Materials

Ceramic Paste for Patching High-Temperature Insulation

Repairs can be performed by use of simple techniques.

Lyndon B. Johnson Space Center, Houston, Texas

A ceramic paste that can be applied relatively easily, either by itself or in combination with one or more layer(s) of high-temperature ceramic fabrics, such as silicon carbide or zirconia, has been invented as a means of patching cracks or holes in the reinforced carbon-carbon forward surfaces of a space shuttle in orbit before returning to Earth. The paste or the paste/fabric combination could also be used to repair rocket-motor combustion chambers, and could be used on Earth to patch similar high-temperature structures.

The specified chemical composition of the paste admits of a number of variations, and the exact proportions of its constituents are proprietary. In general, the paste consists of (1) silicon carbide, possibly with addition of (2) hafnium carbide, zirconium carbide, zirconium boride, silicon tetraboride, silicon hexaboride, or other metal carbides or oxides blended with (3) a silazane-based polymer.

Because the paste is viscous and sticky at normal terrestrial and outer-space ambient temperatures, high-temperature ceramic fabrics such as silicon carbide or zirconia fabric impregnated with the paste (or the paste alone) sticks to the damaged surface to which it is applied. Once the patch has been applied, it is smoothed to minimize edge steps as required [forward-facing edge steps must be ≤0.030 in. (≤0.76 mm) in the original intended space-shuttle application]. The patch is then heated to a curing temperature thereby converting it from a flexible material to a hard, tough material. The curing temperature is 375 to 450 °F (≈190 to 230 °C).

In torch tests and arc-jet tests, the cured paste was found to be capable of withstanding a temperature of 3,500 °F (≈1,900 °C) for 15 minutes. As such, the material appears to satisfy the requirement, in the original space-shuttle application, to withstand re-entry temperatures of ≈3,000 °F (≈1,600 °C).

This work was done by Steven J. Adam, James V. Tompkins, Gordon R. Toombs, Pete Hogensen, and Douglas G. Soden of The Boeing Co. for **Johnson Space Center**.

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act {42 U.S.C. 2457(f)}, to The Boeing Company. Inquiries concerning licenses for its commercial development should be addressed to:

The Boeing Company
PO Box 2515
2201 Seal Beach Blvd.
Seal Beach, CA 90740-1515

Refer to MSC-23942, volume and number of this NASA Tech Briefs *issue, and the page number.*

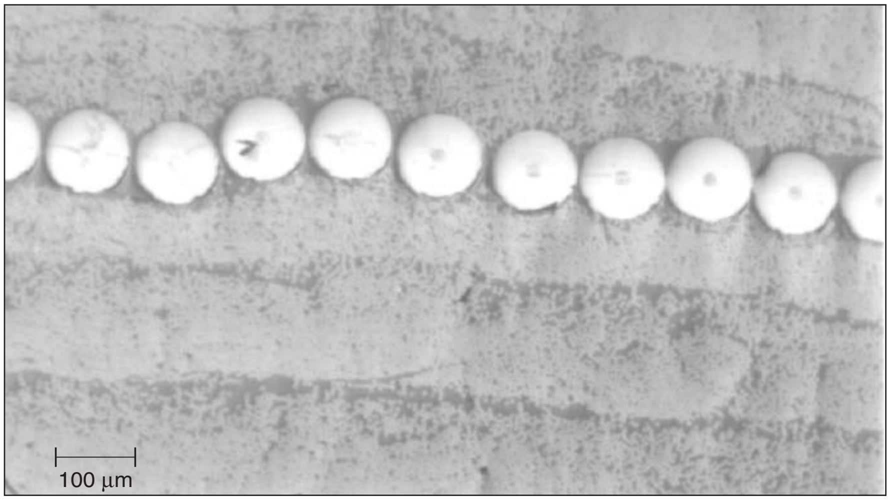
Fabrication of Polyimide-Matrix/Carbon and Boron-Fiber Tape

Production costs can be reduced and compression strengths increased.

Langley Research Center, Hampton, Virginia

The term "HYCARB" denotes a hybrid composite of polyimide matrices reinforced with carbon and boron fibers. HYCARB and an improved process for fabricating dry HYCARB tapes have been invented in a continuing effort to develop lightweight, strong composite materials for aerospace vehicles. Like other composite tapes in this line of development, HYCARB tapes are intended to be used to build up laminated structures having possibly complex shapes by means of automated tow placement (ATP) — a process in which a computer-controlled multiaxis machine lays down prepreg tape or tows. The special significance of the present process for making dry HYCARB for ATP is that it contributes to the reduction of the overall cost of manufacturing boron-reinforced composite-material structures while making it possible to realize increased compression strengths.

The present process for making HYCARB tapes incorporates a "wet to dry" process developed previously at Langley Research Center. In the "wet to dry" process, a flattened bundle of carbon fiber tows, pulled along a continuous production line between pairs of rollers, is impregnated with a solution of a poly(amide acid)



This **Photomicrograph** shows a cross of a specimen containing one layer of boron fibers.

in N-methyl-2-pyrrolidinone (NMP), then most of the NMP is removed by evaporation in hot air. In the present case, the polyamide acid is, more specifically, that of LaRC™ IAX (or equivalent) thermoplastic polyimide, and the fibers are, more specifically, Manganite IM7 (or equivalent) polyacrylonitrile-based carbon filaments that have a diameter of 5.2 µm and are supplied in 12,000-filament tows.

The present process stands in contrast to a prior process in which HYCARB tape was made by pressing boron fibers into the face of a wet carbon-fiber/poly(amide acid) prepreg tape — that is, a prepreg tape from which the NMP solvent had not been removed. In the present process, one or more layer(s) of side-by-side boron fibers are pressed between dry prepreg tapes that have been prepared by the aforementioned "wet to dry" process. The multilayer tape is then heated to imidize the matrix material and remove most of the remaining solvent, and is pressed to consolidate the multiple layers into a dense tape.

For tests, specimens of HYCARB tapes and laminated composite panels made from HYCARB tape were prepared as follows: HYCARB tapes were fabricated as described above. Each panel was made by laying down ten layers of tape, containing, variously, one, two, or three boron-fiber plies and the remainder carbon-fiber-only plies (see figure). Each panel was made by laying down ten layers of tape. Each panel was then cured by heating to a temperature of 225 °C for 15 minutes, then pressing at 200 psi (≈1.4 MPa) while heating to 371 °C, holding at 371 °C for 1 hour, then continuing to hold pressure during cooling. Control specimens that were otherwise identical except that they did not contain boron fibers also were prepared. In room-temperature flexural tests, the HYCARB specimens performed comparably to the control specimens; in room-temperature, open-hole compression tests, the HYCARB specimens performed slightly better, by amounts that increased with boron content.

This work was done by Harry L. Belvin and Roberto J. Cano of **Langley Research Center** *and Monte Treasure and Thomas W. Shahood of Textron Specialty Materials. Further information is contained in a TSP (see page 1).*

This invention is owned by NASA, and a patent application has been filed. Inquiries concerning nonexclusive or exclusive license for its commercial development should be addressed to the Patent Counsel, Langley Research Center, at (757) 864-3521. Refer to LAR-15852.

Protective Skins for Aerogel Monoliths

Viscous polymer precursors are applied, then polymerized before they can percolate in.

John H. Glenn Research Center, Cleveland, Ohio

A method of imparting relatively hard protective outer skins to aerogel monoliths has been developed. Even more than aerogel beads, aerogel monoliths are attractive as thermal-insulation materials, but the commercial utilization of aerogel monoliths in thermal-insulation panels has been inhibited by their fragility and the consequent difficulty of handling them. Therefore, there is a need to afford sufficient protection to aerogel monoliths to facilitate handling, without compromising the attractive bulk properties (low density, high porosity, low thermal conductivity, high surface area, and low permittivity) of aerogel materials. The present method was devised to satisfy this need.

The essence of the present method is to coat an aerogel monolith with an outer polymeric skin, by painting or spraying. Apparently, the reason spraying and painting were not attempted until now is that it is well known in the aerogel industry that aerogels collapse in contact with liquids. In the present method, one prevents such collapse through the proper choice of coating liquid and process conditions: In particular, one uses a viscous polymer precursor liquid and (a) carefully controls the amount of liquid applied and/or (b) causes the liquid to become cured to the desired hard polymeric layer rapidly enough that there is not sufficient time for the liquid to percolate into the aerogel bulk.

The method has been demonstrated by use of isocyanates, which, upon exposure to atmospheric moisture, become cured to polyurethane/polyurea-type coats. The method has also been demonstrated by use of commercial epoxy resins. The method could also be implemented by use of a variety of other resins, including polyimide precursors (for forming high-temperature-resistant protective skins) or perfluorinated monomers (for forming coats that impart hydrophobicity and some increase in strength).

This work was done by Nicholas Leventis, James C. Johnston, Maria A. Kuczmarski, and Mary Ann B. Meador of **Glenn Research Center**. *Further information is contained in a TSP (see page 1).*

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Commercial Technology Office, Attn: Steve Fedor, Mail Stop 4–8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17605-1.