INTERFACIAL SHEAR PROPERTIES OF SILICON CARBIDE COMPOSITES WITH MULTI-LAYERED INTERFACE—T. Nozawa, Y. Katoh, and L. L. Snead (Oak Ridge National Laboratory)

OBJECTIVE

The primary objective of this study is to identify the optimum interlayer design of multiple pyrolytic carbon (PyC) / silicon carbide (SiC) interphase. For this purpose, the effect of interlayer thickness on interfacial shear properties of (non-irradiated) SiC/SiC composites with single PyC interphase and multiple PyC/SiC (ML) interphase is specifically addressed on.

SUMMARY

Interfacial shear properties of ML composites are significantly dependent on the total PyC interlayer thickness, which affects thermally-induced residual radial- and axial-stresses in the fiber. Rough crack surface of ML composites, most of which have an interfacial crack between the fiber and the first PyC layer, also increases frictional shear stress during fiber pull-out due to contribution from roughness of the adjacent SiC sub-layer. Preliminary test results indicate the flexible tailorability of multilayer interphase to provide moderate interfacial shear strength by applying the optimum total PyC thickness, even though each PyC layer remains thin enough.

PROGRESS AND STATUS

Introduction

Silicon carbide matrix composites are being developed for structural applications in fusion systems for their promise of high-temperature performance and low-induced activation. The stability of highly crystalline fibers has led to composites with very good irradiation stability [1, 2]. Specifically, recent report by the authors [3] suggested that the overall composite strength was closely dependent on the neutron irradiation stability of PyC interphase. For non-nuclear applications, a multilayer interphase composed of sequences of thin (< 50 nm) PyC and SiC results in superior composite strength and oxidation resistance [4]. The irradiation effect on interfacial shear properties has been investigated by many authors [5, 6]. However, they did not sufficiently consider the effect of interlayer thickness, which significantly influences the interfacial shear properties. Specifically, the effect of neutron irradiation on ML interphase is still unclear.

Experimental Procedure

All composites were produced by chemically-vapor-infiltration (CVI) process. A highly-crystalline and near-stoichiometric SiC fiber, Hi-Nicalon™ Type-S, was used as reinforcements. Single PyC and multiple PyC/SiC interfaces were formed on the fiber surface by CVI technique. The interlayer thickness of the former was varied from 150 ~ 720 nm. While, ML interphase was composed of five PyC layers of < 50 nm and four SiC sub-layers of ~ 200 nm.

Interfacial shear properties were evaluated by single-fiber push-out test technique. All push-out specimens were obtained from tensile specimens. The tensile specimens were sliced and both surfaces of them were polished with a surface finish of < 1 μm by standard metallographic technique. The sample thickness was 50, 150, and 200 μm. The polished thin films were finally bonded on a push-out specimen holder with ~ 200 μm-wide grooves. Push-out tests were conducted by nano-indentation testing machine. A Berkovich type sharp indenter tip was used. All tests were performed by load control. Push-out data were analyzed by shear-lag based models [7, 8]. Specifically, the effect of the interface thickness was discussed using the double shear-lag model including contribution from the interface [7].

Results and Discussion

Figure 1 shows interfacial debond shear strength with respect to specimen thickness. The interfacial debond shear strength was calculated from debond initiation load extracted from load/displacement curves using a shear-lag model by Hsueh [7]. Interfacial debond shear strength depends on sample thickness; it increases with increasing sample thickness and approaches a constant value. The interfacial debond shear strength also increases with decreasing PyC interlayer thickness. The interfacial debond shear strength of ML composites was very similar to that of single-layered composites with 150 nm PyC interphase, the thickness of which is almost the same with the total PyC thickness of ML composites. There is no data for very thin (< 50 nm) PyC interlayer for Hi-Nicalon™ Type-S composites. However, from the apparent dependence on PyC interphase thickness for other composite system [9], the value would become significantly large with decreasing PyC interphase thickness.

Interfacial friction stress can be obtained from a relationship between maximum push-out stress and sample thickness (Fig. 2). Clamping stress and friction coefficient are extracted by fitting Shetty's model [8] on the maximum push-out stress vs. sample thickness curve, yielding frictional shear stress that is the product of clamping stress and friction coefficient. For thin specimens, which exhibit complete sliding simultaneously when debonding initiates, the frictional stress is also estimated from the slope of the curve at zero stress. Because of limited data, the precise values could not be obtained for ML and 150 nm PyC interlayer composites by Shetty's method. However, a significantly large change of the slope of the maximum push-out stress to the sample thickness of 50 μm indicates that the frictional shear stress of composites with thin PyC interphase would significantly increase. In contrast, frictional shear stress of ML composites was a little higher than that of 150 nm PyC interlayer composites.

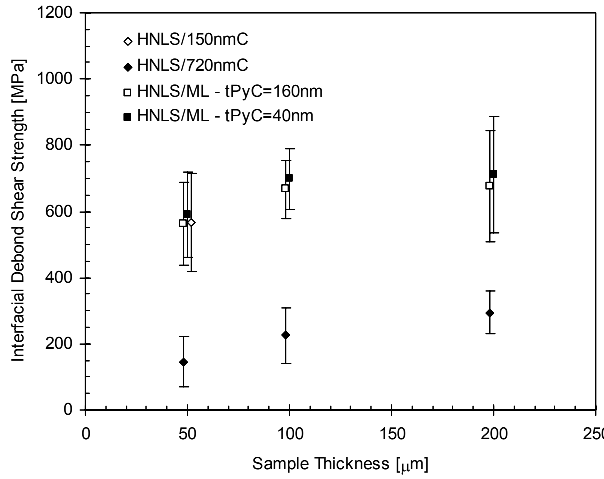

Fig. 1. Interfacial debond shear strength vs. sample thickness. Thicknesses of the first PyC layer and the total interlayer for ML composites were used because this model can not simply apply to multilayered composites. There is no substantial difference when the total interlayer thickness is small enough.

Figure 3 shows thermally-induced residual radial- and axial-stresses with respect to the radial distance from a fiber center predicted by an analytical model. Oel's [10] and Bobet's [11] methods were modified to ML composites in this study. Table 1 lists material properties used in calculation. It is important to note that coefficient of thermal expansion (CTE) of the fiber is assumed to be same as that of the matrix in calculation but very minor differential CTE may results in substantial residual stress. According to Fig. 3, it was found that both residual radial and axial stresses in the fiber of ML composites were clearly dependent on the total PyC interlayer thickness. The residual stresses of ML composites were almost the same with those of

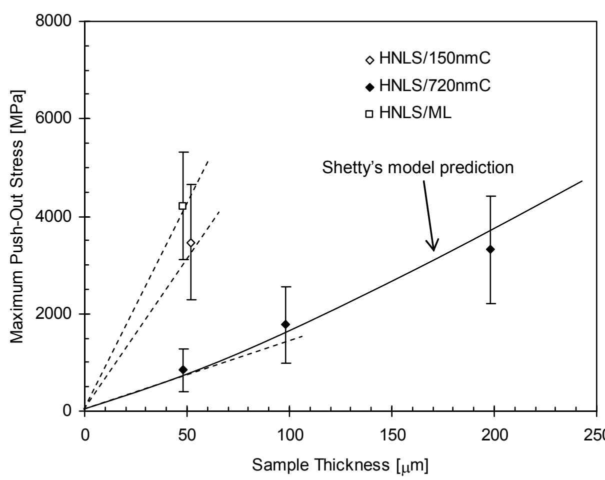

Fig. 2. Maximum push-out stress vs. sample thickness. Shetty's model fitting provides residual clamping stress of 866 MPa and friction coefficient of 0.05 for 720 nm PyC interlayer composites, yielding frictional shear stress of approximately 43 MPa. Frictional shear stresses for 150 nm PyC interlayer composites and ML composites are expected to be ~ 210 and ~ 255 MPa, respectively.

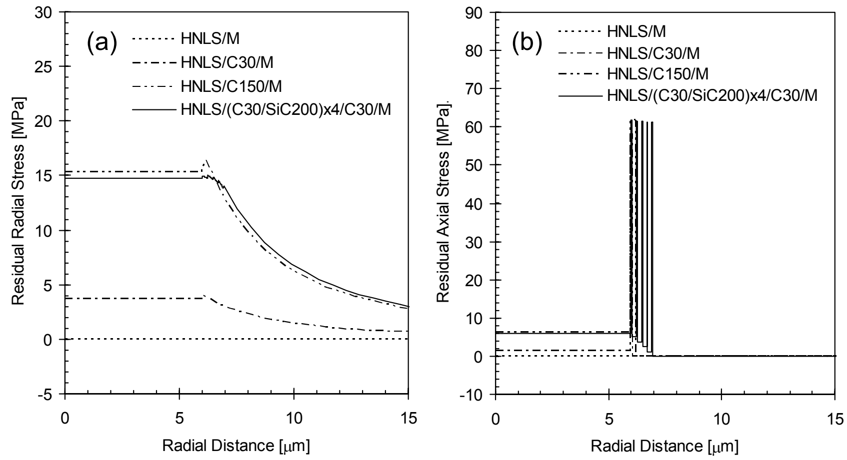

Fig. 3. Calculated thermally-induced residual stresses in (a) radial and (b) axial directions with respect to radial distance from a fiber center for a model composite: a single fiber with interphase is embedded in matrix. In this calculation, CTE of the fiber and the matrix are assumed to be the same because both the Hi-Nicalon™ Type-S fiber (HNLS) and the CVI-SiC matrix (M) are high-crystalline and near- stoichiometric SiC. Pyrolytic carbon is assumed to be isotropic.

Table 1. Material properties used in calculation

Valuables	Hi-Nicalon S	CVI-SiC	PyC
Radius (μm)	6	600	-
Elastic modulus (GPa)	420	460	20
Poisson's ratio	0.2	0.21	0.23

single-layered composites whose PyC thickness was the same with the total PyC thickness of the ML composites. In contrast, the SiC sub-layers hardly affected the stress state in the fiber.

Interfacial shear properties are significantly dependent on the residual stresses in the fiber. These analytical results support the good coincidence of interfacial debond shear strength between ML composites and single-layered composites with 150 nm PyC. A slight difference in frictional shear stress might be explained by different roughness of crack plane, even if the total PyC interlayer thickness becomes the same for both. Most cracks propagated between the fiber and the first PyC layer for ML composites. It is reported that roughness-induced clamping stress and coefficient of friction for thin interlayer composites are increased by contribution of roughness of the adjacent SiC matrix [12]. In a similar manner, ML composites probably exhibit larger frictional shear stress due to contribution from roughness of the adjacent SiC sub-layer.

Generally, very thin carbon interphase provides very strong interface, leading brittle fracture. Hence, the optimum PyC interphase thickness of 100 ~ 200 nm was suggested for non-nuclear applications [9]. Figure 3 implies a great advantage to select thin-layered ML interphase; the moderate interfacial strength and better friction are easily obtained by designing the optimum total PyC interphase thickness, even though each PyC layer remains thin enough.

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