INTERFACE CHARACTERIZATION AND PERFORMANCE FOR THE DEVELOPMENT OF FIBRE-REINFORCED STRUCTURAL COMPOSITES

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ABSTRACT

The present work deals with the characterization of interfacial properties for the development and life prediction of metal matrix and ceramic matrix composites. Advanced non-destructive methods are developed for this endeavour. An innovative micro-mechanics parameter is introduced for evaluating the fibre/matrix interfacial shear stiffness. The interface elastic property both in metal matrix and ceramic matrix composites is determined using the shear-wave back reflectivity (SBR) ultrasonic technique. By means of this approach, the effect of residual radial stresses on the interfacial shear elastic property is quantified. In addition, the interface shear strength is determined in model metal matrix composites by in-situ monitoring of fibre fragmentation using an original ultrasonic technique for imaging fibre breaks of a few micrometers in width. Finally, the interfacial stress at fracture is evaluated in-situ in metal matrix composites using ultrasonic nondestructive monitoring of interfacial deformation and failure under incremental transverse loading of the fibre embedded in the matrix.

Keywords: Metal Matrix Composites, Ceramic Matrix Composites, Consolidation, Interface Elastic Property, Fibre Fragmentation, Interface Transverse Property

1. INTRODUCTION

Introduction of metal matrix and ceramic matrix composites in advanced structural applications require good characterization and evaluation of nascent composite systems in research and developmental stages as well as during eventual production and use. During development of the composite it is critical to evaluate the compatibility of different types of matrix materials with different types of fibres, and the effect of material processing conditions on the formation of the fibre/matrix interphase region so that the composites behave according to the material design criteria [1]. Such a 'designed property' approach is critical to both the cost and the performance of these materials. However, for any designed property approach to be successful, it is imperative to have a method of interface characterization and control of the composite [2, 3]. Also, the suitability of the overall mechanical properties and material behaviour of the material for the intended application are evaluated.

The characterization of interface is of great interest to the developers of new composite systems. In order to determine the optimum interface properties, model monofilament composites are usually used, which have interfaces with tailored properties depending on the choice of fibre and matrix materials, the fibre coating and the processing conditions. Many experimental destructive techniques are employed to characterize the interface properties in model composites including, fibre push-out, fibre fragmentation, transverse loading tests, etc. This paper presents recent developments in novel nondestructive (NDE) techniques for (a) evaluation of micro-consolidation in composites, (b) characterization of elastic properties of the fibre/matrix interface, (c) monitoring fibre fracture during the fragmentation test, and (d) evaluation of interface failure during the transverse test.

2. EVALUATION OF MICRO-CONSOLIDATION IN COMPOSITES

An ultrasonic nondestructive evaluation methodology of consolidation and microstructure characterization of advanced composites has been developed to aid the design and fabrication of composites so that the processing parameters can be suitably adjusted to obtain complete densification around the fibres in both metal matrix composites (MMC) and ceramic matrix composites (CMC), as well as desired microstructure of metallic matrices. In addition, the

methodology can be used to ensure that the composite panels are devoid of any global problems such as fibre movement, ply delamination, and manufacturing anomalies such as voids. Post processing NDE is essential before interfacial characterization is performed.

Composites based on reactive matrices with high melting temperatures such as titanium alloys which are usually processed by solid state diffusion bonding of matrix foils, powders, or sprayed deposits with reinforcements. For example, the processing of continuously reinforced titanium matrix composites by the foil-fibre-foil method typically involves diffusion bonding of rolled matrix alloy foils with reinforcing fibres in the form of woven mats with a cross-weave to hold the fibres in place. The processing conditions are carefully selected in order to achieve complete consolidation and produce acceptable composite material. In practice, the initial processing temperatures are chosen on the basis of known flow characteristics of the matrix alloy at different temperatures and strain rates, and the consolidation of these samples is checked by metallographic examination of polished sections. However, the use of metallography alone is generally inadequate since consolidation often occurs nonuniformly within the composite panels.

The model composites consisting of an SCS-6 SiC fibre in a Ti-14Al-21Nb (wt. %) alloy matrix were fabricated in the form of 2 mm thick panels by diffusion bonding two matrix alloy sheets with a single fibre between them using two different processing conditions: (1) Vacuum hot pressing at 925°C under 5.5 MPa pressure for 30 min followed by hot isostatic pressing (HIP'ing) at 1010°C under 100 MPa pressure for 2 hr (Panel A), and (2) Vacuum hot pressing at 982?C under a pressure of 9.2 MPa for 30 min (Panel B).

The samples made as described above were ultrasonically imaged using two different techniques: (1) Shear wave interrogation and (2) Longitudinal wave interrogation. In the shear wave technique, a 25 MHz focused ultrasonic transducer (6.3 mm diameter, 12.7 mm focal length) has been used in the pulse-echo mode for the imaging of the embedded fibres in composite samples [4]. The ultrasonic wave front was incident on the composite at an angle of 24° (which is between the first and the second critical angles of the matrix material - titanium alloy in this case). As a result, vertically polarized shear waves were incident on the interface between the fibre and the matrix. Back-reflected ultrasound was gated for imaging. Since the wave front was incident at an angle, the received signal was either low amplitude due to back-scattering from the material texture or very strong amplitude due to the back-reflection from the cylindrical fibre (when the wave front was perpendicular to the fibre circumference). As a result, the dynamic range of the image of the fibre was excellent (> 17 dB).

The ultrasonic image of fibres with different degree of consolidation depends on the angle of incidence of the beam and the shape of the cross-section of the poor consolidation area. The reflected amplitude is maximum at the centre of the fibre (which is a cylindrical reflector) and is gradually reduced as the transducer is scanning away from the centre of the fibre. In the case of shear wave interrogation of a fibre with poor consolidation, the embedded reflector in the matrix is non cylindrical in shape, therefore, the wave will be reflected away from the receiver and only a small part of the energy (which is incident

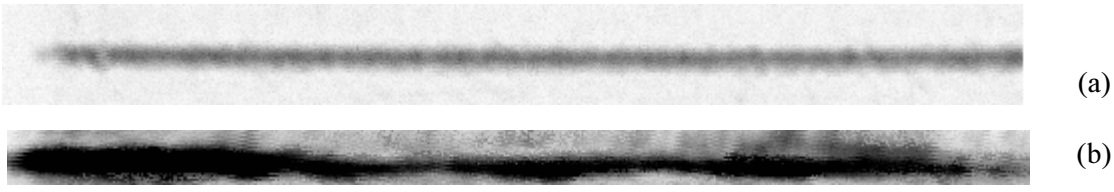

Fig.1: (a) SBR image of a well consolidated fibre/matrix interface; (b) SBR image showing local variations in consolidation quality as indicated by the variations in reflected ultrasonic amplitude and the apparent diameter of the fibre.

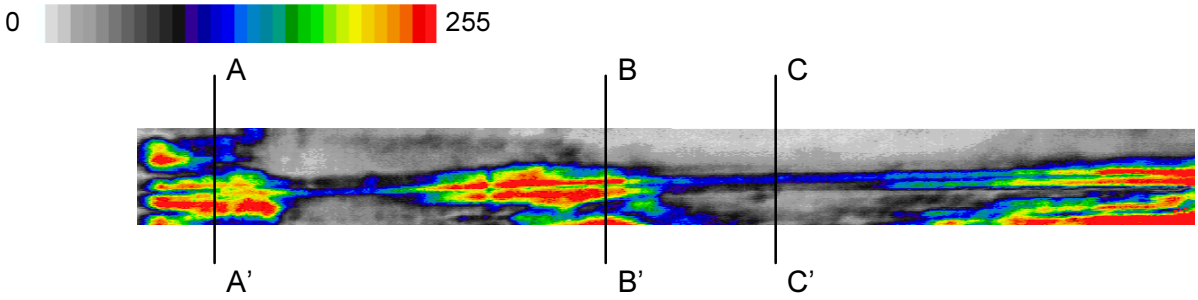

Fig. 2: Ultrasonic image from Ti-14Al-21Nb/SiC single fibre composite (Panel B) using longitudinal wave interrogation. AA', BB', and CC' indicate sections at which metallographic samples were examined and the ultrasonic images were corroborated with metallography (Fig. 3).

on the region close to the top of the fibre) will come back to the transducer thereby distorting the ultrasonic image of the fibre. Consequently, the image of the fibre will appear with a smaller diameter compared to a well consolidated fibre, and also the reflected ultrasonic amplitude will be lower The SBR technique has been very useful in detecting such consolidation micro-defects [5] as shown in Fig. 1.

In case of normal incidence longitudinal wave interrogation, and when a well consolidated fibre is imaged, very small amount of signal will be reflected back to the transducer and the fibre will be barely visible. However, in a situation similar to the configuration presented in Fig. 1 the ultrasonic amplitude reflected back to the transducer will be higher and the image of the fibre will be as shown in Fig. 2. The corresponding metallographic sections are shown in Fig. 3.

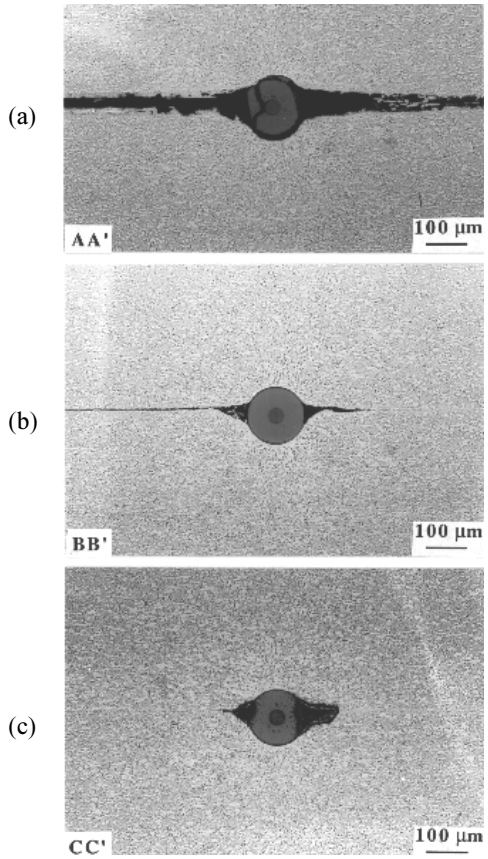

Fig. 3: Optical micrographs of vacuum hot pressed Ti-14Al-21NB/SiC (Panel B) sample showing poor consolidation at sections (a) AA' (b) BB', and (c) CC'.

3. FIBRE/MATRIX INTERFACE ELASTIC PROPERTY CHARACTERIZATION

A novel approach to nondestructively evaluate the elastic load transfer behaviour between the matrix and the fibre is provided here by defining a quantifiable parameter to describe the 'elastic behaviour' of the interface. An analytical model [6] has been developed to microscopically evaluate the elastic properties of the fibre/matrix interface. The model allows for selection of various experimental parameters such as ultrasonic frequency and angle of incidence, and provides the relationship which is necessary to interpret experimental results. The theoretical model considers the reflection of an ultrasonic wave front from a single fibre embedded in a homogeneous isotropic matrix. Although the analysis of the ultrasonic technique is developed for a monofilament composite, the method is equally applicable to study the fibre/matrix interface in the outermost layers of a real, multi-fibre composite system. The ultrasonic characterization of the interface is achieved by the analysis of the back-reflected shear wave signal from the fibre/matrix interface.

The interphase region in the model has been

condensed to represent an 'equivalent elastic interface' which, because of its elastic properties (shear modulus and local thickness) can elastically deform without fracture. The replacement of the interphase region by an equivalent elastic interface eliminates the need for the treatment of the interphase region as a third layer sandwiched between the matrix and the fibre. As a result, the properties such as the density, modulus and the thickness of the interphase zone need not be known for the analysis. A parameter, so-called the interface stiffness coefficient, N_S, which characterizes the interface elastic property has been introduced. The interface between the matrix and the fibre is modelled 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 springs with an equivalent rigidity of N_S [GPa/μm]. From the model the back-reflection coefficient of shear waves is dependent on the properties of the matrix, the properties of the fibre, the diameter of the fibre, the angle of incidence, the ultrasonic frequency of interrogation, and the interfacial stiffness coefficient N_S.

The SBR technique has been used to measure the interface shear stiffness coefficient, which is defined as the shear modulus over the thickness of the interphase region. The experimental procedure for the determination of the shear stiffness coefficient of the fibre/matrix interface consists of (a) measuring the shear wave back reflection coefficient of the interface and, (b) inverting the calculation of the shear stiffness coefficient by using the relationship between N_S and back-reflection coefficient established by using the theoretical model for an appropriate ultrasonic frequency of interrogation. The dependence of N_S on different physical parameters is given in Eq. (1), more details can be found in reference [6]:

$$N_s = f\left(\rho_m, E_m, G_m, \rho_f, E_f, G_f, d_f, \theta, f, D, R\right) \qquad (1)$$

where, ρ_m, E_m, G_m are the density, Young's modulus and shear modulus of matrix material, respectively, ρ_f, E_f, G_f are the density, Young's modulus and shear modulus of the fibre, respectively, d_f is the fibre diameter, θ the angle of incidence of ultrasound, f the ultrasonic frequency of interrogation, D the thickness of the sample, and R is the back-reflection coefficient.

For the measurement of the interface elastic property, a reference A-scan is obtained first. A Fourier transform of this reference A-scan provides the incident wave magnitude at a nominal frequency of 25 MHz. Similarly, A-scans were obtained from two samples, one containing a hole (simulated debond) and another with an embedded fibre. The corresponding reflected magnitudes at 25 MHz were measured using Fourier transform 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 theoretical curve. The calculated values of shear stiffness coefficient based on experimentally measured back-reflection coefficient are listed in Table 1. It should be noted that the shear stiffness coefficient for the hole was found to be zero (Table 1). This value was expected since the hole simulates debond in the matrix material. The fibre/matrix interface and the hole in the matrix were imaged (Fig. 4) to demonstrate the sensitivity of the reflected ultrasonic amplitude to the variability of the interface properties along the fibre axis.

Table 1: Calculated shear stiffness coefficient based on experimentally measured back-reflection coefficient.

Material	Reflection Coefficient	Shear Stiffness Coefficient / GPa/μm
Hole	0.246	0.0
Ti-6Al-4V	0.082	9.4
Ti-24Al-11Nb	0.096	7.3

The above described methodology was also applied to evaluate the interface elastic properties in glass matrix composites. Glass matrix composites are promising materials that could be used in aerospace structural components. Such components can be hot parts of aircraft engines that are subjected to low stresses. By tailoring the interface properties, the

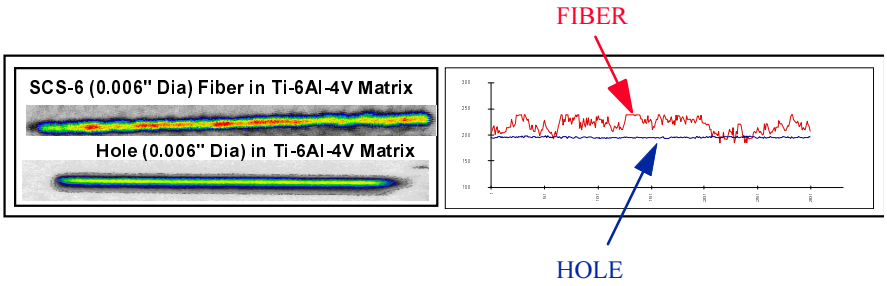

Fig. 4: (a) Ultrasonic back-reflected amplitude image of an SCS-6 fibre embedded in Ti-6Al-4V matrix showing varying interfacial property compared to the image from a hole of the same diameter as that of the fibre. (b) Line plots of the reflected amplitudes along the lengths of the fibre and the hole, respectively, showing the variability of the reflected amplitude from the fibre/matrix interface.

strength of glass matrix composites can be increased thereby making these materials more attractive for advanced applications. Interfacial bond and residual stresses are the two parameters that characterize the interface [7]. Techniques such as the fibre push-out test that measures the force necessary to slip a fibre along its length are widely used to assess the interfacial bond strength. However, fibre push results tend to exhibit a wide data scatter for a given material system.

The residual stresses σ_N at the fibre/matrix interface are given by:

$$\sigma_N = \gamma(\Delta\alpha)(\Delta T) \qquad (2)$$

where, $\Delta\alpha$ is the mismatch in the coefficient of thermal expansion (CTE), ΔT is the temperature variation, and:

$$\gamma = \frac{E_m E_f}{E_f(1+\nu_m) + E_m(1-\nu_f)} \qquad (3)$$

where E_m and E_f are the Young's moduli of matrix and fibre, respectively, and ν_m and ν_f the Poisson's ratios of matrix and fibre, respectively.

The effect of interfacial residual stresses on the amplitude of the back-reflected ultrasonic shear waves from the interface is shown in the results obtained from different types of glass matrix composites wherein six types of borosilicate glasses with virtually identical elastic properties (Table 2) but with different coefficients of thermal expansion were used, thereby producing varying degrees of radial residual stress. Measured properties from glass matrix composites with different matrices and with the same SCS-6 fibres are shown in Table 3. The residual stresses at the interface range from tensile to compressive.

The elastic mechanical properties shown in Tables 2 and 3 were measured using SBR technique. The theoretical curves for a selected frequency of 25 MHz shown in Fig. 5 demonstrate that when the residual stresses are ignored, the ultrasonic back reflected amplitude is very similar for all the types of glass because all these types of glasses have are almost identical elastic properties. However, it was observed substantial differences in the amplitude of shear waves back reflected from the fibre/matrix interface.

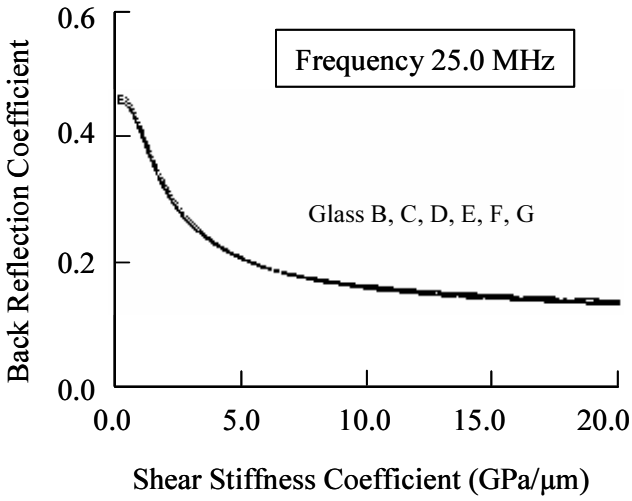

Fig. 5: Amplitude of the back-reflection coefficient vs. shear stiffness coefficient for different composite systems made with various types of glass matrices (B, C, D, E, F, G types) and SCS-6 fibres. The residual stresses are ignored in this calculation.

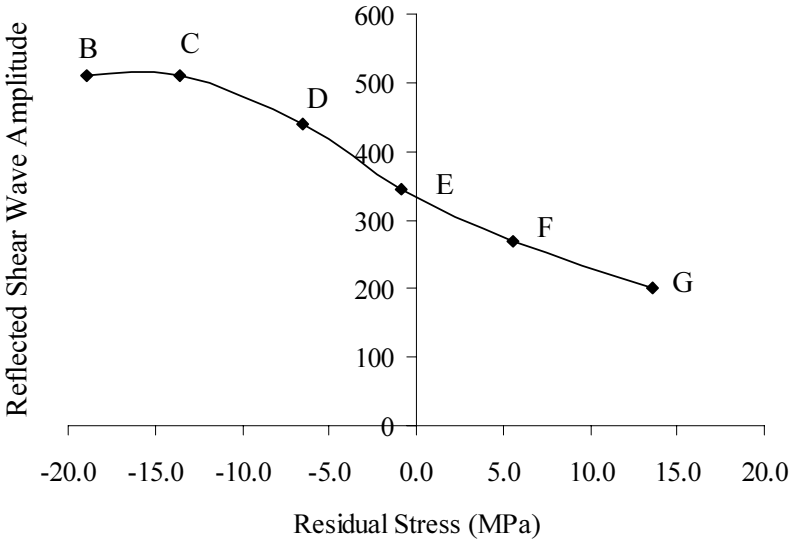

Fig. 6: Linearity of ultrasonic reflection as a function of radial residual stresses. The different letters indicate the respective borosilicate glass types.

Table 2: Elastic properties of various types of custom glasses with similar elastic properties but with different coefficients of thermal expansion due to small differences in chemical composition.

Glass Type	Density * (Kg/m^3)	Longitudinal Velocity (m/s)	Shear Velocity (m/s)	Young's Modulus ** (GPa)	Shear Modulus ** (GPa)	Bulk Modulus ** (GPa)	Poisson's Ratio **	Coefficient of Thermal Expansion (1E-6 / °C)
Glass B	2142	5316.7	3194.3	53.21	21.85	31.40	0.218	3.00
Glass C	2220	5367.2	3270.5	57.22	23.75	32.29	0.205	3.33
Glass D	2171	5391.2	3237.4	55.44	22.76	32.77	0.218	3.66
Glass E	2211	5411.8	3294.7	57.88	24.00	32.76	0.205	3.95
Glass F	2259	5406.8	3303.5	59.27	24.65	33.17	0.202	4.25
Glass G	2260	5479.3	3346.4	60.86	25.30	34.10	0.203	4.61

* Experimentally measured using Archimedes' principle
** Dynamic moduli obtained using ultrasonic velocities

Table 3: Elastic properties, coefficients of thermal expansion and residual stresses of some glass matrix composites with SCS-6 fibres.

Glass Type	Young's Modulus ** (GPa)	Poisson's Ratio **	Coefficient of Thermal Expansion (1E-6 / °C)	Residual Stress at the Interface (MPa)
Glass B	53.21	0.218	3.00	* -18.917
Glass C	57.22	0.205	3.33	* -13.610
Glass D	55.44	0.218	3.66	* -6.547
Glass E	57.88	0.205	3.95	** -0.833
Glass F	59.27	0.202	4.25	*** 5.548
Glass G	60.86	0.203	4.61	*** 13.541
SCS-6 Fiber	410.66	0.150	3.99	

* Tensile residual stress
** Almost zero residual stress
*** Compressive residual stress

The experimentally measured reflected shear wave amplitudes form the fibre/matrix interfaces are plotted in Fig. 6 as a function of the radial interfacial residual stress calculated using Eq. (3) for each type of matrix material. It can be observed from Fig. 6 that the ultrasonic reflected amplitude exhibits a linear dependency on the radial residual stresses as long as the integrity of the interface is intact. Therefore, the elastic shear stiffness coefficient of the fibre/matrix interface can be obtained after calculating the back-reflected shear wave coefficient taken into account the radial residual stresses at the interface. The plateau in the curve (two data points for glass B and glass C) implies ultrasonic signal saturation due to interfacial debond.

The stiffness coefficient of the fibre/matrix interface for SIGMA and SCS-6 fibres embedded in 7040 glass, as well as in glass-E, was determined using the SBR technique. The results are tabulated in Table 4 and were obtained using the theoretical curves from the model [6]. The interface quantification process indicates that the interface for 7040/SIGMA is relatively stiff compared to the other interfaces with the glass-E/SCS-6 interface being the most compliant.

Table 4: Calculated shear stiffness coefficient of CMC based on experimentally measured back-reflection coefficient.

Composite System	Shear Stiffness Coefficient (MPa / μm)
Glass-7040/SIGMA	500
Glass-E/SIGMA	300
Glass-7040/SCS-6	700
Glass-E/SCS-6	400

4. EVALUATION OF INTERFACIAL STRESS TRANSFER BEHAVIOUR DURING FIBRE FRAGMENTATION

During the single fibre fragmentation test, a composite sample made of a single fibre embedded in ductile matrix is subjected to tensile loading along the fibre axis. When the tensile stress, which is transferred from the matrix to the fibre by shear, exceeds the local fibre strength, the single fibre breaks successively into smaller fragments until the fragments become too short to enable further increase in stress level. The measurement of fibre fragment size is critical to the understanding of the load transfer behaviour of the fibre/matrix interface. However, the metallographic approach to measure the fibre fragment size might induce further damage and, therefore, a nondestructive method of imaging would improve the reliability of the measurement of the lengths of the fragments of the embedded fibre.

An ultrasonic technique was used to image fibre fragments in model composite specimens that underwent fibre fragmentation testing [8]. The samples were made of a SCS-6 SiC fibre embedded in Ti-6Al-4V or Ti-14Al-21Nb (wt.%) matrix alloys. Focused ultrasonic transducer was used in pulse-echo mode for imaging the embedded fibre in the sample before and during the fragmentation test. Fig. 7 shows the images of a SiC fibre in an untested composite along with the images of fragmented fibre in Ti-6Al-4V/SiC and Ti-14Al-21Nb/SiC composites. The technique consists in using the SBR setup with a defocused beam incident on the fibre/matrix interface so that the beam diameter, due to defocus, is incident such that it covers a portion of the fibre as well as the crack at the same time. However, since the acoustic impedance of the fibre is higher than that of the matrix while the impedance of the crack is less than that of the matrix, the reflections of the regions of the beam overlapping the fibre and the crack are out of phase with respect to one another. By changing the extent of defocus, which changes the relative areas of the fibre and the crack exposed to the incoming beam, the ultrasonic energy flux (defined as area times the intensity of ultrasound where intensity is the energy per unit area) reflected from the fibre remains constant with the defocus (because larger area reflects lower intensity). However, the energy flux from the crack diminishes with the defocus, because, the area of the crack exposed to the incoming ultrasound remains unaltered while the intensity of ultrasound diminishes. Therefore, by suitable defocusing, it is possible to balance the positive and negative energy fluxes from the fibre and the crack regions thereby cancelling the received ultrasonic amplitude [9]. Hence, the appearance of the crack can be substantially enhanced resulting in a phase sensitive 'microscopy' phenomenon.

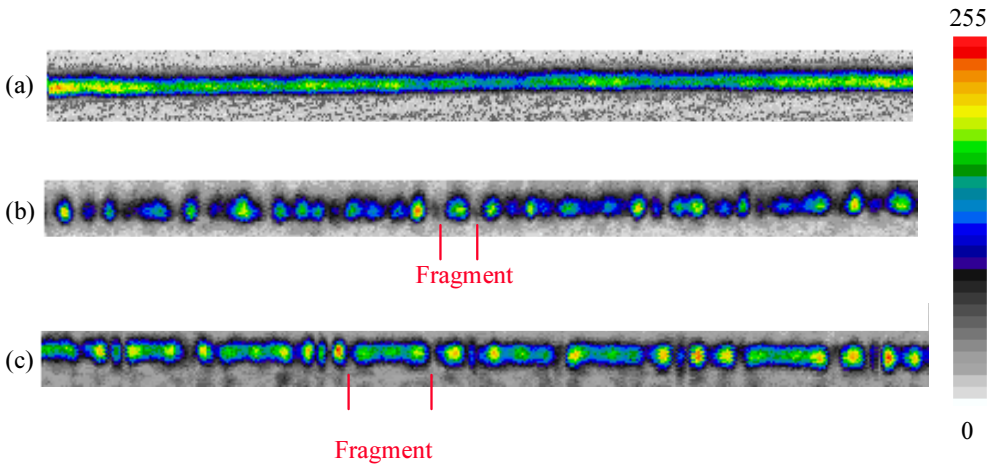

Fig. 7: (a) Ultrasonic image of an untested single fibre sample. (b) Fibre fragmentation image in a Ti-6Al-4V sample with an SCS-6 Fibre. (c) Fibre fragmentation image in a Ti-14Al-21Nb sample with an SCS-6 Fibre.

Fig. 8 shows experimental evidence of this phenomenon. Fig. 8a presents the SBR image obtained when the ultrasonic beam is focused on a fragmented SIGMA SiC fibre embedded in Ti-14Al-21Nb matrix. In this case, the fibre fracture is not clearly defined. However, when the ultrasonic beam is defocused the fibre fragmentation becomes clearly visible (Fig. 8b). If the defocus is further pronounced, the destructive interference between reflected signals from the fibre region and the crack region progressively disappears and a low-sensitivity image similar to Fig. 8a is again obtained.

5. EVALUATION OF TRANSVERSE INTERFACIAL PROPERTIES

The improvement of transverse properties of titanium matrix composites reinforced with continuous silicon carbide fibres requires a basic understanding of the dependence of the fibre/matrix interface deformation and debonding on the residual stresses, on the fibre/matrix bond strength, and on the matrix properties, under transverse loading conditions of the composite [10]. The interfacial stress at fracture was evaluated in-situ using ultrasonic SBR monitoring of interfacial deformation and failure under transverse loading (Fig. 9) which was applied in incremental steps [11]. At each step of loading the fibre/matrix interface was ultrasonically imaged (while holding the sample under that load).

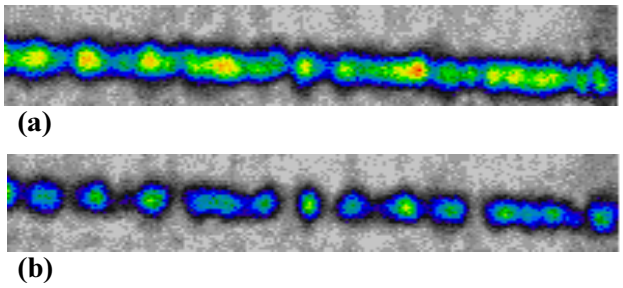

Fig. 8:(a) Ultrasonic image of a SIGMA SiC fibre embedded in Ti-14Al-21Nb matrix; 25 MHz ultrasonic beam is focused on the fibre. (b) Image of the same sample with 1 mm transducer defocus.

Fig. 10 shows the stress-strain diagram for the tensile test of a Ti-6Al-4V/SCS-6 single fibre composite sample under transverse loading, and the corresponding ultrasonic shear wave images at various stress levels of the sample labelled 'A through 'K' in the figure. The image labelled 'A' in Fig. 10 corresponds to the fibre/matrix interface before the commencement of the mechanical loading of the sample. Reflectivity image 'B' indicates the first few points of interface that fracture at about 350 MPa (shown with the maximum amplitude calibrated to red in the colour scale but visible as black in Fig. 10 due to black and white reproduction of colour). These points of interface fracture are located near the two ends of the fibre and in several places at the centre of the fibre. The reflectivity images 'C' through 'J' shows the progression of interfacial damage as the load increases. The image 'K' shows that the entire interface has been fractured at about 700 MPa. An important conclusion drawn from using in-situ SBR imaging of the transverse test contradicts a commonly accepted assumption that the entire interface is likely to fracture almost instantaneously once a sufficiently

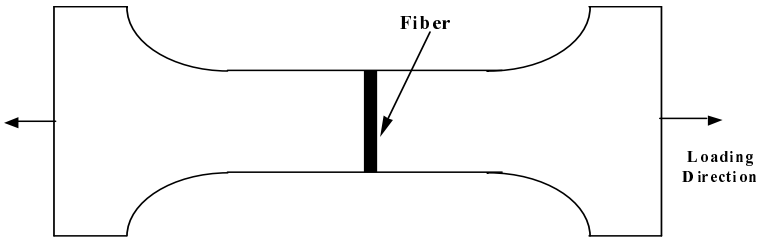

Fig. 9: Experimental configuration showing transverse orientation of the fibre in a sample and the direction of loading.

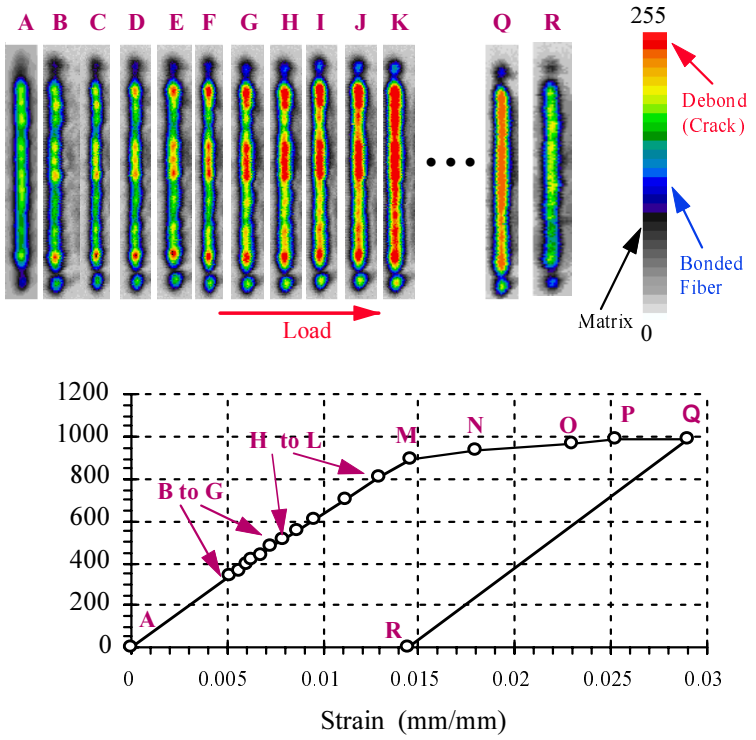

Fig. 10: In-situ ultrasonic SBR imaging of SCS-6 fibre embedded in Ti-6Al-4V matrix during various stages of transverse loading.

high stress level is reached because of the existence of a weak diffusion bond (as contrasted to mechanical bonding).

6. CONCLUSIONS

The development of innovative non-destructive methods enabled for the first time the characterization at the micro-scale of the fibre/matrix interface in fibre reinforced metal matrix and ceramic matrix composites. Using advanced ultrasonic techniques the micro-consolidation in structural composites has been evaluated nondestructively to aid the design and fabrication of composite systems so that the processing parameters can be suitably adjusted to obtain complete densification around the fibres. The interface elastic properties have been determined for a variety of MMCs and CMCs using the shear-wave back reflectivity method. A new micro-mechanics parameter has been introduced to evaluate the fibre/matrix interface shear stiffness and the effect of residual radial stress on the interfacial elastic property has been taken into account. Additionally, the interface stress transfer behaviour was evaluated in model MMCs by in-situ monitoring of fibre fragmentation using ultrasonic NDE. Finally, the interfacial failure process was characterized by in-situ NDE monitoring of interface deformation under incremental transverse loading of SiC fibres embedded in a metallic matrix. These studies greatly contributed to the fundamental understanding of interfacial elastic and fracture behaviour at the micro-scale.

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