

DAMAGE ASSESSMENT OF FIBER/MATRIX INTERFACE IN CERAMIC MATRIX COMPOSITES USING ELASTIC STRESS WAVES[†]

Theodore E. Matikas*, Prasanna Karpur*, and Rollie E. Dutton**

*WL/MLLP/UDRI, 300 College Park Avenue, Dayton, Ohio 45469-0127

**NIST, Wright Laboratory, WL/MLLN, Wright-Patterson AFB, Ohio 45433

ABSTRACT

In this work, nondestructive ultrasonic techniques have been used to characterize the interface as well as to assess the microcracking and interface damage of glass ceramic composites subjected to axial loading. The composites used in this study have been fabricated via tape casting and consist of regular arrays of either TiB₂ coated SIGMA 1240 or carbon coated SCS-6 fibers in a 7040 borosilicate glass matrix. Results from the ultrasonic shear back-reflectivity technique for the evaluation of interfacial shear elastic property are compared to scanning acoustic microscopy observations of matrix microcracking and the associated interface damage in the two composite systems.

INTRODUCTION

Several indirect techniques have been developed in the past to characterize the fiber-matrix interface. However, all those techniques are destructive and deal with the strength or failure modality of the interface although, for mechanics analyses of composites, the elastic behavior of the interface is also essential. Nondestructive techniques based on bulk ultrasonic waves of relatively low frequencies (< 15 MHz) have been successfully developed by Rokhlin et al. [2] to evaluate the global properties and then to deduce the interfacial properties and perform damage assessment. One of the techniques discussed in this paper is based on higher ultrasonic frequencies (≥ 25 MHz) and has been developed by Matikas et al. [4] to assess the local interfacial properties and damage of composite materials. The approach nondestructively evaluates the elastic shear load transfer behavior between the matrix and the fiber by measuring a parameter called the 'shear stiffness coefficient' of the interface. In addition, a technique based on scanning acoustic microscopy (SAM) was developed by Karpur et al. [3] and is used to examine the interfacial damage due to loading in two composite systems with the same matrix but different fibers.

[†] work performed on-site in the Materials Directorate, WPAFB, Contract No. F33615-94-C-5213.

To the extent authorized under the laws of the United States of America, all copyright interests in this publication are the property of The American Ceramic Society. Any duplication, reproduction, or republication of this publication or any part thereof, without the express written consent of The American Ceramic Society or fee paid to the Copyright Clearance Center, is prohibited.

reflectivity (SBR) technique) from the fibers. The experimental procedure for the measurement of the interfacial shear stiffness coefficient was carried out in several steps. First, a reference A-scan was obtained from a sample made with the matrix material. A Fourier transformation provided the incident magnitude of reflected ultrasound at 25 MHz (the frequency of 25 MHz was selected based on the information obtained by the modeling [4]). Similarly, A-scans were obtained from the fibers and the corresponding reflected magnitudes at 25 MHz were measured using Fourier transformation and the reflection coefficient was calculated.

The calculated reflection coefficient was used to inverse calculate the shear stiffness coefficient of the equivalent elastic interface using the theoretically generated curves as shown in Figure 1. The elastic properties of the constituents of the composites used in the calculations as well as the interfacial shear stiffness coefficients are listed in Table 1.

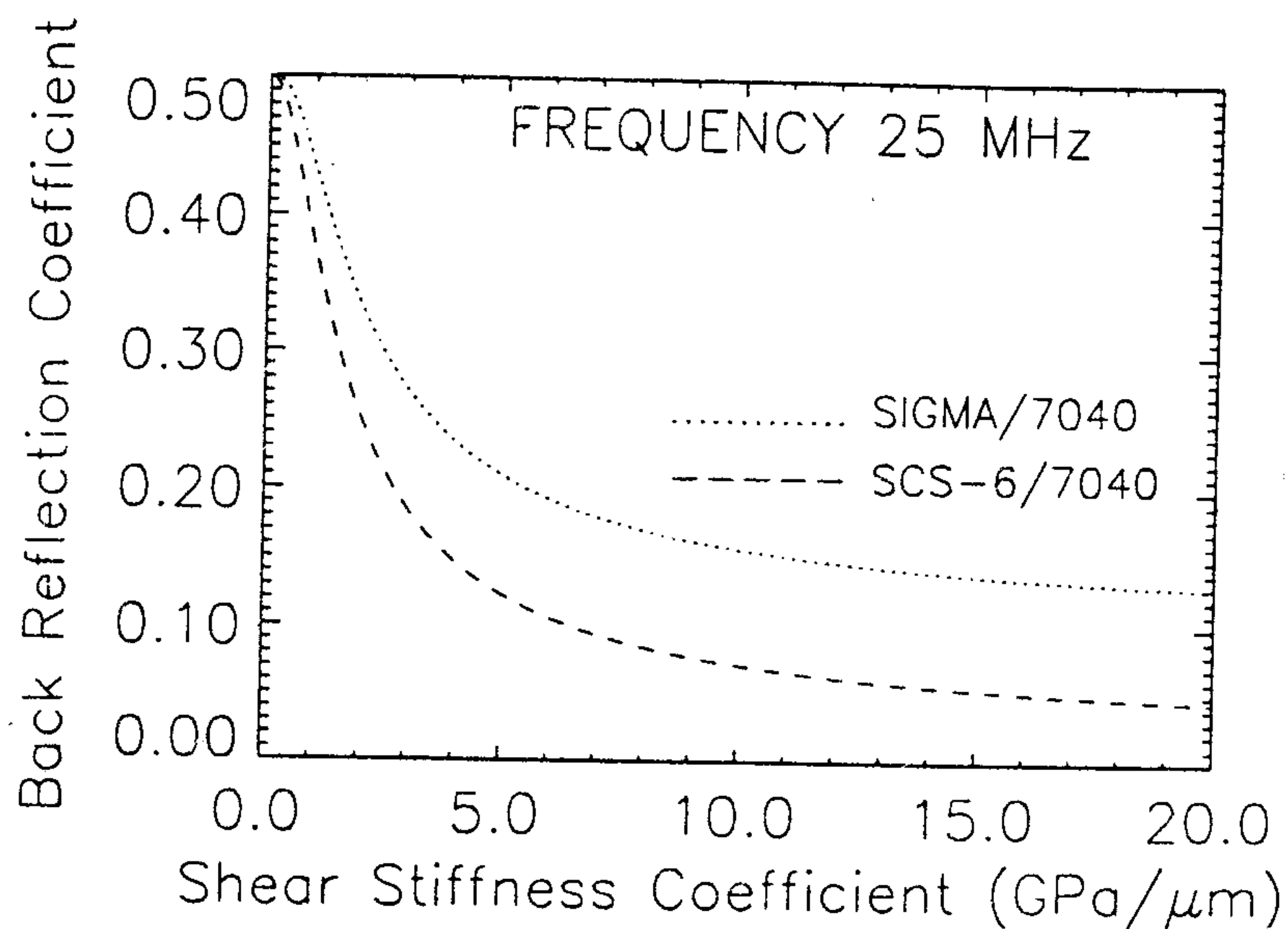


Figure 1 Theoretical curves showing the dependence of the shear back-reflection coefficient on the interfacial shear stiffness coefficient for the two types of composites used in this study.

Table 1 Measured elastic properties of composite constituents and theoretically calculated shear stiffness coefficient of CMC's based on experimentally measured back-reflection coefficient.

Material	Density g/cm ³	Long. Velocity m/s	Shear Velocity m/s	Poisson's Ratio	Young's Modulus GPa	N _s MPa/μm
7040	2.24	4980	3050	0.2	50	-
SCS-6	3.17	11690	7156	0.2	325	483 ± 35
SIGMA	3.65	9940	6087	0.2	390	812 ± 19

MODELING OF THE INTERFACIAL SHEAR STIFFNESS COEFFICIENT

A theoretical model was developed which aids in the determination of various experimental parameters such as the frequency of ultrasound and angle of incidence while providing the vital relationships necessary to interpret the experimental results. The theoretical model considers the reflection of an ultrasonic wave front from fibers embedded in a ceramic matrix. For the development of the theoretical model, the interface between the matrix and the fiber is modeled by: (i) assuming continuity of normal and shear stresses and normal displacements at the interface, and (ii) by allowing the discontinuity of shear displacements at the interface. It is assumed that the vibration is transmitted instantaneously from one medium to the other by weightless shear springs with an equivalent rigidity of N_s (MPa/ μm) which defines the shear stiffness coefficient of the interface. Accordingly, the interface conditions are:

$$\begin{aligned} \{\sigma^P\} = 0, \quad \{\sigma^T\} = 0 \quad \text{and} \quad [u^P] = 0 \\ \sigma^T = N_s \cdot [u^T] \end{aligned} \quad (1)$$

where the superscripts P and T denote the normal and tangential displacements/stresses respectively; the square brackets denote the jump of a function across the interface, and the curly brackets denote the vectorial resultant of stresses at the interface. In this study, the above model has been used to assess the interfacial properties in several composite systems.

In this analysis, the ultrasonic beam is considered to be incident on the composite such that the refracted wave is always normal to the fiber circumference [4]. The angle of incidence is such that the refracted shear waves are incident on the fibers and reflected back to the transducer. The reflection can be quantified by the shear back-reflection coefficient from the fiber (SBRC) as shown in Equation (2) below. This can be calculated after solving the wave equation using the boundary conditions in Equation (1) above:

$$\text{SBRC} = T_M R_F T_W \quad (2)$$

where the terms on the right hand side of the equation are functions of the properties of the matrix and the fiber, the diameter of the fiber, the angle of incidence, the frequency of interrogation of ultrasound, and the interfacial shear stiffness coefficient (N_s). Further details of this method can be obtained from literature [4].

EXPERIMENTAL DETERMINATION OF INTERFACIAL SHEAR STIFFNESS COEFFICIENT

Ultrasonic quantification of the shear stiffness coefficient can be performed [5] by experimental measurement of the back-reflected ultrasonic shear waves (shear back-

INTERFACIAL DAMAGE ASSESSMENT USING SCANNING ACOUSTIC MICROSCOPY

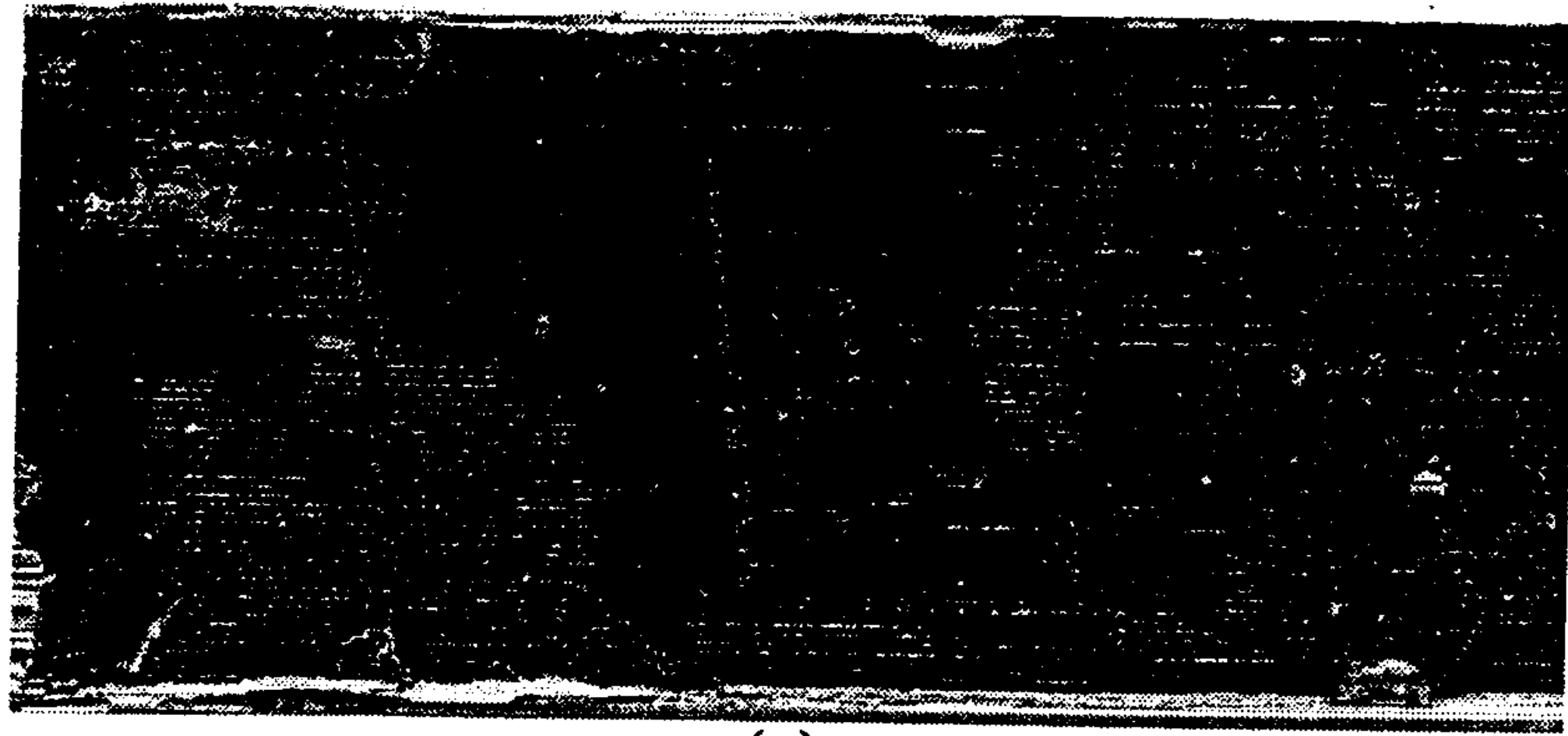
The principle of operation of a SAM transducer is based on the production and propagation of surface acoustic waves (SAW) as a direct result of a combination of the high curvature of the focusing lens of the transducer and the defocus of the transducer into the sample [3]. The contrast of the images obtained using SAM is based on the attenuation and reflection of SAW. In addition, the sensitivity of the SAW signals to the surface and subsurface features depends on the degree of defocus. This has been well documented in the literature as the $V(z)$ curves [6]. The defocus distance also has another important effect on the SAW signal obtained by the SAM transducer: the degree of defocus dictates whether the SAW signal is well separated from the specular reflection or interferes with it. Thus, depending on the defocus, the SAM technique can be used either to map the interference phenomenon in the first layer of subsurface fibers or to map the surface and subsurface features (reflectors) in the sample.

A 50 MHz scanning acoustic microscopy transducer has been used in this study for the imaging and evaluation of advanced composites. All the images have been produced by exploiting the surface wave component of the ultrasonic signals from the scanning acoustic microscope because of the higher sensitivity of surface waves to both surface/subsurface cracks. The results shown in this paper provide an illustration of the potential of SAM for Fiber-Matrix evaluation in advanced composites such as ceramic matrix composites.

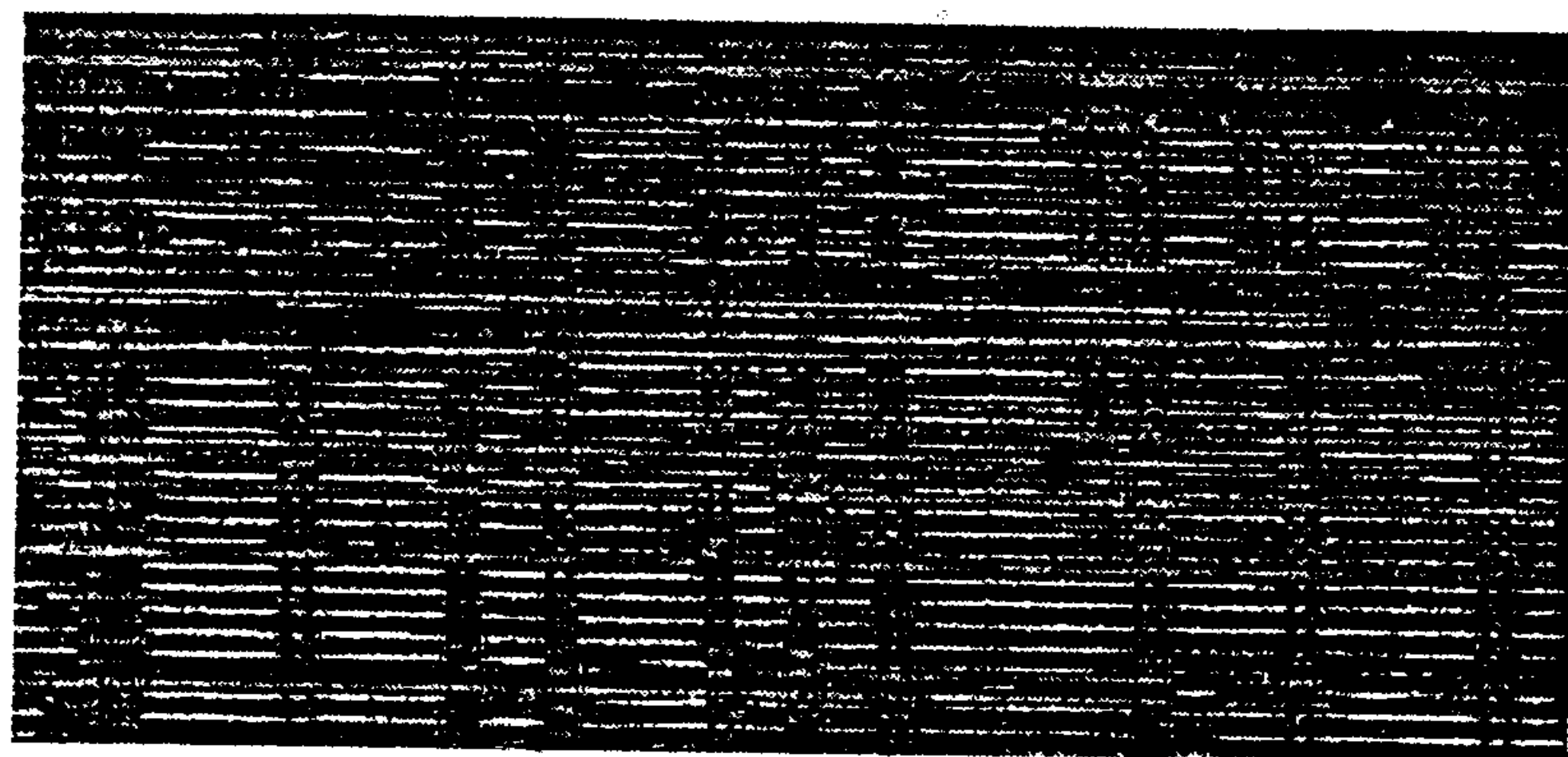
In this work unidirectional SiC fiber reinforced, 7040 borosilicate glass matrix composites with controlled spacing [1] were fabricated by tape casting. The glass used for the composites was supplied by Corning as an $\sim 8\mu\text{m}$ powder. The composition of the glass was such that the thermal expansion coefficient mismatch between the fiber and the matrix resulted in compressive residual radial thermal stresses after processing. Two different fibers were utilized: a TiB_2 coated SIGMA 1240 fiber with a diameter of 102 μm and a SCS-6 fiber (carbon coated) with a diameter of 142 μm . The borosilicate glass strongly wets the TiB_2 coating of the SIGMA 1240 fiber while it weakly wets the carbon coating of the SCS-6 fiber. This allowed the investigation of the effects of bonding at the fiber/matrix interface and the resultant variations in interfacial damage upon the initiation of matrix cracking due to externally applied tensile loading of the composite samples.

The composites were processed by tape casting the glass powder into a green tape with a relative density of 50%. The green tapes were cut to size and laminated with fiber mats of the desired fiber spacing (27 or 47 fibers per cm). The volume fraction of fibers in the composites was varied by altering the thickness of the green tape and using the two different fiber spacings. After lamination the composites were inserted into a tube furnace and vacuum sintered at 710° C for one hour. The samples were then hot isostatically pressed at 650° C for 30 min. with an applied pressure of 35 MPa to remove the residual porosity ($\sim 2\%$). The resulting samples were approximately 10 cm long by 2 cm wide with a thickness of 0.2 cm.

The samples were loaded to induce microcracking of the composites as a part of an extensive study to assess the microcracking behavior of glass matrix composites. The samples were then evaluated using the SAM technique. The results are shown in Figures 2(a) and 2(b) where in the images are those of samples with SCS-6 fibers and SIGMA fibers, respectively.



(a)



(b)

Figure (2) (a) Scanning acoustic microscopy image of a SCS-6/7040 composite showing matrix cracking and extensive fiber/matrix interface debonding. (b) SAM image of a SIGMA/7040 composite showing matrix cracking and fiber/matrix interface debonding limited to the proximity of the matrix cracks.

DISCUSSION

The results of the shear back-reflectivity technique shown in Table 1 indicate that the fiber/matrix interface of SCS-6/7040 composite is compliant compared to the SIGMA 1240/7040 composite because the measured shear stiffness coefficient of the first composite is about half of that of the second composite. Acoustic microscopy results shown in Figures 2(a) and 2(b) indicate that the interface of the SCS-6/7040 composite substantially debonds along the fiber/matrix interface (Figure 2(a)) after matrix cracking induced by uniaxial loading. Comparatively, the SIGMA 1240/7040 composite with higher interfacial stiffness coefficient shows minimal interfacial debonding in the proximity of the matrix cracking (Figure 2(b)). Further investigation on the extent of debonding (which appears in Figure 2(b) to be about one fiber diameter) will be conducted. While the SBR technique measures

an elastic parameter, N_s , it appears to correlate well with the strength of the fiber/matrix interface. That is, the SIGMA 1240 fibers strongly wet by the glass matrix and the resulting interface has an N_s of 812 ∓ 19 MPa/ μm , and exhibits minimal debonding after matrix cracking. In contrast, the SCS-6 fibers are weakly wet by the glass, have an N_s of 483 ∓ 35 MPa/ μm , and exhibit extensive debonding after matrix cracking. It is reasonable to expect that the degree of wetting will influence the degree of bonding at the fiber/matrix interface and hence its elastic and strength properties.

CONCLUSIONS

In this paper, ultrasonic nondestructive methods have been shown to be effective for the evaluation of damage in ceramic matrix composites. First, the shear back-reflectivity technique has been used to evaluate the interface shear stiffness coefficients of two glass matrix composite systems made with the same matrix material and different fibers. Further, another technique based on acoustic microscopy has been used to image the matrix microcracking and interface damage. From the results, an apparent correlation has been established between interface stiffness and the corresponding interfacial debonding due to uniaxial loading of the composites.

REFERENCES

- [1] Gustafson C. M., R. E. Dutton, R. J. Kerans, "Fabrication of Glass Matrix Composites by Tape Casting", *J. Am. Ceram. Soc.*, 78(5), pp. 1423-24, (1995).
- [2] Chu Y. C., S. I. Rokhlin, "Determination of Macro- and Micromechanical and Interfacial Elastic Properties of Composites from Ultrasonic Data", *J. Acoust. Soc. Am.*, 92(2), pp. 920-931, (1992).
- [3] Karpur P., T. E. Matikas, M. P. Blodgett, J. R. Jira, D. Blatt, "Nondestructive Crack Size and Interfacial Degradation Evaluation in Metal Matrix Composites Using Ultrasonic Microscopy", *Special Applications and Advanced Techniques for Crack Size Determination*, J. J. Ruschau, J. K. Donald, Eds., (American Society for Testing and Materials, Philadelphia, 1995), ASTM STP 1251, pp. 130-146.
- [4] Matikas T. E., P. Karpur, "Ultrasonic Reflectivity Technique for the Characterization of Fiber-Matrix Interface in Metal Matrix Composites", *Journal of Applied Physics*, 74(1), pp. 228-236, (1993).
- [5] Karpur P., T. E. Matikas, S. Krishnamurthy, "Ultrasonic Characterization of the Fiber-Matrix Interphase/Interface for Mechanics of Continuous Fiber Reinforced Metal Matrix and Ceramic Matrix Composites", *Journal of Composites Engineering*, (1995), in print.
- [6] Briggs G. A. D., *Acoustic Microscopy*, (Oxford Univ. Press, Oxford, 1992).