§4. Evaluation of Interfacial Shear Properties by Microindentation Technique

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Ceramic matrix composites (CMCs) are expected to be applied to advanced energy systems such as high thermodynamic efficiency gas cycles and fusion systems because of their high temperature mechanical properties and low activation. The importance of interfacial fracture behavior between fiber and matrix on mechanical properties of CMCs has long been emphasized. The recent progress in fabrication technique by CVI method made it possible to form various controlled fiber/matrix interphase such as multiple C/SiC layers. In order to understand interfacial shear properties including clamping stress and frictional stress and interfacial fracture behavior, microindentation technique was applied, since the SiC fiber diameter is approximately 10 μm and thickness of most fiber/matrix interphase is less 0.5 μm. As for SiC/SiC composites, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) observation showed that almost always, the interfacial crack propagated at the interface between the fiber and the interfacial shear stress control layer (ISSCL), such as pyrolitic C layer. This behavior was not limited only to C layers. Multiple C/SiC layers and 'porous' SiC layers, which are expected for nuclear applications, showed the same behavior. Fracture surface of fibers was smooth. This led to low frictional stress of debonded interface and made crack propagation easy. This fracture behavior is attributed to a smooth fiber surface and weaker bond between ISSCL and fiber. In order to get strong bond between fiber and ISSCL, SiC was coated on fibers, for the interfacial bond between ISSCL and SiC matrix was stronger than the interfacial bond between fiber and ISSCL. The objective of this work is to understand the relationship between interfacial microstructure and interfacial fracture behavior by micro indentation technique and to improve SiC/SiC composites for nuclear application.

Materials used in this study were flat-woven Hi-Nicalon™ SiC fibers reinforced SiC matrix composites. SiC/SiC composites were fabricated by chemical vapor infiltration (CVI) method. As the fiber surface treatments, fibers were coated with SiC, whose thickness was 0.25 μm, by CVI method, prior to deposition of ISSCL of C with 0.13 μm thickness, and matrix SiC deposition. The reference material, which has 0.15 μm thickness ISSCL of C without any surface treatment, was also fabricated. Microstructure of the composites was examined by optical microscopy, SEM and TEM. The thin film for TEM observation was prepared by focused ion beam. Fracture surfaces after mechanical tests were observed by SEM. Pull-out fibers surfaces were analyzed by EDS equipped with SEM and by optical interferometric profilometry to evaluate roughness. Interfacial shear properties were obtained by single fiber push-out tests. Specimens were sliced into 500 μm-thick samples. After mechanical polishing, specimens were reduced its thickness to approximately 50 μm. Specimens were put on the holder with a groove of 50 μm in width. Isolated fibers with the fiber direction perpendicular to holder surface on the groove were selected with a video microscope. They were pushed out by a Berkovich type pyramidal diamond indenter tip with maximum load of 1 N. Both pushed in side and pushed out side were observed by SEM after the tests.

Loading curves of push-out tests are parabolic at first as the indenter penetrates into a fiber. Then the gradient change of the curve by fiber 'push-in' which indicates the initiation of interfacial debonding at surface occurs. The crack between a fiber and matrix propagated, and a fiber was deformed elastically and pushed in. Then, the whole interface was debonded, and a fiber was pushed out. 'Push-in' load does not depend on specimen thickness, but on fiber diameter, since 'push-in' load is used to debond the interface near the surface, although it is impossible to measure the depth of the debonded region at 'push-in'. 'Push-in' load was divided by fiber diameter to normalize data. Interfacial shear stress was obtained from 'push-out' load. 'Push-out' load was divided by debonded area. Effects of fiber surface treatments on interfacial shear properties are summarized in Table 1. Although 'push-in' load of the sample with SiC coating was slightly less than the reference sample without SiC coating, 'push-out' load of the material with SiC coating was larger than one of the reference sample without SiC coating. The value, where 'push-in' load is divided by fiber diameter, depends on clamping stress of interface. Interfacial shear stress includes clamping stress and frictional stress at debonded region. These results indicate that the frictional stress of interface was increased by the fiber SiC coating, although the clamping stress of the fiber SiC coating sample was little lower than the reference sample without surface treatment. The crack propagated at the interface between the fiber and C layer in the case of the reference samples without fiber surface treatment, while crack propagated within the C layer in the case of the fiber SiC coating samples. This fracture behavior corresponded to that of tensile tests. The pull-out fiber surface of the fiber SiC coating sample was rougher than one of the reference sample. Tensile properties were significantly improved by fiber SiC coating.

Table 1. Effect of fiber SiC coating on interfacial shear properties

Interphase	Push-in load/ Diameter	I.S.S.* (MPa)
SiC/C	3.01	280
C	3.32	212

* Interfacial shear stress (push-out load/interfacial area).

References

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