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SURFACE ROUGHNESS CHARACTERIZATION OF VARIOUS CERAMIC FIBERS USING AFM AND LOW VOLTAGE SEM

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The effect of interfacial properties in fiber-reinforced ceramic matrix composites is critical to the overall mechanical behavior of the composite material. The creation of a relatively weak fiber/matrix interface allows for the beneficial actions of debonding and fiber pull-out to occur, thus improving the fracture toughness and, in many cases, the ultimate strength of the composite.^{1,2} To date, the best room temperature interfacial properties have been achieved by coating the fibers with either carbon or boron nitride.³ There are several factors which contribute to the interfacial properties of a composite, including the residual stress (clamping stress) present at the fiber/matrix interface, which is a result of differences in thermal expansion, and the fiber surface roughness.⁴ In this study, the surfaces of several ceramic fibers have been characterized qualitatively using a Hitachi S-4500 FEG SEM operated at low voltages and quantitatively using a Topometrix atomic force microscope (AFM). This study is part of an overall program relating fiber surface roughness to the interfacial shear stress.⁵

The fibers examined in this study were primarily Nicalon (a SiC fiber) and Nextel (an aluminosilicate fiber), both of which have diameters less than $\sim 15 \mu\text{m}$. Since the fiber diameters were so small and the fibers themselves very flexible, it was necessary to prepare the specimens for both SEM and AFM carefully to ensure that the native fiber surfaces were being characterized (as opposed to mounting wax or binder). The fibers were first subjected to standard cleaning procedures to remove binder material. Individual, small lengths of the fibers were then mounted on carbon conducting tape on the SEM stubs. Although the fibers were ceramic, they were not coated with a conducting material which could mask fine-scale surface features. SEM was thus performed at 1-2 kV to minimize charging effects. Several fibers were mounted on a sample mounting disc using a low melting point wax for AFM analysis. Proper fiber mounting techniques were extremely important - wicking of the wax up the sides and onto the top surface was avoided. The AFM cantilever with a Topometrix supertip was oriented perpendicular to the long axis of the fiber and was scanned across the surface as shown in Fig. 1.

A low voltage SEM image of the Nicalon fiber surface is shown in Fig. 2. Small, irregularly shaped surface features, most of which are less than 50 nm, are evident. A higher magnification AFM image of a Nicalon fiber surface is shown in Fig. 3. The AFM data has been used to quantitatively define the surface roughness of the Nicalon fibers. In order to accurately measure the roughness of a fiber surface, the surface must be "levelled" to remove the effects of inherent fiber curvature. To remove the curvature, a fitted second order polynomial is subtracted from the data. The resulting levelled image is then used for surface roughness measurements using both line profiles and area analyses. For comparative purposes, the primary roughness parameter determined was R_a , which is the arithmetic mean of the absolute values of the departures of the roughness profile from the mean line. The value of R_a for the Nicalon fibers averaged over several line scans was $R_a=3.4 \text{ nm}$ and R_a determined from an area analysis was $R_a=3.9 \text{ nm}$. This surface roughness corresponded well with the $\sim 4.0 \text{ nm}$ average SiC grain size in Nicalon fibers. An AFM image of a Nextel 312 aluminosilicate fiber is shown in Fig. 4. The Nextel fibers have an oval shape rather than cylindrical. The Nextel fibers are slightly rougher than Nicalon fibers and have an average $R_a=4.8 \text{ nm}$ as measured from several line profiles. This also corresponds well with the average grain size of $\sim 5.0 \text{ nm}$ in Nextel. Additional surface features were found in the Nextel fibers which were not observed in the Nicalon fibers, as arrowed in Fig. 5. These "grooves" ran the entire length of the Nextel fibers (note that there is also a groove in Fig. 4) and were found to be approximately 100 nm in

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depth. Work is ongoing to characterize additional ceramic fiber surfaces and to relate the surface roughness to interfacial properties.⁵

1. R.W. Rice, *Ceram. Eng. Sci. Proc.*, (1981) 2(7-8), 561-701.
2. D.B. Marshall and A.G. Evans, *J. Amer. Cer. Soc.*, (1986) 68(5), 225-231.
3. K.L. More and R.A. Lowden, *Proc. Ann. EMSA Meeting*, (1989) 47, 556-557.
4. C.H. Hsueh, *J. Amer. Cer. Soc.*, 76(12), 3041-3050.
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FIG. 1-- AFM configuration for characterization of fiber surfaces.

FIG. 2-- Low voltage (2 kV) SEM image of Nicalon surface.

FIG. 3-- AFM image of Nicalon fiber surface.

FIG. 4-- AFM image of Nextel fiber surface.

FIG. 5-- AFM image of Nextel fiber surface showing surface grooves.

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