MECHANICAL PROPERTIES OF HIGH PURITY SiC FIBER-REINFORCED CVI-SiC MATRIX COMPOSITES

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Mechanical properties of silicon carbide composites reinforced with highly crystalline fibers and fabricated by the chemical vapor infiltration method were evaluated. Materials used were SiC/SiC composites reinforced with unidirectional Hi-Nicalon Type-S fibers and unidirectional Tyranno SA fibers with various fiber/matrix interphase. Also, SiC/SiC composites reinforced with plain weave Tyranno SA fibers with carbon or multilayers of silicon carbide and carbon interphase were evaluated. In-plane tensile, transthickness tensile and interlaminar shear properties were evaluated by the in-plane tensile test, the transthickness tensile test, the diametral compression test and the compression test of double-notched specimens.

The elastic modulus and proportional limit stress were improved by using high purity silicon carbide fibers. The in-plane tensile properties were insensitive to carbon interphase thickness for a range of thicknesses between 30 and 230 nm. It was found that the in-plane tensile strength of composites containing multilayers of silicon carbide and carbon coating of fibers and fiber bundles was superior to that of composites with carbon alone. Transthickness tensile strength and shear strength of high purity silicon carbide composites were successfully evaluated.

I. INTRODUCTION

Silicon carbide (SiC) can be used in harsh environments due to its thermal, mechanical and chemical stability. SiC also provides exceptionally low radioactivity under neutron irradiation.[1] The intrinsic features of SiC make SiC fiber-reinforced SiC matrix composites (SiC/SiC composites) attractive structural materials for nuclear applications, since SiC/SiC composites can overcome the inherently brittle fracture behavior of monolithic SiC ceramics.[2] SiC/SiC composites usually exhibit higher fracture toughness and less scatter of mechanical properties than SiC ceramics. At the same time, the excellent intrinsic features of SiC ceramics are retained.

SiC/SiC composites fabrication processes such as chemical vapor infiltration (CVI),[3-5] reaction sintering,[6] liquid phase sintering,[7] polymer impregnation and pyrolysis,[8] chemical vapor reaction[9] and their combined processes[10,11] are being developed. The CVI process produces a high performance SiC/SiC composite. The major advantage of CVI over other processing routes is the low thermal and mechanical stress achieved during the densification process, in large part due to the lower deposition temperature. The process imparts little mechanical stress to the preform. In addition, CVI produces a stoichiometric, crystalline β-SiC which is fairly stable to neutron irradiation.[12] The excellent control of the fiber/matrix interphase formation, which significantly affects mechanical properties,[13] is also an advantage of the method. The relatively high production cost and long fabrication time compared with the other fabrication methods are disadvantages. Nevertheless advanced SiC/SiC composites fabricated by the CVI method play an important role in the advanced research and the establishment of reference properties for the other methods.

The physical and mechanical properties of continuous fiber-reinforced ceramic matrix composites (CFCCs) depend on the properties of their various constituents, their geometry and concentration (e.g., volume fraction of fibers, fiber/matrix interphase structure, fiber weave architecture and matrix properties). In particular, the reinforcing fibers and the fiber/matrix interphase control the in-plane tensile strength and the fracture behavior of the composite.[14]

Significant degradation of interfacial bonding between the fiber and matrix of neutron-irradiated SiC/SiC composites limited mechanical performance.[15] This

Table I. Properties of Nicalon ™ and Tyranno™ SiC-based fibers

SiC Fiber	C/Si Atomic Ratio	Oxygen Content (wt%)	Tensile Strength (GPa)	Tensile Modulus (Gpa)	Elongation (%)	Density (Mg/m^3)	Diameter (μm)
Nicalon	1.31	11.7	3.0	220	1.4	2.55	14
Tyranno Lox M	1.37	11	3.3	187	1.8	2.48	11
Hi-Nicalon	1.39	0.5	2.8	270	1.0	2.74	14
Tyranno TE	1.59	5.0	3.4	206	1.7	2.55	11
Hi-Nicalon Type-S	1.05	0.2	2.6	420	0.6	3.10	12
Tyranno SA	1.08	<1	2.8	380	0.7	3.10	10, 7.5

degradation has been attributed primarily to shrinkage in the SiC-based fibers due to irradiation-induced grain growth of microcrystalline fibers,[16,17] irradiation-assisted oxidation,[18] and potentially large dimensional changes of the C interphase,[17] while matrix swells a little by irradiation-induced point defect. Fiber shrinkage leads to fiber/matrix debonding[19] and a decrease in elastic modulus and fracture strength. Therefore, there is a critical need to optimize the microstructure of SiC/SiC composites (i.e. fiber, fiber/matrix interphase and matrix) to retain the fiber/matrix interfacial shear strength. To mitigate radiation effects, the recent trend in SiC fiber development is toward lower oxygen content, reduced free carbon (C) and enhanced crystallinity. The development of more radiation-resistant SiC composites is based on the use of highly crystalline SiC fibers and the fiber/matrix interphase with reduced C or multilayer of reduced C and SiC. Recently, highly crystalline SiC fibers have been developed including Hi-Nicalon™ Type-S[20] (Nippon Carbon Co., Ltd), Sylramic™[21] (Dow Corning Co.) and Tyranno™ SA[22] (Ube Industries, Ltd). Therefore the evaluation of the SiC/SiC composites reinforced with the highly crystalline fibers is desired. Properties of representative SiC fibers reported by the manufactures are summarized in Table I.

It is difficult to obtain reliable mechanical properties of SiC/SiC composites by flexural tests, although mechanical properties of SiC/SiC composites have been evaluated mostly by flexural tests due to the simplicity of conducting these tests. Flexural loading includes tensile stresses on the tensile side of the specimen, compressive stresses on the compression side and shear stresses across the thickness of the beam. Despite the experimental simplicity of this test, interpretation of the results is difficult because of stress redistribution through thickness following matrix cracking, which is neither linear nor symmetrical through thickness, [23] and asymmetric behavior between tension and compression of SiC/SiC composites.[24] To appreciate mechanical properties of SiC/SiC composites, in-plane tensile is preferred since it is easier to model and analyze data in contrast to flexural data.

The objective of this work is to develop or implement more advanced quantitative mechanical tests to assess properties of SiC/SiC composites for nuclear application.

II. MATERIALS

The materials used were unidirectional (UD) SiC fiber-reinforced SiC matrix composites fabricated by isothermal chemical vapor infiltration (ICVI) at Hyper-Therm High-Temperature Composites, Inc. and plain weave (P/W) SiC fiber-reinforced SiC matrix composites fabricated by the forced-flow, thermal gradient chemical vapor infiltration (FCVI) at Oak Ridge National Laboratory (ORNL). Fibers used were Hi-Nicalon Type-S and Tyranno SA for ICVI and Tyranno SA for FCVI. Prior to matrix infiltration the fibers were coated with either C with various thickness or multilayer C/SiC by CVI. Details of the material and its fabrication can be found elsewhere.[3,25]

III. IN-PLANE TENSILE BEHAVIOR

Fracture behavior of composites is completely different from those of monolithic materials. The underlying phenomenology involves matrix cracking, fiber/matrix interfacial sliding and fiber failure. Figure 1 shows typical tensile stress-strain curves for a SiC/SiC composite reinforced with UD Hi-Nicalon Type-S fibers with a C coating and a fiber volume fraction (V_f) of 29 %, and a reference SiC/SiC composite reinforced with UD Hi-Nicalon fibers with a C coating and a fiber volume fraction of 40 %. The curves consist of linear elastic region up to proportional limit stress (PLS) (a)-(b) and nonlinear inelastic region (b)-(c). Matrix cracks initiate and interact with intact fibers where the crack is deflected along the interface. This crack deflection causes the fiber/matrix to debond and slide. As a result the residual stress distribution changes. Frictional dissipation occurs at the fiber/matrix interfaces and depends upon the type of fiber coating, the fiber morphology, and topography of the fiber surface. By varying the magnitude of the interfacial frictional stress, the prevalent damage mechanism and the resultant nonlinear stress-strain behavior can be dramatically modified.

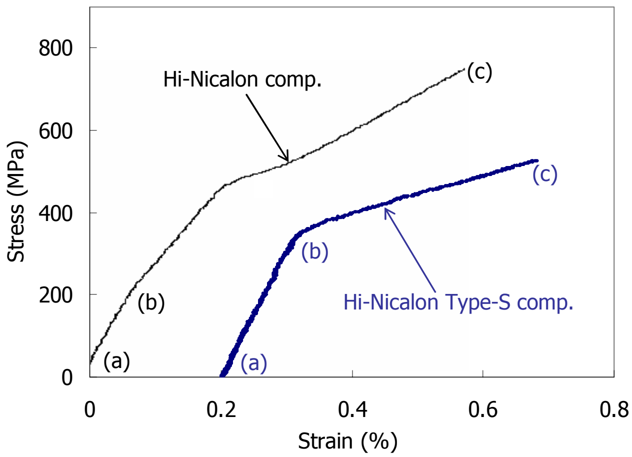

Figure 1. Typical stress-strain curves obtained from in-plane tensile tests of UD SiC/SiC composites reinforced with Hi-Nicalon (V_f: 40%) or Hi-Nicalon Type-S fibers (V_f: 29%).

Theoretical studies describing the energy dissipation by fiber pullout and fracture characteristics of CFCCs have been presented.[26,27] According to Curtin, the ultimate tensile strength (UTS) (σ_u) of UD CFCCs is;[15]

$$\sigma_u = f\sigma_c \left(\frac{2}{m+2}\right)^{1/m+1} \left(\frac{m+1}{m+2}\right) \quad (1)$$

$$\sigma_c = \left(\frac{\sigma_0^m \tau L_0}{r}\right)^{1/m+1} \quad (2)$$

where f is the fiber volume fraction, σ_c is the relevant fiber strength, m is the Weibull modulus describing the variability in fiber strengths, σ_0 is the mean strength of the fiber in a length of L_0, τ is the sliding resistance at fiber/matrix interface and r is the radius of the fiber. As described in Eq. (1) and (2), the UTS is dominated by fiber volume fraction, fiber properties and fiber/matrix interfacial properties. These theoretical models are basically for an UD composite, but they can be applied to 2D and 3D composites with f replaced by the fiber volume fraction of 0º plies (f_e) (e.g., f_e=f/2 in a 0º/90º 2D composite), when a stress concentration attributed to cracks originated in the 90º plies is small. In 2D and 3D composites, the UTS depends primarily on the fiber volume fraction of 0º plies.[28]

The matrix cracking stress, σ_{mc}, is described by the following equation;[26]

$$\sigma_{mc} = \left(\frac{6\tau\Gamma_m}{r} \frac{f^2 E_f E^2}{(1-f)E_m^2}\right)^{1/3} \quad (3)$$

where Γ_m is the matrix fracture energy, and E_f, E and E_m are the elastic moduli of fiber, composite and matrix, respectively. The elastic modulus of a composite with no porosity is determined by the following equation;

$$E = fE_f + (1\text{-}f)E_m \quad (4)$$

The matrix cracking stress, below which composites show elastic behavior, is the most important factor, because it is a critical stress for fatigue and creep. The matrix cracking stress is directly related to PLS obtained from stress-strain curve.

Minor revision including porosity is required to apply Eq. (3) for CVI SiC/SiC composites. Eq. (3) shows that a higher sliding resistance, a smaller fiber radius, a larger fiber volume fraction, and a larger fiber and composite elastic modulus increase the matrix cracking stress. Pores decrease the matrix cracking stress, because they decrease the composite elastic modulus. Also pores may cause stress concentration.

Highly crystalline SiC fibers have advantage of stability at high temperature and under neutron irradiation. In addition, most mechanical properties of composites reinforced with the highly crystalline fibers should be superior to those of composites reinforced with the less crystalline fibers. The elastic modulus of composites reinforced with highly crystalline fibers should be larger than that of composites reinforced with the other fibers, because the elastic moduli of highly crystalline fibers are larger than those of less crystalline fibers. The matrix cracking stress of composites reinforced with the highly crystalline fibers also should be larger than that of composites reinforced with the less crystalline fibers. The matrix cracking stress depends on fiber radius and elastic moduli of fiber and composite according to Eq. (3). Typically highly crystalline fibers have smaller radii and the elastic moduli of fibers and composites are larger than those of composites reinforced with less crystalline fibers. The frictional resistance at fiber/matrix interface of composites reinforced with highly crystalline fibers tends to be large due to rougher fiber surface attributed to larger grain size. The UTS depends on mean fiber strength, fiber radius, interfacial frictional resistance and Weibull modulus. Tensile strength of the highly crystalline fibers is smaller than that of the less crystalline fiber, while radii of highly crystalline fibers are smaller. The interfacial frictional resistance of the highly crystalline fibers is larger than that of the less crystalline fibers.

Typical results of effect of fiber properties are shown in Figure 1, although fiber volume fraction and thickness of C interphase are different. The elastic modulus and PLS of the composite reinforced with Hi-Nicalon Type-S fibers are larger than those of the composite reinforced with Hi-Nicalon fibers, while the UTS of the composite reinforced with Hi-Nicalon Type-S fibers is smaller than that of the composite reinforced with Hi-Nicalon fibers due to the difference in fiber volume fraction. The UTS is directly proportional to fiber volume fraction according to Eq. (1). If both of the UTS are normalized by fiber volume fraction (e.g. UTS is divided by fiber volume fraction), UTS of composites reinforced with Hi-Nicalon Type-S and Hi-Nicalon is 1814 MPa and 1875 MPa, respectively.

Fiber/matrix interfacial properties also are important to achieve improved mechanical properties of composites. Normally C or BN is used as fiber/matrix interphase. Some investigations of the effect of fiber/matrix interphase on mechanical properties of SiC/SiC composites have been reported.[29-31] However the effect of C interphase thickness on tensile properties of composites reinforced with highly crystalline fibers have not been reported. Figure 2 shows the effect of C interphase thickness on PLS and UTS of SiC/SiC composites reinforced with P/W Tyranno SA fibers. Tensile properties of the composites were not affected by C interphase thickness in the range of 30 through 230 nm thickness at ambient temperature. These results are different from the flexural results of composites reinforced with less crystalline fibers. It is known that the mechanical properties of interphase and composite are affected by C interphase thickness. Interfacial shear strength is reduced by increasing thickness of the interphase. Too thin an interphase induces brittle fracture behavior, while too thick an interphase decrease mechanical properties with less load transfer at the interphase. Flexural properties might be more sensitive to interfacial properties than tensile properties. The fiber surface of highly crystalline fiber is rougher than that of less crystalline fiber. Interfacial frictional resistance of composites reinforced with highly crystalline fiber is relatively large and might be more insensitive to the thickness of interphase. The elastic moduli of highly

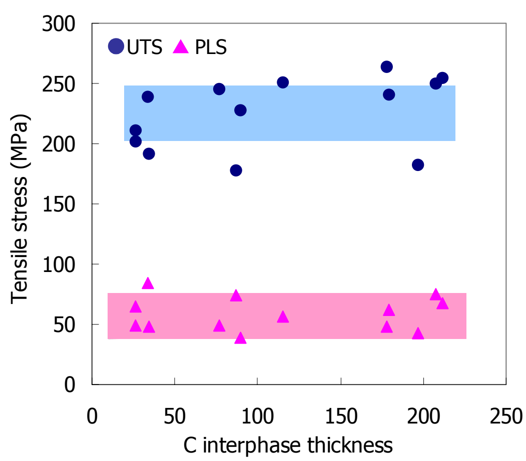

Figure 2. The effect of C interphase thickness on tensile properties of SiC/SiC composites reinforced with plain weave Tyranno SA fibers.

crystalline fibers are similar to those of the CVI matrix. The role of the interphase for composites reinforced with highly crystalline fibers for load transfer isn't as important as that of composites reinforced with less crystalline fibers. It is encouraging and important result that the C interphase can be reduced to 30 nm thickness or perhaps even smaller. For applications in a high temperature air environment and the fusion environment, the C interphase is the weakest link due to oxidation and deformation by neutron irradiation. Mechanical properties in a severe environment can be improved by using reduced C interphase.

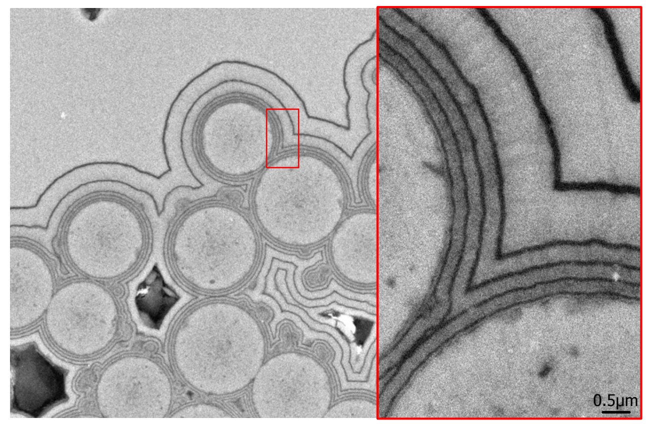

Figure 3. SEM images of composites with SiC/C multilayer interphase showing effective individual fiber and fiber bundle coating.

Another approach to improve the interfacial mechanical properties is the multilayer interphase of SiC and thin C. Figures 3 show SEM images of SiC/C multilayer interphase. The first thin SiC layer (50 nm) is to strengthen bond between fiber and interphase to prevent crack propagation between fiber and interphase and induce crack propagation within interphase.[14] The next three SiC layers (200 nm) are for multiple interfacial fractures of fibers. The next two thick SiC layers (500 nm) are for

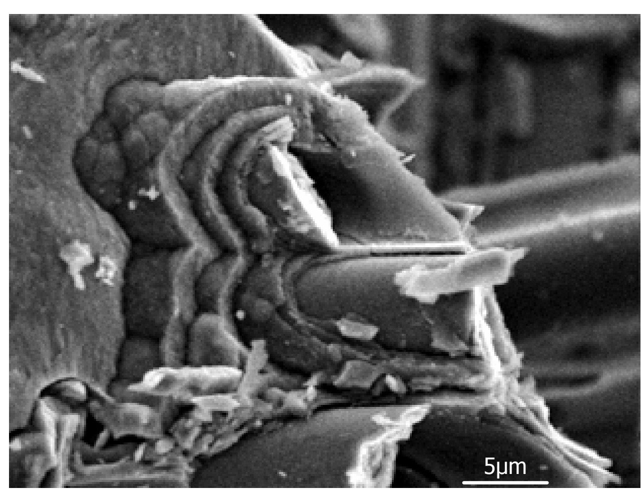

Figure 4. Tortuous fracture surface through the fiber/matrix interphase for composite with SiC/C multilayer.

multiple fractures of fiber bundles. Thin C layers (50 nm) are used just to separate SiC layers. The composites with the multilayer SiC/C interphase showed multiple fracture layers in the interphase as shown in Figure 4. The tensile strength of the 2D composites with the multilayer was about 300 MPa and nearly 30 % higher than the 2D composites with single C interphase.

IV. EVALUATION OF TRANSTHICKNESS TENSILE STRENGTH

The tensile strength perpendicular to the lay-up planes of 2D laminated composites (transthickness tensile strength: TTS) is typically much lower than the strength of the composite on the lay-up plane. Figure 5 schematic shows the relationship of fiber direction, in-plane tensile stress and transthickness tensile stress.

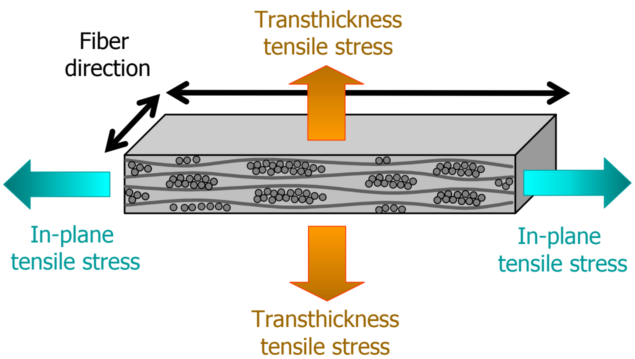

Figure 5. Schematic showing alignment of tensile stress for the in-plane and transthickness tensile test.

Recently, ASTM standardized a test method to evaluate the TTS of CFCCs (ASTM C1468). Because this test method relies on the use of adhesively-bonded extenders to transfer load to the specimen, its applicability is limited by the properties of the adhesive. Some experiments failed due to debonding at the adhesive. Figure 6 shows the stress-displacement curves obtained from transthickness tensile tests of a CVI-SiC/SiC composite reinforced with UD Hi-Nicalon Type-S fibers with 0.52 μm thick of C coating and that reinforced with UD Tyranno SA fibers with 0.56 μm thick of C coating, a picture of a specimen after the test and schematic of the test. Fiber volume fractions of the Hi-Nicalon Type-S composites and the Tyranno SA composites were 29% and 21% respectively. The specimens were square in size (5 × 5 mm) and 1.5 mm thick. The cross head displacement vs. stress curves obtained from transthickness tensile testing were slightly parabolic up to the peak load which was followed by a sudden load drop when the specimens failed. The average TTS of the Hi-Nicalon Type-S composites and the Tyranno SA composites was 26.9 MPa and 20.2 MPa, respectively. It was found that in this test the crack propagated through fiber/matrix interphase between large pores in the matrix.

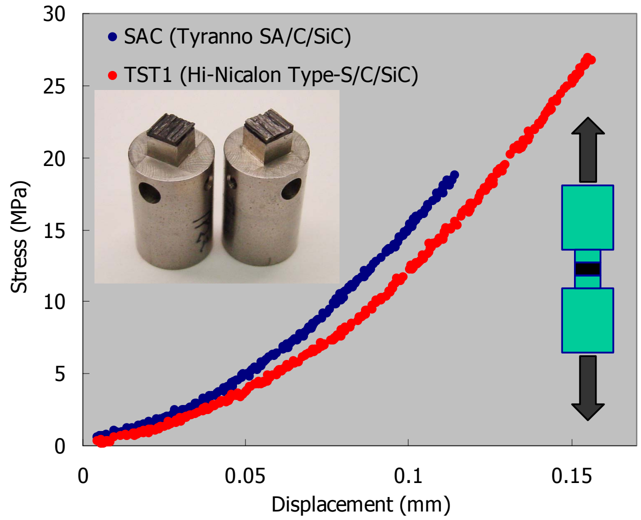

Figure 6. Schematic and typical loading curves of transthickness tensile test of two types of SiC/SiC composite.

In addition to the difficulty of the experiment, the transthickness tensile test can only be used at low temperatures. The diametral compression test,[32] also known as Brazilian test, overcomes the limitations imposed by the adhesive and, therefore, can be applied at high temperatures. This test method is based on the fact that tensile stresses develop when a circular disk is compressed by two diametrically opposed forces as shown in Figure 7. The maximum tensile stresses exist perpendicularly to the loading direction and are proportional to the applied compressive force. The preparation of test specimens and the actual tests are relatively straightforward, making this test method amenable for use. Figure 8 shows a typical stress-displacement curve obtained from a transthickness

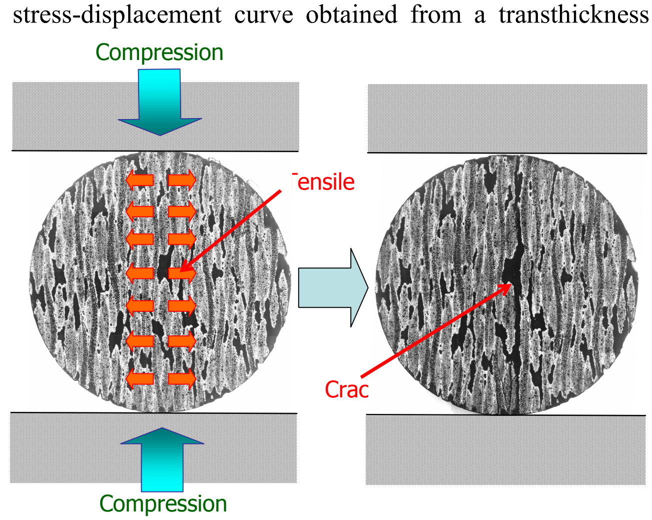

Figure 7. Schematic of diametral compression test showing optical microscope images of a typical 2D-SiC/SiC specimen before and after the test.

tensile test of a CVI-SiC/SiC composite reinforced with P/W Tyranno SA fibers with dual coating of 0.08 μm thick of SiC and 0.57 μm thick of C. The load increased monotonically to a peak value, which was followed by an abrupt drop and an audible indication that the sample had failed. Every specimen failed by a crack that propagated along the loaded diameter, along an interlaminar region through large pores, and along the fiber/matrix interphases. The TTS (σ_T) was determined according to Eq. (5).

$$\sigma_T = \frac{2P}{\pi dt} \tag{5}$$

where P is the load at failure, d is the diameter, and t is the thickness of the specimen. However, this relationship between the TTS and the failure load is only valid for isotropic materials and, therefore, it needs to be corrected to account for the transverse isotropy of the material evaluated. This work is in progress and will be reported in the future. The average TTS of the composites reinforced with Tyranno SA fibers obtained by the diametral compression test was 23.8 MPa.

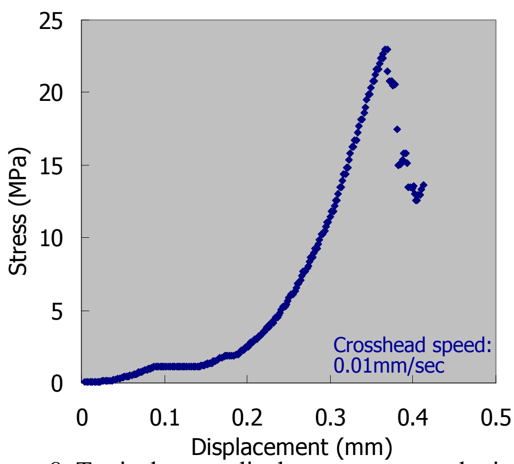

Figure 8. Typical stress-displacement curve obtained from Diametral Compression test.

V. EVALUATION OF INTERLAMINAR SHEAR STRENGTH

SiC/SiC composites are expected to be used at high temperatures, where most of the stress may be thermal stress. While tensile stress arises from thermal gradients, interlaminar shear stress also arises due to the difference of temperature between the surface with heat flux and another side (i.e. cooling side in blanket application).

Interlaminar shear strength of CFCCs can be evaluated by compression of double-notched specimen (DNS) as described in ASTM C1292 and ASTM STP 1309.[33]

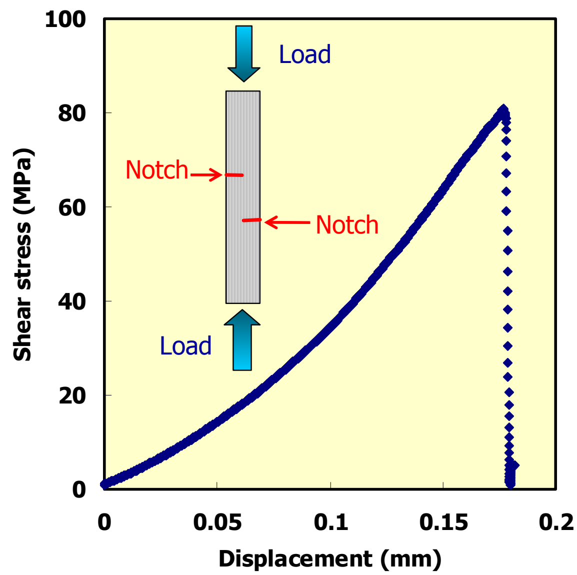

Figure 9. A typical stress-displacement curve by compression of Double-Notched Specimen.

Interlaminar shear strengths of CVI-SiC/SiC composites reinforced with UD Hi-Nicalon Type-S or UD Tyranno SA fibers and C or C/SiC multilayer coating was evaluated by the compression of DNS with the dimension 25 mm (long) × 4.0 mm (wide) × 1.5 mm (thick) and contained two centrally-located notches, 6 mm apart, that were machined halfway through the thickness. Figure 9 shows a schematic of the test and a typical stress-displacement curve. The curve was slightly parabolic up to the peak load which was followed by a sudden load drop when the specimens failed. The apparent shear strength (τ) was determined from Eq.

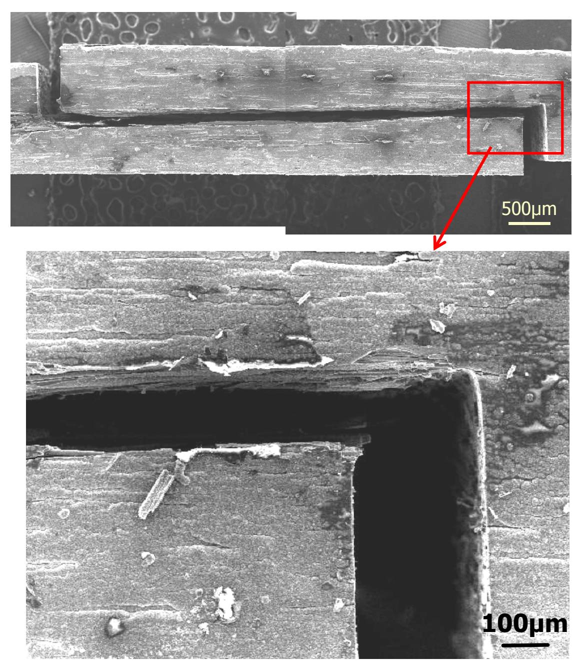

Figure 10. SEM images of double-notched specimen after a compression test.

(6), as the ratio of the peak load, P, divided by the surface area of the imaginary plane between the notches.

$$\tau = \frac{P}{wL} \qquad (6)$$

where w is the specimen width and L is the notch separation. Figures 10 show SEM images of a DNS after a test. Cracks propagated at fiber/matrix interphase. It was found that there were no significant differences among the shear strength values obtained for the composites evaluated. The average shear strength of the UD composites with various fiber and coatings ranged between 54 and 63 MPa.

VI. CONCLUSIONS

Mechanical properties of CFCCs depend on the properties of constituents (i.e. fiber, matrix fiber/matrix interphase) and volume fraction of fiber and matrix. SiC/SiC composites with highly crystalline fibers exhibited improved elastic modulus and PLS. The UTS and PLS obtained from tensile tests of 2D CVI-SiC/SiC composites reinforced with Tyranno SA were independent of the fiber/matrix interphase thickness of C over the range of 30 through 230 nm thickness. Carbon interphase is the weakest link for high temperature application and nuclear application due to oxidation and deformation by neutron irradiation. The primary beneficial result is that the C interphase can be reduced to less than 30 nm without degradation of mechanical properties. The tensile strength of SiC/SiC composites reinforced with Tyranno SA fibers and SiC/C multilayer coating of fibers and fiber bundles was improved by multiple fractures of fiber and fiber bundle coating.

The transthickness tensile test was used to evaluate TTS of UD CVI-SiC/SiC composites reinforced with Hi-Nicalon Type-S and Tyranno SA. The average TTS was 26.9 MPa in Hi-Nicalon Type-S composites and 20.2 MPa in Tyranno SA composites. In this test, the crack propagated through the fiber/matrix interphase between large pores in the matrix.

The diametral compression test also was applied to evaluate TTS of 2D CVI-SiC/SiC composites reinforced with Tyranno SA. It was found that every specimen failed by a crack that propagated along the loaded diameter, along an interlaminar region through large pores, and along the fiber/matrix interphases. The average TTS of the composites reinforced with Tyranno SA fibers obtained by the diametral compression test was 23.8 MPa. The diametral compression test is preferred to the transthickness tensile test, because tests are straightforward without failure of experiments and the test can be carried out at high temperature.

Interlaminar shear strengths of CVI-SiC/SiC composites reinforced with either UD Hi-Nicalon Type-S fibers or UD Tyranno SA fibers with various coatings were evaluated by the compression of DNS. The average shear strength of the UD composites with various fiber coatings were over the range between 54 MPa and 63 MPa. It was found that there were no significant differences among the shear strength values obtained for the composites evaluated.

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