THE EFFECT OF NEUTRON IRRADIATION ON INTERFACIAL SHEAR PROPERTIES OF SILICON CARBIDE COMPOSITES WITH MULTILAYER INTERPHASE—T. Nozawa, Y. Katoh, and L. L. Snead (Oak Ridge National Laboratory)

OBJECTIVE

This study aims to evaluate shear properties at the fiber/matrix interface of silicon carbide (SiC) composites with the multiple pyrocarbon (PyC)/SiC interphase by the fiber push-out test technique. Specifically, the effect of neutron irradiation on interfacial shear properties was emphasized on.

SUMMARY

Fiber push-out test results indicate that neutron irradiation decreased both the interfacial debond shear strength and interfacial friction stress. The mechanism of interphase property decrease for the multilayer interphase composite is due primarily to the changing interfacial cracking path. The primary crack propagated within PyC before irradiation provided a very rough fiber surface, resulting in high interfacial shear properties. In contrast, the cracking at the comparatively smooth fiber/PyC interface upon irradiation significantly decreased the interfacial shear properties. The effects of irradiation-induced dimensional change, associated with microstructure and mechanical property changes, and irradiation creep need to be further investigated.

PROGRESS AND STATUS

Introduction

Silicon carbide matrix composites are candidate structural materials for nuclear fusion and advanced fission energy applications. Previous work [1] has shown that, even though fiber and matrix properties remain unchanged after neutron irradiation, the composite performance can potentially degrade. This potential degradation can be directly linked to change in interfacial properties and indirectly to the crystallinity of the SiC fiber. The high-performance fiber/matrix interphase is therefore important for further stability under neutron irradiation.

In non-nuclear applications, a multilayered interphase composed of sequences of very thin (< 50 nm-thick) PyC and SiC has several advantages. First, the very thin PyC layer enhances interfacial shear strength, resulting in improved debond strength [2]. Second, multilayered interfacial structures promotes multiple crack deflections between layers [3] and the resulting tortuous crack path contributes effectively as a toughening mechanism [4]. Additionally, the very thin layered structures are more resistant to oxidation [5]. However, a fundamental question is whether the irradiation-induced swelling of carbon layers, albeit very thin layers in multilayered interphase, will impact composite performance. Presently, no quantitative data are available to evaluate irradiation effects on interfacial shear properties.

Experimental Procedure

A unidirectional Hi-Nicalon™ Type-S fiber reinforced chemically vapor infiltrated (CVI) SiC matrix composite was fabricated. A multiple PyC/SiC interphase was formed on the fiber surface by the CVI technique. The multilayer interphase was composed of five layers of ~ 20 nm-thick PyC and four sub-layers of ~ 100 nm-thick SiC (Fig. 1).

Neutron irradiation was performed in the High Flux Isotope reactor (HFIR) at Oak Ridge National Laboratory as a part of the RB-14J capsule irradiation experiment. The neutron fluence was 7.7 dpa (assuming that 1 dpa corresponds to 1.0×10^{25} n/m^2 (E > 0.1 MeV)), and the irradiation temperature was 800°C.

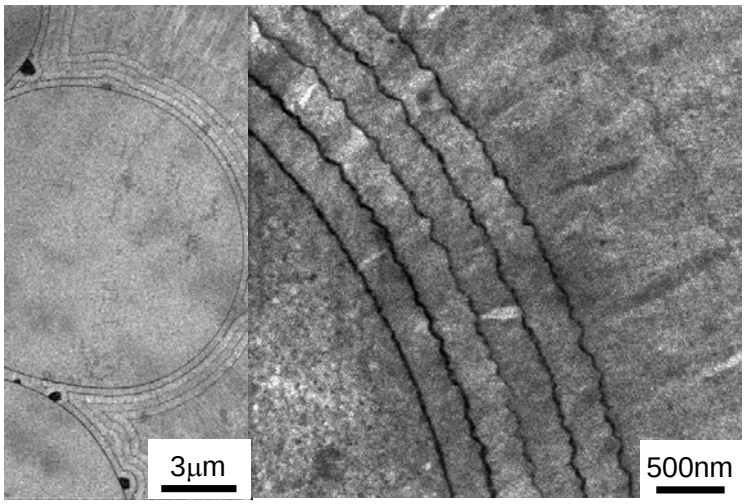

Fig. 1. Typical cross-sectional images of multilayer interphase SiC/SiC composites.

Interfacial shear properties were evaluated by the fiber push-out technique. The specimens were polished with a surface finish of < 1 μm by a standard metallographic technique. The sample thickness was ranged in 50~200 μm. Push-out tests were conducted using a nano-indentation test machine equipped with a Berkovich indenter tip. The loading rate was 0.05 s^{-1}. The pushed-out fiber surface was observed by scanning electron microscopy.

Push-out data were analyzed by non-linear shear-lag models [6, 7]. Two important push-out parameters: an interfacial debond shear strength, which is a critical shear stress to initiate a primary crack at the perfectly bonded interface, and an interfacial friction stress for the debonded interface were calculated from compressive stresses applied on the fiber surface at the debond initiation and the complete debonding, respectively (Fig. 2).

Results and Discussion

Interfacial debond shear strength

Figure 3 shows an experimental debond initiation stress with respect to the sample thickness and fitted curves were obtained using the double shear-lag model originally proposed by Hsueh [6]. Error bars correspond to ± one standard deviation. Interfacial debond shear strengths of 710 MPa (non-irradiated) and 590 MPa (irradiated) were obtained. In calculation, both PyC and SiC were assumed to be isotropic. Thermally-induced residual clamping stress was considered to be negligibly small. For simplicity, the effects of irradiation-induced swelling and irradiation creep were ignored because of uncertainity of irradiation effects on the constituents. Preliminary test results indicate that the slight decrease of the interfacial debond shear strength was obtained upon neutron irradiation up to 7.7 dpa at 800°C.

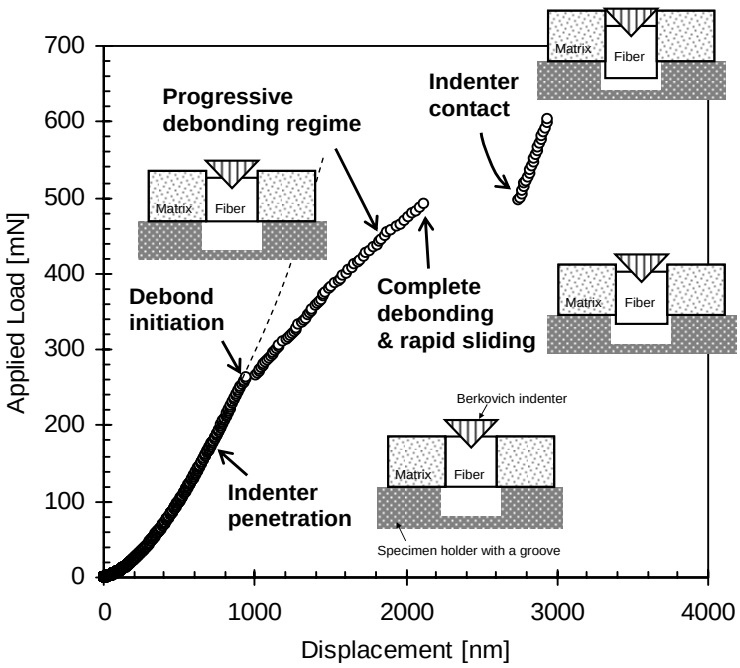

Fig. 2. Typical nano-indentation fiber push-out behavior of composites: 1) penetration of the sharp indenter tip, 2) crack pop-in, 3) progressive debonding regime, 4) complete debonding and rapid sliding, and 5) indenter contact with the SiC matrix.

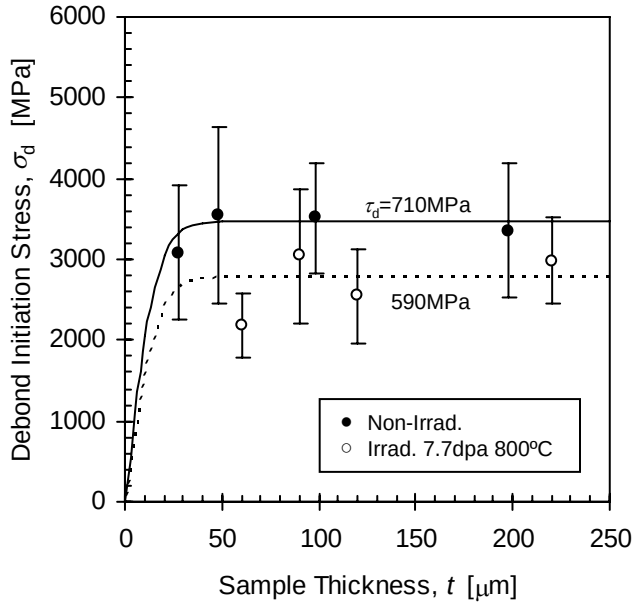

Fig. 3. Debond initiation stress vs. sample thickness, yielding interfacial debond shear strength of 710 MPa (non-irradiated) and 590 MPa (irradiated).

Interfacial friction stress

Figure 4 shows a complete debonding stress with respect to the sample thickness before and after neutron irradiation. According to the Shetty's model [7], for the very thin specimens, the complete debonding stress should be proportional to the sample thickness. Then, the slope of the figure gives estimates of interfacial friction stress: 288 MPa (non-irradiated) and 110 MPa (irradiated). Approximately 60% decrease was apparent in the multilayer interphase upon neutron irradiation up to 7.7 dpa at 800°C. However it is strongly emphasized that the interfacial friction stress of neutron-irradiated multilayer interphase composites was still sufficiently high.

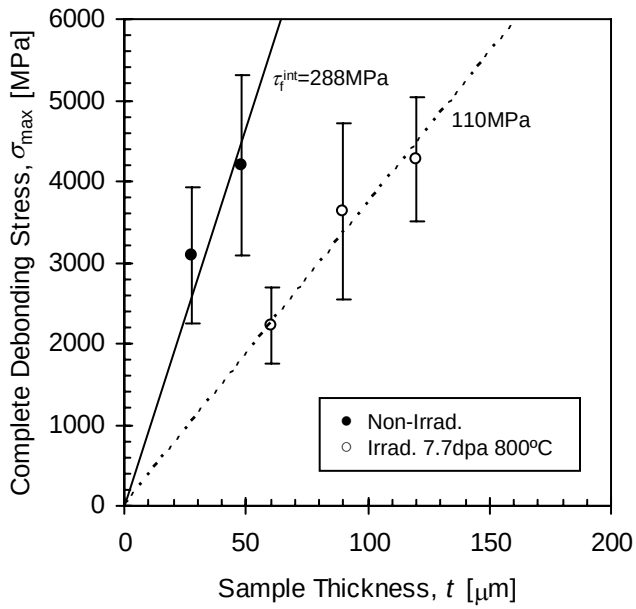

Fig. 4. Complete debonding stress vs. sample thickness, yielding interfacial friction stress of 288 MPa (non-irradiated) and 110 MPa (irradiated).

Effect of fiber surface roughness

Figure 5 shows typical micrographs of pushed out fiber surface. Apparently a rough fiber surface was observed before irradiation. The rough fiber surface indicates that the primary crack probably propagated within the PyC interphase, potentially providing very high clamping stress for the debonded interface. In contrast, the fiber surface for the neutron-irradiated composites, which was an original Hi-Nicalon™ Type-S fiber surface, was comparatively smooth. In this case, the primary crack might propagate at the fiber/PyC interface, resulting in the lower friction stress.

The primary mechanism for the changing interfacial cracking path induced by neutron irradiation is currently unknown. Irradiation-induced swelling, associated with the changes of mechanical property and microstructure, and irradiation creep may influence. The graphitic structure of the carbon interphase, which is preferred to form near the fiber surface, may also impact on interfacial shear properties and the irradiation effect.

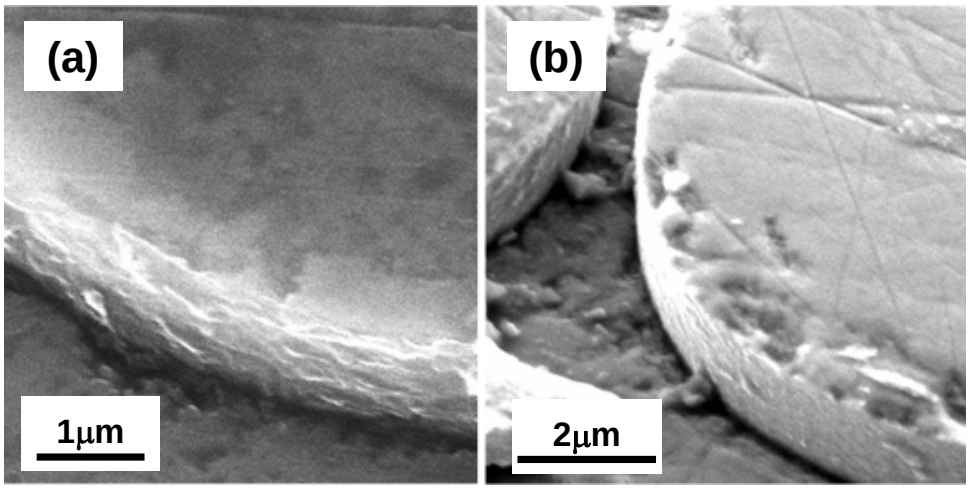

Fig. 5. Typical pushed-out fiber surface images of the multilayer interphase composites: (a) non-irradiated and (b) neutron irradiated up to 7.7 dpa at 800°C.

Acknowledgement

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