Thin-Film Zeolite Membrane Formed by Pulsed-Laser Deposition

Researchers at Dow Chemical Company and Los Alamos National Laboratory have deposited zeolite onto a thin ceramic using pulsed-laser deposition to provide zeolite membranes that maintain an efficient porous structure. Rob Dye, Paul Reis, and Nathaniel Peachy used a laser aimed at a rotating zeolite target in a vacuum chamber. The laser loosens a plume of particles from the target. The plume deposits as a thin film on the ceramic materials. Since the deposited zeolite is so thin and the ceramic substrate is so porous, the material can be used in a chemical process stream.

Industry wants zeolite membranes that can separate gases in a process stream. The zeolite-and-ceramic membranes allow some gases to flow through them, but the zeolites prevent larger complex molecules from passing through.

Zeolites have large, cage-like structures that either physically or chemically trap large chemical molecules within their lattice, allowing the chemicals to react and form other products. The researchers used pulsed-laser deposition to make thin films that have the zeolite's porosity.

Gelled Colloidal Silica and Polysiloxane Barriers Stop Spread of Underground Hazardous Waste

Scientists from Bechtel Corporation and Lawrence Berkeley Laboratory (LBL) collaborated in a field test to demonstrate a process that contains and prevents the spread of underground hazardous waste. To immobilize waste, researchers drilled a string of wells around and beneath the perimeter of the area that was to be contained. They then injected two fluids, a colloidal silica and a polysiloxane fluid, into the wells. The fluids were to permeate the ground before later gelling and forming an impermeable barrier that surrounds the contaminated site.

The colloidal silica performed satisfactorily, while the polysiloxane exceeded expectations. When injected into the earth, polysiloxane has a viscosity similar to water. A catalyst causes it to turn into a strong, rubber-like gel and controls how quickly this occurs. Soil chemistry, which can cause a compound to gel prematurely, apparently has no effect on polysiloxane.

George Moridis, earth scientist at LBL, said, "Not only did we see a uniform, almost symmetrical plume, but everything was grouted uniformly, everything from very large pores to the small pores in clays."

Precisely how long a gel containment would last in the earth must still be determined, although the life expectancy of similar products is 30 to 50 years. During remediation, according to the researchers, even a barrier that lasts only for months can be useful, helping to contain or redirect groundwater flows.

SHS Process Used for Production of TiB_2 and TiB_2-Al_2O_3

Researchers at the Georgia Institute of Technology have developed a self-propagating high-temperature synthesis (SHS) process for manufacturing high-purity titanium diboride (TiB_2) and (titanium-diboride)-alumina composite (TiB_2-Al_2O_3).

Kathryn V. Logan, associate director of Georgia Tech's School of Materials Science and Engineering, said, "Compared to conventionally produced titanium diboride, the chemically based SHS technique yields TiB_2 material with smaller particles, and allows the composite to be produced in final form within molds." Conventional TiB_2 synthesis relies on a solid-state diffusion process that heats boron carbide (B_4C) and titanium dioxide (TiO_2) to a tempera-

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ABSTRACT DEADLINE: AUGUST 15, 1995

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ture of 2000°C. Some carbide material remains as a contaminant in the powder after synthesis, and the process produces large particles that must be ground into finer powder in cobalt-bonded tungsten carbide mills, which add more contaminants at grain boundaries of the TiB_2 particles. Parts made from the contaminated carbothermic TiB_2 must then be hot-pressed at high pressures and temperatures for extended periods to produce the density needed for many applications.

By comparison, the SHS system uses powdered metal of either Mg or Al, TiO_2, and B_2O_3. The materials are mixed and placed into a high temperature crucible. The mixture is then ignited and the reaction between the two oxides and the metal powder proceeds to completion. Once started, the self-sustaining reaction reaches temperatures of more than 2000°C.

The reaction produces TiB_2 dispersed within either MgO or Al_2O_3, depending on the metal powder used. The MgO can be leached from that mixture, leaving only pure TiB_2 in particles that average 0.5 µm. The small particles do not have to be grounded, and because they are not contaminated by carbide materials, can be hot-pressed into finished products at lower temperatures in less time than the conventional materials, thus reducing manufacturing costs.

The TiB_2-Al_2O_3 does not need further processing and offers many of the same properties of the pure material but at a lower melting point. Logan said, "Processing conditions can be varied to produce either a dense composite material or a porous foam into which materials such as molten metals can be infiltrated."

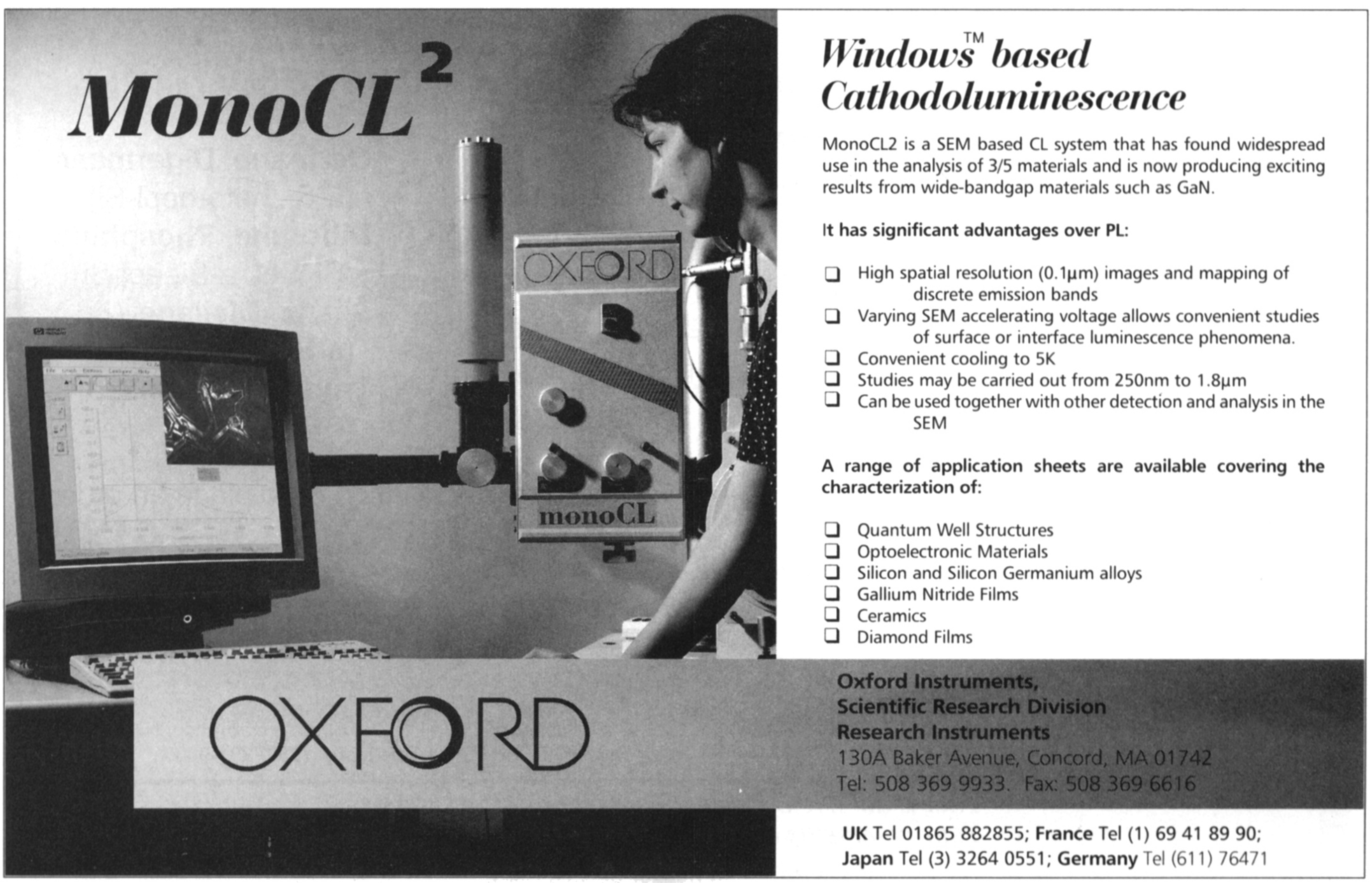
Both TiB_2 and TiB_2-Al_2O_3 offer properties such as electrical conductivity, wear resistance, high compressive and mechanical strength, resistance to chemical reactions, molten metal and thermal shock, high thermal conductivity, and the ability to withstand high temperatures because of its 3000°C melting point. The high wear-resistance of TiB_2 may also benefit companies that now use dies and cutting tools made from materials such as hardened steel, tungsten carbide, or diamond.

Rubbing Rearranges Polymer Strands on LCDs

A team of scientists from IBM's Almaden Research Center and The Pennsylvania State University published a report in *Nature*, specifying that a swipe with a soft cloth over a hard, glassy polymer film similar to that used as a base for liquid-crystal displays (LCDs) causes a substantial realignment of its near-surface molecules. "The rubbing action aligns the jumbled long-chain molecules near the surface," said Tom Russell, polymer scientist at IBM.

In their experiments, the researchers aimed x-rays at thin films of polymers at angles less than a tenth of a degree above the film surface. While nearly all the x-rays bounced off the surface, a few penetrated and collided with atoms within the top 60 Å of the film. The x-rays then bounced away at angles that indicated both the orientation and structure of the near-surface molecules they encountered.

They tested films that were 2,000 and 60 Å thick both before and after a single swipe with a velour cloth. Before rubbing,



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both films showed no surface-molecule orientation. After rubbing, most of the molecules near the surface of the thicker films and nearly all the molecules in the thinner film were realigned within 10 degrees of the rubbing direction.

Russell said, "This is another case where a material's surface properties can be strikingly different from its bulk properties."

Without rubbing, the liquid-crystal molecules are oriented in many different directions, resulting in a useless flat-panel screen. Researchers want to control molecular patterning at the surface, enabling production of displays that use thin LCD films, have fast response and high contrast, and present a clear image.

Pan American Advanced Study Institutes to Begin in 1995

A series of Pan American Advanced Study Institutes, patterned after the successful NATO-ASI series, will be initiated in 1995. The first Institute, "Synthesis and Properties of Advanced Materials," will be held in Merida, Mexico during August 13–25, 1995. This Institute is supported by the Office of Basic Energy Sciences; Department of Energy; the National Science Foundation; the University of Tennessee—Knoxville; Consejo Nacional de Ciencia y Tecnologia (CONACYT), Mexico; the Mexican Academy of Materials; the University of Yucatan, Mexico; and Conselho Nacional de Desenvoluimento Cientifico e Tecnologico (CNPq), Brazil. Additional support from other Pan American sources is expected.

The Advanced Study Institute is a graduate-level teaching activity covering material that has not yet been formally incorporated into the typical university curriculum. The main lectures are tutorial in nature. Additional presentations may be solicited to treat current research activities in a given field. The objective of these institutes are dissemination of the latest knowledge in the subject field, development of international cooperative research efforts, and development of collegial relationships within the Pan American scientific and engineering community.

The program of "Synthesis and Properties of Advanced Materials" will treat the subjects of Diamond and Related Materials, Nanostructured Materials, Biomaterials, Advanced Cementitious Systems, Structural Ceramics, Composites, Advanced Polymeric Materials, Advanced Magnetic Materials, and High-Temperature Superconductors. The faculty consists of five lecturers from each of the United States and Mexico and one from Canada. The Fellows attending will be advanced graduate students, post-doctoral students, and faculty or industrial investigators who wish to broaden their background and/or establish contacts with other investigators in the Western Hemisphere. Attendance will be limited to approximately 75 Fellows. Scholarships are available to assist graduate and post-doctoral students.

Further information can be obtained from Carl J. McHargue, Director, Center for Materials Processing, 102 Estabrook Hall, University of Tennessee—Knoxville, Knoxville, TN 37996-2350; phone 615-974-7680, fax 615-974-4995, e-mail CRL @utkvx.utk.edu.

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Near-Infrared Spectroscopy Detects Counterfeits

Don Burns from Los Alamos National Laboratory applied for a patent to use near-infrared spectroscopy to distinguish genuine from counterfeit American currency and turquoise stones found in Southwest jewelry. With this technique, light, just beyond the visible spectrum, shines through a fiber optic cable onto a sample that reflects the light back to sensors that feed the information to a monochromator, then to a computer.

The monochromator acts like a prism and breaks up the light into many wavelengths. The makeup of every material creates its own reflectance pattern, much like a fingerprint. The computer then analyzes the pattern, matches it against stored patterns, and provides an analysis of the material's composition.

In order to recognize a pattern that relates to a particular material, Burns has built a software computer library stocked with information gathered through experimentation. To build the library, Burns places a known substance in an enclosed chamber, such as a certain grade of plastic. The plastic reflects the light from the device and sends a specific pattern of absorbances to the monochromator. The computer's memory permanently records the pattern for that item.

The technology tested on currency exhibited dramatic differences in patterns of counterfeit and genuine bank notes. This technology also readily exposed differences between fake turquoise made from plastic and real stones fixed with resin stabilizers, which are used to make the stones workable. Burns plans to test stones with known levels of various resins against the pure plastic gems to determine whether the device can distinguish between differing grades.

Bonding Model Illuminates Fusion Bonding Mechanisms in Thermoplastic Composites

Christine A. Butler, graduate student at the University of Delaware, has developed a bonding model that can be used to optimize fusion bonding processes at polymer-polymer interfaces. Butler's coupled bonding model accounts for variability in initiation time for healing due to a growth in the area in intimate contact. Butler said, "Previous healing models give an idealized healing based on total intimate contact. These theories are based on the assumption that the entire weld surface is fully contacted initially and that healing is therefore uniformly initiated. In reality, the amount of area in contact increases with processing time, resulting in a *distribution* of initiation times for healing across the weld interface."

A key feature of Butler's model is a scaling parameter that distinguishes between intimate-contact-dominated and healing-dominated processes but can also describe conditions under which the mechanisms are coupled. The model has been experimentally verified in resistance welding of high-temperature amorphous polyimide thermoplastic laminates (Avimid®K3B/IM7 coated with a resin-rich layer of Avimid®K3A, DuPont) and the tow-placement process using AS-4/PEKK prepreg tow.

Butler said, "The welding process is intimate-contact dominated, in which case the coupled model reduces to a more simplified form. The processing region

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for two placement, on the other hand, falls within the coupled regime." The ability to rigorously determine the controlling mechanisms is critical in accurately modeling strength development during any fusion-bonding process.

Roy L. McCullough, professor of chemical engineering at the University of Delaware, said the model can be used "to assess the quality of a bond made through various joining techniques and optimize the bonding process."

Butler's work has also provided information on the effects of crystallinity on bonding during high-speed processes such as tow placement. According to Butler, debonding may occur as a result of crystalline growth near the interface, pulling previously diffused chains back across the interface.

—Modified from *Composite Update*, a publication of the Center for Composite Materials at the University of Delaware.

Recently Announced CRADAs

Kent Development Corporation (Houston, Texas) and **Los Alamos National Laboratory** (Los Alamos, New Mexico) signed two $100,000 agreements to characterize a new, automated heat treatment machine for metal parts and powders used in medical prostheses, jet engines, and other products.

Lawrence Berkeley Laboratory (Berkeley, California) and **Air Techniques, Inc.**, (Hicksville, New York) signed a $100,000 agreement to develop a dental electronic readout device, by using a cesium ionide scintillator instead of film, minimizing patients' exposure to x-ray radiation.

Microwave-Affected Driving Force for Electrochemical Diffusion Enhances Ionic Transport in Ceramics

Researchers at the University of Wisconsin-Madison have identified a new driving force for ionic transport in ceramic solids that uniquely occurs when the material is irradiated with high power microwave fields. Graduate student Sam Freeman and his professors John Booske from the Electrical and Computer Engineering Department and Reid Cooper from the Department of Materials Science and Engineering observed the effect while measuring ionic currents in halide salts. The experiments were designed to measure whether microwave radiation enhances ionic mobility in ceramics as compared with conventional furnace heating.

This notion of enhanced ionic mobility due to microwave radiation had been previously hypothesized based on numerous claims by other researchers that microwave heating of ceramics and glasses results in apparent activation energy reductions (in comparison with conventional furnace heating) for sintering, bonding, and ionic diffusion of radiogenic tracers. The Wisconsin researchers discovered that although ionic transport is enhanced by microwave radiation, the phenomenon is due to an additional microwave-affected driving force for electrochemical diffusion and that the microwave fields apparently do not affect the concentrations or intrinsic mobilities of the lattice defects responsible for diffusion. Illustrative results were recently reported in *Physical Review Letters* based on experiments with NaCl as well as with AgCl, reported at the 1995 American Ceramic Society Meeting. The experimental measurements are consistent with a recent theoretical model for an effective "surface pressure" that results from the interaction of the microwave radiation field with the near-surface space charge layers associated with mobile point defects such as vacancies.

Follow-up experiments have been designed to evaluate the effect that this new driving force has on bulk or grain boundary diffusion as well as for the overall reaction kinetics of (pseudo)binary compound formation. The results of these experiments will be directly applicable to understanding whether (and how) microwaves accelerate solid-state chemical and microstructural reactions in comparison to conventional furnace heating, and how best to employ the effect of the processing of commercial ceramics and glasses. □

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