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# Influence of Interfacial Shear Strength on the Mechanical Properties of SiC Fiber Reinforced Reaction-Bonded Silicon Nitride Matrix Composites

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# INFLUENCE OF INTERFACIAL SHEAR STRENGTH ON THE MECHANICAL PROPERTIES OF SiC FIBER REINFORCED REACTION-BONDED SILICON NITRIDE MATRIX COMPOSITES

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## SUMMARY

The influence of fiber/matrix interface microstructure and interfacial shear strength on the mechanical properties of a fiber-reinforced ceramic composite has been evaluated. The composite consisted of ~30 vol % uniaxially aligned 142  $\mu\text{m}$  diameter SiC fibers (Textron SCS-6) in a reaction-bonded  $\text{Si}_3\text{N}_4$  matrix (SiC/RBSN). The interface microstructure was varied by controlling the composite fabrication conditions and by heat treating the composite in an oxidizing environment. Interfacial shear strength was determined by the matrix crack spacing method. The results of microstructural examination indicate that the carbon-rich coating provided with the as-produced SiC fibers was stable in composites fabricated at 1200 °C in a nitrogen or in a nitrogen plus 4 percent hydrogen mixture for 40 hr. However this coating degraded in composites fabricated at 1350 °C in  $\text{N}_2+4\%\text{H}_2$  for 40 and 72 hr and also in composites heat treated in an oxidizing environment at 600 °C for 100 hr after fabrication at 1200 °C in a nitrogen. It was determined that degradation occurred by carbon removal which in turn had a strong influence on interfacial shear strength and other mechanical properties. Specifically, as the carbon coating was removed, the composite interfacial shear strength, primary elastic modulus, first matrix cracking stress, and ultimate tensile strength decreased, but the first matrix cracking strain remained nearly the same.

## INTRODUCTION

For advanced heat engine applications, specifically for hot section components, there has been considerable interest in recent years in developing fiber-reinforced ceramic composites. These materials offer the potential for retaining the useful properties of monolithic ceramics such as low density, high temperature strength, and oxidation resistance while providing microstructural mechanisms required for improved material toughness and graceful failure characteristics. Several fiber-reinforced ceramic composites are currently being developed (refs. 1 to 3). One system which shows promise for advanced propulsion components is SiC fiber-reinforced reaction-bonded silicon nitride (ref. 3). In the as-fabricated condition, SiC/RBSN composites display a metal-like stress-strain behavior, graceful failure beyond matrix fracture, and strength properties superior to that of unreinforced RBSN of comparable density. These improvements in properties are obtained because of the use of high modulus, high strength, high purity, carbon-coated SiC fibers that did not degrade or react with the matrix to form a strong chemical bond in the thermal and environmental conditions associated with composite fabrication.

In general, the mechanical performance of a fiber-reinforced ceramic composite depends on the properties of the constituents, and on the nature and strength of the fiber/matrix interface (ref. 4). If the interface bonding between the fiber/matrix is strong, the composite displays no strain capability beyond matrix fracture because the stress intensity ahead of a propagating matrix crack is high enough to fracture the fibers and the composite fails in a brittle and catastrophic manner; a behavior similar to that of an unreinforced ceramic. On the other hand, if the bonding is too weak, the composite displays poor mechanical performance because of poor load transfer between the fiber and the matrix. In contrast, if the interface bonding is optimum, it provides both fiber-matrix load transfer and microstructural mechanisms for matrix crack deflection. In other words, the matrix crack formed at the instant the matrix fractures are able to propagate around the fibers and not through them. If the fibers are strong, they can carry the load previously carried by the matrix. With continued loading, the matrix block on either side of the main crack can load up until they fracture. This process continues and the resulting composite fracture appears as a series of macrocracks that are bridged by the reinforcing fibers. The ultimate composite failure occurs when the fiber bundle sharing load through the interface and matrix fractures at a strain typically much greater than that of the matrix fracture strain. Therefore, by engineering the microstructure and interface strength, it is possible to develop strong and tough fiber reinforced ceramic composites.

The shear strengths of interfaces have been measured or estimated by at least four different techniques: matrix crack spacing (ref. 5), fiber push-in (ref. 6), fiber push-out (refs. 7 and 8), and single fiber pull-out (ref. 8). These techniques measure either the interfacial debond strength,  $\sigma_b$ , which reflects the degree of chemical bonding between fiber and matrix, or the interfacial frictional strength,  $\sigma_f$ , which relates to slippage of fibers in the matrix, or both. The theoretical basis for each technique and its advantages and disadvantages are discussed elsewhere (ref. 4).

The objective of this study was to determine the influence of interfacial microstructure and shear strength on the important mechanical properties of unidirectionally reinforced SiC/RBSN composites. This basic understanding is necessary to develop and evaluate current micromechanics models as well as to optimize composite properties. The interfacial microstructure in the composite was varied by changing the fabrication conditions and by heat treating the composites in an oxidizing environment. The interfacial shear strength was determined from the matrix crack spacing method.

## EXPERIMENTAL

### Composite Fabrication

The SCS-6 SiC monofilaments and high purity silicon powder were used as starting materials for SiC/RBSN composite fabrication. The SiC fibers of nominal diameter 142  $\mu\text{m}$  and the silicon powder of average particle size 3  $\mu\text{m}$  were obtained from Textron Specialty products and Union Carbide, respectively. A detailed description of the composite fabrication method is reported elsewhere (ref. 10). Briefly, the composites were fabricated by a three step process. In the first step, SiC fiber mats and silicon cloth were prepared using polymer fugitive binders. In the second step, alternate layers of SiC fiber mats and

silicon cloths were stacked in a metal die and pressed in a vacuum hot press under an applied stress from 27 to 200 MPa for up to 1 hr in the temperature range from 600 to 1000 °C. In the third step, the consolidated SiC/Si preforms were then heat-treated in a high purity (>99.99 percent) nitrogen environment or in a N<sub>2</sub>+4%H<sub>2</sub> mixture in the temperature range from 1000 to 1400 °C for up to 100 hr to convert them to SiC/RBSN composites. The typical dimensions of the as-nitrided composite panels were 150 by 50 by 2.2 mm.

### Specimen Preparation and Testing

Tensile test specimens from the composite panels nitrided under the table I conditions were prepared by cutting and grinding them with a diamond impregnated abrasive wheel. Nominal dimensions of the specimens were 127 by 12.7 by 2.0 mm. To evaluate oxidation effects on interfacial microstructure, tensile specimens from panels nitrided in nitrogen only were heat treated in flowing oxygen at 600 °C for 100 hr. To prepare all specimens for tensile testing, glass fiber reinforced epoxy tabs were adhesively bonded at the specimen ends leaving 50 mm as the test gauge length. A wire wound strain gauge was adhesively bonded to the specimen gauge section for monitoring axial strains. The tensile stress-strain behavior for each specimens was measured at room temperature in an Instron machine at a crosshead speed of 1.3 mm/min. For each treatment condition, at least five specimens were tested and the average values of composite modulus, first matrix cracking stress, and ultimate strength were determined. The fractured specimens were examined using optical and SEM microscopy to determine fiber-matrix interfacial shear strength from matrix crack spacing method (ref. 5) and composite failure behavior.

### Microstructural Characterization

Polished cross sections of composites were prepared for microstructural analysis by grinding the specimens successively on 40-, 30-, 15-, 10-, 6-, and 3- $\mu$ m diamond impregnated metal disks and finally by polishing them in a vibratory polisher on microcloth using 0.03  $\mu$ m diamond powder. The polished specimens were ultrasonically cleaned in alcohol, dried, and sputter coated with thin layer of pyrolytic carbon. These specimens were examined in a scanning electron microscope equipped with an energy dispersive x-ray spectrometer (EDAX).

## RESULTS

### Microstructural Analysis

A representative photomicrograph of a polished cross-section is shown in figure 1(a) for a 30 fiber vol % SiC/RBSN composite specimen that was fabricated at 1200 °C in nitrogen for 40 hr. The composite matrix contained ~30 vol % porosity and displayed local density variations around the fibers. The SCS-6 fiber itself can be considered as a microcomposite and consists of a pyrolytic carbon core, SiC sheath, and carbon-rich surface coating. The SiC sheath contains two zones; the inner zone, referred to as A in figure 1(b),

contains carbon-rich SiC and the outer zone, referred to as B in the same figure, is essentially stoichiometric SiC (ref. 11). The outer surface of the SiC sheath contains two layers of a carbon-rich coating.

Representative secondary electron, and carbon and oxygen elemental x-ray images of the cross sections of composites fabricated in nitrogen, in nitrogen plus 4 percent hydrogen, and in nitrogen, but oxidized in oxygen at 600 °C for 100 hr are shown in figure 2. Due to cracking of the fibers during specimen preparation, microstructural analysis was not performed on composites fabricated in  $N_2+4\%H_2$  for 72 hr. Figure 2 shows that the carbon-rich surface coating is stable in composites fabricated in nitrogen but those fabricated in  $N_2+4\%H_2$  at 1350 °C for 40 hr or heat treated in oxygen at 600 °C for 100 hr showed loss of carbon in the coating by reaction with hydrogen or with oxygen, respectively.

### Mechanical Properties

Typical room temperature tensile stress-strain curves are shown in figure 3 for the various treatment conditions. In general, the curves show either two linear regions separated by a nonlinear region or a initial linear region only. In all cases the deviation from linearity in the stress-strain curve originated with the onset of a through thickness matrix macrocrack normal to the fibers. The stress corresponding to the deviation from the first linear region is considered as the first matrix cracking stress. With continued loading, additional matrix cracks were formed at regular intervals along the gauge length. Comparison of the stress-strain curves in figure 3 shows that the first matrix cracking stress value and the modulus or slope of the initial linear region for the composites fabricated at 1350 °C in a nitrogen plus 4 percent hydrogen and for the composites heat treated in oxygen were significantly lower than the values measured for the composites fabricated at 1200 °C only. Moreover, with continued loading beyond their maximum load carrying capability, these specimens showed continuous loss in load, resulting in a long descending tail to the stress-strain curve. In contrast, the 1200 °C specimens demonstrated strain capability beyond matrix fracture and very high ultimate tensile strength.

Examination of fractured tensile specimens by optical and scanning electron microscopy showed that the composites fabricated at 1200 °C fractured in a broom-like manner with very little matrix around the fibers. On the other hand, composites fabricated at 1350 °C for 40 hr or heat treated in oxygen failed with extensive fiber pull out and widely spaced matrix microcracks. Those fabricated at 1350 °C for 72 hr fractured with single matrix crack and fiber pull out from the grip. The optical photographs of representative fractured specimens of composites are shown in figure 4.

To determine the influence of interface microstructure on fiber/matrix bond integrity, the interfacial shear strength,  $\sigma_f$ , was calculated using the Aveston, Cooper and Kelly (ACK) theory (ref. 5), the measured values of the matrix crack spacing, the first matrix cracking stress, and the elastic modulus values of  $E = 390$  GPa (ref. 12) and  $E = 110$  GPa (ref. 3) for the SiC fibers and RBSN matrix, respectively. The average matrix crack spacing was determined from direct optical measurement after the tensile specimens were loaded well beyond the first matrix cracking stress--typically to 75 percent of the average

ultimate strength of the composites--and then unloaded. The average values of the first matrix cracking stress, matrix crack spacing, interfacial shear strength, and other mechanical properties are shown in table I. Error indications are for one standard deviation.

Using the table I data, the primary elastic modulus, and first matrix cracking stress are plotted as a function of interfacial shear strength in figures 5 and 6, respectively. Both these figures show that as the interfacial shear strength increases the primary elastic modulus and the first matrix cracking stress increases. Also shown in figure 6 is the predicted variation of first matrix cracking stress with interfacial shear strength based on Marshall and Evan's theory (ref. 13) using a fracture toughness value range between 2 and 2.5 measured for RBSN containing ~30 vol % porosity (ref. 14).

## DISCUSSION

In general, interfacial shear strength in a ceramic composite depends on the degree of chemical interaction between the fiber and matrix, the interfacial microstructure, the nature and magnitude of radial residual stresses acting on the interface, and the elastic properties of the constituents. Since the SiC/RBSN composite preforms and fully nitrided composite panels contain interconnected porosity, we were able to modify the interface microstructure during fabrication or by post fabrication oxidation treatments without significantly affecting the matrix properties or the residual stresses acting on the interface.

Results of this study have clearly indicated that the interface microstructure of SiC/RBSN composites, i.e., the microstructure of the carbon-rich coating on SCS-6 fiber, has significant influence on the interfacial shear strength which in turn affects the mechanical properties of the composites. If this coating is degraded during processing or during environmental exposures, the composite shows loss in properties. The effect of interfacial shear strength on the four key mechanical properties, namely, primary elastic modulus, first matrix cracking stress and strain, and ultimate tensile strength can be understood from composite mechanics and the interfacial microstructural observations.

### Elastic Modulus

The primary elastic modulus of a unidirectionally reinforced composite loaded along the reinforcement direction depends on the elastic moduli and volume fractions of the constituents, and the nature of bonding between the fiber and matrix. If we assume that adequate load transfer occurs between the fiber and matrix, then the elastic modulus should be equivalent to that predicted from the rule of mixtures, and should be independent of interfacial shear strength. The fact that the elastic modulus values of SiC/RBSN composites having interfacial shear strength values greater than 10 MPa were similar to a value of 193 GPa predicted from the rule of mixtures indicates good load transfer between fiber and matrix (cf., fig. 5). On the other hand, the elastic modulus values of composites showing interfacial shear values lower than 10 MPa were significantly lower than the rule of mixtures value suggesting that the physical contact or the bonding between the fiber and the matrix is affected.

Microstructural results presented in figure 3 reveal that the loss of bonding is due to a loss of the carbon coating during fabrication in hydrogen or during secondary heat treatment in oxygen.

### First Matrix Cracking Stress and Strain

The first matrix cracking stress is affected by the interfacial shear strength, the elastic properties of the matrix and the fiber, and the fracture toughness of the matrix. The fracture mechanics theories (refs. 4 and 12) predict that the first matrix cracking and strain should increase with increasing interfacial shear strength. For the SiC/RBSN composites showing interfacial shear strength greater than 10 MPa, the predicted values of first matrix cracking stress were in reasonable agreement with the measured values (cf., fig. 6). On the other hand, for the composites yielding interfacial shear strength values less than 10 MPa, the predicted first matrix cracking stress values were significantly lower than the measured values. These theories (ref. 12) also predict that first matrix fracture strain should follow a behavior similar to that of the first matrix cracking stress with increasing interfacial shear stress. However, this behavior was not observed in SiC/RBSN where varying interfacial shear strength had no significant effect on first matrix fracture strain. Explanations for these deviations between experiment and theory have yet to be developed.

### Ultimate Tensile Strength

The ultimate tensile strength of the composite is primarily controlled by the bundle strength of the fibers. The bundle strength is also a function of gauge length and decreases with increasing gauge length. Any factors which affect individual fiber properties generally also affect fiber bundle strength. Certainly, the loss of ultimate tensile strength of SiC/RBSN composites fabricated or heat treated under different conditions might be attributed to loss of bundle strength of fibers due to hydrogen or oxygen reaction. However, the data shown in table I indicate that ultimate tensile strength of SiC/RBSN composites decreases significantly with decreasing interfacial shear strength, suggesting that the loss in interfacial strength may be more important than the loss in individual fiber strength. Although the theoretical reasoning for such a relationship is not fully understood as yet, one possible interpretation is that loss of interfacial shear strength increases the effective gauge length of the fiber bundle in the composite which in turn has a direct influence on the ultimate tensile strength of the composite.

### SUMMARY OF RESULTS

The effect of interface microstructure and interfacial shear strength on the mechanical properties of SiC/RBSN composites have been determined. The major findings are as follows:

1. Interfacial shear strength of SiC/RBSN composites is controlled by the integrity of the carbon-rich fiber surface coating. Under fabrication and heat treatment conditions where the carbon-rich coating remains intact, SiC/RBSN composites display strong and tough composite behavior.



2. Fabrication of composites in hydrogen containing nitrogen or oxidation of composites at low temperature causes degradation of the carbon-rich surface coating which in turn causes degradation in interfacial shear strength and other important mechanical properties, such as, composite modulus, first matrix cracking strength, and ultimate tensile strength.

3. To maintain strong and tough composite behavior in SiC/RBSN composite after fabrication or after post-fabrication heat treatments, an interfacial shear strength value greater than 10 MPa is desirable.

#### CONCLUSION

For the fabrication and maintenance of strong, tough SiC fiber reinforced RBSN, a stable carbon-rich interface coating with an interfacial shear strength of 10 MPa minimum is required. Because of potential environmental degradation of the current carbon based fiber surface coating during processing or use, an alternative coating system is needed.

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TABLE I. - MECHANICAL PROPERTY DATA FOR SiC/RBSN COMPOSITES ( $V_f \sim 0.3$ )

Treatment	Primary elastic modulus, GPa	First matrix cracking stress, MPa	First matrix cracking strain, percent	Average matrix crack spacing, mm	Interfacial shear strength, MPa	Ultimate tensile strength, MPa
Fabricated at 1200 °C in N <sub>2</sub> for 40 hr	193±7	227±40	0.12	0.8±0.2	18±4	692±150
Fabricated at 1200 °C in N <sub>2</sub> +4%H <sub>2</sub> for 40 hr	190±8	217±30	.11	1.0±.4	14±3	440±110
Fabricated at 1350 °C in N <sub>2</sub> +4%H <sub>2</sub> for 40 hr	150±6	176±35	.12	7.0±.5	1.8±5	320±56
Fabricated at 1350 °C in N <sub>2</sub> +4%H <sub>2</sub> for 72 hr	90±8	46±10	.05	-----	-----	46±10
Fabricated at 1200 °C in N <sub>2</sub> for 40 hr and then treated in oxygen at 600 °C for 100 hr	140±10	154±30	.11	12±2	0.8±.4	230±50

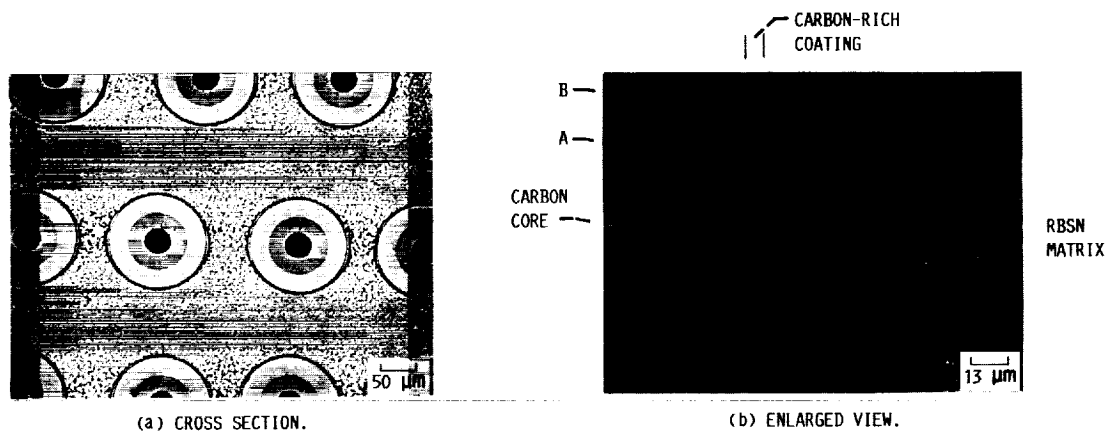
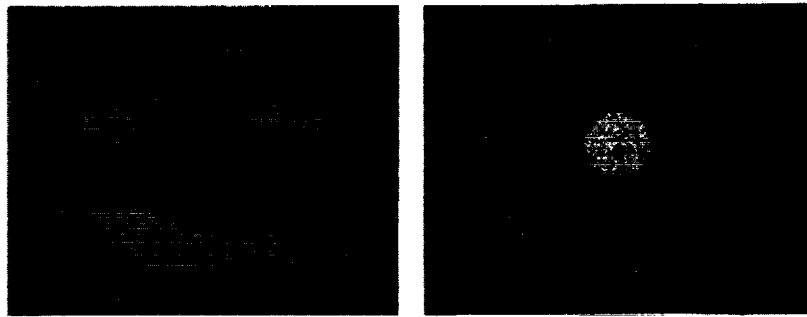
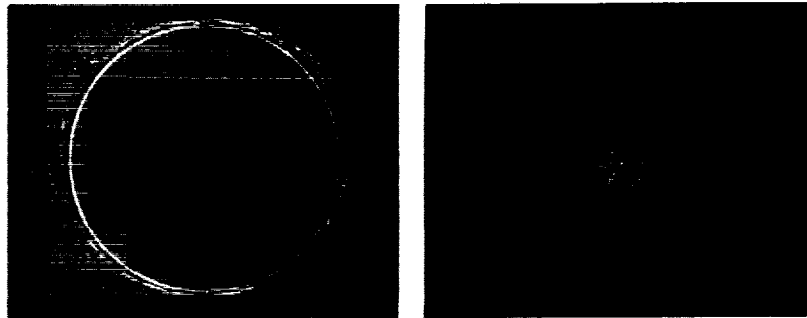


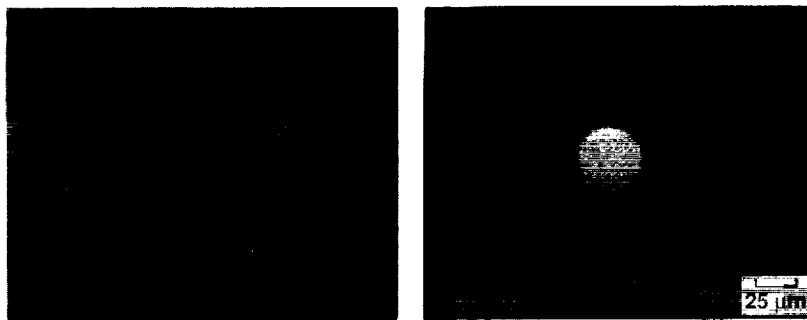
FIGURE 1. - TYPICAL CROSS SECTION OF SiC/RBSN COMPOSITE FABRICATED AT 1200 °C IN NITROGEN SHOWING FIBER DISTRIBUTION AND MATRIX POROSITY (~30 FIBER VOLUME PERCENT) AND ENLARGED VIEW OF FIBER-MATRIX INTERFACE.



(a)

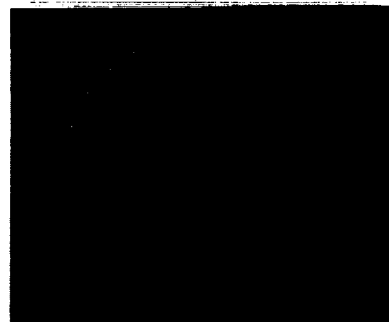


(b)



SECONDARY ELECTRON

CARBON X-RAY



OXYGEN X-RAY

(c)

FIGURE 2. - SEM MICROGRAPHS AND ELEMENTAL X-RAY IMAGES FOR SiC/RBSN COMPOSITES. (a) FABRICATED IN NITROGEN AT 1200 °C FOR 40 HR. (b) FABRICATED IN A N<sub>2</sub>+4%H<sub>2</sub> MIXTURE AT 1350 °C FOR 40 HR. (c) FABRICATED IN CONDITION (a) AND THEN HEAT TREATED IN OXYGEN AT 600 °C FOR 100 HR.

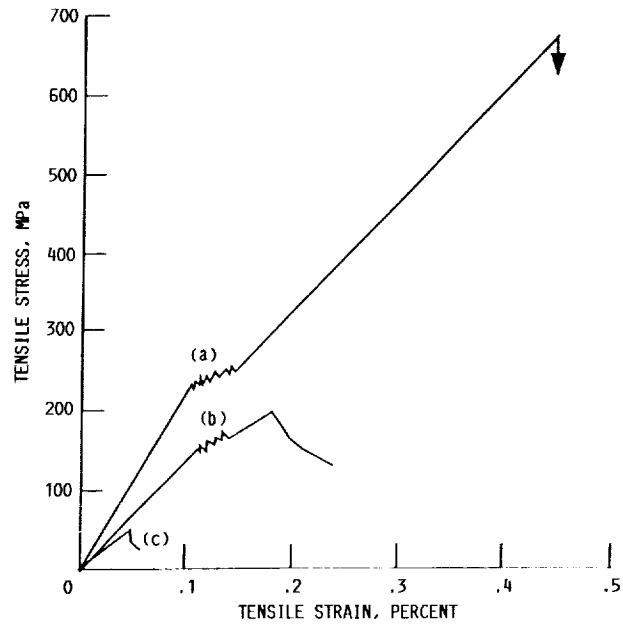


FIGURE 3. - ROOM TEMPERATURE STRESS-STRAIN CURVES FOR SiC/RBSN COMPOSITES TREATED UNDER VARIOUS CONDITIONS (a) FABRICATED IN A NITROGEN ENVIRONMENT AT 1200 °C FOR 40 HR., (b) FABRICATED IN A N<sub>2</sub>+4%H<sub>2</sub> MIXTURE AT 1350 °C FOR 40 HR., OR FABRICATED IN CONDITION (a) AND THEN HEAT TREATED IN OXYGEN AT 600 °C FOR 100 HR., (c) FABRICATED IN N<sub>2</sub>+4%H<sub>2</sub> MIXTURE AT 1350 °C FOR 72 HR.

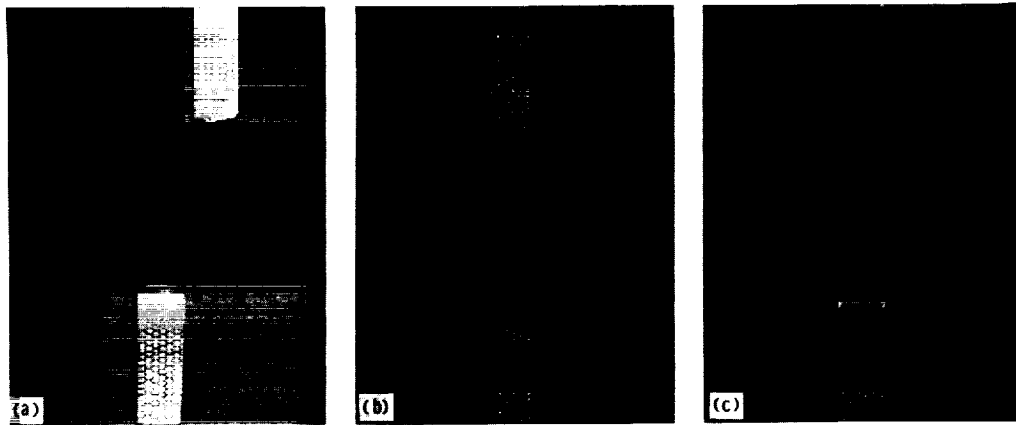


FIGURE 4. - FRACTURED TENSILE SPECIMENS OF SiC/RBSN COMPOSITES, (a) FABRICATED IN NITROGEN AT 1200 °C FOR 40 HR., (b) AND (c) FABRICATED IN N<sub>2</sub>+4%H<sub>2</sub> MIXTURE AT 1350 °C FOR 40 HR AND 72 HR., RESPECTIVELY.

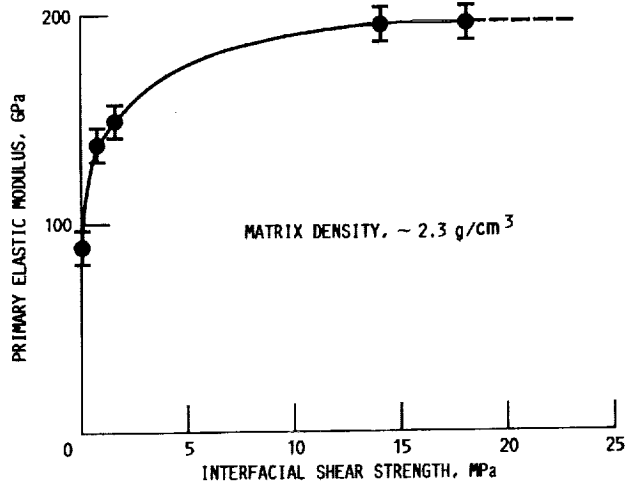


FIGURE 5. - VARIATION OF PRIMARY ELASTIC MODULUS WITH INTERFACIAL SHEAR STRENGTH.

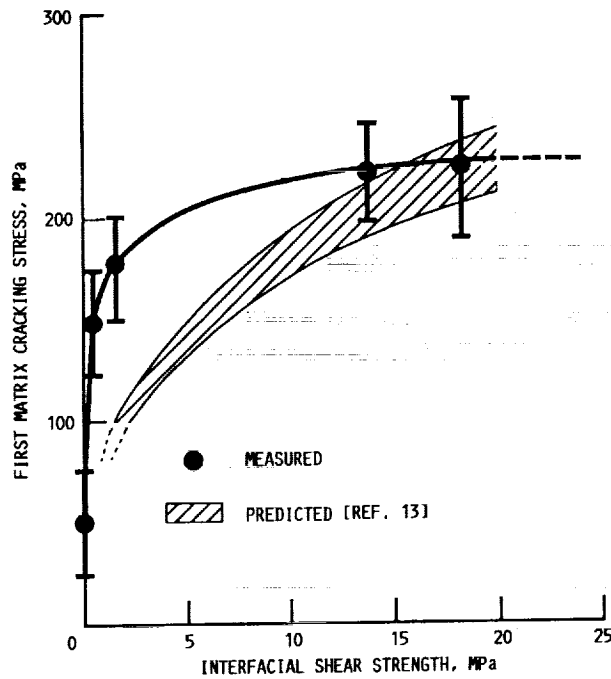


FIGURE 6. - VARIATION OF FIRST MATRIX CRACKING STRESS WITH INTERFACIAL SHEAR STRENGTH.

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