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# Silicon Effects on Properties of Melt Infiltrated SiC/SiC Composites

Ramakrishna T. Bhatt
U.S. Army Research Laboratory, Glenn Research Center, Cleveland, Ohio

John Z. Gyekenyesi
Ohio Aerospace Institute, Brook Park, Ohio

Janet B. Hurst
Glenn Research Center, Cleveland, Ohio

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# SILICON EFFECTS ON PROPERTIES OF MELT INFILTRATED SiC/SiC COMPOSITES

Ramakrishna T. Bhatt
U.S. Army Research Laboratory
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

John Z. Gyekenyesi
Ohio Aerospace Institute
Brook Park, Ohio 44142

Janet B. Hurst
National Aeronautics and Space Administration
Glenn Research Center
21000 Brookpark Rd
Cleveland, OH 44135

## ABSTRACT

Silicon effects on tensile and creep properties, and thermal conductivity of Hi-Nicalon SiC/SiC composites have been investigated. The composites consist of 8 layers of 5HS 2-D woven preforms of BN/SiC coated Hi-Nicalon fiber mats and a silicon matrix, or a mixture of silicon matrix and SiC particles. The Hi-Nicalon SiC/silicon and Hi-Nicalon SiC/SiC composites contained ~24 and 13 vol% silicon, respectively. Results indicate residual silicon up to 24 vol% has no significant effect on creep and thermal conductivity, but does decrease the primary elastic modulus and stress corresponding to deviation from linear stress-strain behavior.

## INTRODUCTION

SiC/SiC composites fabricated by the melt infiltration (MI) approach are candidate materials for next generation turbine components because of their performance potential and complex shape fabrication capability [1,2]. However, current state-of-the-art MI SiC/SiC composites are limited to 1200 $^0$C applications partly due to the SiC fibers and partly due to the SiC matrix that contains residual silicon ranging from 10 to 30 vol.%. Residual silicon in SiC/SiC composites is considered to be detrimental for two reasons: First, the possibility of silicon sweating out from rotating components at temperatures >1400 $^0$C during hot streak conditions and damaging the metallic attachments; second, the possibility of mechanical property degradation of SiC/SiC composites due to diffusion of residual silicon through the composite constituents after long term exposure at temperatures below 1400 $^0$C.

Even in monolithic reaction bonded silicon carbide (RBSC), silicon effects have not been clearly understood. It is hypothesized in the ceramic literature that in unstressed conditions, isolated regions of silicon in the SiC matrix may not have a major effect on properties, but in stressed conditions, silicon may enhance crack growth at temperatures above 1200 $^0$C [3-6]. In the case of SiC/SiC composites fabricated by the MI process, silicon is present as a continuous phase and thus may affect themo-mechanical properties. However, varying the silicon content in the SiC/SiC composites is difficult because of significant variation in the vol % of the chemical vapor infiltrated (CVI) silicon carbide coating, closed porosity in the preform, and SiC particle distribution within a batch as well as between batches of the composite specimens.

The long-term objective of this study is to determine the influence of residual silicon on the thermo-mechanical properties of SiC/SiC composites. In this paper, however, the preliminary results of silicon effects on tensile and creep properties, and thermal conductivity are reported.

## EXPERIMENTAL PROCEDURES

Preforms of 2-D woven, 5 harness satin (5HS), BN/SiC coated Hi-Nicalon™ SiC fibers, and 5 HS Hi-Nicalon SiC/SiC composites were purchased from Honeywell Composites Inc., Delaware. The preforms contained 8 layers of 2-D woven SiC fiber mats, an ~0.5 μm thick BN layer, and a 3-5 μm thick SiC layer on the SiC fibers. The Nippon Carbon Company, Japan fabricated the Hi-Nicalon fibers and Albany International Techniweave Inc. Rochester, prepared the fiber mats. The nominal dimensions of the preforms after CVD coating were 229-mm (L) x 152-mm (W) x 2-mm (T). The as-received preforms contained ~40 vol. % SiC fibers, ~18 vol% BN/SiC coatings, and 20-40 vol% of interconnected open porosity. In the SiC/SiC composites, the vol% SiC fibers and the BN/SiC coatings were similar to that in the preform and the SiC matrix contained ~13 vol% silicon.

The preforms and the composite panels were cut into specimens of dimension 152-mm (L) x 12.5-mm (W) x 2-mm (T) using a diamond impregnated metal bonded cut-off wheel. The cut specimens were degreased with a solvent and then dried at 100 $^0$C.

The SiC/silicon composites were fabricated by infiltrating electronic grade silicon into SiC preform specimens. The melt infiltration was performed at

1420 $^0$C in a vacuum furnace equipped with a graphite fixture and a graphite-heating element.

Some preform specimens were also infiltrated with epoxy. Baseline strength data of the epoxy infiltrated preform specimens were compared with those of SiC/silicon and SiC/SiC composites to monitor possible degradation of the preforms during the melt infiltration process.

### Specimen preparation

Some of the specimens were sectioned, mounted in a metallographic mold, ground successively on 40-μm down to 1-μm diamond particle impregnated metal disks, and polished in a vibratory polisher on a micro cloth using 0.3 μm diamond powder paste. The mounted specimens were coated with a thin layer of carbon or palladium in a vacuum evaporator to avoid charging during observation in a scanning electron microscope (SEM).

For tensile tests, dog-bone shaped specimens were machined from the composite block by using an ultrasonic SiC slurry impact machine. At each specimen end, two glass fiber-reinforced epoxy tabs of dimension 37 mm x 12 mm x 1 mm were bonded, leaving ~60 mm for the gage section. A spring-loaded clip-on gauge (25 mm gage length) was attached to the gage section of the specimen to monitor the strain during the tensile test. The specimens were tested at a crosshead speed of 1.3 mm/min in a servo-controlled tensile testing machine equipped with self-aligning grips until failure. Testing was performed in air at temperatures from 25 to 1400 $^0$C. A testing procedure similar to that described in reference [7] was followed.

The creep testing was conducted in an Instron 4502 machine according to an ASTM procedure (ASTM C1337-96). Specimen dimensions were similar to those used for tensile testing. Strain was monitored by attaching a spring-loaded clip-on gage to the 25-mm gage section. Testing was performed in air at 1200 $^0$C at stress levels of 35, 65, and 103 MPa.

A commercial vendor performed thermal diffusivity measurements by the laser flash method from 25 to 1400 $^0$C in argon. ASTM procedure ASTM 1461-92 was used. The thermal conductivity was calculated knowing the density and heat capacity data.

## RESULTS

### Microstructure

The optical photographs of the cross-section of the SiC/silicon and SiC/SiC composites are shown in fig 1. The light regions in the figure are silicon, the grayish regions are the SiC fibers or CVI SiC coating or SiC particles, and the black region around the SiC fibers is the CVI BN coating. In both composites no reaction between the SiC coating and silicon was noticed, but isolated closed porosity can be observed.

**Tensile properties**

The room temperature tensile stress-strain curves for the 5HS SiC/Epoxy, SiC/silicon, and SiC/SiC composites are shown in Fig. 2. All three composites displayed similar stress-strain behavior: an initial linear region followed by a non-linear region. The values of stress corresponding to deviation from linearity (DFL) and primary elastic modulus appear to increase from the SiC/Epoxy to the SiC/SiC composites. Literature reported values for room temperature elastic modulus of SiC, silicon and epoxy matrix are 345 [8], 112 [9], 1 GPa, respectively. Comparison of the matrix modulus and the corresponding

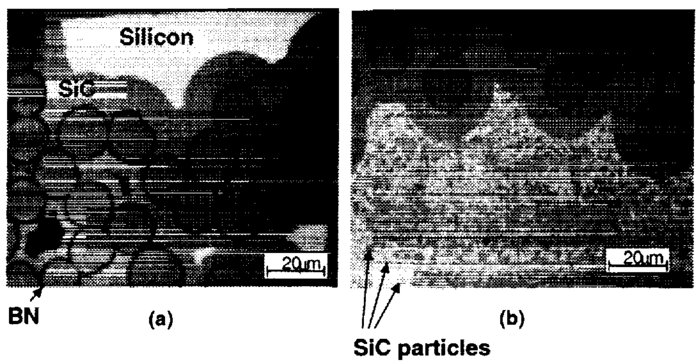


Fig. 1 Optical micrographs of the cross-section of the melt infiltrated 5 HS Hi-Nicalon SiC/SiC composites: (a) silicon matrix (b) SiC+silicon matrix

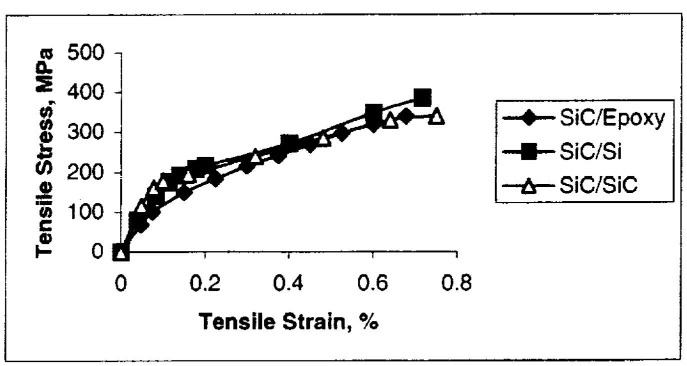


Fig. 2 Room temperature tensile stress-strain behavior of the 5HS Hi-Nicalon SiC/Epoxy, SiC/silicon, and SiC/SiC composites

DFL values for the composite suggests that the DFL value is influenced by the matrix modulus. The values of the secondary elastic modulus and the ultimate tensile strength (UTS) for the three composites appear to be similar, indicating that no significant degradation of the preforms during MI processing condition. Variation of the primary tensile elastic modulus with temperature for the SiC/silicon and SiC/SiC composites is shown in Fig.3. As indicated in this figure, the primary elastic modulus of the SiC/silicon composites is lower than that of

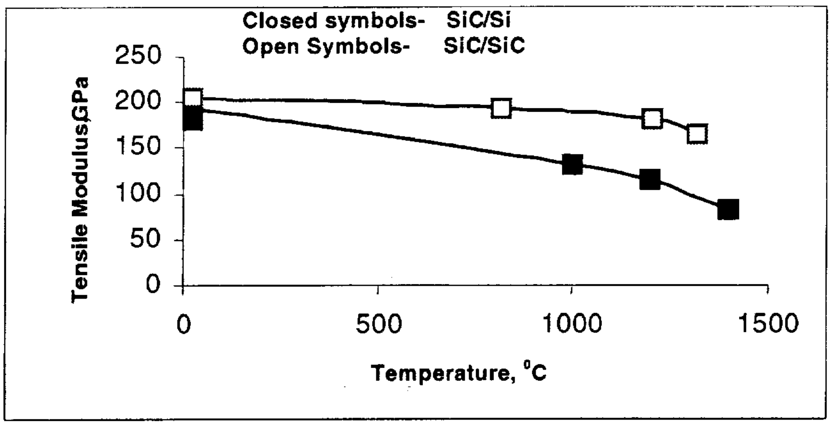


Fig. 3 Variation of primary tensile elastic modulus with temperature for the Hi-Nicalon SiC/silicon and SiC/SiC composites tested in air.

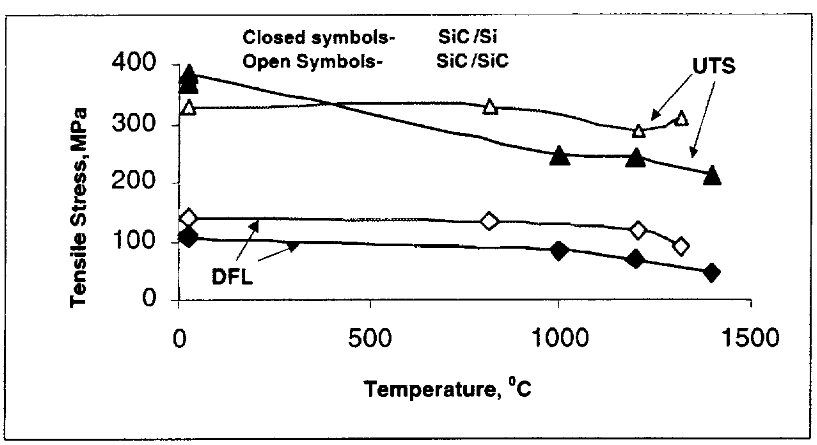


Fig. 4 Variation of stress corresponding to deviation from linearity and ultimate tensile strength with temperature for the Hi-Nicalon SiC/silicon and SiC/SiC composites tested in air.

the SiC/SiC composites. As the test temperature increases the elastic modulus for the SiC/SiC composite remains nearly the same up to 800 $^{0}$C. Beyond this temperature, it decreases gradually. On the other hand, the elastic modulus value for the SiC/Silicon composites appears to decrease continuously with increasing temperature. Figure 4 shows the variation of DFL and UTS with increasing test temperature for the two composites. Across the test temperature range, the DFL values for SiC/SiC composites are ~20% higher than those for the SiC/silicon composites. The UTS of SiC/silicon at room temperature is higher than that for the SiC/SiC composites, but above 1000 $^{0}$C, UTS for SiC/silicon composites are lower than that for the SiC/SiC composites.

**Creep properties**

The tensile creep measurements were performed at 1200 $^{0}$C in air and at stress levels below the DFL value to avoid through-the-thickness matrix cracks and environment assisted creep damage of the SiC fibers. Both of these factors are known to complicate the understanding of the creep mechanism. Figure 5 shows the tensile creep behavior for the Hi-Nicalon SiC/silicon and SiC/SiC composites tested at 35 MPa in air. It is clear from this figure that the total creep strain after 100-hr exposure for the SiC/silicon composites is significantly lower than that for the SiC/SiC composites. Figure 6 shows the total tensile creep strain accumulated at 35, 59, and 103 MPa stress levels in 100-hr creep tested specimens for both composites. According to Fig. 7, the 100-hr creep strain at the three stresses for the SiC/SiC composites is half an order-of-magnitude higher than that for the SiC/Silicon composites. Two possible reasons for the surprisingly higher creep rate in SiC/SiC composites are to partial damage to the CVI SiC coating during processing, or a thinner CVI SiC coating.

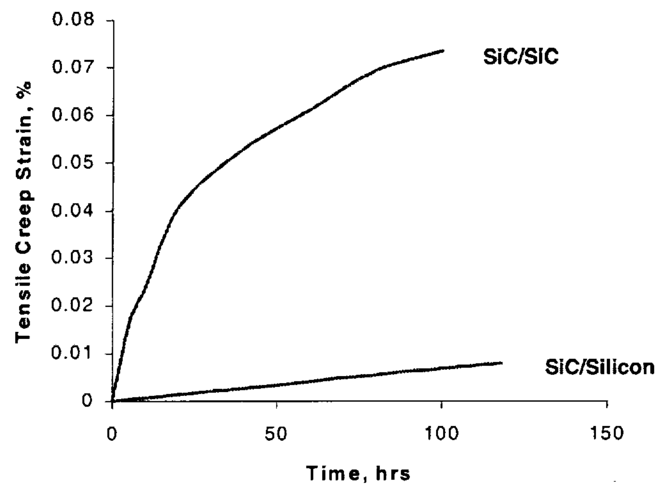


Fig. 5 Tensile creep behavior for the Hi-Nicalon SiC/silicon and SiC/SiC composites tested in air at 35 MPa.

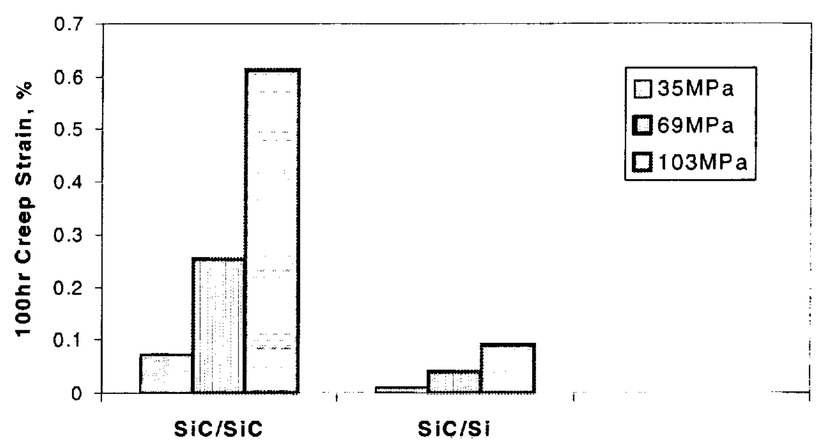


Fig. 6 Comparison of 100-hr tensile creep strain for the Hi-Nicalon SiC/SiC and Hi-Nicalon/silicon composites tested at 1200 $^{0}$C in air.

**Thermal conductivity**

Variation of the transverse thermal conductivity with temperature in argon is plotted in Fig. 7 for the Hi-Nicalon SiC/silicon and SiC/SiC composites. At room temperature, the transverse thermal conductivity for the SiC/SiC composites is higher than that for the SiC/silicon composites. As test temperature increases, the difference between the thermal conductivity of these two materials decreases, and beyond 1100 $^0$C, the thermal conductivity of both materials is nearly the same.

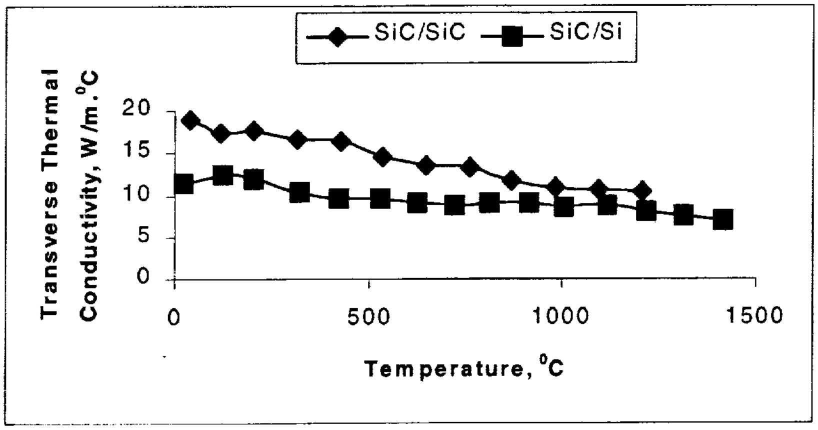


Fig. 7 Variation of transverse thermal conductivity with temperature for the Hi-Nicalon SiC/silicon and SiC/SiC composites tested in argon.

## SUMMARY OF RESULTS

Based on the limited thermo-mechanical data generated on the Hi-Nicalon SiC/silicon and SiC/SiC composites, we conclude the following.

(1) Residual silicon appears reduce to the primary elastic modulus and stress corresponding to deviation from linearity in SiC/SiC composites primarily due to lower elastic modulus of the silicon.

(2) At 1200 $^0$C, the tensile creep (at stress levels below DFL) and transverse thermal conductivity of the Hi-Nicalon SiC/SiC composites are not adversely affected by matrix residual silicon levels up to 24 vol%.

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