Technology Focus: Composites & Ceramics

improving Thermomechanical Properties of SiC/SiC Composites A heat treatment increases thermal conductivity and increases creep resistance.

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Today, a major thrust toward improving the thermomechanical properties of engine components lies in the development of fiber-reinforced silicon carbide matrix composite materials, including SiC-fiber/SiC-matrix composites. These materials are lighter in weight and capable of withstanding higher temperatures, relative to state-of-the-art metallic alloys and oxide-matrix composites for which maximum use temperatures are in the vicinity of 1,100 °C. In addition, the toughness or damage tolerance of the SiC-matrix composites is significantly greater than that of unreinforced silicon-based monolithic ceramics.

For successful application in advanced engine systems, the SiC-matrix composites should be able to withstand component service stresses and temperatures for the desired component lifetimes. Inasmuch as the high-temperature structural lives of ceramic materials are typically limited by creep-induced growth of flaws, a key property required of such composite materials is high resistance to creep under conditions of use. Also, the thermal conductivity of the materials should be as high as possible so as to minimize component thermal gradients and thermal stresses.

A state-of-the-art SiC-matrix composite is typically fabricated in a three-step process: (1) fabrication of a componentshaped architectural preform reinforced by thermally stable high-performance fibers, (2) chemical-vapor infiltration (CVI) of a fiber-coating material such as boron nitride (BN) into the preform, and (3) infiltration of an SiC-based matrix into the remaining porosity in the preform. Generally, the matrices of the highest-performing composites are fabricated by initial use of a CVI SiC matrix component that is typically more thermally stable and denser than matrix components formed by processes other than CVI. As such, the initial SiC matrix component made by CVI provides better environmental protection to the coated fibers embedded within it. Also, the denser CVI SiC imparts to the composite better resistance to propagation of cracks, enhanced thermal conduc-



Figure 1. Room-Temperature Thermal Conductivities were measured on unannealed and annealed specimens of composite panels containing SiC matrices formed in various ways as described in the last paragraph of the main text.



Figure 2. Creep Strains were measured on unannealed and annealed specimens of composite panels containing SiC matrices like two of those represented in Figure 1.

tivity, and higher creep resistance.

For adequate deposition of CVI SiC matrix material into a preform, it is typically necessary to perform the infiltration at a temperature below 1,100 °C. The use of an infiltration temperature in this range typically results in the formation of an SiC matrix that is fairly dense, but contains metastable atomic defects and is non-stoichiometric in that it contains a small excess of silicon. These defects typically exist at the matrix grain

boundaries, where they can act as scatterers for thermal phonons and degrade matrix creep resistance by enhancing grain-boundary sliding.

In order to make it possible to eliminate these defects and improve the thermomechanical properties of ceramic composites that contain SiC matrices, researchers at Glenn Research Center (GRC) have developed a high-temperature heat or annealing treatment that can be performed after deposition of a CVI SiC matrix into a fiber preform. Using (1) SiC fibers of a type developed by GRC and denoted "Sylramic-iBN" and (2) BN-based fiber coatings, which are both stable in their functions under the treatment conditions, the GRC researchers have observed minimal loss of strength in composite panels formed from two-dimensional architectural preforms and various contents of CVI SiC. More importantly, the NASA treatment significantly increased panel thermal conductivity and creep resistance, as indicated in Figures 1 and 2, respectively.

For the treated panels, the volume fractions of the fibers and BN coatings were about 36 percent and 8 percent, respectively. The first case represented in Figure 1 is that of a panel containing 50 volume percent SiC formed by CVI only. In the second case, the remaining open porosity in the 35-percent CVI SiC matrix was filled by a process denoted PIP, which involved repeated infiltration and pyrolysis of an SiC-yielding polymer (hybrid CVI + PIP). For the third case represented in Figure 1, the remaining open porosity in a 35-percent CVI SiC matrix was filled by a process that involved repeated melt infiltration (MI) of silicon at a temperature near 1,400 °C (hybrid CVI + MI). For the first two cases, the heat treatment was performed after final matrix formation; for the last case, the treatment was performed after CVI and before MI. In all cases, panel thermal conductivity was significantly increased by the NASA heat treatment. On an absolute scale, Figure 1 also shows the detrimental effect of trapped porosity on composite conductivity for the matrices formed by CVI only or by hybrid CVI + PIP, and the beneficial effects of the hybrid CVI + MI approach that more effectively fills the CVI SiC pores. However, Figure 2 shows that in the case of the matrix fully formed by CVI only, creep resistance and temperature capability were greater than in the case of the matrix formed by hybrid CVI + MI. Thus, because of their lack of elemental silicon, the long-term use temperature of the CVI-only matrix (as well as the hybrid CVI + PIP matrix) could exceed 1,400 °C, which is well above the long-term use temperature for panels containing matrices made by hybrid CVI + MI.

This work was done by James A. DiCarlo of Glenn Research Center and Ramakrishna T. Bhatt of the Army Research Laboratory. 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, Innovative Partnerships Office, Attn: Steve Fedor, Mail Stop 4–8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-17595-1.

Aerogel/Particle Composites for Thermoelectric Devices Shrinkage is reduced through addition of titania powder.

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Optimizing solution chemistry and the addition of titania and fumed silica powder reduces shrinkage. These materials would serve to increase thermal efficiency by providing thermal insulation to suppress lateral heat leaks. They would also serve to prolong operational lifetime by suppressing sublimation of certain constituents of thermoelectric materials (e.g., sublimation of Sb from CoSb₃) at typical high operating temperatures. [The use of pure silica aerogels as cast-in-place thermal-insulation and sublimation-suppression materials was described in "Aerogels for Thermal Insulation of Thermoelectric Devices" (NPO-40630), NASA Tech Briefs, Vol. 30, No. 7 (July 2006), page 50.]

A silica aerogel is synthesized in a solgel process that includes preparation of a silica sol, gelation of the sol, and drying of the gel in a solvent at a supercritical temperature and pressure. The utility of pure silica aerogel is diminished by a tendency to shrink (and, therefore, also to crack) during the gelation and supercritical-drying stages. Moreover, to increase suppression of sublimation, it is advantageous to make an aerogel having greater density, but shrinkage and cracking tend to increase with density.

A composite material of the type



Shrinkages of Representative Aerogels of various densities and of aerogel/titania composites were measured.

under investigation consists mostly of titania oxide powder particles and a small addition of fumed silica powder, which are mixed into the sol along with other ingredients prior to the gelation stage of processing. The silica aerogel and fumed silica act as a binder, gluing the titania particles together. It is believed