Boron Nitride Matrix Used for Heat-Resistant Material

A recent study of boron nitride composites showed them to be significantly better than composites of carbon fibers in amorphous carbon as heat-resistant products, a University of Illinois scientist reports. James Economy, leader of the study, and his group found the boron nitride matrix stronger, resistant to higher temperatures, and quicker to make.

For 25 years, industries have depended on the lightweight, strong, and heat-resistant carbon/carbon material, but baking a carbon matrix around carbon fibers can require weeks or months of repetitive charring and impregnation during the manufacturing process. Such composites also tend to oxidize, and thus deteriorate, during repeated use at high temperatures.

Economy's group found that carbon fibers in a boron nitride matrix can be created in a single baking that takes less than two days. The resulting composite can withstand temperatures roughly twice as high as carbon/carbon composites before oxidizing. While much stronger, the boron nitride matrix is also relatively transparent to a range of probing electromagnetic beams, thus permitting the design of composites that are difficult for electronic sensing devices to detect. Such heat-resistant composites would be desirable in the making of advanced military aircraft.

Another advantage of the new matrix material is the way it oxidizes, according to Dongpyo Kim, postdoctoral research associate. Silicon carbide often is applied to carbon/carbon composites to prevent oxidation. If a scratch or pinhole develops in the coating, the coating deteriorates rapidly. But in the carbon/boron nitride composite, oxidized boron nitride forms boron oxide, a viscous liquid that fills the pinhole, thus protecting against further deterioration, Kim said.

Borazine, the substance from which boron nitride is derived, yields more than 90 percent boron nitride when baked, a finding that means multiple processing steps are unnecessary. Borazine, however, is available only in small quantities.

The University of Illinois has applied for a patent on the process and the composite. The work has been supported by the Defense Advanced Research Project Administration and the Office of Naval Research.

Lone Peak to Develop Materials Lab Kit

Lone Peak Engineering, Inc. has received a Phase I SBIR grant from the National Science Foundation to develop an "Interactive Materials Science and Engineering Lab Kit" to help educate

https://doi.org/10.1557/S0883769400047047 Published online by Cambridge University Press

students in materials science and engineering labs. The kit will allow students to compare the theoretical strength and actual strength of different high-technology materials. It will provide students with ready-to-use test bars designed to help them better understand the strength characteristics of linear elastic brittle materials, such as ceramics. This approach will be combined with an interactive computer program that will graphically present the scientific theories behind the hands-on work performed by the students.

This interactive learning style will give students quick, concise, well-defined information about how the size, shape, and position of a flaw would affect the strength of various materials.

Uniform and consistent test bars will be produced out of advanced materials that contain controlled flaws. Students will then be able to relate flaw characteristics to both theoretical and practical strengths and work with materials they may encounter in future careers. This kind of mechanical property evaluation is now conducted in university laboratories with simple materials, such as glass rods, which would not be considered structural materials in high-technology applications.

Interested persons can contact JoDee Daufenbach, Lone Peak Engineering, Inc., 1270 West 2320 South Suite F, West Valley City, UT 84119; telephone (801) 975-7979.

Researchers to Develop High-Temperature Composites of Molybdenum Disilicide and Silicon Carbide

Researchers from Los Alamos National Laboratory (LANL) and Advanced Refractory Technologies, Inc. (ART) will work together on new high-temperature composite materials of molybdenum disilicide and silicon carbide whiskers that could result in better industrial furnaces, incinerators, gas turbines for aircraft, automobiles and power generation, and other components operating in high temperatures. Under a cooperative research and development agreement, "Los Alamos and ART plan to develop a commercially viable process for producing composites with the best properties of both materials," said ART president Keith Blakely. Under the agreement, ART will provide Los Alamos with silicon carbide whiskers and blends of silicon carbide whiskers and molybdenum disilicide, along with related materials from the company's Buffalo plant. Los Alamos will process, fabricate, test, and evaluate the blends and related materials for potential commercial applications.

Molybdenum disilicide is an ordered

compound with shared metallic and ceramic properties. Although brittle at room temperature, the compound is highly resistant to oxidation and capable of deforming without breaking at high temperatures. By dispersing silicon carbide throughout the molybdenum disilicide matrix, Los Alamos scientists in 1989 created a new class of high-temperature composites that remain intact in environments destructive to other materials. These high-temperature structural silicides combine the strength, oxidation resistance, low density, and low thermal expansion of ceramics with the high-temperature ductility and fracture resistance of metals.

Molybdenum disilicide deforms at high temperatures because atoms at dislocations in the crystal move when a stress is applied. Silicon carbide increases strength by pinning the atoms at dislocations when they are stressed. The Los Alamos composites are much stronger than superalloys based on nickel and cobalt and more ductile than structural ceramics. Blakely said that they think the composites will have significant potential for applications in oxidizing environments at temperatures between 1200 and 1600°C. The composites also will be less expensive to manufacture than silicon-based ceramic materials and can be alloyed fairly easily with other metals to improve their properties, Blakely said.

Commercial use of molybdenum disilicide has been limited due to the deformation or "creep" of the material at high temperatures. By reinforcing the material with silicon carbide whiskers, ART expects to improve the resistance to creep as well as other properties of molybdenum disilicide, such as its fracture toughness at room temperature.

Steel for Use in Fusion Reactors Studied

Using tungsten, vanadium, and tantalum, instead of molybdenum and niobium, as alloying elements in steel, researchers from Oak Ridge National Laboratory (ORNL) are developing radiation-resistant steels in which the radioactivity decays more quickly than in conventional steels. Testing numerous steels in radioactive environments over the past 20 years, the researchers studied two obstacles. First, irradiation causes nuclear transmutation reactions that induce radioactivity in the steel, incurring long-term radiation, and second, irradiation causes the steels to become brittle.

Radioactivity in the new steels decays to low levels in tens of years instead of thousands of years, as in conventional steels. Ronald Klueh, a researcher with ORNL's Metals and Ceramics Division, said, "These steels can then be considered for shallow land burial after a reactor is dismantled, instead of the much more expensive deep geological disposal."

The new steels were irradiated in fission reactors to simulate a fusion environment, since no fusion reactors are available for testing materials. Tests following irradiation showed that the new steels become less brittle by irradiation than the conventional steels they were developed to replace.

Sintered Polycrystalline Diamond Maintains Thermal Stability at 1400°C

Japan's National Institute for Research in Inorganic Materials (NIRIM) of the Science and Technology Agency (STA) and Mitsubishi Materials Corporation have jointly developed a sintered diamond (produced by high-pressure, high-temperature sintering using natural diamond powder) that is capable of withstanding temperatures as high as 1400°C.

Two and a half years ago, the research team of NIRIM, headed by Minoru Akaishi, succeeded in developing a series of nonmetallic catalysts (carbonates, sulfates, and hydroxides) for diamond synthesis which completely differ from the conventional types of metallic catalysts developed by General Electric.

It was found that carbonates (Mg, Ca, and Sr) of alkaline earth metals are highly effective as sintering agents among 11 inorganic compounds. Diamond powder was subsequently laminated onto these carbonates and then sintered for 30 minutes at 2300°C under a pressure of 7.7 GPa, providing sintered diamond with a homogeneous microstructure.

Scanning electron micrographs show that it is difficult to discriminate the grain boundaries of the diamond particles, and that the mode of fracture is based mostly on intergrain fracture, suggesting that numerous direct bonds are formed between the diamond grains. Further, no abnormal growth has been observed in the sintered body, indicating that it is a sintered diamond with a homogeneous microstructure. The average Vickers hardness is over 70 GPa (normal load: 19.6 N), which is almost equal to that of a single-crystal diamond.

In order to investigate its heat-resistance capability, the sintered body was heat-treated for 30 minutes at 1200°C. It was subsequently confirmed that no graphitization or cracking could be observed. Although many cracks and large amounts of graphitization have been observed on conventional sintered bodies with a heat treatment of 900°C for 30 minutes, no graphitization or cracking was detected on the new sintered diamond, despite a heat treatment at over 300°C higher than the conventional heat-treatment temperature.

After displaying such heat resistance at 1200°C, the new sintered body was heat-treated for 30 minutes at 1400°C with no graphitization or cracking observed. The heat resistance of the sintered diamond was significantly improved by using carbonate as a sintering agent in the production process, without any use of a metallic sintering agent such as Co. This shows that the new sintered diamond—with its small amount of carbonate—has a superior resistance to both cracking and graphitization. Although the carbonate decomposes into oxide and carbon dioxide upon high-temperature treatment, the oxide formed eliminates any graphitization of the diamond.

The new sintered diamond was used to fabricate cutting tools, which feature a higher wear resistance and fine cutting surfaces, and were found suitable for practical high-speed cutting of high Si-Al alloys (18 wt% Si). They are also applicable to the practical cutting of WC-Co alloys (6 wt% Co). Expectations are that the new sintered diamond also can be applied to the cutting of other hard ceramic materials.

It is expected that high Si-Al alloys will become more commonly used in the automotive industry to reduce weight, creating a demand for cutting tools capable of working with these materials. Thus the new sintered diamond which uses nonmetallic carbonate as a sintering agent has the potential to gain wide acceptance as a material for producing diamond cutting tools which must provide excellent thermal resistance.

F.S. Myers

IES Forms Committee to Communicate Environmental Effect on Technology Transfer

The Product Reliability technical division of the Institute of Environmental Sciences (IES) has formed a Commercial Applications Technical Committee (CATC) to collect and disseminate information about the effect of the environment on the transfer of technology developed for government and military use to commercial products. CATC scheduled its first organizational meeting for May 5 at the IES 39th Annual Technical Meeting and Exposition in Las Vegas.

The new committee will concentrate on the shift of military and government applications to commercial product development in such areas as consumer electronics and appliances; automobiles; business equipment; safety, security and health devices; airline and general aviation electronics; and communications equipment.

With the increased emphasis on environmental disciplines in the commercial sector, the CATC's purpose is twofold: to help product designers develop high-quality products by giving them information about the effect of environmental stress on their designs, and to assist manufacturers of equipment for government and military use in adapting products to the commercial arena. Some of the CATC's specific areas of interest include:

- Streamlining product development by tailoring environmental stresses;
- evaluating the effectiveness of environmental stress applications from product development through the manufacturing process;
- adapting equipment developed for government and military applications for use in commercial applications; and
- characterizing end-use (consumer) environments for commercial products.

For more information regarding the Commercial Applications Technical Committee, or IES, call (708) 255-1561, or fax (708) 255-1699.

Coherent, IBM to Work on Blue-Laser Devices

Coherent, Inc. and International Business Machines Corporation (IBM) have announced an agreement relating to the development of frequency-doubled semiconductor diode laser devices operating at blue wavelengths. Under the terms of the agreement, IBM will provide Coherent with a license to use blue-laser technology developed at the IBM Research Division's Almaden Research Center, along with the rights to use some of IBM's patents. In addition, Coherent and Almaden have agreed to work closely with each other to move this technology from the laboratory to the marketplace. Bernard Couillaud, vice president and general manager of Coherent's Laser Group, said, "We believe that this type of blue-laser device can be used for analytical and medical instrumentation, inspection and measurement, printing, and optical data storage applications."

Over the past three years, IBM's Almaden scientists have invented and developed a new technology for efficiently converting the output of an infrared diode laser to blue light. The device prototype can produce 50 mW of 428 nm blue light with a beam quality suitable for high-density optical data-storage applications. In this design, 856-nm infrared light from a commercial semiconductor diode laser passes through a special crystal, efficiently doubling the frequency of the infrared light, converting it into blue light.

Because blue light can be focused to a spot having half the diameter of the infrared light used in current optical data storage devices, its use offers the possibility of quadrupling the areal density at which data can be stored on an optical disk. The collaboration with Coherent may also lead to new blue-laser devices for less demanding analytical and medical instrumentation, inspection, and measurement, as well as printing applications.

ASME Calls Thermal Engineering Research "Critical"

By strengthening support for thermal engineering research, the federal government can help shed light on global change issues, help improve the manufacture of superconducting materials, and aid in the design of cleaner, more efficient electrical generating plants, according to a task force of the American Society of Mechanical Engineers (ASME). "Thermal engineering, which deals with the flow of fluids and the transfer of heat and mass, is of fundamental importance to the future of the United States," says a policy statement prepared by experts from the ASME Heat Transfer Division and sent to Congress and relevant federal agencies in mid-January. "If the United States is to maintain or enhance its competitive position in the world marketplace," says the ASME, "it is critical that the importance of thermal engineering be recognized for its vital role as a fundamental component

https://doi.org/10.1557/S0883769400047047 Published online by Cambridge University Press

of current and future research, development, and design initiatives."

Significant advances in thermal engineering research can contribute to critical technologies in energy and the environment, manufacturing and materials processing, and bioengineering and biotechnology. Major thermal research areas identified by the task force as having an impact on each of these critical technology fields are multiphase thermal phenomena; thermal processing of engineered materials; and ultralarge- and ultrasmall-scale technology. "Systems containing more than one phase (solid, liquid, or vapor) or more than one component contain interfaces between the phases or between the components," the statement notes. "Advances in our knowledge of the behavior of such systems will result in higher-efficiency energy production systems, improved heat exchanger design, safer nuclear reactors, enhanced treatment of hazardous wastes, and more cost-effective clean-up of chemical spills."

Creating advanced materials with special thermal properties is crucial for the health of the aerospace industry, for the development of improved heat exchangers and industrial furnaces, and for use as environmentally benign refrigerants. While in most cases, thermal processing alone will not give the desired results, the statement says that "in many engineering applications the thermal properties of the selected materials are of critical importance."

In addition to brief discussions of the three cross-cutting areas of thermal engineering research, the statement lists significant research areas for each cross-cutting field, arranged by critical technology. Copies of the statement are available free of charge from Government Relations, ASME Washington Center, 1828 L Street, N.W., Suite 906, Washington, DC 20036-5104, or by calling (202) 785-3756.

ISTEC Develops Improved YBCO Josephson Junction

In Japan, the ISTEC group headed by Katsumi Suzuki has developed a superconducting Josephson junction in which the I_cR_n value (I_c is the critical current, and Rn is the normal resistance) is two orders larger than that previously reported. The I_cR_n of this new structure junction (an important parameter used in electronics devices) is reproducibly acquired at between 10 and 50 mV.

This new structure is different from previous ones in that it has a flat, lateral junction on a flat surface of the substrate, not the usual step-, groove-, layer-type structure; also, the crystal lattice orientation in the junction region is the same as that of electrodes contacting the junction, as compared to the bicrystal substrate-type structure.

The Josephson junction implemented in $YBa_2Cu_3O_{7-\delta}$ (YBCO) microstipline is fabricated on a flat surface of a MgO substrate by utilizing a focused ion beam (50-nm-beam diameter). In the fabrication process, $Ga+$ ions are implanted 100 nm wide and 10 um long) into a substrate such as MgO, 300-nm-thick YBCO film is deposited on the substrate by pulsed laser deposition (PLD), then the YBCO film is patterned to be a 5-μm-wide stripline over the implanted region.

The researchers said that satellite broadcasting should be enhanced as a result of this accomplishment. This research was published in Advances in Superconductivity V, Proceedings, ISS'92, Kobe, Japan (Springer, 1993) ADP-014; it will also be published in *Physica-C* and *Japanese Journal of Applied Physics* sometime soon.

F.S. Myers

Alfred Establishes Frechette Chair

Van Derck Frechette's achievements in ceramic engineering and glass science have been recognized by the New York State College of Ceramics at Alfred University. Alfred, where Frechette has spent more than 50 years, first as a student and then as a professor and researcher, has created the Van Derck Frechette Professorship in Ceramic Science, an endowed chair in the College of Ceramics.

"Van Derck Frechette's contributions to Alfred University and the New York State College of Ceramics are consider-

https://doi.org/10.1557/S0883769400047047 Published online by Cambridge University Press

able," said James W. McCauley, dean of the College of Ceramics, citing the quality of Frechette's teaching and research.

Arriving at Alfred from Ottawa in 1936, Frechette earned his BS degree in ceramic engineering and technology, then left Alfred to pursue master's and doctoral degrees in ceramic engineering at the University of Illinois. He then spent two years as a research physicist at the Corning Glass Works. In 1944, Frechette returned to Alfred University as a professor of ceramic technology, spending the next 43 years in the classroom. Although he officially retired in 1987, Frechette continues his research and occasionally teaches.

A pioneer in the field of fractrography of glasses and ceramics, Frechette was frequently called on as an expert witness in court trials involving the breaking and shattering of glass. His research in advanced ceramic materials, sponsored by the Air Force in the 1950s, was among the first research projects to receive outside grants. As an educator, Frechette saw the need to expand the horizons of ceramic engineering students, and began setting up international exchange programs in the 1950s. Until his retirement, Frechette continued to oversee the college's Study Abroad program.

More Flexible Composite Fiber Developed

A Georgia company has begun producing a prepreg—a thermoplastic-impregnated composite in continuous fiber form—that will lower the cost of manufacturing complex composite components, improve the performance of finished parts, expand the range of a material's applications—and reduce environmental concerns. Developed and patented by the Georgia Institute of Technology and licensed to Custom Composite Materials, Inc. (CCMI), the prepreg is flexible enough to be woven, braided, or wound into three-dimensional shapes, using automated textile manufacturing techniques.

Tim L. Greene, manager of applications for CCMI, said that the material can be processed with conventional textile production techniques, rather than by more costly manual methods. After preforming, the prepreg is placed in a mold under pressure to form a consolidated part which may be stronger and lighter than a similar metal part. Previously such composites were more costly due to the labor and exacting production processes involved.

In the CCMI process, a spread bundle of continuous fiber is passed though an electrostatically-charged cloud of powdered resin. The resin adheres to the bundle, which then passes through an oven to fuse the resin onto the fiber. The resulting material can be processed with standard textile equipment or even tied in a knot. Once woven, braided, or otherwise formed, composite structures made with the prepreg are finished through a consolidation process that melts the resin around the fibers to produce the desired solid part. Other processes result in stiffer materials, require comingling of the resin and structural fibers, or pose environmental and storage problems because they use solvents. The new process results in improved production-line flexibility, where the types of resins and fibers can be quickly changed to suit the product.

Because the powdered resin thoroughly infiltrates the fiber bundle, the new process can improve the quality of composite components by minimizing voids left in composite structures and by ensuring uniform mixing of the resin with the fiber, said John Muzzy, a Georgia Tech chemical engineer and a developer of the process. Cycle times are reduced and processing temperatures and pressures can be cut, leading to significant energy savings. The new technique provides "broad material utilization capabilities with this one basic technology," he said, noting that it can be used with any thermosetting resin that can be ground into a powder, and will even allow the use of materials that now cannot be processed with conventional composite methods. □

Send BULLETIN News to:

Editor, *MRS Bulletin*, Materials Research Society
9800 McKnight Road, Pittsburgh, PA 15237
FAX: (412) 367-4373

Erratum

An inaccurately drawn figure appeared in the article "Quantitative Auger and XPS Analysis of Thin Films," by J.M. Slaughter, W. Weber, G. Güntherodt, and C.M. Falco (December 1992, Vol. XVII, No. 12). Below is the correct version of Figure 6, which supports the conclusion drawn in the text that "Apparently an exponential ansatz cannot be fit to the data points, whereas a fit with straight line segments works much better."

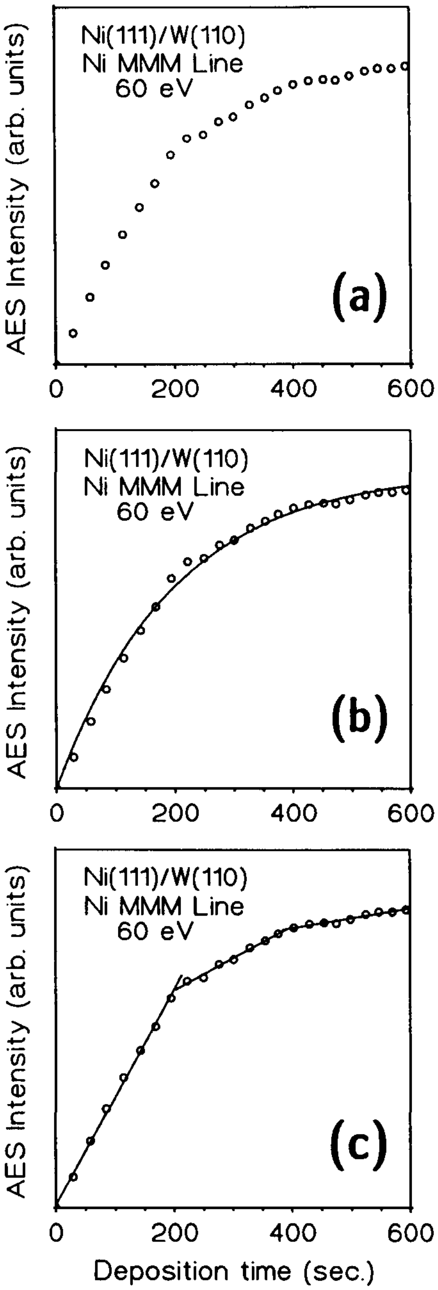

Figure 6. Auger electron spectroscopy (AES) intensity of the Ni MMM line (60 eV) as a function of the deposition time. (a) Raw data, (b) raw data plus exponential fit, and (c) raw data plus straight line fit.

https://doi.org/10.1557/S0883769400047047 Published online by Cambridge University Press