has been that metal atoms "roll around like marbles" on the existing bumpy surface until they group together into compact little islands, sometimes moving to new positions by finding an energetically low point (analogous to a pass in a mountain range) and "hopping" over it.

Investigations by Sandia National Laboratories' physicists Gary L. Kellogg and Pe-



Field emission in 1971. *FEI's first emission cathodes were for this kind of gun.*

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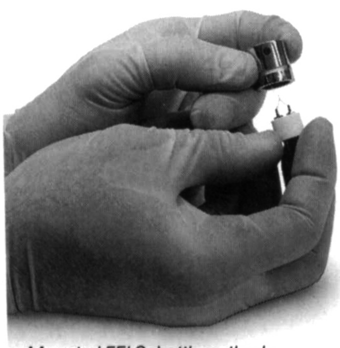
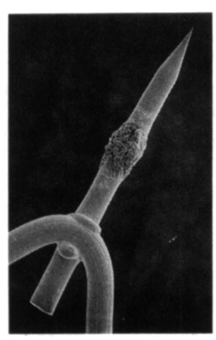


Mounted FEI Schottky cathode with suppressor removed.



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ter J. Feibelman have established that the dominant mode of movement on certain surfaces is, instead, "a concerted exchange/displacement process," markedly different in certain consequences and effects from the hopping process previously assumed. An atom newly brought into interaction with the surface moves by displacing another atom in the existing crystalline surface below it. The underlying atom is then forced to the surface. That atom then repeats the process with another atom in a nearby position.

Feibelman's computed model indicates that the exchange occurs because substitution minimizes the degree of chemical bond rupture required by surface atom motion. Movements of individual atoms on a surface now have to be viewed more as a chemical exchange phenomenon than as simply a physical, mechanical movement, said Feibelman.

Feibelman's initial calculations suggested that the energies required for a single atom (specifically, aluminum) to "hop about" were far higher than would be expected. The calculations showed that only one-third as much energy would be needed if the atoms moved about by exchanging their positions with other atoms in sequence. So he predicted that experimental evidence would show that exchange/displacement was the dominant mechanism.

The exchange/displacement mechanism requires that atoms move only in certain directions and patterns so that a single atom's movements can be predictably mapped. The pattern for aluminum, for example, would be a square with sides parallel to two predicted directions.

Looking at data he had previously gathered with a field-ion microscope, Kellogg noted the same predicted square pattern for platinum atoms. The sites visited by a single platinum atom moving about on a smooth platinum surface formed a predicted square pattern. Similar experiments at Pennsylvania State University showed the same for iridium atoms diffusing on iridium surfaces.

The exchange/displacement effect has been established for platinum, iridium, and nickel atoms. Studies have also shown the effects apply on heterogeneous surfaces, for instance platinum atoms on a nickel surface, and also that they occur at particularly low temperatures, to 105 K.

Another study shows that two-atom pairs also migrate by a series of exchange displacements, doing so even more easily than single atoms. Three-atom clusters, however, move by a combination of exchange and hopping displacements. "This is really a nice demonstration that we have to look at the movements of atoms on surfaces in terms of chemical bonding," said Kellogg.

The findings show that it may not be

possible to grow certain atomically sharp interfaces, suggesting it may be difficult or impossible to build some new, otherwise desirable, crystal structures with alternating layers of atomic composition. Also, the work has shown that the interaction between diffusing atoms can develop small structures not previously predicted, helping scientists understand the limiting size of engineered structures.

Sandia to Lead Center of Excellence for Synthesis and Processing of Advanced Materials

As a result of a National Research Council study showing that the United States was falling behind in the synthesis and processing of advanced materials, Congress has appropriated funds for a Department of Energy (DOE) Center of Excellence for the Synthesis and Processing of Advanced Materials, led by Sandia National Laboratories. The distributed center will involve research at 11 DOE laboratories. Initial funding of $5 million is provided by DOE's Office of Basic Energy Sciences.

The Center aims to determine the principles that control the synthesis and processing of materials, develop specialized equipment and sensors, reduce the time span and cost of commercializing materials, document the principles learned, and transfer new technologies to industry. Targeted research areas include:

- Atomic structuring. Researchers will concentrate on design at the atomic level for producing devices such as solar cells, infrared detectors, high-frequency transistors, and optical modulators.
- Complex polymers. Scientist will study polymer growth, cross-linking, and fractal structures. Processing these complex materials depends on the interplay between chemical reactions and physical processes.
- Ceramic synthesis and processing. Scientists will explore new ways to control the properties of ferroelectric ceramics as well as develop new advanced ceramics like cubic boron nitride.
- Nanophase materials. Clusters of a few tens to thousands of atoms with unique properties determined by their small particle size, nanophase materials have potential applications in catalysis, pollution control, hazardous waste isolation, and electronics.

Another goal is to involve industry and universities in the Center. Agreements are either in place or being initiated with several institutions including Dow-Corning, Eveready, Hewlett-Packard, Nanophase Technologies, Norton, Raytheon, Saphikon, SEMATECH, Massachusetts Institute of Technology, University of Cincinnati, Cornell University, and the State University of New York at Stony Brook.

"Organoceramics" Contain Molecular-Level Polymers

Composites that contain polymers interspersed at the molecular level in synthetic

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ceramic crystals have been developed by University of Illinois researchers Samuel I. Stupp and Phillip B. Messersmith. Called "organoceramics" by the researchers, the composites could lead to tougher ceramics for such uses as artificial bones or in-body drug delivery systems.

"Traditional composites do not disperse polymers at the molecular level," Stupp said. "This level of dispersion gives you greater potential for achieving new structures with new properties." High-strength materials consisting of inorganic crystals toughened by threaded or layered organic polymer chains could result in composites with the high fracture resistance of polymers, which can also be processed at low temperatures, he said.

One organoceramic was synthesized by sandwiching polymers between layers of calcium aluminate. In soluble form, organoceramics could deliver organic molecules or drugs to an environment, such as in the body. Another synthetic material was based on apatites, the natural mineral of bone and teeth, and holds promise as a hard-tissue replacement.

Changing the nature of the dispersed organic component of the composite could produce other functional materials, such as conducting or piezoelectric materials. "Alternatively, 'smart' materials could be produced that function as sensors of external signals or that react with cells in the body," Stupp said.

Researchers Evaluate Superconducting Magnetic Energy Storage

A workshop convened at the Naval Postgraduate School in Monterey drew suggestions and evaluations from scientists and engineers on possible uses of the Superconducting Magnetic Energy Storage (SMES) Engineering Test Model (ETM) after the co-sponsors, the Department of Defense and the Electric Power Research Institute, make initial tests on the facility.

The ETM is being developed as an energy storage device for electric utility and military applications. According to conceptual designs, the superconducting coil could provide useable magnetic fields from a maximum of about one tesla (10,000 gauss) close to the coil, to a few gauss at the proposed fence line.

The workshop's primary aim was to determine the effectiveness of the ETM for research purposes, and addressed three general areas: materials, electrical/electronic systems and applications, and biological uses. The many potential applications enumerated during the meeting would utilize either the dc nature and large volume of the ETM's magnetic field or the high discharge power capability of the ac-dc converter.

One of the first large-scale applications of high-T_c superconductors will likely be the power leads that carry current from room temperature to a low-T_c device. It was determined that two designs could apply, but many at the meeting believed that existing high-T_c leads would be inadequate because of their limited critical current capability in the combined ETM and self field. (Current density in small samples has little significance for large-scale uses.)

The group recommended the ETM design include space for a pair of high-T_c leads, at 200 gauss in both designs. Characteristics of the leads set a minimum requirement for the development of high-T_c materials for operation at 77 K.

Other suggestions for materials research included use of the large volume of magnetic field for large instrument testing, imaging of large, nonmetallic objects, or processing of magnetically-aligned materials. The high power capability of the power conversion systems for the two proposed ETM designs could be used in applications needing pulsed power such as electromagnetic pulse supplies, plasma reactors, plasma processing, lightning simulators, and flashover breakdown studies.

Research biologists were highly interested in the ETM because the facility could present a unique environment—a controllable dc magnetic field with adequate space and time for experimentation.

Proceedings of the workshop are being prepared for publication and distribution by the Electric Power Research Institute.

Polymer Binding Clue Found

One highly sticky polymer can pin dozens of less sticky ones to a surface, says University of Illinois materials scientist Steve Granick. This insight into why polymers bind or lubricate could aid in solving problems as diverse as the clogging of artificial hearts by blood proteins and the sometimes low efficiency of lubricating oils. Granick used simple polymers in an idealized system to show that polystyrene, a weakly sticky polymer, would be bound to a surface by polymethyl methacrylate, a stickier polymer.

Engineers have traditionally dealt with scum buildup in pipes and clogging in artificial hearts—examples of polymer massing—on an *ad hoc* basis, without a strict research model. Lubricants, for instance, may have more than 100 ingredients, so specific interactions are difficult to isolate. Granick's work, showing that a less adhesive simple polymer could be bound to a surface by a highly sticky polymer, offers a new principle to explain why such chains bind to surfaces, as well as a direction to look for a method to release them.

Other areas that could benefit from this kind of materials research are thin film lubrication between computer hard disk drives and their heads, adhesives, and paints, in which adhesion of pigment polymers to each other is undesirable.

AST Receives Grant to Develop Coatings for Implantable Biosensors

Advanced Surface Technology (AST), Inc., has received a $250,000 Small Business Innovation Research (SBIR) Phase II grant from the National Science Foundation, to develop fluorinated parylene coatings for implantable biosensors.

During Phase I research demonstrating the feasibility of producing fluorinated parylene coatings by plasma-enhanced parylene deposition, the coatings were found to outperform conventional parylene coatings against body fluids. Phase II research will continue with *in-vitro* biocompatibility studies and will focus on the development, evaluation, and optimization of these protective thin coatings on implantable electronics, especially cables and wires, for use with nonhermetically sealed, implantable devices.

Commercial applications for implantable biosensors will be the enhancement of intelligent cardiac pacemakers as well as aids in neurological research. Successful plasma-enhanced parylene coatings would speed the development of sensors for such uses as mobility aids for artificial limbs, artificial kidney assists, cochlear implants, and aids for incontinence.

Single Deposition Source Promises New Generation of Superconducting Thin Films

A thin-film deposition system developed at Argonne National Laboratory uses either a laser or an ion beam under computer control to deposit thin layers of superconducting materials to produce complex multicomponent and multilayered thin films for microelectronic devices.

The ion/laser beam deposition system controls the thickness of each layer from as thin as 0.2 Å average thickness to several thousand angstroms. By controlling the layer thickness, deposition rate, and substrate temperature, scientists can create a layered structure that can either be retained or homogenized to a uniform multicomponent film.

According to developer Alan Krauss, "Because only one deposition source is used regardless of the number of materials being deposited, the production cost is lower than with typical multisource deposition systems. This difference is especially significant for the new high-temperature superconducting materials which may contain four or five metallic elements."

The entire deposition systems is computer-controlled and can be automated. The deposition sequence can be reprogrammed easily, and the sequence can consist of hundreds of individual steps, permitting the production of small numbers of highly specialized devices, prohibitively expensive using more traditional technologies.

Prototype Particle Reducer Aims for Energy Efficient, Low Cost Powder Production

Tests of a prototype particle reducer have shown that in one pass a variety of materials up to one-half inch can be reduced to fine powders. The process works for materials ranging from cement clinker to high purity metallic oxides, producing closely controlled particle sizes both inexpensively and efficiently. Researchers say they can produce particles uniformly sized to a maximum of one or two microns in diameter, and that the prototype unit can easily be scaled up for commercial production and to accept larger feedstock. Tests performed on oil shale, coal, and different minerals showed that material could be reduced to a grain size of 3.4 microns.

Researchers at Cryo Quench Plus suggest potential for their process in any technologies requiring powders, including ceramics, cement, superconductors, polymers, coal, minerals and ores, and even food.

The process is less damaging to the machine and less heat intensive than more traditional methods employing ball mills, hammer mills, roll crushers, grinders, and bar mills. Some materials, such as tires, plastics and biomass, may need to be treated cryogenically to be efficiently reduced, but the complete apparatus need not be cooled. Materials pass through the reduction process and exit before they return to ambient air temperature.

Low power consumption is a key advantage, say the developers. During test runs in the prototype model, 12 pounds of cement clinker were ground to fine powder in one minute using 16.5 kW. This result is equivalent to 13.1 kW hours per ton. Present cement industry ball mills require 36 to 38 kW hours per ton.

Unlike most conventional reduction processes, the prototype technology requires neither residual material reclassification nor reprocessing. In addition, high purity ultrafine reductions and reductions requiring special atmospheres such as argon have been found to be practical using the new process.

Details of the technology, which works on a principle not presently used in industry, are confidential until the patent is granted.

Permacharge, Sandia to Pursue Microcellular Foam Research

Permacharge Corporation and Sandia National Laboratories have signed a cooperative research and development agreement aimed at expanding the commercial uses of microcellular foams developed by Sandia. Permacharge, an Albuquerque-based manufacturer of electret foams for filtration applications, is using Sandia's microcellular foam process to produce filter media in three market areas: (1) high efficiency particulate air filters for hospitals, semiconductor cleanrooms, and computer disk drives; (2) liquid prefilters to extend the life of a membrane filter; and (3) battery separators for improved automobile batteries under development. □

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