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Effects of High Temperature Argon Heat Treatment on Tensile Strength and Microstructure of BN/SiC Coated SiC Fiber Preforms

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EFFECTS OF HIGH TEMPERATURE ARGON HEAT TREATMENT ON TENSILE STRENGTH AND MICROSTRUCTURE OF BN/SiC COATED SiC FIBER PREFORMS

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ABSTRACT

Preforms of BN/SiC coated Hi-Nicalon and Sylramic SiC fibers were heat treated under 0.1 MPa argon pressure between 1000^0 to 1800 ^{0}C for 1 and 100h. The effects of high temperature exposure on physical dimensions, weight, room temperature tensile strength, and microstructure of preforms have been studied. Both preforms showed shrinkage and weight loss, and microstructural changes beyond 1000 ^{0}C. After 100 hr exposure, the Hi- Nicalon preforms showed strength degradation beyond 1200 ^{0}C. The mechanisms of the strength degradation appear to be grain growth of the SiC fibers and crystallization of the BN coating. After 100 hr, the Sylramic preforms heat treated to 1000 ^{0}C retained their as-produced strength while some of those heat-treated between 1000^0 and 1800 ^{0}C showed strength degradation and others did not. At 1800 ^{0}C, the ultimate tensile strength decreased with increasing time of exposure. Reasons for strength degradation of Sylramic preforms are being investigated.

INTRODUCTION

Preforms of boron nitride/silicon carbide (BN/SiC) coated Hi-Nicalon and Sylramic SiC fibers are used as building block for the fabrication of silicon carbide based composites by the melt infiltration (MI) approach. The composite fabrication involves two steps. In the first step the preform is infiltrated with carbon foam derived from a polymer, or with a SiC slurry. In the second step the filled preform is infiltrated with molten silicon. The MI fabrication approach offers two main advantages over other methods of fabricating SiC/SiC composites: Minimal fiber damage during fabrication, and near net shape capability. The current generation SiC/SiC composites show high thermal conductivity and have performance potential up to 1320 ^{0}C.

However, because of the large scatter in mechanical properties, poor oxidation resistance of the BN interface coating at intermediate temperatures, high surface recession rate in combustion environments, and the presence of high amounts of residual silicon in the SiC matrix, current generation SiC/SiC composites are restricted to below 1200 ^{0}C applications [1]. The large scatter in mechanical properties is partly due to variation in mechanical properties of the preforms, or to poor microstructural control during processing. Since performance and properties of the composites are controlled by the preforms, the factors that affect preforms will have a major effect on reliability, reproducibility, and maximum use capability of the composites.

The objective of this study is to determine the microstructural and strength stability of commercially available BN/SiC coated SiC fiber preforms in argon to simulate processing and application temperature exposures.

EXPERIMENTAL

The BN/SiC coated Hi-Nicalon™ and Sylramic™ SiC fiber preforms were purchased from AlliedSignal Composites, Delaware. Hi-Nicalon™ and Sylramic™ are the trade names of the SiC fiber manufactured by The Nippon Carbon Company, Japan, and The Dow Corning Company, Midland, Michigan, respectively. For conciseness, henceforth, these two preforms will be referred to as Hi-Nicalon and Sylramic preforms. The preforms were prepared by stacking eight layers of 2-D woven 5-harness-satin (5HS) SiC fiber mats (152 mm x 229 mm) and then infiltrating the assembly with the BN and SiC coatings by a proprietary chemical vapor infiltration (CVI) process. Typical dimensions of the as-processed preforms were 152 mm x 229 mm x 2 mm. The preforms consist of ~40 vol. % SiC fiber, ~3-8 vol. % BN, ~18-30 vol. % SiC, and ~20-40 vol. % open porosity. The rectangular specimens of dimensions 152mm x 13mm x 2mm were cut from the larger preforms using a diamond impregnated metal bonded cut-off wheel.

The specimens were heat-treated in a box furnace in 0.1 MPa argon for 1 and 100 hrs at 200 ^{0}C temperature intervals between 1000 ^{0}C and 1800 ^{0}C. At each exposure condition at least 3 to 5 specimens were heat-treated. The specimen dimensions and weight were measured before and after heat treatment at room temperature.

To avoid possible delamination and compressive failure within the grip area during tensile testing, the entire heat-treated preform was infiltrated with a low strength, low modulus epoxy. An infiltration procedure similar to that developed for ceramographic specimens was used. The epoxy infiltrated specimens were machined to dog-bone shape in the gauge section using an ultrasonic impact machine and SiC slurry. For tensile testing, two glass fiber reinforced epoxy tabs of dimensions 38mm x 13mm x 2mm were glued at each specimen end. A spring loaded contact extensometer was attached to the specimen in the gauge section to monitor strain. The tensile tests were conducted at room temperature at a cross-head speed of 1.3 mm/sec.

For the preparation of transmission electron microscope (TEM) specimens, the preform specimens were sectioned using a diamond saw, vacuum degassed in a mixture of epoxy, and sandwiched between two silicon wafers. The sandwich specimens were sliced to a size of 2.5mm x 2mm x 0.5mm. These specimens were thinned to electron transparency using tripod

polishing and argon ion-beam milling. Diffraction contrast images, selected-area and micro diffraction data were recorded using a Philips CM200 electron microscope operated at 200 kV. The TEM is coupled to an energy dispersive X-ray (EDX) spectrometer for chemical identification. The present transmission electron microscopy results include analyses from cross-sections of preforms. In this mode, the fibers were observed 'end-on'.

RESULTS

The optical micrographs of the cross-section of the epoxy infiltrated Hi-Nicalon and Sylramic fiber preforms are shown in Figs. 1 and 2. These cross-sections show similarities and

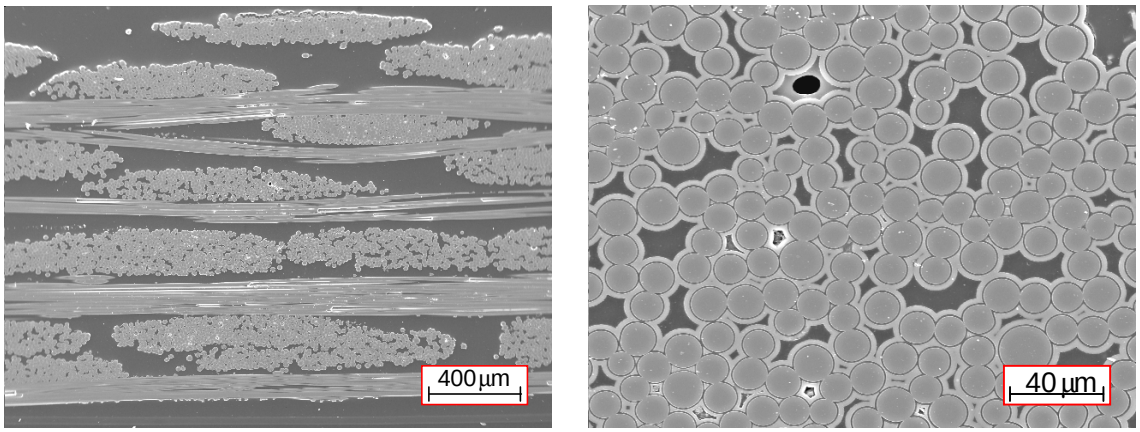


Fig. 1: Optical photographs of the cross-section of an epoxy infiltrated BN/SiC coated Hi-Nicalon SiC preforms showing fiber distribution and CVI coating thickness variation.

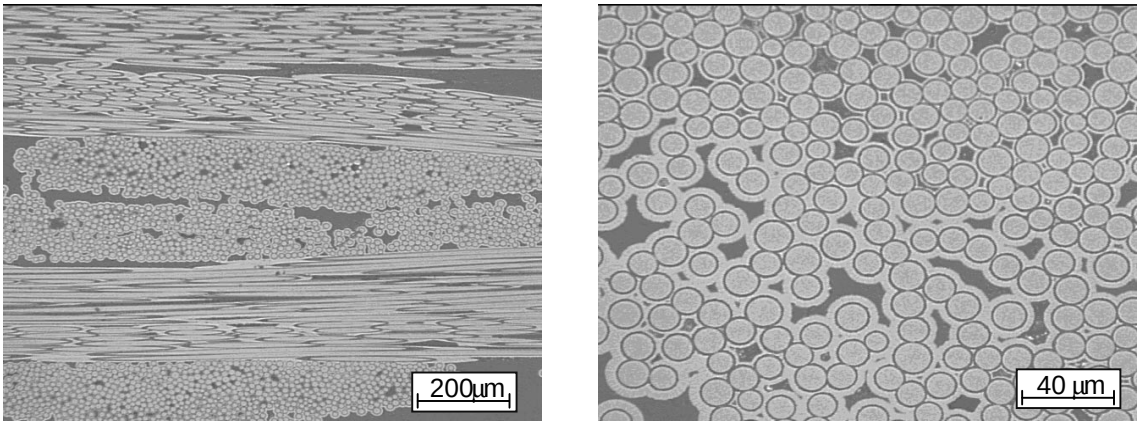
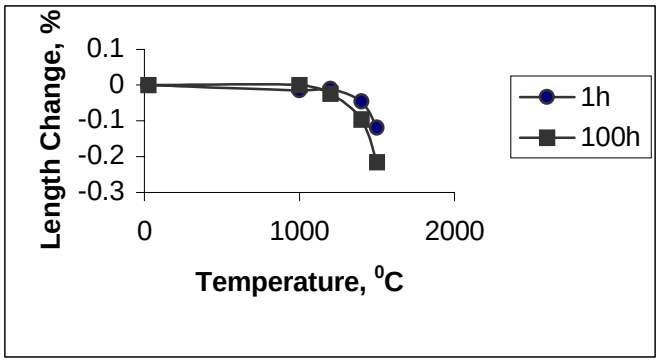


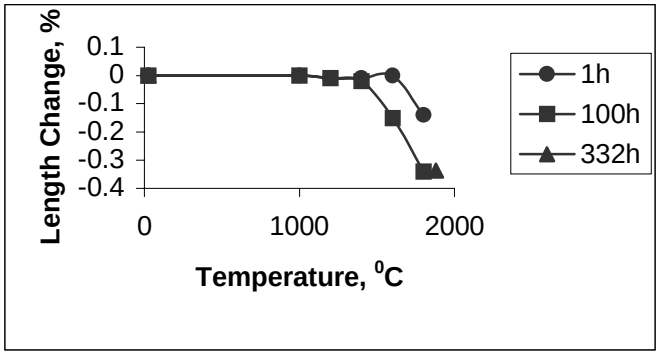
Fig. 2: Optical photographs of the cross-section of an epoxy infiltrated BN/SiC coated Sylramic SiC preforms showing fiber distribution and CVI coating thickness variation.

differences between preforms. In these figures, the dark ring around the fibers is the BN coating and the light grayish region on top of the BN coating is the SiC coating. For both Hi-Nicalon and Sylramic preforms, the SiC coating thickness varies from tow to tow as well from the middle to the periphery of the tow. There are also several differences noticed between preforms. The fiber diameters in the Hi-Nicalon preforms are ranging from 9 to 15 μm, while those in Sylramic preforms are all ~10 μm. The fiber tows in Hi-Nicalon preforms are loosely woven, whereas in Sylramic, they are tightly woven. Closed porosity in Hi-Nicalon preforms is <2 vol. %, and ~2-5 vol. % in Sylramic preforms. The Sylramic preforms show a thick layer of SiC on the outer periphery of the tows.

Both types of preforms showed shrinkage in length and weight loss at high temperatures. Shrinkage observed in preforms after 1 and 100 hr heat treatments in argon at temperatures to 1800 ^{0}C is plotted in Fig. 3 along with a specimen heat treated at 1800 ^{0}C for 332 hr. According to figure 3(a) the length of the Hi-Nicalon preforms remained nearly the same up to 1200 ^{0}C. Beyond this temperature, the length decreased with increasing heat treatment temperature at a



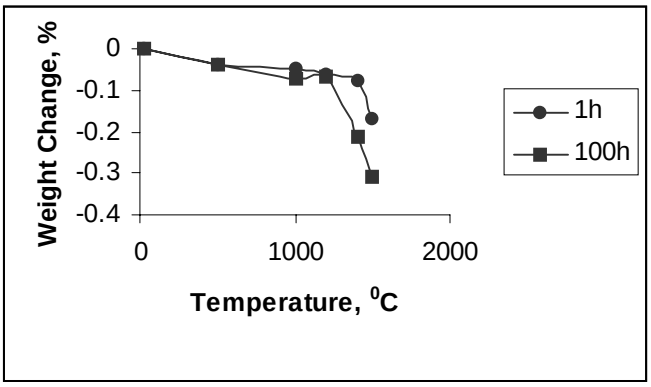
(a)



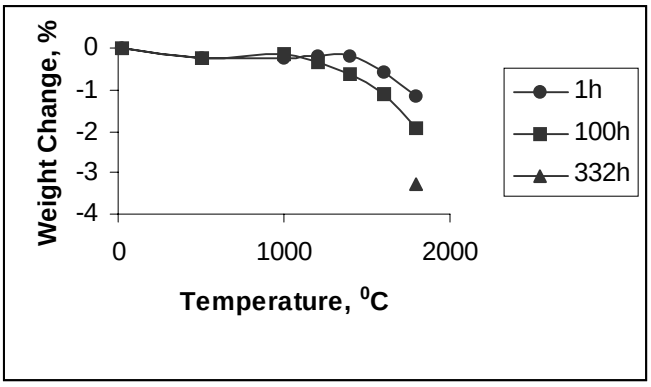
(b)

Fig. 3: Variation of length change with temperature for the BN/SiC coated SiC preforms. (a) Hi-Nicalon; (b) Sylramic.

fixed exposure time, or with increasing time at a given temperature. The Sylramic preform showed shrinkage in length beyond 1600 ^{0}C (Fig. 3(b)). Figure 4 shows variation of weight loss with heat treatment temperature. Two weight loss regions were observed in the figure: An initial region where weight loss is independent of exposure condition followed by a region where weight loss is dependent on time and temperature. The initial region lasted up to 1200 ^{0}C for both preforms. In this region ~ 0.07 % and ~0.29 % weight losses were observed respectively, in Hi-Nicalon and Sylramic preforms. Above 1200 ^{0}C, the weight loss increased with increasing temperature at constant time, or with increasing time at a given temperature.



(a)

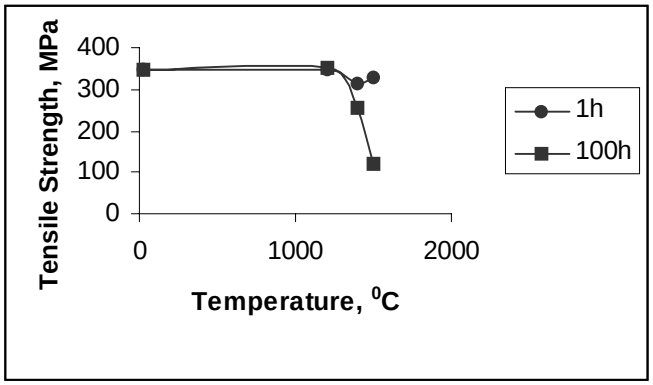


(b)

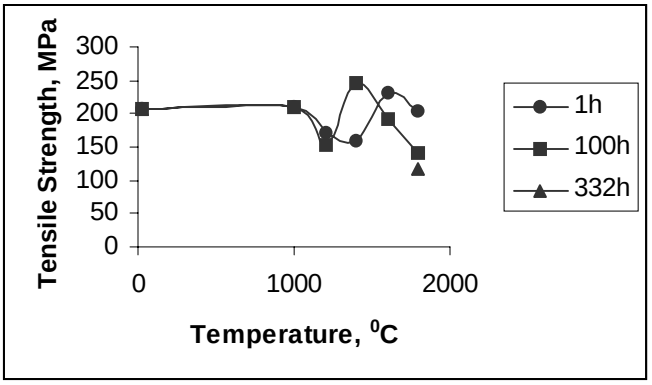
Fig. 4: Variation of weight loss with temperature for the BN/SiC coated SiC preforms. (a) Hi-Nicalon; (b) Sylramic.

The effects of heat treatment temperature and time on ultimate tensile strength of the Hi-Nicalon and Sylramic preform specimens are plotted in figure 5. Each data point represents an average of 3 to 5 specimens. The scatter in the strength data is ~ 25 MPa. The Hi-Nicalon preforms heat treated at temperatures to 1500 ^{0}C for 1hr and those heat treated for 100 hr at temperatures to 1200 ^{0}C showed no loss in ultimate tensile strength when compared with the UTS values of the as-received specimens. Between 1200^0 and 1500 ^{0}C, the UTS values of the 100hr heat-treated specimens decreased with increasing exposure temperature. On the other hand, the Sylramic

preforms are relatively stable up to 1000 ^{0}C after 1 and 100h. Between 1000 and 1600 ^{0}C both at 1 and 100 hr exposures, some preforms showed strength degradation and others did not. In addition, the UTS of the preforms appears to decrease with increasing time of exposure at 1700^0 and at 1800 ^{0}C.



(a)

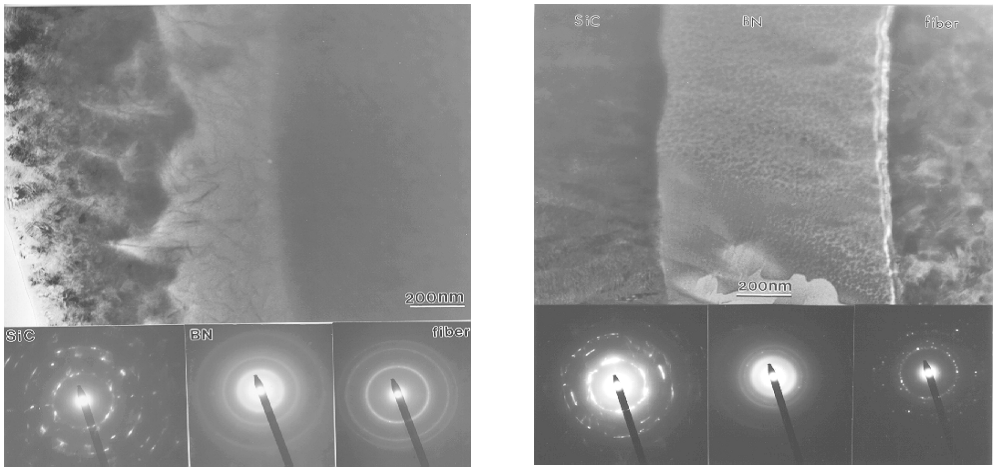


(b)

Fig. 5: Effect of heat treatment temperature and time on ultimate tensile strength of the BN/SiC coated SiC preforms. (a) Hi-Nicalon; (b) Sylramic.

The TEM micrographs and selected area diffraction (SAD) pictures of the as-fabricated Hi-Nicalon and Sylramic preforms are shown in Fig. 6. Results indicate that for as received condition, the BN interface is nearly amorphous and the SiC coating is crystalline in both preforms. The SiC grains are columnar and grow radially. The boundary between the SiC fiber and BN coating or between the BN and SiC coatings is sharp and distinct suggesting no interdiffusion between the zones. However in few cases a graded zone of fine grained SiC and amorphous BN was noticeable at the BN/SiC coating interface as shown in figure 6(a). The EDX analysis shows that the BN coating contains small amounts Si, C, and O. The Hi-Nicalon fiber is microcrystalline with an average grain size of 2 nm (Fig. 6(a)). The Sylramic SiC fiber contained randomly oriented SiC grains of nominal diameter of ~100 nm, and TiB_2 particles.

After argon heat treatment beyond 1000 ^{0}C, the Hi-Nicalon and Sylramic preforms showed coarsening of grains in the SiC fibers and crystallization of the BN coating. However only major microstructural changes observed in the heat-treated preforms will be described here. The TEM micrograph of the Hi-Nicalon preform heat treated at 1500 ^{0}C for 100 hr is shown in Fig. 7.



(a) *(b)*

Fig. 6: TEM micrographs of the cross section of as-received BN/SiC SiC coated preforms showing interface microstructures. (a) Hi-Nicalon; (b) Sylramic.

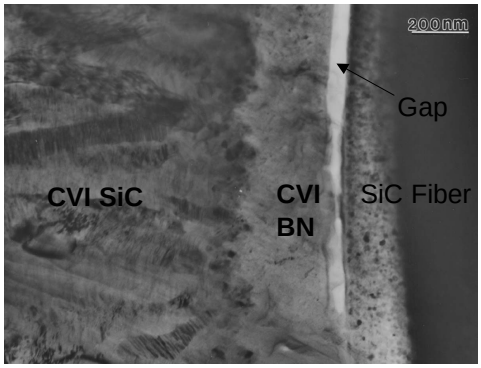
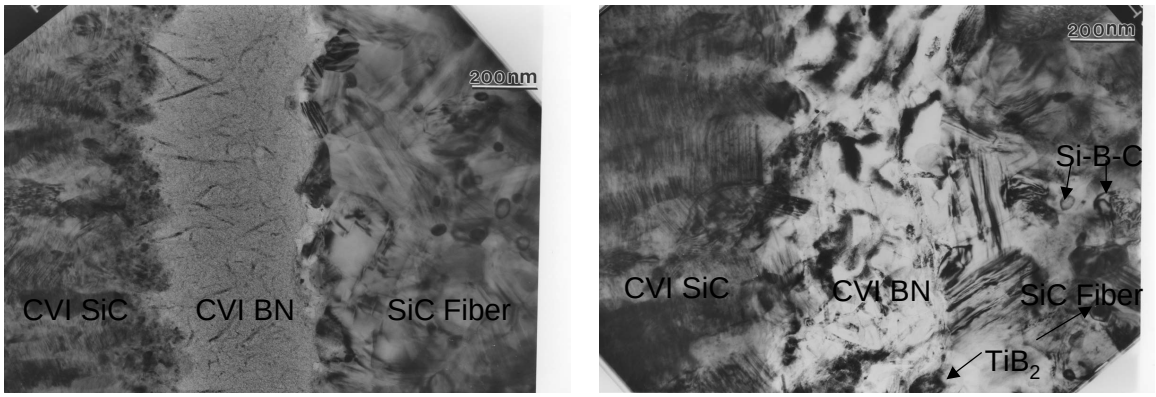


Fig. 7: TEM micrograph of the interface zone of the BN/SiC coated Hi-Nicalon preform heat treated at 1500 ^{0}C for 100 hr in argon showing grain growth in SiC fiber.

Several microstructural changes are noticed, when comparing Fig. 7 with Fig. 6(a): Crystallization of the BN interface, grain growth in SiC fibers, coarsening of SiC grains in the CVI SiC coating, and a gap at the BN/SiC fiber interface. Gap formation could be due to fiber shrinkage or due to differential etching during TEM specimen preparation. The EDX analysis across the interface indicates O_2 enrichment in the BN coating and at the interface of the BN/SiC coating, and carbon segregation at the surface of the SiC fiber. Figure 8(a) shows the

TEM micrograph of the Sylramic preform heat-treated at 1420 ^{0}C for 2 h in argon. Noticeable features in the micrograph are coarsening of SiC grains in the fiber, and partial recrystallization of amorphous BN coating. The SiC grains grew from ~100 nm (as-fabricated condition) to ~200 nm (heat-treated preforms). The BN coating contains rod like BN grains. Figure 8(b) is the TEM micrograph of the preform heat treated for 1 h at 1800 ^{0}C. When compared with the BN interface in Fig. 8(a), Fig. 8(b) shows greater amounts of the crystallized grains in the BN coating, larger SiC grains, migration of TiB_2 particles from the interior of the SiC fibers to the BN/SiC fiber interface, and Si-B-C islands in the Sylramic fibers.



(a) *(b)*

Fig. 8: TEM micrographs of the interface zone of the heat treated BN/SiC coated Sylramic preforms showing crystallization of BN coating and coarsening of SiC grains in the fibers. (a) 1420 ^{0}C for 2 hr; (b) 1800 ^{0}C for 1 hr.

DISCUSSION

Results of this investigation indicate that heat treatment of Hi-Nicalon and Sylramic preforms in argon above 1000 ^{0}C resulted in shrinkage, weight loss, microstructural changes in the constituents, and strength degradation. Although preform strength degradation mechanisms are not clearly understood, an attempt is made to explain weight loss and shrinkage based on the composition, microstructure, and processing temperatures of the preform constituents.

The Hi-Nicalon SiC fibers used in the preforms were processed by the melt spinning of polycarbosiline, followed by radiation curing and pyrolysis at ~ 1200 ^{0}C [2,3]. Based on the elemental analysis, the chemical composition of fiber can be represented as 91 wt% SiC and 9 wt% C [4 . The reported value of the C/Si atomic ratio is 1.3 [4]. Naslain etal [5] reported that the fiber contains β-SiC as 2-20 nm crystallites and excess elemental carbon. This is in agreement with the TEM results shown earlier in Fig. 6(a). On the other hand, the Sylramic fiber is processed at a temperature > 1600 ^{0}C and contains 95 wt% SiC, 3 wt% TiB_2, 1.3 wt% B_4C and 0.7 wt% O [5]. The fiber contains 0.1 to 0.5 μm grains of β-SiC. The BN/SiC interface coatings of the Hi-Nicalon and Sylramic preforms were deposited by CVI at <1200 ^{0}C [6]. Physical property characterization results indicate that the preforms contain ~40 vol.% SiC fibers,

3-5 vol.% BN and 18-32 vol.% SiC coatings, and 20-40 vol.% open porosity. The BN coating is porous and the SiC coating is fully dense.

Microstructure of the constituents, physical dimensions, and strength of Hi-Nicalon and Sylramic preforms after 1 and 100 hr heat treatment up to 1200^0 C remained nearly the same as those of the as-fabricated preforms. The only change observed under these exposure conditions is a small weight loss. This weight loss is probably related to moisture desorption. Large amounts of interconnected open porosity, and the porous BN coating in the preforms favor moisture adsorption. Although physical dimensions and pore volume are nearly the same, the Sylramic preforms show a greater tendency for moisture adsorption than the Hi-Nicalon preforms (cf Fig. 4). One possible reason is that TiB_2 and other silicon borides present in the fibers are more susceptible to moisture adsorbtion and dehydration than the SiC and carbon.

Upon heating the preforms above the processing temperature of the BN/SiC coatings or the SiC fibers, microstructural changes can occur. Time and temperature dependent shrinkage and weight loss, crystallization of the BN coating, and slight coarsening of grains in the SiC fibers and the SiC coating were the observed changes after exposing the preforms in argon at temperatures > 1000 ^{0}C. Some of the suggested mechanisms for the weight loss and shrinkage are dissociation of chemically bound O_2 from the BN coating as volatile B_2O gas, dissociation of silicon carbide into silicon vapor and carbon on the surface of the SiC coating, and sintering of the constituents. Previous heat treatment studies have reported shrinkage, and strength degradation in Hi-Nicalon SiC fibers heat treated in inert environments for 1 hr at temperatures >1200 ^{0}C [7,8]. The strength degradation in these fibers is primarily attributed to grain growth. On the other hand, the Syramic fibers heat-treated in argon up to 1550 ^{0}C for 10 hr do not show weight loss or strength degradation [5]. The weight loss seen in both preforms at least up to 1500 ^{0}C appears to be related to dissociation of the BN/SiC coating by mechanisms as suggested earlier. Based on limited data, the strength degradation mechanism of Hi-Nicalon preforms appears to be related to grain growth in the fibers and crystallization of the BN interface. Although microstructural changes, weight loss, and shrinkage were observed in the Sylramic preforms heat-treated above 1000 ^{0}C, only some preforms show strength degradation. Therefore, correlation between microstructure and strength is least understood in these preforms.

SUMMARY OF RESULTS

Microstructural characterization, and room temperature physical and tensile property measurements were performed on BN/SiC coated Hi-Nicalon and Sylramic SiC preforms heat treated in argon at temperatures to 1800 ^{0}C for 1 and 100 hr. Key findings are as follows:

(1) The Hi-Nicalon preforms are stable up to 1200 ^{0}C. Beyond this temperature, the preforms showed weight loss, shrinkage, and strength degradation. The property degradation increased with increasing temperature and time. Probable causes of strength degradation are grain growth in the SiC fibers and crystallization of the BN coating.

(2) The maximum heat treatment temperature without strength loss for Sylramic preforms can reach to 1600 ^{0}C. However, between 1000^0 and 1600 ^{0}C some preforms showed strength

degradation and others did not. Preforms also showed dimensional changes and weight loss above 1400 ^{0}C.

(3) In both preforms, microstructural changes were detected in the fiber and the BN interface for heat treatment above 1000 ^{0}C. With increasing temperature or time of exposure, the degree of crystallinity in the BN interface, and the grain size of SiC in the fibers also increased. The CVI SiC coating on top of the BN coating is stable after 1h heat treatment at 1800 ^{0}C. For longer time exposures at the same temperature, the morphology of the SiC coating converted from columnar to equiaxed grains.

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13. ABSTRACT ***(Maximum 200 words)***

Preforms of BN/SiC coated Hi-Nicalon and Sylramic SiC fibers were heat treated under 0.1 MPa argon pressure between 1000° to 1800 °C for 1 and 100h. The effects of high temperature exposure on physical dimensions, weight, room temperature tensile strength, and microstructure of preforms have been studied. Both preforms showed shrinkage and weight loss, and microstructural changes beyond 1000 °C. After 100 hr exposure, the Hi- Nicalon preforms showed strength degradation beyond 1200 °C. The mechanisms of the strength degradation appear to be grain growth of the SiC fibers and crystallization of the BN coating. After 100 hr, the Sylramic preforms heat treated to 1000 °C retained their as-produced strength while some of those heat-treated between 1000° and 1800 °C showed strength degradation and others did not. At 1800 °C, the ultimate tensile strength decreased with increasing time of exposure. Reasons for strength degradation of Sylramic preforms are being investigated.

14. SUBJECT TERMS
SiC fibers; Preform; Microstructure; Tensile strength; BN/SiC interface

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