ACOUSTICAL AND DYNAMIC MECHANICAL CHARACTERIZATION OF FIBER-MATRIX INTERFACE BONDS IN CERAMIC COMPOSITES

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INTRODUCTION

Ceramic matrix composites presently being developed are potentially well suited for high temperature structural applications. The character of the fiber-matrix bond plays a significant role in determining the fracture toughness of the material and thus its performance. Increased toughness is achieved by phenomena such as interface debonding and fiber slip or pull-out, which improve material toughness by increasing the energy required to propagate a crack [1]. In a bond that is too weak, the toughening mechanisms are not significant. However, a bond that is too strong permits a crack to propagate directly through a fiber-matrix interface without being significantly affected, resulting in brittle fracture. As a result, care is required in the manufacture of these materials to achieve optimum fiber-matrix bonding [2]. The objective of this work is to develop and evaluate techniques to nondestructively characterize the fiber-matrix interface bonds. The techniques being investigated include ultrasonic velocity and attenuation, acousto-ultrasonic response, and internal dynamic mechanical damping.

DESCRIPTION OF MATERIAL AND TEST SAMPLES

The samples used for this investigation were fabricated and supplied by Oak Ridge National Laboratory (ORNL). The material is a continuous fiber reinforced SiC matrix composite containing a 2-d weave Nicalon (Nippon Carbon Co., Tokyo, Japan) cloth layered in a 30-60-90 pattern. The matrix was formed using a chemical vapor infiltration (CVI) process [2]. The samples are right cylinders 45 mm in diameter and 11.5 mm high with 40 vol% fiber and 85% of theoretical density.

Samples with strong (Sample 260) and weak (Sample 265) interface bond strengths were fabricated. The weak interface bond was produced by coating the Nicalon fibers with carbon prior to infiltration. Following infiltration, a heat treatment of 850°C for 2 h in flowing oxygen was used to oxidize the carbon interlayer, effectively weakening the bond. The strong interface bond was produced by not applying any fiber precoat or subsequent heat treatment. A silica layer which forms on the surface of the uncoated Nicalon fibers during initial heating during the CVI process bonds strongly to both the fiber and the deposited matrix.
Other samples prepared in identical fashions to Samples 260 and 265 were destructively tested by ORNL [3]. Interfacial frictional stress and flexure measurements confirmed that the carbon precoat and subsequent oxidation produced a weak interface bond in comparison to the alternate fabrication technique with no precoat or oxidation. Using the indentation method, the strong interface bond had a 48 MPa interfacial stress while that of the weak interface bond was too low to measure. Flexure tests resulted in brittle fracture for the material with the strong interface (flexure strength of 82 MPa), while the weak interface failed with significant fiber pullout (flexure strength of 189 MPa).

TECHNIQUE EVALUATION

Ultrasonic Velocity and Attenuation

The velocity and attenuation of an ultrasonic wave are strongly influenced by the microstructure of the medium through which it propagates. For the case of a ceramic matrix composite, the fiber-matrix interface contributes to both the amount of acoustic scatter and absorption. With composites using similar materials for both matrix and fibers, as is the case here, it seems that as the strength of the fiber matrix interface bond decreases, an increase in acoustic absorption and scatter should be observed. This would result in an increase in attenuation and a decrease in the velocity.

Ultrasonic velocity and attenuation measurements were made using a through-transmission technique. Matched broadband contact transducers were dry-coupled to the samples using sheets of thin polyvinyl chloride to prevent errors due to uptake of liquid couplants. A 1.5 MHz pulsed oscillator was used to drive the transmitting transducers (center frequencies of 2.25 MHz longitudinal, 5.0 MHz shear). The received signals were digitized at 25 MHz and stored for processing.

Table 1 summarizes the longitudinal and shear wave velocity measurements made transverse and parallel to the plies of the fiber reinforcing cloth. Significantly lower velocities for Sample 265 (weakly bonded interface) compared to Sample 260 (strongly bonded interface) are evident. These velocities should not be interpreted as being the definitive values for this material as they represent measurements on only two samples; because of the complexity of the material, a good deal of statistical scatter is expected for measurements from nominally identical samples. Nevertheless, it is felt that the measured differences in the two samples are indicative of the difference in fiber-matrix bonding. It should also be noted for Sample 265, the measured shear wave velocity perpendicular to the plies was apparently greater than the longitudinal wave velocity, an unusual, if not physically impossible, result. However, the wave velocities were difficult to make for this sample because of its high attenuation and distorted wave forms which resulted in a large experimental measurement error. This is still under study. As illustrated in Figure 1, Sample 265 also demonstrates a significantly higher signal attenuation. Since both samples are similar in density and fiber content, the measured differences in acoustic properties are attributed to the differences in interface bonding.

Acousto-Ultrasoundics

The acousto-ultrasonic (AU) technique was developed by Vary and coworkers [4] to characterize the mechanical properties of materials. Because the technique measures the response of the material to acoustic


<table>
<thead>
<tr>
<th>Sample</th>
<th>Strong bond</th>
<th>Weak bond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Propagation Direction</td>
<td>↓</td>
<td>↑</td>
</tr>
<tr>
<td>Relative to Plies</td>
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<td></td>
</tr>
<tr>
<td>Longitudinal Velocity (mm/μs)</td>
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<td>10.3</td>
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<td>Shear Velocity (mm/μs)</td>
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<td>6.6</td>
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<tr>
<td>Polarization</td>
<td></td>
<td>to Plies</td>
</tr>
<tr>
<td>Shear Velocity (mm/μs)</td>
<td>5.2</td>
<td>6.5</td>
</tr>
<tr>
<td>Polarization ↓ to Plies</td>
<td></td>
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Fig. 1. Ultrasonic attenuation measured for longitudinal waves.

stresses in directions which lie in the laminate plane [5], it is potentially capable of characterizing the fiber-matrix bond. The name of the technique derives from the fact that it is basically a hybrid between ultrasonics and acoustic emission. Two piezoelectric transducers are placed on the same side of a material sample. An excitation pulse to one transducer generates acoustic waves in the sample which are received by the other transducer, amplified, and recorded in a manner similar to conventional transmit-receive ultrasonics. The difference is that, instead of detecting a well-defined acoustic pulse traveling between the transducers, as is the case with conventional ultrasonics, a complicated superposition of wave modes and sample reverberations which resembles an
acoustic emission signal is generated and detected. The received signals are processed in a manner similar to acoustic emission signals. Usually the total number of signal "counts" (number of times the signal exceeds a fixed threshold) or the total energy content of the signal is calculated and used to characterize the sample.

The AU measurement system used for this work is shown schematically in Figure 2. The transducers were Valpey-Fisher "pinducers," which are high sensitivity, small diameter (2.4 mm) piezoelectric transducers. These were chosen primarily for their small size, which makes them ideal for point measurements, and because they minimize perturbations to the vibration modes of the samples.

Recorded AU signals for Samples 260 and 265 are shown in Figure 3. Figure 4 shows Fourier transforms of the signals. As can be seen, there are significant differences in both amplitude and frequency content between the two signals. The total energy contained in the signal response from the well-bonded sample is over 100 times greater than that of the poorly-bonded sample. Based on measurements made on other samples of this material, acoustic property differences of this magnitude would be expected only for material samples having significantly different porosity content. However, since the densities of these samples are approximately the same, the different AU responses are assumed to be caused by the differences in fiber-matrix bonding.

**Dynamic Mechanical Damping**

Dynamic vibration response measurements are also being used to characterize fiber-matrix interface bonds. One of the parameters directly measured by this technique is the internal damping of vibrational energy that can occur at the fiber-matrix interfaces of a composite [6]. The technique has an advantage over ultrasonic attenuation measurements in which the actual absorption of elastic energy is often dominated by the scattering of elastic waves by material discontinuities such as porosity and reinforcing fibers.

![Diagram of experimental setup for acousto-ultrasonic and internal damping measurements.](image)

Fig. 2. Diagram of experimental setup for acousto-ultrasonic and internal damping measurements.
Fig. 3. Recorded acousto-ultrasonic signals. The energies contained in the signals are given in arbitrary units.

Fig. 4. Frequency spectra of the acousto-ultrasonic signals of Figure 3.
A measure of the internal damping can be obtained by the diffuse field decay rate method [7]. This analysis is based upon the premise that a diffuse ultrasonic field in an isolated sample will decay due only to absorption mechanisms and that the contributions of damping by air, transducers, and fixturing are minimal. The damping is measured through determination of the volume averaged decay rate of the ultrasonic field as a function of frequency and time. A greater decay rate indicates a greater amount of internal damping or energy absorption through internal friction. The technique can be implemented using the same experimental setup as was used for the AU measurements (Figure 2). Mechanical isolation of the sample is accomplished by weakly coupling the transducers to the sample, which is set on point supports. Determination of the decay rate is accomplished by dividing the recorded waveform into a number of time windows for which Fourier transforms are performed. The resulting spectrums are then broken into frequency bins and the mean square spectral amplitude is calculated for each bin. This provides a measure of the signal decay rate for individual frequency bins as a function of time.

Diffuse decay rate analysis was performed using the recorded AU responses for Samples 260 and 265. The results were obtained using 16 μs windows in conjunction with 0.98 MHz wide frequency bins. To avoid initial field transients, the first 48 μs of the recorded AU waveforms were not used. Typical results for one frequency bin (0.98 - 1.95 MHz) are presented in Figure 5. The different slopes for the two curves represent different decay rates with Sample 265 having the largest decay rate. From several measurements made on each sample, average decay rates of 0.24 1/μs ±0.04 1/μs and 0.47 1/μs ±0.07 1/μs were calculated for Samples 260 and 265, respectively. This indicates that the weakly bonded fiber-matrix has a greater amount of internal damping.

Fig. 5. Diffuse field decay with time in the frequency band 0.98-1.95 MHz for the signals of Figure 3.
SUMMARY AND CONCLUSIONS

Measurable ultrasonic and acoustic differences were observed between the extreme cases of strong and weak fiber-matrix interface bonding in SiC/SiC ceramic composites. Significant differences exist between the ultrasonic shear and longitudinal velocities, magnitude and frequency dependence of the attenuation, acousto-ultrasonic responses, and internal damping of the two samples. Additional work on material samples having intermediate bond strengths will be required to fully determine the capability of the techniques to measure fiber-matrix bond strengths.

ACKNOWLEDGMENTS

This work was supported by the U.S. Department of Energy Assistant Secretary for Fossil Energy, Office of Technology Coordination, under DOE Contract No. DE-AC07-76ID01570.

REFERENCES