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# ULTRASONIC CHARACTERIZATION OF THE FIBER-MATRIX INTERPHASE/INTERFACE FOR MECHANICS OF CONTINUOUS FIBER REINFORCED METAL MATRIX AND CERAMIC MATRIX COMPOSITES

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Abstract-This paper presents a novel approach to evaluate the elastic properties and the behavior of the interphase region formed by a chemical reaction between the matrix and the fiber materials in metal matrix and ceramic matrix composites. Contrary to the traditional approach which does not allow any relative displacement at the interface without fracture, this paper considers elastic deformation of the interphase zone between the matrix and the fiber by replacing the zone by an "equivalent elastic interface". The elastic behavior of the equivalent elastic interface describes the local elastic rigidity and deformation of the interphase zone and can be quantified by a mechanics parameter called "shear stiffness coefficient" which is proportional to the ratio of the shear modulus to the local thickness of the interphase material. This paper also outlines an ultrasonic reflectivity modeling that can be used for the experimental measurement of the interfacial shear stiffness coefficient along the length of an embedded fiber. Further, an experimental method of measurement of the shear stiffness coefficient is presented and experimentally measured values are tabulated. The significance of the quantification of such a parameter is that the elastic property of the interface obtained can be used as a common basis among material scientists designing and developing the composite systems, and groups studying material behavior for life prediction. Also, the parameter can be used by production engineers to assure that the designed properties of the composite are being achieved, and by the end users to ensure that the designed and produced properties are being retained in use.

#### INTRODUCTION

Many types of structural materials, such as continuously reinforced metal matrix and ceramic matrix composites, are being designed and developed for high strength and high temperature aerospace applications. The fiber and the matrix in these composites are selected based on several factors including their mechanical properties to impart high strength, high toughness, high impact resistant properties and high modulus to the composite in its finished form. However, the resulting properties of the composite are also dependent on the chemical reactivity between the matrix and the fiber, the elastic behavior of the fiber-matrix interface and thermomechanical compatibility between the matrix and the fiber.

Continuously reinforced composites are generally fabricated at high temperatures and pressures. Under these processing conditions, the chemical reaction between the matrix and the fiber often produces a thin "interphase zone" which generally has mechanical properties different from the two phases producing it (Metcalfe, 1974). Although the interphase region between the fiber and the matrix materials is generallly very thin, the properties of the composite system are dominated and determined by the properties and the mechanical behavior of the interphase region (Clyne and Watson, 1991; Evans *et al.*, 1991). It is through the interphase region that the load is transferred between the matrix and the fiber, and that the toughness of the composite is determined (Park *et al.*, 1989; Clyne and Watson, 1991; Evans *et al.*, 1991). As a result, the interphase is of great interest for the mechanics of composites analyses.

Several indirect techniques (Kerans and Parthasarathy, 1991; Karpur *et al.*, 1992; Waterburry *et al.*, 1994) such as fiber push-in, fiber push-out, fiber pull-out, and fiber fragmentation, have been developed in the past to characterize the fiber-matrix interface. However, all the techniques mentioned above 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 as essential as the failure strength obtained by the above methods. Hence, the objective of this paper is to provide a novel approach to non-destructively evaluate the elastic load transfer behavior between the matrix and the fiber by defining a quantifiable parameter to describe the "elastic behavior" of the interface. The definition of the parameter will be accomplished by describing the behavior of an "equivalent elastic interface" which replaces the "interphase region" consisting of one or several "layers" of different compositions.

Modeling in mechanics of composites is generally accomplished by either considering an interface as a perfect boundary between the matrix and the fiber (Wright et al., 1990) or by including several "interphase layers" with ideal (perfect) interfaces between them. Analyses in the area of fracture mechanics of composites model the interface as either an ideal boundary where continuity of tractions and displacements exists if the interface is intact (Clyne and Watson, 1991; Evans et al., 1991) or a complete discontinuity when a fracture of the interface occurs because of a crack deflection at a weak interface. However, the treatment of the interface from only the fracture point of view does not consider the possibilities of various elastic properties of the interphase region obtained due to the processing of the composite and the necessary design of the interphase material such that the interface behaves in the way desired by the fracture mechanics requirements. Since the elastic properties of the interphase material are critical to the overall mechanical properties and the fracture behavior of the composite, it is essential to consider the elastic properties of the interphase zone in the elasticity modeling. However, since the elastic/mechanical properties as well as the thickness of the interphase material are generally variable, unknown, and difficult to measure, inclusion of the interphase region as a third (or multiple) layer poses serious difficulties for the analyses. As a result, the concept of an "equivalent elastic interface" is presented in this paper to recognize and incorporate the finite thickness of the interphase, as well as the associated elastic property without having to explicitly measure either the thickness or the elastic property of the interphase material.

## MODELING THE INTERPHASE REGION AS AN EQUIVALENT ELASTIC INTERFACE BETWEEN THE MATRIX AND THE FIBER

The interphase region is modeled here by an "equivalent elastic interface" possessing a specific "elastic behavior". The "equivalent elastic interface" must not be confused with the traditional "interface" which is an ideal boundary between two solid materials (the matrix and the fiber) because the traditional interface has a precise mechanical property (implying no relative displacement between its sides without fracture). In contrast, the "equivalent elastic interface" is an "elastic boundary" between the matrix and the fiber that takes into account the elastic properties of the interphase region and has a precise elastic behavior which is pictorially represented in Figs 1a and 1b.

Figure 1a shows the interphase region between the matrix and the fiber. The gradation of gray scale in the interphase region has been drawn to represent the possibilities of either multiple interphase layers or an interphase region of variable properties along its thickness. The interfaces "A" and "B" in Fig. 1a would be the ideal traditional interfaces as used in current mechanics literature. As a result, the pairs of arrows of equal lengths drawn at these interfaces represent the continuity of displacements at the respective interfaces. However, to build such a behavior into any model, an *a priori* knowledge of the elastic properties of the interphase region is essential. Since interphase material properties are usually difficult to obtain, there is a need for a model and an experimental procedure that take into account the relative elastic displacement across the interphase zone by quantifying the shear behavior of the interphase region. In Fig. 1b, the interphase region has been condensed to represent the

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Fig. 1. (a) Schematic of the interphase region between the matrix and the fiber. The gradation of gray scale in the interphase region has been drawn to represent the possibilities of either multiple interphase layers or an interphase region of variable properties along its thickness. (b) Schematic showing the "equivalent elastic interface" between the matrix and the fiber materials. (c) Schematic showing an intermittent bonding between the matrix and the fiber materials. Such a condition of intermittent bonding is different from the "elastic bonding" defined in this paper as in Figs 1a and 1b.

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"equivalent elastic interface" which, because of its elastic properties (shear modulus and the local thickness) can elastically deform without fracture. Thus, there can be an elastic relative displacement across the "equivalent elastic interface" as shown in Fig. 1b wherein the arrows representing the displacements are the same displacements depicted at the interfaces on either side of the interphase region in Fig. 1a. Therefore, contrary to the notion of the traditional "interface" which will have to fracture to provide differential displacement across it, the "elastic behavior" of the "equivalent elastic interface" allows, by virtue of its elastic deformation, a relative displacement between its side (matrix and fiber) without fracture.

It is obvious from this description of equivalent elastic interface and its elastic behavior that the proposed model is different from the "intermittent bonding" model (Fig. 1c) available in the literature (Margetan *et al.*, 1992). Although intermittent bonding is a valid method of modeling the interface when the composite has been made with poor consolidation on either macroscopic or microscopic levels, it is irrelevant to this paper because a well consolidated composite is assumed for the analysis to be presented in the next section. Such an assumption of good consolidation is realistic for a functional composite and is feasible because of ultrasonic nondestructive techniques previously developed (Matikas *et al.*, 1992; Krishnamurthy *et al.*, 1994) to assure proper consolidation.

# SIGNIFICANCE OF EQUIVALENT ELASTIC INTERFACE

The use of the words "interphase" or "interface" and the associated meanings, although traditionally not interchangeable, are dependent on the point-of-view of the different scientific/engineering groups involved in the development, fabrication, testing, and modeling of the composite.

The materials processing point-of-view involves the formation (Metcalfe, 1974) of a "reaction zone", i.e. an interphase that may impart bonding between the matrix and the fiber. It is this reaction zone that is modeled in this paper as the equivalent elastic interface. The modeling will be used to guide the experimental measurement of the elastic property of the equivalent elastic interface which is dependent on the properties of the interphase material that are in turn dependent on the fiber-matrix material compatibility determined by the chemical reactivity (Fig. 2) as well as the materials processing parameters such as pressure, temperature, and duration. In addition, the equivalent elastic interface between the matrix and the fiber can have various behaviors due to no contact (void or porosity at the fiber-matrix interface) or mere contact (implies just physical contact with no chemically formed bonding and hence no transmission of displacements and stresses across the interface), perfect bonding (implying no relative displacement across the equivalent elastic interface), or a gradation of elastic properties of the interphase region ranging from "no contact" or "mere contact" to "perfect bonding". Therefore, a parameter which describes this elastic behavior of the interface will also have a range of possible valid values.



Fig. 2. The elastic behavior of the interphase region is affected by the chemical bonding due to reactivity between the matrix and fiber materials.

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# Characterization of fiber-matrix interphase/interface

The implication of a range of possible values for the parameter characterizing the elastic behavior of the interphase region is that the load transfer behavior of the interphase is directly linked to a range (due to the choice of the matrix and fiber materials and the composite processing conditions) of shear modulus and thickness of the interphase region which would modify the overall mechanical properties of the composite. As a result, a well understood and controlled interphase development is needed to optimize the mechanical behavior of the composite systems. However, since there is no method of quantification of the progressive formation of the interphase, achieving an interphase with desired properties is difficult at best. Hence, a quantifiable measurement parameter based on the principles of mechanics will provide the bridge between the materials processing concepts of an interphase and the mechanics concepts of an interface while providing a tool for the monitoiring and control of the composite development process.

Environmental effects and the high temperature behavior of MMCs and CMCs necessitate the recognition of the degradation of the interface in addition to the fracture behavior of the interface (Russ et al., 1991; Blatt et al., 1993; Karpur et al., 1995). The mechanical properties of the composite are usually altered due to the effects of the elevated temperature in a given environment even before an extensive fracture of the interfaces in the composite occurs (Karpur et al., 1995). Such a change in the properties of the composite occurs primarily due to the chemical and material modifications of the interphase layer (in addition to the modifications of the matrix and the fiber properties). This view is supported in the literature (Blatt et al., 1993; Karpur et al., 1995) wherein it has been shown that it is the interphase region that starts to deteriorate first well before any noticeable deterioration of the matrix and fiber properties. The characterization and quantification of the elastic behavior and the degradation process of the interphase zone could be accomplished through the quantification and monitoring of a parameter (to be defined later in this paper) associated with the equivalent elastic interface. Such a parameter could be used for both the mechanics modeling of composites and the characterization of the interphase layer. This parameter can also be used as a tool for the monitoring of the material behavior of composites during life prediction studies. This approach would complement, while deviating from, the present methodology of indirect evaluation of the interface behavior and deterioration through experimental procedures such as monitoring of crack initiation and propagation (Marshall and Evans, 1985; McCartney, 1987) and analysis of micro-mechanical stresses (McCartney, 1990; Pagano, 1991).

# A NEW PARAMETER FOR THE CHARACTERIZATION OF THE EQUIVALENT ELASTIC INTERFACE

A newly introduced parameter for the interphase/interface characterization should serve two purposes: (a) the parameter should be quantifiable to monitor the progressive formation and changes in the interphase during the manufacturing process so that the composite can be made with the specified interphase properties for the designed material behavior requirements, and (b) the parameter should be well defined to be used in mechanics modeling as a tool to evaluate the mechanical behavior of the interfaces and to account for changes during usage such as fracture, environmental degradation, etc. As a result, the parameter to be developed should be based on physical/mechanical quantities that could be both theoretically modeled and experimentally measured. At the same time, the experimental measurement technique should be nondestructive so that the composite can be either used for other material behavior and life prediction experiments during its developmental stages, or as a structural member after the research and development stages. The parameter can be defined and measured using ultrasonic elastic stress waves to characterize the interphase/interface.

# Wave mechanics modeling for ultrasonic evaluation of the interface

Ultrasonic stress waves as used in nondestructive evaluation produce extremely small elastic displacement amplitudes of a fraction of an Angstrom. Hence, the objective of

using ultrasonic characterization would be to study the elastic property of the interphase region through the quantification of the equivalent elastic interface constituted by the interphase region (and not to measure the strength of the interfacial bonding which is not possible using elastic waves).

The interphase zone, by virtue of its elastic behavior in response to the small amplitude ultrasonic waves, acts as a coupling between the matrix and the fiber. The ultrasonic stresses incident on the interphase can be generally of two modalities: dilatational (or longitudinal) waves and tangential (or shear) waves. The interphase behaves differently to the two incident modes of ultrasonic waves. When the compressional waves are incident from the matrix to the fiber through the interphase, the stresses and displacements are transmitted as if a "compressional spring" exists (Fig. 3a) in place of the interphase. However, because of the nature of the incident compressive stresses, even a mere contact with a complete absence of chemically formed interphase zone is sufficient to transmit the stresses and displacements from the matrix to the fiber, especially in the presence of radial compressive residual stresses. Therefore, it is generally not feasible to measure the response of the "compressional spring" using ultrasonic methods. On the other hand, when elastic (ultrasonic) shear waves are incident from the matrix to the fiber through the interphase region, the interphase acts like a "shear spring" as shown in Fig. 3b. The incident shear stresses and the resulting displacements are transmitted through the shear spring only when a chemically formed bonding (interphase) exists. In the absence of chemical reaction (bonding), especially when residual stresses are also negligible, the shear spring will be unable to transfer any stresses and thus a complete "slip" occurs at the boundary between the matrix and the fiber. Consequently, ultrasonic methods can be used to study the behavior of the "shear spring" at the interface. A new



Fig. 3. (a) Dilatational or compressive spring behavior of the interphase/interface zone when longitudinal elastic waves are incident on the equivalent elastic interface. (b) Shear spring behavior of the interphase/interface zone when shear elastic waves are incident on the equivalent elastic interface.

method of ultrasonic characterization has been developed (Matikas and Karpur, 1993a) for this purpose and will be presented next. Although similar nondestructive techniques based on bulk ultrasonic waves of relatively low frequencies (<15 MHz) have been successfully developed by Rokhlin and co-workers (Rokhlin and Wang, 1989, 1992; Chu and Rokhlin, 1992; Hefez and Rokhlin, 1992; Chu *et al.*, 1993a, b) to evaluate the global properties and then to deduce the interfacial properties and damage assessment, the methodology being proposed here deviates from Rokhlin's method because of the localized property measurement using higher ultrasonic frequencies.

The interphase formulated as a spring, either compressional, shear, or both, defines the fiber-matrix interface. However, due to the insensitivity of the compressive spring to the variability of the interface elastic behavior, only the shear springs and the associated stiffness coefficient will be used in this study as a quantifying parameter to characterize the interface.

The geometry of the ultrasonic characterization of the equivalent elastic interface is shown in Fig. 4. For incident shear stress waves at the interface, the following boundary conditions exist:

- continuity of stresses, both normal,  $\sigma_L$ , and shear:  $\sigma_T$ :  $\{\sigma_L\} = 0$  and  $\{\sigma_T\} = 0$  (1)
- continuity of normal displacements,  $u_L$ :  $[u_L] = 0$  (2)
- discontinuity of shear displacements,  $u_T: [u_T] \cdot N_S = \sigma_T$  (3)

where [] denotes a jump across the interface and {} denotes the vectorial resultant of stresses at the interface. In eqn (3) above, the product on the left hand side shows the linearity possible because of the small amplitudes of vibration and relatively long wavelengths of ultrasound, and  $\sigma_{\rm T}$  is the shear stress at the interface due to inertia-free spring forces. We introduce a new parameter,  $N_{\rm S}$ , which is the stiffness coefficient of an



Fig. 4. Schematic of the geometry of the interface characterization modeling using ultrasonic wave propagation theory.

inertia-free shear spring proposed here to quantitatively characterize the interphase/ interface in the mechanics of composites analyses. Although somewhat similar boundary conditions can be found in the literature (Jones and Whittier, 1967; Hashin, 1990; Jasiuk *et al.*, 1992; Jun and Jasiuk, 1993), the boundary conditions introduced here are significant for the ultrasonic analysis because of the explicit treatment of the slip condition of the shear spring even when mere contact exists, however with no chemical bond and also by the facet that the compressional spring is allowed to have only two values, zero and infinity. This two valued treatment of the compressional spring is necessary and valid for ultrasonic analyses because of the reasons discussed in the literature (Matikas and Karpur, 1993a).

The replacement of the interphase region by an equivalent elastic interface characterized by the stiffness coefficient of an inertia-free shear spring eliminates the need for the treatment of the interphase region as a third layer sandwiched between the matrix and the fiber. As a result, the properties such as the density, modulus and the thickness of the interphase zone need not be known for the analysis. However, although an explicit usage of a third layer has been alleviated, the shear stiffness coefficient does indeed incorporate the properties in an indirect form as shown by the following analysis wherein the implicit assumption is that the interphase layer is homogeneous. Such an assumption is valid because of the ultrasonic wavelength which is very large compared to the interphase thickness and also because the interest is to evaluate the load transfer between the matrix and the fiber without evaluating the load transfer among different materials/phases making up the interphase. However, since the interphase might generally have variable properties along its thickness (h), the shear modulus of the interphase zone (G) and the shear stiffness coefficient  $(N_S)$  are integrals over the thickness and represent statistical average values.

From eqn (3), the shear stiffness coefficient is given by:

$$N_{\rm S} = \sigma_{\rm T} / [u_{\rm T}] = (\sigma_{\rm T} / f) / ([u_{\rm T}] / f) = G / h \tag{4}$$

where, for small elastic shear differential displacements, shear strain is given by  $f = \delta u/h$ wherein  $\delta u$  is the jump in displacement  $[u_T]$ . As a result, from eqns (3) and (4), the shear stiffness coefficient of the interface, which is defined here as the shear stress transmitted across the interface/interphase per unit of differential elastic displacement, is a measure of the ratio of shear modulus and the local thickness of the interphase zone.

The preceding discussion and the related equation (4) assume that the interphase region is homogeneous and made from a single material. However, because of the use of long wavelengths of ultrasound, the technique is applicable to the interphase region which is likely to be either made up of several sublayers of different compositions or comprised of a medium with gradually varying properties as represented in Fig. 5a (which is a modification of Fig. 1a). In Fig. 5a, the absence of clear boundaries (interfaces) for the matrix-interphase and interphase-fiber combinations is represented. Such an interphase region is evident in both metal matrix composites and ceramic matrix composites. For example, in metal matrix composites with a titanium based alloy matrix and silicon carbide fiber with carbon coating, the formation of the interphase is dependent on the extent of the diffusion of carbon into the matrix and titanium into the fiber leading to a reaction zone with different layers of TiC, Ti<sub>3</sub>Si or Ti<sub>5</sub>Si<sub>3</sub> and other components, and is modulated by the alloying composition of the matrix and the processing parameters (Baumann et al., 1990; Rhodes, 1992). Similarly in Nicalon<sup>TM</sup> reinforced glass ceramic matrix composites, an interphase layer of graphitic carbon forms as a product of the fiber-matrix reaction (Cooper and Chyung, 1987). Degradation of these ceramic matrix composites under oxidizing conditions has also been demonstrated (Luh and Evans, 1987).

In Fig. 5b the elastic behavior of both the multilayered and graded interphase regions is pictorially represented. In the case of composites with a multilayered interphase region having well defined boundaries between the interphase layers, Fig. 5b shows the linearity of elastic deformation in each layer (because of its homogeneity). Also, the elastic linearity, and hence the shear properties, of each layer is different as shown by solid thin lines o  $0_4$  in F respec corresp Howey matrix extrem deform

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Fig. 5. (a) Schematic representation of the interphase region comprised of a medium with gradually varying properties. The figure shows the absence of clear boundaries (interfaces) between the matrix and the interphase region as well as between the interphase and the fiber. (b) Schematic representation of the interphase region made up of several sublayers of different compositions.

lines of different slopes. Further, the shear displacement at each interface  $(0_1, 0_2, 0_3)$  and  $0_4$  in Fig. 5b) is continuous as represented by an arrow of equal size on either side of the respective interface. When the interphase is a graded region as shown in Fig. 5a, the corresponding elastic shear deformation is represented in Fig. 5b by dotted curves. However, since the point of interest here is the overall load transfer behavior between the matrix and the fiber in the elastic regime of the interphase zone, and also because of the extremely small elastic deformations produced by ultrasound, the total elastic deformation of the interphase has been linearized for both multilayered and graded

interphase regions (as shown by the dashed lines in Fig. 5b). Thus, the analysis allows for elastic differential displacement between the matrix and the fiber which is represented in Fig. 5b by the two displacement arrows of unequal lengths at  $0_{1^-}$  and  $0_{4^+}$ . Hence, the entire region (Fig. 5b) between  $0_1$  and  $0_4$  is the equivalent elastic interface, "0", as shown in Fig. 1b.

### Solution of the wave equation

For the development of the theoretical model, consider a plane elastic wave propagating in the positive direction  $z_i$ , and obliquely incident at an angle  $\theta$  on a model composite immersed in water, made with a cylindrical isotropic and homogeneous fiber embedded in a ceramic matrix. 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 (shear-wave back reflection (SBR) technique (Matikas and Karpur, 1992, 1993a)), without the loss of the generality, the cylindrical fiber can be replaced with an infinitely extended isotropic and homogeneous layer of thickness equal to the diameter of the fiber (shown as dotted lines in Fig. 5a). Moreover, since the matrix is relatively thin for this application, the effects of attenuation and diffraction are omitted.

The shear back-reflection coefficient from the fiber (SBRC) is calculated after solving the wave equation using the boundary conditions in eqns (1)-(3):

$$SBRC = T_M R_F T_W$$
(5)

where

$$T_{\rm M} = \frac{-2(c_{2\rm S}/c_{2\rm L})^2 \sin 2\theta_{2\rm L}}{(c_{2\rm S}/c_{2\rm L})^2 \sin 2\theta_{2\rm L} \sin 2\theta_{2\rm S} + \cos^2 2\theta_{2\rm S} + (\rho_1 c_1/\rho_2 c_{2\rm L})(\cos \theta_{2\rm L}/\cos \theta)}$$
(6)

is the transmission coefficient of mode converted shear waves from water to the matrix material,

$$T_{\rm W} = \frac{2\rho_1 c_1 \cos \theta_{2\rm L} \sin 2\theta_{2\rm S}}{((c_{2\rm S}/c_{2\rm L})^2 \sin 2\theta_{2\rm L} \sin 2\theta_{2\rm S} + \cos^2 2\theta_{2\rm S} + (\rho_1 c_1/\rho_2 c_{2\rm L})(\cos \theta_{2\rm L}/\cos \theta))\rho_2 c_{2\rm L} \cos \theta}$$
(7)

is the transmission coefficient of mode converted shear waves from the ceramic matrix back to water, and,  $R_F$  is the reflection coefficient of the matrix-fiber interface given by the equation:

$$R_{\rm F} = \frac{N_{\rm S}(A_{\rm S}\Omega - B_{\rm S}) - iA_{\rm S}B_{\rm S}}{N_{\rm S}(A_{\rm S}\Omega + B_{\rm S}) - iA_{\rm S}B_{\rm S}}E_{\rm S}^{\rm P}$$
(8)

with

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$$\Omega = \frac{1 + \Phi E_{\rm S}}{1 - \Phi E_{\rm S}} \tag{9}$$

$$\Phi = \frac{N_{\rm S}(B_{\rm S} - A_{\rm S}) - iA_{\rm S}B_{\rm S}}{N_{\rm S}(B_{\rm S} + A_{\rm S}) - iA_{\rm S}B_{\rm S}}$$
(10)

$$A_{\rm S} = 2\pi\rho_2 c_{\rm 2S} f \tag{11}$$

$$B_{\rm S} = 2\pi\rho_3 c_{\rm 3S} f \tag{12}$$

$$E_{\rm s} = {\rm e}^{({\rm i}k_{3{\rm S}}\,2d')} \tag{13}$$

$$E_{\rm s}^{\rm P} = e^{(ik_{2\rm s}2d_{\rm s})} \tag{14}$$

$$d_{\rm S} = \frac{1}{2} \left( \frac{\bar{d}}{\cos 2\theta_{2\rm S}} - d' \right). \tag{15}$$

From the above equations ((8)-(15)), the shear back-reflection coefficient in eqn (5) is dependent on:

- —the properties of the matrix (density,  $\rho_2$ , longitudinal,  $c_{2L}$ , and shear,  $c_{2S}$ , velocities),
- —the properties of the fiber (density,  $\rho_3$ , longitudinal,  $c_{3L}$ , and shear,  $c_{3S}$ , velocities),
- —the diameter of the fiber (d'),
- —the angle of incidence  $(\theta)$ ,
- —the frequency (f) of interrogation,
- —the interfacial shear stiffness coefficient  $(N_s)$ ,
- $-\bar{d}$  the thickness of the sample.

Further, if all the necessary (as in eqns (8)-(15)) properties and dimensions of the matrix and the fiber are known, a curve as shown in Fig. 6 can be generated from eqns (5)-(15)wherein the back-reflection coefficient (theoretical) of the shear waves (y-axis in Fig. 6) is shown as a function of the shear stiffness coefficient (x-axis in Fig. 6) of the fiber-matrix interface. Therefore, using the experimental approach outlined in the next few paragraphs, the shear stiffness coefficient can be obtained from Fig. 6 after the experimental measurement of the back-reflection coefficient (y-axis).

# Experimental measurement of the interfacial shear stiffness coefficient

Ultrasonic quantification of the shear spring stiffness coefficient can be performed (Matikas and Karpur, 1992, 1993a) by the experimental measurement of the back-reflected ultrasonic shear waves from the fibers. The incident stress wave (Fig. 7) induces a displacement of the matrix at the interface which is partially transmitted to the fiber due to the elastic deformation of the shear spring. The degree of discontinuity of the displacements and the associated partial transfer of stresses across the interface is a function of the interfacial shear stiffness coefficient as discussed above. However, due to the conservation of energy requirements (continuity of stresses), the remaining part of the incident ultrasonic energy will be reflected back to the transducer. As a result, after experimentally measuring the incident and reflected ultrasonic energies, the part of the stress reflected from the fiber can be calculated as a percentage of the incident energy (back-reflection coefficient). This back-reflection coefficient is also a measure of the part of the stresses transmitted across the interface to the fiber because of the continuity of stresses at the interface. Therefore, the ultrasonic shear back-reflection coefficient can be







Fig. 7. Continuity of shear stresses and discontinuity of displacements at the interface.

used to calculate the shear stiffness coefficient of the interface. It should be noted that the experimentally determined shear stiffness coefficient will be an average over the ultrasonic beam diameter (which is related to the incident wavelength) at the interface along the circumference of the fibers.

The experimental sample block for measuring the shear wave back-reflection coefficient of the interface and the subsequent inverse calculation of the shear stiffness coefficient was fabricated with three targets: (1) since the calculation of the reflection coefficient requires the measurement of the wave amplitude incident on the interface, a sample was designed as shown in Fig. 8 wherein the machined angle is the same as the refracted shear wave angle and air was sealed in behind the angle surface as shown; (2) the sample also had a drilled hole with the same diameter as that of the fiber  $(142 \,\mu\text{m})$ . The drilled hole was sealed to keep out the coupling fluid (water) and was used as a target to simulate an unbonded interface; (3) an SCS-6 fiber was also embedded in the matrix material (either Ti-6Al-4V or Ti-24Al-11Nb).

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 the angled surface as shown. 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 (Matikas and Karpur, 1993a, b)). Similarly, A-scans were obtained from the hole and the fiber 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. The values are listed in Table 1. It should be noted that the shear stiffness coefficient for the hole was zero (Table 1) as expected (because the hole simulates a debond). Figure 9 shows the mapping of the reflection coefficient along the length of the fiber as well as the hole indicating that the technique is very sensitive to the local elastic behavior of the fiber-matrix interface.





### Characterization of fiber-matrix interphase/interface

Table	1.	Theoretically	calculated	shear	stiffness	coefficient	of	MMCs	based	on	experimentally
			mea	sured	back-refle	ection coeff	ïcie	ents			

	(GPa/µm)
0.246	0.0
0.082	9.4
0.096	7.3
	0.246 0.082 0.096



Fig. 9. Reflected amplitude of ultrasound along the lengths of the embedded fiber and a hole of the same diameter as the fiber drilled at the same depth as the fiber in the matrix. A constant value has been subtracted from the reflected amplitude from the hole for displaying on the same scale as the fiber. The interfacial property variations along the length of the fiber can be clearly seen from the figure.

 

 Table 2. Theoretically calculated shear stiffness coefficient of CMCs based on experimentally measured back-reflection coefficients

Material	Reflection coefficient	Shear stiffness coefficient (GPa/µm)				
7040/SIGMA	0.242	0.7				
Glass-E/SIGMA	0.231	0.6				
7040/SCS-6	0.318	0.7				
Glass-E/SCS-6	0.208	0.4				

Similar to the measurement of the interfacial shear stiffness coefficient, the interface properties were quantified for a few different glass matrix composite systems. The results are shown in Table 2 wherein Glass-E and Glass-7040 indicate designation of two different types of glasses used for the matrix of the composites.

### SUMMARY AND CONCLUSIONS

Utilization of metal matrix and ceramic matrix composites for aerospace applications requires a good characterization and evaluation of nascent composite systems at the research and developmental stages as well as during eventual production and use. Many of these needs during the various stages of the composite life cycle can be satisfied by the characterization of the fiber-matrix interphase region because of its contribution toward the overall mechanical properties of the composite system. During the research and developmental stages of the composite, it is critical to evaluate the compatibility of different types of matrix materials with different types of fibers and the characteristics of the resultant interphase region so that the interphase formation can be controlled through the modulation of material processing conditions. Also, the effect of variability in interfacial elastic behavior because of the varying interphase properties and its

implications on the overall mechanical properties and material behavior of the composite for the intended application will have to be evaluated. On the other hand, after the composite and its fiber/matrix interface have been designed and developed, it is imperative to ensure that the designed properties are being achieved during production and retained during use. As a prelude to meet these goals, this paper introduces a novel mechanical parameter called the interfacial shear stiffness coefficient wherein the interphase is modeled by an "equivalent elastic interface" that represents the elastic behavior of the interphase zone. The fiber-matrix interface is characterized here by quantifying the "shear stiffness coefficient". A methodology and the necessary material behavior concepts are provided here for using an ultrasonic nondestructive technique to quantify the interfacial shear stiffness coefficient through the measurement of the shear wave back-reflectivity. Experimentally measured shear stiffness coefficients for various titanium matrix and glass matrix composites with different fibers are tabulated.

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#### REFERENCES

Baumann S. F., Brindley, P. K. and Smith, S. D. (1990). Reaction zone microstructure in a Ti<sub>3</sub>Al + Nb/SiC composite. *Metall. Trans. A* 21A, 1559-1569.

Blatt, D., Karpur, P., Stubbs, D. A. and Matikas, T. E. (1993). Observations of interfacial damage in the fiber bridged zone of a titanium matrix composite. Scripta Metall. Mater. 29, 851-856.

Chu, Y. C. and Rokhlin, S. I. (1992). Determination of macro- and micromechanical and interfacial elastic properties of composites from ultrasonic data. J. Acoust. Soc. Amer. 92(2), 920-931.

Chu, Y. C., Baaklini, G. Y. and Rokhlin, S. I. (1993a). Assessment of damage in ceramics and ceramic matrix composites using ultrasonic techniques, NASA.

Chu, Y. C., Rokhlin, S. I. and Baaklini, G. Y. (1993b). Ultrasonic assessment of interfacial oxidation damage in ceramic matrix composites. J. Engng Mater. Technol. 115(July), 237-243.

Clyne, T. W. and Watson, M. C. (1991). Interfacial mechanics in fibre-reinforced metals. Comp. Sci. Technol. 42, 25-55.

Cooper, R. F. and Chyung, K. (1987). Structure and chemistry of fibre-matrix interfaces in silicon carbide fibre-reinforced glass-ceramic composites: an electron microscopy study. J. Mater. Sci. 22, 3148-3160.

Evans, A. G., Zok, F. W. and Davis, J. (1991). The role of interfaces in fiber-reinforced brittle matrix composites. Comp. Sci. Technol. 42, 3-24.

Hashin, Z. (1990). Thermoelastic properties of fiber composites with imperfect interface. Mech. Mater. 8, 333-348.

Hefez, M. and Rokhlin, S. I. (1992). Thermal shock damage assessment in ceramics using ultrasonic waves. J. Amer. Ceram. Soc. 75(7), 1839-1845.

- Jasiuk, I., Chen, J. and Thorpe, M. F. (1992). Elastic moduli of composites with sliding inclusions. J. Mech. Phys. Solids 40(2), 373-391.
- Jones, J. P. and Whittier, J. S. (1967). Waves at a flexibly bonded interface. Trans. ASME: J. Appl. Mech. December, 905-909.

Jun, S. and Jasiuk, I. (1993). Elastic moduli of two-dimensional composites with sliding inclusions-a comparison of effective medium theories. Int. J. Solids Struct. 30(18), 2501-2523.

- Karpur, P., Matikas, T. E. and Krishnamurthy S. (1992). Matrix-fiber interface characterization in metal matrix composites using ultrasonic imaging of fiber fragmentation. American Society for Composites, pp. 420-429. Pennsylvania State University, University Park, PA.
- Karpur, P., Matikas, T. E., Blodgett, M. P., Blatt, D. and Jira, J. (1995). Nondestructive crack size and interfacial degradation evaluation in metal matrix composites using high frequency ultrasonic microscopy. ASTM Symposium on Special Applications and Advanced Techniques for Crack Size Determination, Vol. ASTM STP 1251 (Edited by J. J. Ruschau and J. K. Donald), pp. 130-146. ASTM, Philadelphia, PA.

Kerans, R. J. and Parthasarathy, T. A. (1991). Theoretical analysis of the fiber pullout and pushout tests. J. Amer. Ceram. Soc. 74(7), 1585-1596.

Krishnamurthy S., Matikas, T. E., Karpur, P. and Miracle, D. B. (1995). Ultrasonic evaluation of the processing of fiber-reinforced metal matrix composites. J. Comp. Sci. Technol., accepted for publication.

Luh, E. Y. and Evans, A. G. (1987). High-temperature mechanical properties of a ceramic matrix composite. J. Amer. Ceram. Soc. 70(7), 466-469.

Margetan, F. J., Thompson, R. B., Rose, J. H. and Gray, T. A. (1992). The interaction of ultrasound with imperfect interfaces: experimental studies of model structures. J. Nondestr. Eval. 11(3/4) 109-126.

Marshall, D. B. and Evans, A. G. (1985). Failure mechanisms in ceramic-fiber/ceramic-matrix composites. J. Amer. Ceram. Soc. 68(5), 225-231.

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- Matikas, T. E. and Karpur, P. (1992). Matrix-fiber interface characterization in metal matrix composites using ultrasonic shear-wave back-reflection coefficient technique. *Review of Progress in Quantitative Nondestructive Evaluation, Vol. 12B* (Edited by D. O. Thompson and D. E. Chimenti), pp. 1515-1522. Plenum Press, La Jolla, California.
- Matikas, T. E. and Karpur, P. (1993a). Ultrasonic reflectivity technique for the characterization of fiber-matrix interface in metal matrix composites. J. Appl. Phys. 74(1), 228-236.
- Matikas, T. E. and Karpur, P. (1993b). Micro-mechanics approach to characterize interfaces in metal and ceramic matrix composites. 20th Annual Review of Progress in Quantitative Nondestructive Evaluation, Vol. 13B (Edited by D. O. Thompson and D. E. Chimenti), pp. 1477-1484. Plenum Press, Brunswick, Maine.
- Matikas, T. E., Karpur, P. and Krishnamurthy S. (1992). Metal matrix microstructural characterization using reflectivity techniques in a model composite. ASNT Fall Conference, pp. 258-260. Chicago, IL.
- McCartney, L. N. (1987). Mechanics of matrix cracking in brittle-matrix fibre-reinforced composites. Proc. R. Soc. Lond. A409, 329-350.
- McCartney, L. N. (1990). New theoretical model of stress transfer between fibre and matrix in a uniaxial fibre-reinforced composite. *Proc. Roy. Soc. Lond.* A425.
- Metcalfe, A. G. (1974). Interfaces in Metal Matrix Composites, Vol. 1. Academic Press, New York.
- Pagano, N. J. (1991). In Local Mechanics Concepts for Composite Material Systems (Edited by J. N. Reddy and K. L. Reifsnider), pp. 1-26. IUTAM Symposium, Blacksburg, VA.
- Park, H. S., Zong, G. S., Brown, L. D., Rabenberg, L. and Marcus, H. L. (1989). In Metal Matrix Composites: Testing, Analysis, and Failure Modes (Edited by W. S. Johnson), pp. 270-279. American Society for Testing and Materials, Philadelphia, PA.
- Rhodes, C. G. (1992). Characterization of fiber/matrix interfaces by transmission electron microscopy in titanium aluminide/SiC composites. Mater. Res. Soc. Symp. Proc. 273, 17-29.
- Rokhlin, S. I. (1992). Recent advances in waves in layered media. J. Physique IV 2(April), C1-819-C1-825.
- Rokhlin, S. I. and Wang, W. (1989). Critical angle measurement of elastic constants in composite material. J. Acoust. Soc. Amer. 86(5), 1876-1882.
- Rokhlin, S. I. and Wang, W. (1992). Double through-transmission bulk wave method for ultrasonic phase velocity measurement and determination of elastic constants of composite materials. J. Acoust. Soc. Amer. 91(6), 3303-3312.
- Russ, S. M., Nicholas, T., Bates, M. and Mall, S. (1991). In Failure Mechanisms in High Temperature Composite Materials, Vol. AD, Vol. 22/AMD, Vol. 122, pp. 37-43. ASTM, Philadelphia, CA.
- Waterburry, M. C., Karpur, P., Matikas, T. E., Krishnamurthy S. and Miracle, D. B. (1994). In-situ observation of the single fiber fragmentation process in metal matrix composites by ultrasonic imaging. J. Comp. Sci. Technol. 52(2), 261-266.
- Wright, P. K., Nimmer, R., Smith, G., Sensmeier, M. and Brun, M. (1990). The influence of the interface on mechanical behavior of Ti-6Al-4V/SCS-6 composites. International Conference on Interfaces in Metal-Ceramics Composites, at the TMS Annual Meeting (Edited by R. Y. Lin, R. J. Arsenault, G. P. Martins and S. G. Fishman), pp. 559-581. TMS, Anaheim, CA.