

R-CURVE RESPONSE OF SILICON CARBIDE WHISKER-REINFORCED ALUMINA: MICROSTRUCTURAL INFLUENCE

E. Y. Sun, C. H. Hsueh, and P. F. Becher

Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6068

ABSTRACT

Rising fracture resistance with crack extension (*R*-curve response) can lead to improvements in the mechanical reliability of ceramics. To understand how microstructures influence the *R*-curve behavior, direct observations of crack interactions with microstructural features were conducted on SiC whisker-reinforced alumina. The contribution of the dominant toughening mechanisms to the *R*-curve behavior of these composites is discussed using experimental and theoretical studies.

INTRODUCTION

Rising fracture resistance with crack extension (*R*-curve response) has been observed in SiC whisker-reinforced alumina and explained by crack-bridging mechanisms [1-9]. *R*-curve behavior predicted using constitutive models based on crack-bridging mechanisms agrees well with experimental results [4-9]. However, because of the experimental difficulty in measuring several parameters critical to modeling, a certain degree of ambiguity always exists in identifying the important toughening parameters, especially in the short-crack region ($< 50 \mu\text{m}$). The objective of the present study was to examine the fracture behavior of SiC whisker-reinforced alumina at a micro-scale level, and to obtain a better understanding of the toughening mechanisms in these materials by *in-situ* observations of crack interactions with microstructural features when subjected to an applied stress.

EXPERIMENT PROCEDURE

Materials

SiC whisker-reinforced alumina composites were prepared by mixing the $\sim 0.8 \mu\text{m}$ diameter SiC whiskers (Advanced Composite Materials Corporation, Greer, SC) with alumina powder suspended in hexane in a shear mill, followed by rapid evaporation of the media to form a granulated, friable mixture. The mixtures were hot pressed in graphite dies at a temperature of 1800°C to obtain composites with densities $\geq 98\%$ of theoretical. Densification was controlled to maintain a grain size of $1-2 \mu\text{m}$ in final composites containing $\sim 20 \text{ vol.}\%$ whiskers.

R-Curve Measurement and Direct Observation of Fracture Behavior

R-curve responses of SiC whisker-reinforced alumina composites were investigated using an applied moment double cantilever beam (DCB) geometry with the crack plane oriented parallel to the hot-pressing axis. The DCB blanks had a dimension of $10 \times 35 \times 2.5 \text{ mm}$. One of the wide surfaces ($10 \times 35 \text{ mm}$) contained a $\sim 1 \text{ mm}$ deep by 1.5 mm wide centerline groove parallel to the length while the opposite surface was mechanically polished to facilitate observation of the crack. One end of the specimen was notched and precracked using a TiB_2

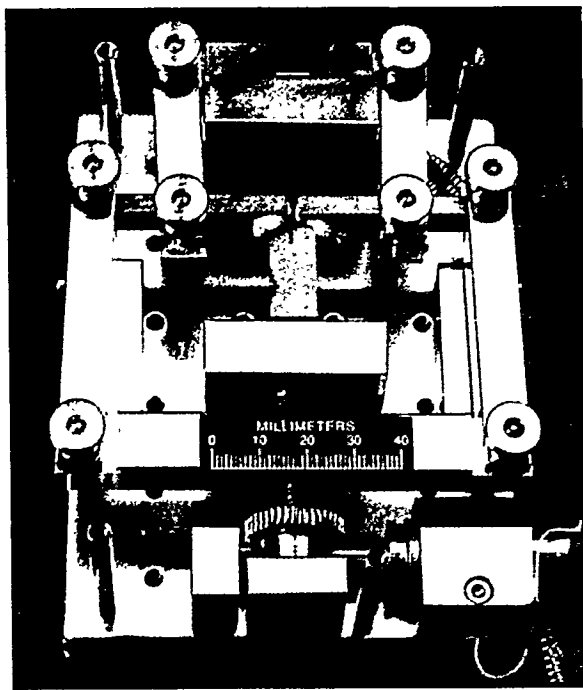


Figure 1. Applied moment DCB test module and the sample. The testing stage can be placed on an optical microscope stage or housed in the chamber of an SEM.

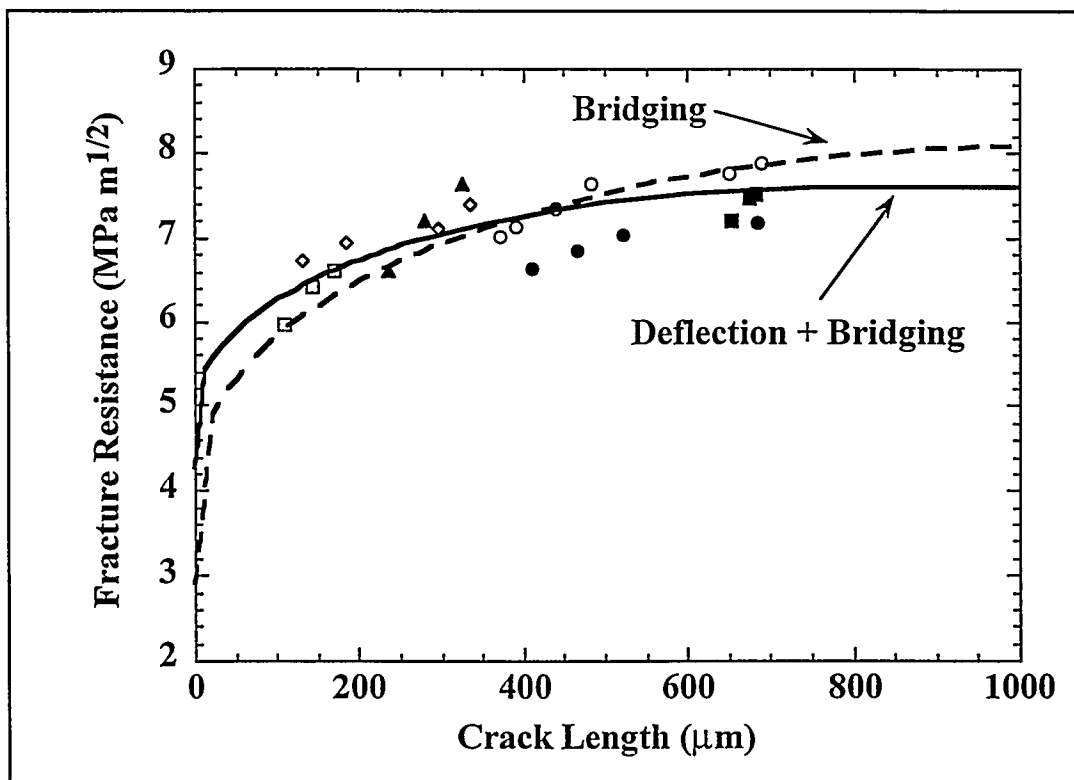
wedge. The wake of the precrack was removed with a low-speed diamond saw. The initial crack lengths employed in the present study were between 100-200 μm . Stainless steel loading arms were attached to the DCB specimens using an epoxy adhesive. Samples were carbon coated.

Samples were tested using a DCB test module, as shown in Figure 1. The testing stage employs a DC drive motor to generate the various applied loads, which are monitored by a semiconductor load cell. The load is applied through a bending moment on the loading arms. With this geometry, the applied stress intensity depends on the load but not on the crack length [10]. During the experiments, the testing stage was either placed on an optical microscope stage or housed in the chamber of a scanning electron microscope to enable both direct observation of the crack interaction with microstructures and measurement of crack opening displacements under applied loads. Details of sample preparation and testing procedures are reported in Reference 9.

RESULTS

Typical R -curve responses for the present SiC whisker-reinforced aluminas are shown in Figure 2(a). (Comparison between the experimental results and the prediction based on different models is discussed in the next section.) The initial crack (typically 100-200 μm in length) started to propagate at $\sim 6 \text{ MPa}\sqrt{\text{m}}$ and a steady-state toughness ($\sim 8 \text{ MPa}\sqrt{\text{m}}$) was approached at crack lengths of $\sim 700 \mu\text{m}$. Direct observation of crack propagation revealed that the crack tip was frequently deflected at whisker surfaces and then propagated along the whisker/matrix interface. Crack deflection was an operative process in these composites. If the embedded length of the whisker on one side of the crack plane is short, the crack then continued around the whisker-matrix interface and into the matrix ahead of the whisker. Otherwise, the crack tip appeared to arrest as it encountered the interface and then reappeared in the matrix ahead of the whisker, leaving the whisker intact and functioning as a bridging ligament. An example is shown in Figure 3(a) where the whisker is $\sim 5 \mu\text{m}$ behind the tip. (The crack propagated from right to left in all the pictures). This type of bridging by intact whiskers has been defined as "frictional bridging" in previous studies [7-9]. It has been predicted by the bridging model that the R -curve rises quickly due to frictional bridging during the initial stages of crack extension and the size of the bridging zone is $< 50 \mu\text{m}$. *In-situ* observations revealed that a large fraction of such whiskers underwent "partial" debonding and then fractured without experiencing any pull-out, as shown in Figures 3(a) and 3(b). In Figure 3(a), debonding along the whisker/matrix interface is only observed at the right-side of the whisker. No debonding appears along the interface on the left-side of the whisker though it may have been covered by the carbon coating deposited on the specimen surface. In Figure

(a)



(b)

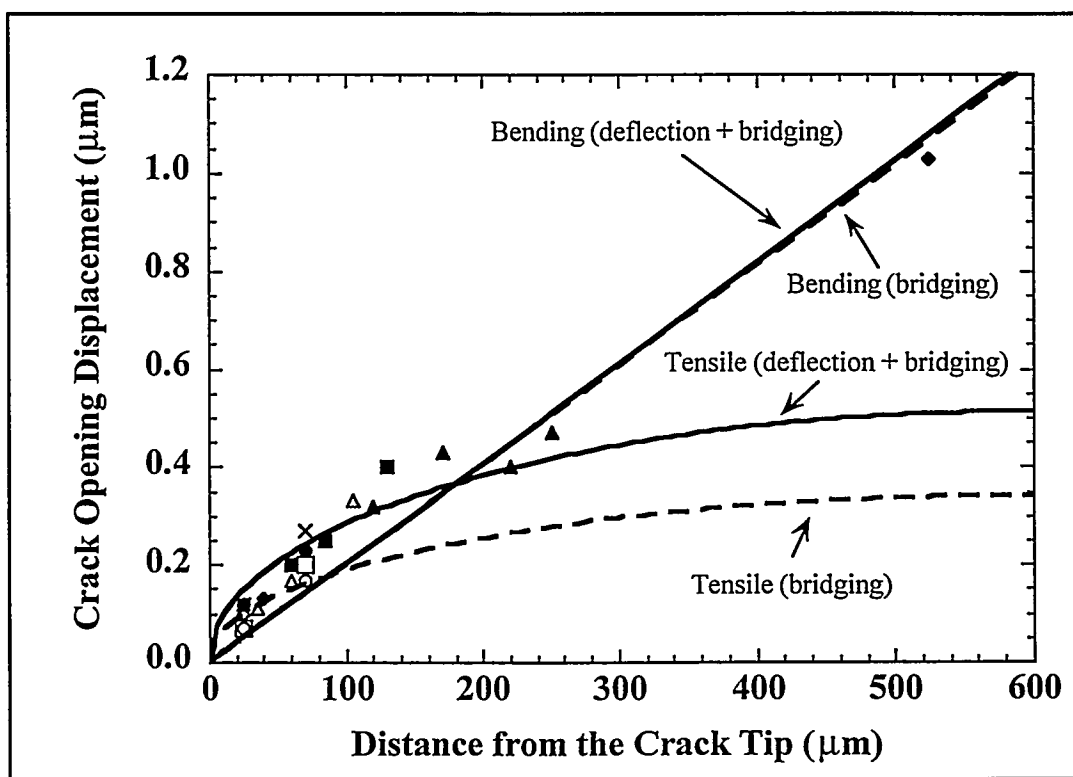


Figure 2 (a) *R*-curve response of SiC whisker-reinforced alumina and (b) crack opening displacements under dynamic loading. (Symbols are experimental data and curves represent calculations based on different toughening mechanisms described in Discussion: dashed lines are for solely bridging processes and solid lines include crack deflection contribution as well.)

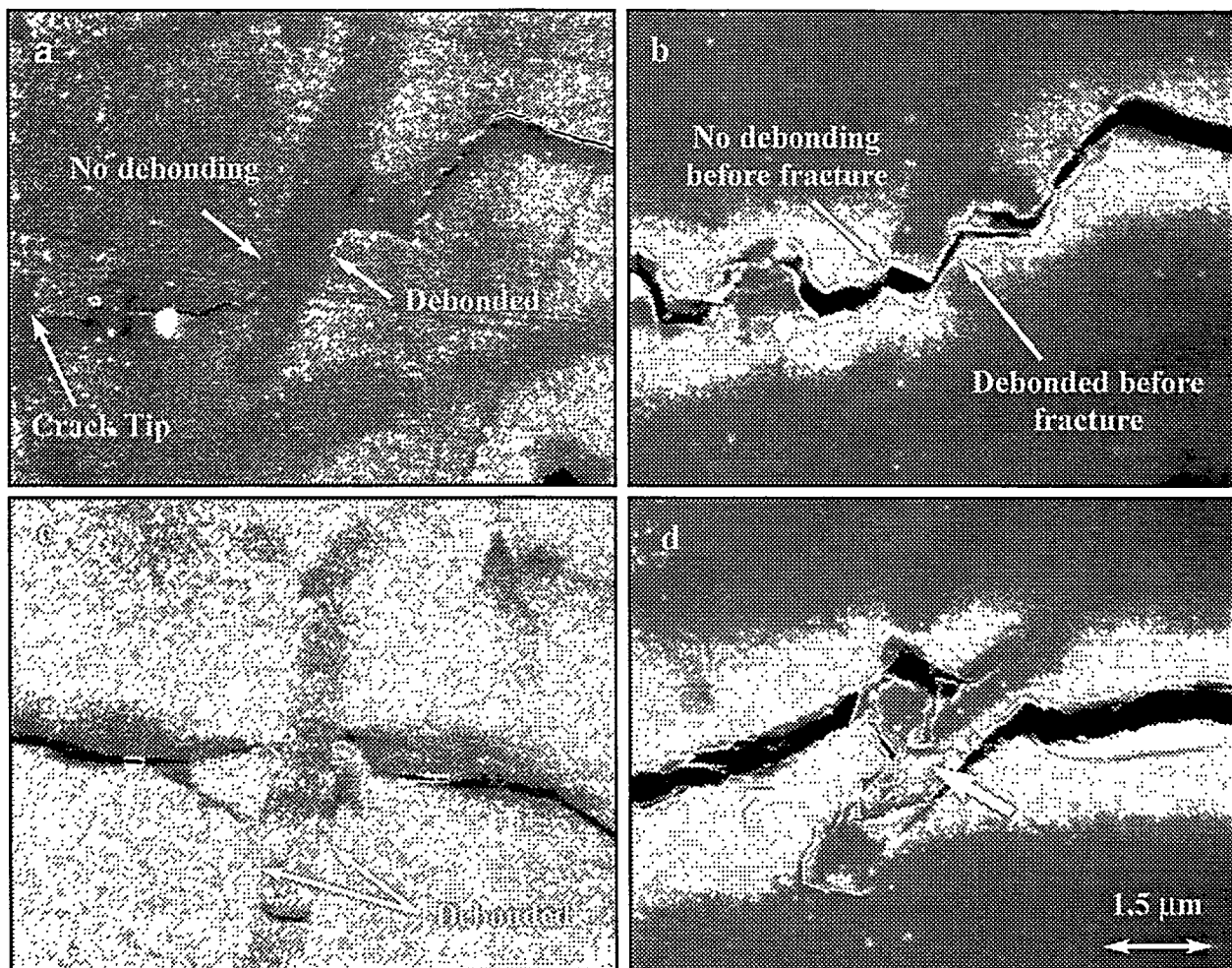


Figure 3. Fracture behavior of SiC whisker-reinforced alumina: (a) crack deflected and bridged by an intact whisker; (b) whisker fractured under higher applied stress; (c) crack-bridging via whisker pull-out; and (d) whisker subjected to bending stresses during pull-out. (Crack propagated from right to left in all pictures.)

3(b), this particular whisker fractured under a higher applied stress. Apparently debonding had only occurred on the right-side interface but not on the left-side one before it failed. Whiskers underwent this type of debonding contributed to the rising fracture resistance only in the short-crack region. Only whiskers debonded along the entire circumference contributed to the *R*-curve behavior in the crack wake beyond the “friction bridging” zone. Energy can be dissipated either via frictional pull-out process (Figure 3(c)), or via bending and rotation due to local stress field (Figure 3(d)). The fraction of whiskers participating in the “pull-out” process was found to be smaller than that in the “friction bridging” process.

Crack opening displacements (COD) were measured by monitoring certain areas along the crack under different applied stress levels. The COD data plotted as a function of the distance behind the crack tip (x) is shown in Figure 2(b). A linear relationship between COD and x is exhibited. This phenomenon is discussed later. It is also noted that the fracture resistance approached its steady-state value when the COD value at the end of the crack was about twice of the whisker diameter.

DISCUSSION

From the experimental evidence, it can be concluded that crack deflection, "frictional bridging" and pull-out process are active in silicon carbide whisker-reinforced alumina. However, it should be realized that among these three mechanisms, crack deflection increases the "intrinsic" toughness value but has no influence in the crack wake. In other words, crack deflection would shift the entire R -curve up-and-down. Based on this basic interpretation, the previously developed bridging model has been modified to incorporate a crack-deflection contribution to the material's toughness. Specifically, a significant increase in the K_{I0} value (the toughness at the crack tip) is used to reflect the increase in toughness by crack-tip deflection.

In addition, some other changes have been made in the modeling work based on the *in-situ* observations. First, in the previous calculation [9], debonding was assumed to occur at the interface of a particular whisker on both sides of the crack plane. However, the present experiments indicate that the whiskers more likely only debond on one side of the crack. Secondly, the expression of the crack profile is changed. A $u(x)$ function for simple uniaxial tensile stress condition was used in the previous work, where u is half of the COD value and x is the distance from the crack tip [9]. However, it is found that the COD values calculated using this method are much smaller than that observed experimentally, especially at large distance from the tip, as shown by the curves labeled as "tensile" in Figure 2(b). Thus a new expression of $u(x)$ is derived by analyzing the deformation of a beam under an applied moment, which is more appropriate for the stress condition experienced by a DCB sample. The COD values calculated as a function of x using this new expression is plotted in Figure 2(b) and labeled as "bending".

With these modifications, the R -curve for the present 20 vol.% SiC whisker-reinforced alumina is calculated using both the "deflection + bridging" model and the "bridging" model. The K_{I0} value used in the "deflection + bridging" model is $4.2 \text{ MPa m}^{1/2}$ instead of the $2.8 \text{ MPa m}^{1/2}$ used earlier in the "bridging" model [9]. This 50% increase in K_{I0} is estimated from Faber and Evans' work [11]. In both calculations, it is assumed that 50% of the whiskers participate in the frictional bridging and pull-out processes, considering the random orientation of whiskers in planes perpendicular to the hot-pressing axis. Also, 0.25 is used as the pull-out length to debonding length ratio in both calculations to account for (1) the fact that a bridging whisker can fracture at any point along the debonded portion; and (2) the smaller fraction of whiskers participating in the pull-out as compared to the frictional bridging process. It is found that a higher whisker strength is required in the "bridging" model (9.2 GPa) than that in the "deflection + bridging" model (7.0 GPa) to obtain similar toughness values in the long-crack region. (All the other parameters are remained the same in both calculations except the whisker strength and the K_{I0} value). Comparing the two curves in Figure 2(a), apparently the major difference lies in the short-crack region. Because of the lack of experimental data in the short-crack region ($< 50 \text{ }\mu\text{m}$), it is difficult to justify the actual contribution of crack-deflection to the toughness increase at the crack tip. Future work will focus on development of new techniques to investigate fracture resistance in the short-crack region.

CONCLUSIONS

The R -curve responses of SiC whisker-reinforced aluminas were investigated by direct observation of crack interactions with microstructural features. It is found that crack-deflection and crack-bridging are the operative toughening mechanisms in the composites, with crack-