High-Temperature Strength Achieved Using Fiber-Reinforced Ceramic

Japan's National Aerospace Laboratory (NAL) under the Science and Technology Agency (STA) announced in May the development of a heat-resistant composite material that can withstand a stress of 3.5 tons per cm^2 at temperatures as high as 1200°C. Research on the lightweight oxidation-resistant fiber-reinforced ceramic was reported at ICCM-9 (9th International Conference on Composite Materials), held in Madrid, Spain in July.

This research was carried out by groups headed by Takashi Ishikawa of the Airframe Division of NAL; Takemi Yamamura, Director of the Inorganic Fiber and Composites Group, Inorganic Materials Research Laboratory, Ube Industries, LTD; and Tetsuro Hirokawa of the R&D Department, Industrial Textile Division, Shikibo, LTD. NAL was in charge of defining the basic idea and evaluating the mechanical properties (such as tensile strength, fracture toughness, and interlaminar shear strength); Ube Industries, LTD provided the fibers and conducted consolidation of the matrix; Shikibo, LTD wove these fibers into three dimensional reinforcing fabrics.

According to Ishikawa, it may be possible to commercialize the ceramic—which is highly suitable for use in jet engines and space shuttles, such as the planned Japanese space shuttle HOPE—within two or three years. The material is made of 9-micron-thick silicon carbide, which contains titanium (registered under the tradename Tyranno, a patented product of Ube Industries). The fibers are first woven into three-dimensional fabrics, then dipped into a kind of melted poly-titano-carbo-silane and baked at a temperature greater than 1000°C. This process is repeated in order to create densified ceramic composites. The density of the ceramic is about one-fourth the density of the conventional heat-resisting nickel alloys currently used in jet engines.

According to Ishikawa, oxidation-resistant ceramic/ceramic composites made of Si-Ti-C-O fibers (Tyranno) and three-dimensional fabric reinforcement have been developed. Two types of matrix consolidation processes, polymer conversion (PC) and chemical vapor infiltration (CVI), have been attempted. The room-temperature ultimate tensile strength of one type of PC material reaches an exceptionally high (average) value of 400 MPa, which is more than twice that of the preceding record CMC systems produced by SEP (a French company). Ishikawa says that it is very significant that a PC material has realized the best performance, due to the incredibly high fabrication cost of a CVI material.

In addition to the high strength, the CMC also shows a very large ultimate strain of about 0.9%. Its density is 1.8 g/cm^3. Preliminary high-temperature strength tests at 1200°C have revealed strengths almost as high as those at room temperature, unless they fail in the shear-out mode. Fracture toughness tests have exhibited a K_{IR} as high as 40 MPa $m^{1/2}$. Also, the material shows a great advan-

tage in interlaminar shear strength due to its three-dimensional reinforcement structure. An intensive effort to apply this CMC to future space transportation systems is presently being undertaken in Japan. Other possible applications would be for hypersonic aircraft, supersonic engines, and efficient energy generators. Aircraft-related applications include nose sections, thermal protection systems, or even some primary structures, says Ishikawa. This material is considered superior to the present ceramic heat shields of the U.S. space shuttle, which are several centimeters thick, often fracture, and flake off during entry into the Earth's atmosphere.

F.S. Myers

ERATO Announces New Projects

Every June, the Science and Technology Agency's Research Development Corporation of Japan announces its new projects, which will start up the following fall. Funded at about U.S. $3 million per year for a five-year period, the ERATO program is the most prestigious and highly financed individual research program in Japan, and perhaps in the world. Potential project leaders do not apply, but are scouted and selected by a talented group of bureaucrats and a very broad community of well-respected scholars from government, industry, and academics.

Each project is centered around a project leader who continues his/her present job while assembling and stimulating an international collection of young and talented researchers. Although all projects end after five years, successful research is usually resurrected in a metamorphosed form under another sponsorship. JRDC also provides the expertise to further develop and commercialize any potentially interesting results.

This year, JRDC selected four new projects, three involving materials science and one concerning biological science. One project is the Yamamoto Quantum Fluctuation Project, headed by Hoshihisa Yamamoto (see *MRS Bulletin*, February 1993, page 10). Yamamoto, who has received numerous awards and has worked in Japan, Europe, and the United States, has been on leave from the Musashino Electrical Communications Laboratories at NTT since 1992, serving as a professor of applied physics and electrical engineering at Stanford University.

Over the next five years, the Yamamoto Quantum Fluctuation Project will research the relationships between the quantum fluctuations of semiconductor laser light and the electron processes that produce this light, based on the tradeoff between the intensity noise and phase noise, the relationship of which is defined by Heisenberg's uncertainty principle. One research group will look at the so-called "squeezing" of light, while exploring the use of microcavities to control this phenomenon. It will also study the interactions between squeezed light and atoms while working toward the development of accurate atomic spec-

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troscopy and wavelength standards. A second group will study the nondestructive measurement of quantum systems. Techniques will be explored using the optical soliton and electron interferometry while extending experimental research to wave compression and quantum interferometry. A third research group will be seeking to control the injection of single electrons into microscopic semiconductor junctions while conducting research toward the production and control of single photons emitted from these junctions.

Another project is the Tanaka Intersolid Fusion Project, headed by Shunichiro Tanaka, a member of Toshiba Corporation's Materials and Device Laboratory. The main focus of Tanaka's project will be the basic physics of interfaces between differing solids. One research group will investigate the atomic processes that occur when solids are fused together, while looking for chemical reactions at the interfaces, and at how atoms rearrange themselves during fusion. The group will also seek ways to control the fusing interface, using applied stresses as well as electric and magnetic fields. A second group will explore the theory behind the fusion interface, using computer calculations to better understand electronic and other processes that take place during fusion. Based on this understanding, an effort will be made to simulate the fusion process and, thereby, begin to design solid-state interfaces.

The third materials project is the Hashimoto Polymer Topophase Project, which will concentrate on designing and synthesizing new block polymers and other materials. Real-time measurements will be undertaken to understand the physics and processes that occur when these complex liquids separate into differing phases. The influence of external conditions—such as electric and magnetic fields, processing flows, and solvents—on the formation of these special structures will also be pursued.

The new Hirohashi Cytoadaptation Project involves elucidating the shapes of cells as expressions of their inner genetics and of the influences of surrounding cells. One group will look at the genes that affect shape and binding, and another group will look at the processes by which the genes achieve this end.

F.S. Myers

Diamond Produced by Heating Polymers

A team of Penn State chemists have made a polymer that readily turns into diamond and diamondlike carbon when it is heated to the 200–400°C range, according to Patricia A. Bianconi, assistant professor of chemistry. "I believe this is the first method for making diamond from a soluble, solid-phase, nondiamond material, and it works at normal atmospheric pressure with relatively low temperatures," she said.

Bianconi's method involves a new class of carbon-based polymers that she and graduate student Glenn T. Visscher developed. She says the polymer, which looks like brown sugar, has a natural tendency to turn into diamond when it is heated slightly because some of its atoms are already in a diamondlike arrangement. Bianconi and Visscher, along with graduate student David C. Nesting and assistant professor of chemistry John V. Badding, describe their work in the June 4, 1993 issue of the journal *Science*.

Bianconi said the new polymer is also the first protodiamond material that can be dissolved in a solvent. She speculates that objects could be coated with this polymer solution and then heated, covering them with a protective diamond film. The polymer could be used in manufacturing a wide variety of products, including superhard abrasives, wear-resistant protective coatings, and computer chips with superdense circuits. Diamond has the highest melting point of any known material, so more circuits could be packed onto a diamond-based chip without the risk of heat damage. In addition, Bianconi said, "People who work in this field say that if you want to build a faster computer chip, you should make it out of diamond, which is an extremely quick carrier of electrical pulses."

Manufacturers could eventually transform the polymer with the heat from a laser beam to "directly draw diamond circuits onto polymer-coated computer chips," Bianconi said, adding that this method, if it proves feasible, would be less likely to damage the chips than some conventional diamondmaking techniques.

The atomic structure of the new polymer, which the Bianconi team has named polyphenylcarbyne, consists of an interlinked base of carbon atoms with R groups, forming an interlocking network structure similar to diamond. Bianconi said this is the first carbon polymer whose base is a tetrahedral network rather than a linear string of atoms. "Because it is already tetrahedral, it doesn't need much energy to convert to the tetrahedral diamond structure," she said.

Heating this polymer to between 200 and 400°C breaks the bonds to the R groups, which fall away, leaving a core of carbon atoms that are each missing a single bond. As these carbon atoms link up with each other, they form the tetrahedral crystal structure of diamond. Heating ordinary carbon polymers that have exactly the same formula but a different geometry from the new polymer produces only graphite.

Microscopically, the material reveals a patchwork of transparent granular areas interspersed with solid black areas. Raman spectroscopy showed that the black areas are microcrystalline or amorphous diamond coated with graphite and the transparent regions are diamond crystals. "We think the graphite is coming from stray double-bonded carbon atoms left over from some of the R groups," Bianconi said. The double bonds predispose the atoms to form graphite rather than diamond.

"We purposely used the most crude processing methods to demonstrate that the structure of this tetrahedral carbon polymer primes it to turn into a tetrahedral carbon solid, the most perfect form of which is diamond. We are experimenting with a polymer whose R groups have no carbon-carbon double bonds at all, which we hope will give us a lot less graphite," Bianconi said.

Drzal to Receive Adhesion Society Award During Special Session

The Adhesion Society Award for Excellence in Adhesion Science for 1994, sponsored by 3M Company, will be presented to Lawrence T. Drzal at the annual meeting of the Society in Orlando, Florida in February. Drzal is professor of chemical engineering and materials science and mechanics, and director of the Composite Materials and Structures Center at Michigan State University. He was cited "for advancing the understanding of adhesion phenomena in fiber-reinforced composites and for the development of methods to quantify these phenomena."

Drzal's research focuses on surface and interfacial aspects of adhesively bonded joints and fiber-matrix adhesion in composite materials. The thrust of his research is directed at understanding how fiber-matrix adhesion affects mechanical properties, composite processing, and long-term performance. An important contribution has been the development and application of the "interphase" concept to the study of fiber-matrix adhesion. Results from his laboratory have shown that fiber-matrix adhesion is due to the cooperative interplay of the fiber structure in the surface region, the fiber surface chemistry, and the polymeric matrix interaction with the fiber surface.

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Invited and contributed presentations will be given at the Adhesion Society Annual Meeting in recognition of Drzal's understanding of fiber-matrix adhesion. Contributions are sought from individuals describing the influence of Drzal's work on their own investigations involving interfacial phenomena in composites. Abstracts are still being accepted. Interested parties should contact: Dr. Larry Peebles, 8204 Mockingbird Drive, Annandale, Virginia 22003; phone (703) 323-0113.

Lasers Considered for Strengthening Teeth, Removing Decay

Researchers from the University of California–San Francisco and the Lawrence Livermore and Sandia National Laboratories are studying the effects of lasers on teeth. Their work could lead to new ways of strengthening the dentin, a vulnerable layer beneath the tooth enamel. Lasers might also be used instead of drills to remove decayed tissue, leaving more of the tooth intact, instead of removing both healthy and decayed material as contemporary drills do.

Medical research with lasers for soft-tissue work uses wavelengths and frequencies that interact with water or other fluids. When the liquid is superheated, it produces a "microburst," or small shock wave, which can, for example, clear blockage from an artery without scorching the tissue. "But with the hard-tissue procedures," said John Kinney, principal investigator for the Livermore research program, "we don't want to interact with the fluids but with the hard tissue itself."

The hard tissues, enamel and dentin, enclose soft pulp tissue which contain blood vessels and nerves. Work is under way to find the type of laser light that is best absorbed by the hard surface tissue of the tooth, but doesn't interact with the pulp. Another difficulty is that properties—such as absorption—change as the surface heats up. The researchers are using atomic force microscopy to observe changes on the tooth surface, and an "integrating sphere spectrophotometer" to determine the optical properties of teeth as they change with temperature.

Dentin is a tough mixture of mineral and fiber that makes up the bulk of the tooth and tooth roots. "As we get older, the gum tissues recede and expose more of the roots," Kinney said. "The dentin is not able to resist decay as well as the enamel. Bacteria in the mouth produce lactic acid and the root surface is very vulnerable to decay. Root decay is a leading cause of tooth loss," he said. If the porous dentin can be sealed with lasers, cavities can be halted, Kinney believes. To do this, the laser must be able to alter the mineral of the dentin to produce a synthetic enamel that is decay resistant.

The effects of laser beams on the inner tooth materials will be examined using x-ray tomographic microscopy (XTM), which allows scientists to image the interior of materials nondestructively. XTM will be used in clinical studies to monitor the progress of cavities *in vitro* to evaluate the treatment's effectiveness.

Kinney plans to treat one part of an extracted tooth, while leaving the other part in its natural state. The entire tooth will then be placed in a decay-inducing acidic broth. Images of both sections will be recorded and the changes over time compared to show the effect of the laser treatment.

The researchers involved in the study are Sally Marshall, Bill Marshall, and Joel Gray of UCSF; Monte Nichols and Tom Breunig of Sandia; and Kinney, Mehdi Balooch, Allyn Saroyan, David Haupt, Walt Bell, and Warren Massey of Lawrence Livermore.

Technique Developed to Disperse Metal Particles in Ceramics

Cornell University researchers have developed a process for making ceramic grains with a fine distribution of metal particles. These materials are potentially useful for the next generation of high-performance aircraft engine turbines. The technique allows the *in-situ* formation of metals and ceramics on a microscopic scale. Oxide phases are reduced to form mixtures of a ductile metal and a hard ceramic, yielding unique microstructures.

The researchers have produced ceramic grains that contain a fine distribution of metal particles—essentially, a ceramic that is both very stiff and resistant to failure. The researchers used ductile nickel particles in aluminum oxide, a high-melting-temperature ceramic. Typically, ceramics crack easily. But a crack that meets a ductile, metal particle is blunted, so propagation stops. The material is expected to be useful to at least 1000°C and may endure temperatures as high as 1200°C.

Stephen L. Sass, Cornell professor of materials science and engineering, said, "What is new is that no one thought of bringing a metallic phase out of the oxide as we do, except in very academic research that had quite different goals. By use of reduction reaction, you can put the metal right where you want it. And when you do that, you get all kinds of intriguing and, hopefully, useful microstructures."

According to Sass, this advance is a step toward the development of new materials that weigh much less than current metal alloys and can survive the high temperatures, high stresses, and hot gases encountered inside a jet turbine.

"We may, and I stress, *may*, be able to achieve an improvement of nearly a factor of three compared to nickel alloys that are flying today," Sass said. "If you can get a material to withstand the same temperatures at half the weight, that's a tremendous advantage. And, if this technique works as we hope, we will also have a material that is cost-effective to produce."

Cornell Research Foundation has filed two patents on the technique. Other researchers involved in the project include Rüdiger Dieckmann, Emmanuel P. Giannelis, and Carol S. Nichols in materials science and engineering, C.Y. Hui, S. Leigh Phoenix, and Alan T. Zehnder in theoretical and applied mechanics, Paul R. Dawson in mechanical and aerospace engineering, and James M. Burlitch in the Chemistry Department.

R.N. Parkins Is New President of NACE

Redvers N. Parkins, professor emeritus of metallurgy at the University of Newcastle upon Tyne, assumed the office of president of NACE International on March 10, 1993 at CORROSION/93, NACE's (National Association of Corrosion Engineers) annual conference.

A NACE member since 1970, Parkins has been active in many facets of the association, making his mark through his work in several standing committees and technical committees. Parkins was the first overseas member elected to the board of directors, and served on the executive committee from 1988 until now. He has received NACE's R.A. Brannon Award and the association's W.R. Whitney Award. Among his other awards are the Cavallaro Medal of the European Federation of Corrosion, the U.R. Evans Award of the Institute of Corrosion, and the Kroll Medal and Prize of the Institute of Metals.

Parkins received his BS degree in metallurgy and a doctorate for his thesis, "Stress Corrosion Cracking of Mild Steels," from the University of Durham, and a doctorate of science from the University of Newcastle upon Tyne. □

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