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Push-out testing of SiC monofilaments with a TiC based functionally graded coating

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Metal matrix composites consisting of SiC monofilaments in Ti-based matrices show great promise for aerospace applications. This work is on a novel graded coating system for SiC monofilaments used to reinforce Ti matrices. SiC Sigma monofilaments were coated with a functionally graded (FG) TiC-based coating (SiC_f/C/(Ti,C)/Ti) using close field un-balanced magnetron sputtering. The coated fibres were incorporated into Ti matrices using hot isostatic pressing. The interfacial properties of the bulk composites were then evaluated using pushout testing of thin composite samples, in order to asses the effect of the graded coating. The tests were repeated on the same samples after heat treatment. The samples were subsequently analysed using scanning electron microscopy (SEM) equipped with both secondary and backscattered electron analysis modes to identify the region of failure. The test results indicate that the FG coating offers an improvement on composite fracture toughness over the un-coated fibres by approximately 70% before heat treatment, approximately 35% after heat treatment.

1. Introduction

Continuous fibre reinforced Ti-based alloys are suitable materials for replacement of monolithic components within the aerospace industry. This is because they posses high specific stiffness and high specific strength. Ti alloys reinforced with continuos Silicon Carbide (SiC) monofilaments are especially suitable for gas turbine compressor applications [1], where their use can result in a 75% weight saving over equivalent monolithic components. The main problem with the SiC/Ti system is the complex interaction between SiC and Ti, which manifests itself at the fibre matrix interface [2].

The fabrication of Ti-based composites usually requires consolidation at elevated temperatures (900 °C), and under these conditions formation of a reaction layer at the fibre/matrix interface is favourable. In the case of SiC/Ti systems, these reaction products (e.g., titanium carbides and titanium silicides) are usually brittle and degrade the composites mechanical properties. Coating the fibres prior to incorporation in the matrix is the favourable method of inhibiting reaction product growth while still allowing the fibres to stiffen the matrix. This involves the selection of an appropriate coating system that fulfils the requirements of the fibre/matrix interface.

Numerous coatings have been investigated such as TiB_2, TiC, and TiN [7–9], as well as other coatings such as Y_2O_3 and duplex Y/Y_2O_3 coatings [3–6]. A suitable coating that fulfils all the requirements has not been found. This has driven the development of functionally graded (FG) coated fibres, carbon fibres being one example [10]. FG materials possess a gradual change in composition and microstructure, and thus properties, across the material [12]. A graded titanium boride coating has been used to coat SiC fibres using physical vapour deposition [11]. The FG coating applied to the fibres in this work was a graded Ti/C coating {C/(Ti,C)/Ti} for SiC monofilaments which has been previously used to coat Sigma SiC fibres by Choy *et al.* [13]. The coating has been reported to be able to minimise the interfacial problems. These include formation of a brittle reaction layer, and residual stresses set up due to thermal expansion mismatch encountered in SiC/Ti. In addition the coating has been reported to conserve the strength of the as-received SiC fibre, and provide effective protection for SiC fibres in Ti-matrices at elevated temperatures (e.g. 900 °C).

However, there is a lack of understanding of the role of the fibre/matrix interface during failure of FG coated fibre reinforced composite structures. Interfacial shear strength can be used to characterise composite interfacial properties. A low interfacial strength allows easy debonding at the fibre/matrix interface, allowing sliding of the fibre within the matrix and favouring crack deflection along the interface. The opposite is the case for systems where the fibre is strongly bonded to the matrix, resulting in a high interfacial shear strength. The pushout test has been successfully used to measure the interfacial strength of SiC fibres in Ti-based matrices [15–19]. This work describes testing carried out on Ti

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matrices reinforced with SiC fibres, with the graded coating system described above, before and after heat treatment, and on composites containing as-received fibres as a comparison.

2. Experimental

The FG coating was first deposited on the fibres using a closed-field unbalanced magnetron sputtering method, which is described in detail elsewhere [13]. The composites were manufactured by first depositing a thick matrix coating on top of the FG coating and then hot isostatic pressing (HIPing) to consolidate the composites fully [14]. Push-out test pieces were then machined by first sectioning a thin slice ($\sim$250 μm) from each composite sample perpendicular to the fibre direction using a low speed diamond blade. Each sample was then polished to a 1 μm finish on both sides before mounting on a stub with two 200 μm wide grooves machined into it. The spacing between fibre centres was between 250 and 300 μm in an approximately hexagonal array, and there were 50 to 60 fibres per specimen. The sample descriptions are summarised in Table I. The fibres above each groove were indented with the aim of achieving approximately 10 pushouts per sample. The testing was carried out using a MicroMaterials microhardness tester. The apparatus allowed precision alignment ($\pm$3 μm) of the Berkovitch diamond used to indent samples by the use of a video camera.

As many fibres as possible were indented on each sample and the load extension data for each run collated. From each run it was possible to extract a critical load when the fibre/matrix interface debonded. From this the interfacial shear strength was calculated [20] using Equation 1.

$$\sigma_{app} = \frac{2\tau L}{r} \quad (1)$$

where, σ_{app} is the applied stress, L the fibre length or sample thickness, r the fibre radius and τ is the interfacial shear strength.

In addition the samples were analysed in the scanning electron microscope (SEM) in both secondary and backscattered modes, to determine the region of failure.

3. Results and discussion

The aim of this work was to investigate the effect of the presence of a compositionally graded TiC coating on the strength of the fibre/matrix interface. Table II shows the test data from the four samples.

The samples were not of uniform thickness due to the manual grinding and polishing of the samples. However, Equation 1 takes the length of the fibre into account allowing samples of different thicknesses to be compared. Fig. 1 shows a typical load/displacement plot of a successful test. There are four regions on the graph, which are designated 1–4. In region 1 the load increases to a point when a critical load is reached (pushout load) at which point the interface fails and the fibre slides. The displacement in region occurs due

TABLE I Sample descriptions

Sample	Description
1	$SiC_f/C/(Ti,C)/Ti_{matrix}$
2	SiC_f/Ti_{matrix}
3	$SiC_f/C/(Ti,C)/Ti_{matrix}$, (heat treated for 100 h, at 800 °C, in Argon)
4	SiC_f/Ti_{matrix}, (heat treated for 100 h, at 800 °C, in Argon)

TABLE II Average test data for the four samples tested in pushout

Sample	Pushout load (N)	Stress (MPa)	Sample thickness (μm)	Shear strength (MPa)
1	5.86 ± 2.5	745 ± 318	224 ± 36	83 ± 35
2	15.67 ± 3.6	1994 ± 458	182 ± 13	274 ± 63
3	13.31 ± 4.7	1694 ± 598	208 ± 4	204 ± 72
4	15.76 ± 3.6	2006 ± 458	160 ± 12	313 ± 72

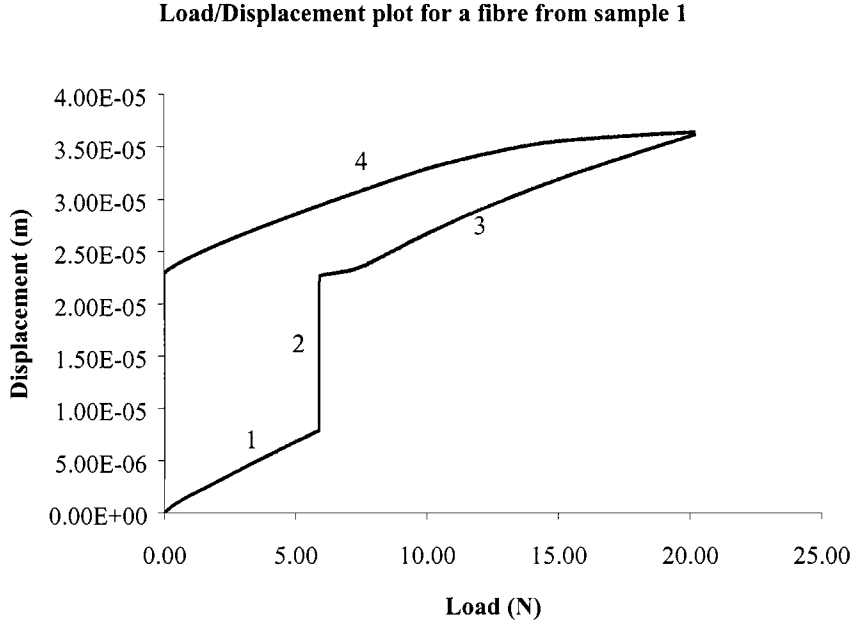

Figure 1 Load/displacement curve for a successful pushout test. The four regimes during the pushout test are designated 1–4.

to elastic deformation of the matrix with the interface remaining intact. Failure of the interface is manifested by a large increase in displacement with no increase in load, shown in region 2. In this case the fibre was observed to slide until the diamond impinged on the matrix. At this point the load increases again as the diamond indents the matrix, shown in region 3. Each test continued until either a maximum load or a maximum displacement of the diamond was reached. Region 4 shows the automated reduction in load to zero once the maximum load has been reached.

Although the above analysis (Equation 1) does not take into account the variation in shear stress along the fibre length during the test [20], it is sufficient for the analysis of experimental data from similar systems. It also neglects the presence of axial thermal residual stresses generated as a result of the difference in the coefficient of thermal expansion between SiC and Ti. Work has been done on the effect of thermal residual stresses on the failure of the fibre/matrix interface [15]. With no residual stresses present the application of a load to the fibre generates a stress profile such that interfacial shear stresses are greatest at the top surface of the sample where the load is applied. However, the strength of the interface at the top surface is increased by Poisson expansion of the fibre. At the back surface of the sample, due to bending of the sample at the edge of the support groove, radial tensile stresses are set up across the interface. This tends to favour opening of the interface and favours crack initiation at the back surface and propagation along the interface.

If just residual stresses are considered, tensile radial stresses result at the top surface and back surfaces. In addition, interfacial shear stresses are greatest at the top and back surfaces. When a load is applied to a thermally stressed system, the stresses generated by application of a load tend to counter the thermal residual stresses at the top surface. The shear stresses are opposite in direction and the Poisson expansion of the fibre in the radial direction counters the thermal residual radial tensile stress. However, at the back surface the opposite is the case. The thermal residual stresses are reinforced by those generated by application of a load to the fibre end at the top surface. Thus the above test geometry when applied to the SiC/Ti system favours initiation of failure of the fibre/matrix interface at the back surface, and crack propagation long the fibre/matrix interface towards the top surface.

It can be seen from the data in Table II that prior to heat treatment the application of the graded coating results in a drop in the measured shear strength from 274 ± 63 to 83 ± 35 MPa, a decrease of approximately 70%. Typical values for Sigma 1240 fibres prior to heat treatment have been reported as a σ_{app} of 1030 ± 36 MPa, and a τ of 127 ± 15 MPa [19, 20]. This shows that the measured values are of the correct

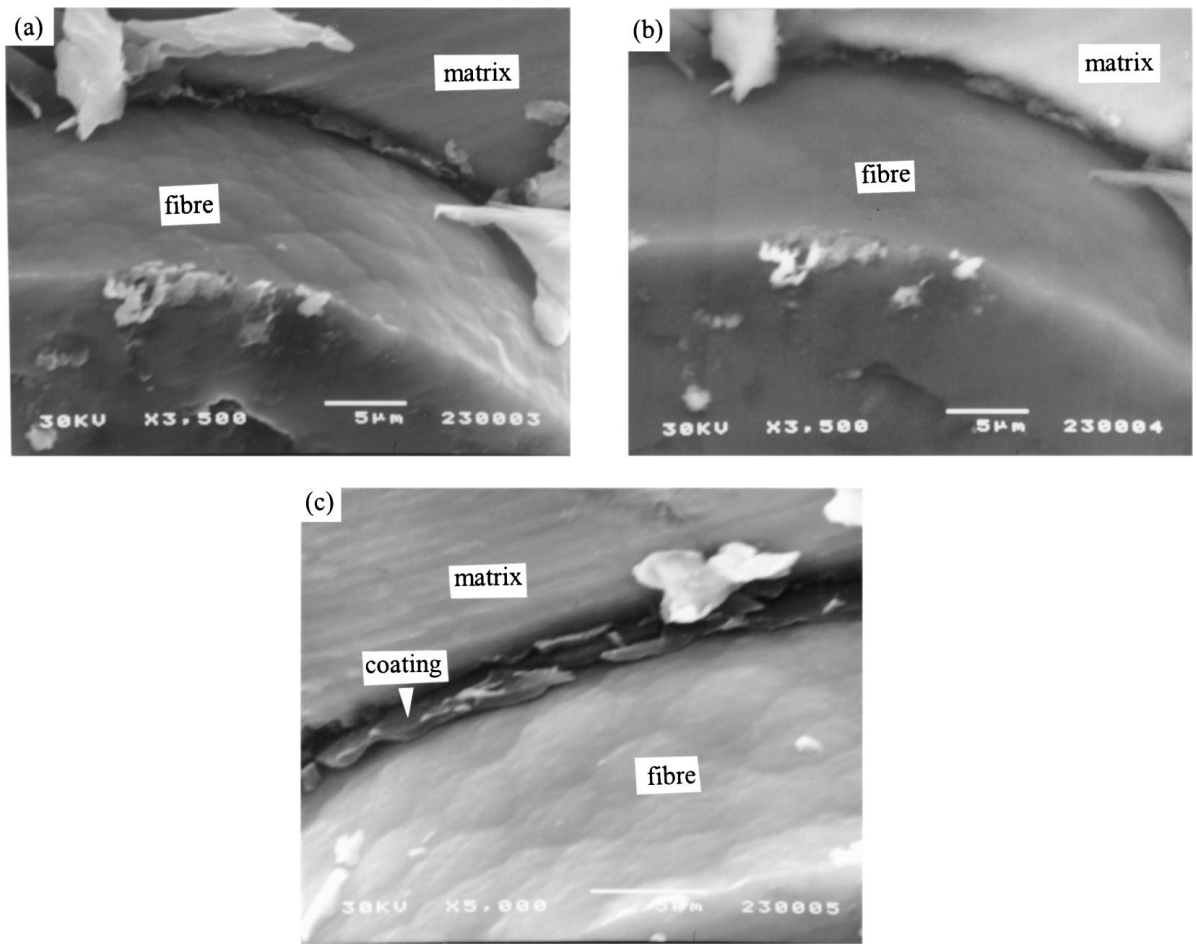

Figure 2 Micrographs from sample 1: (a) Secondary electron image of pushed-out fibre; (b) Backscattered electron image of same fibre; and (c) Secondary electron image at base of pushed-out fibre.

order of magnitude. A higher value for the 1040 fibres than those reported for 1240 fibres would be expected, as the 1040 fibres were un-coated, which was observed. Fig. 2 shows electron micrographs of pushed-out fibres from sample 1. It can be seen that the coating system has adhered better to the matrix than to the fibre from the secondary electron image, due to the lack of any observed coating on the pushed-out fibre. This is reinforced by backscattered electron imaging of the same fibre and closer examination at the base of the protruding fibre where some coating can be seen. This suggests that the carbon layer within the coating system is weakly bonded to the fibre. As expected, the as-received fibres were well bonded to the Ti resulting in a correspondingly high value for interfacial shear strength. The above observations are reinforced by axial tensile testing of the composite system, where mechanisms such as crack bridging and fibre pullout were observed during failure of sample 1 composites, but not in sample 2 composites [21].

After heat treatment the shear strength for the FG coated fibre interfaces increased to 204 ± 72 MPa (an

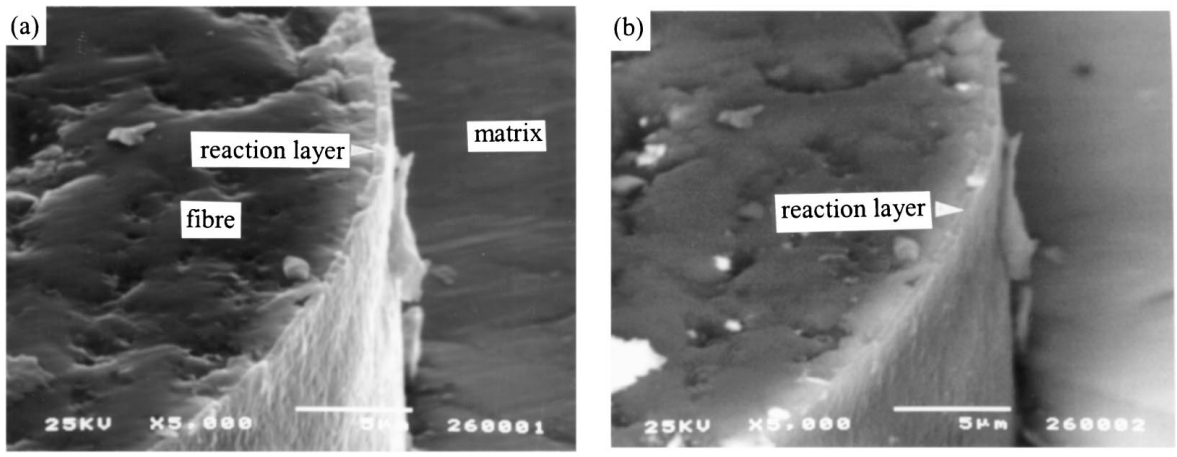

Figure 3 Micrographs from sample 3: (a) Secondary electron image of pushed out fibre; and (b) Backscattered electron image of the same fibre showing presence of a reaction layer on fibre surface.

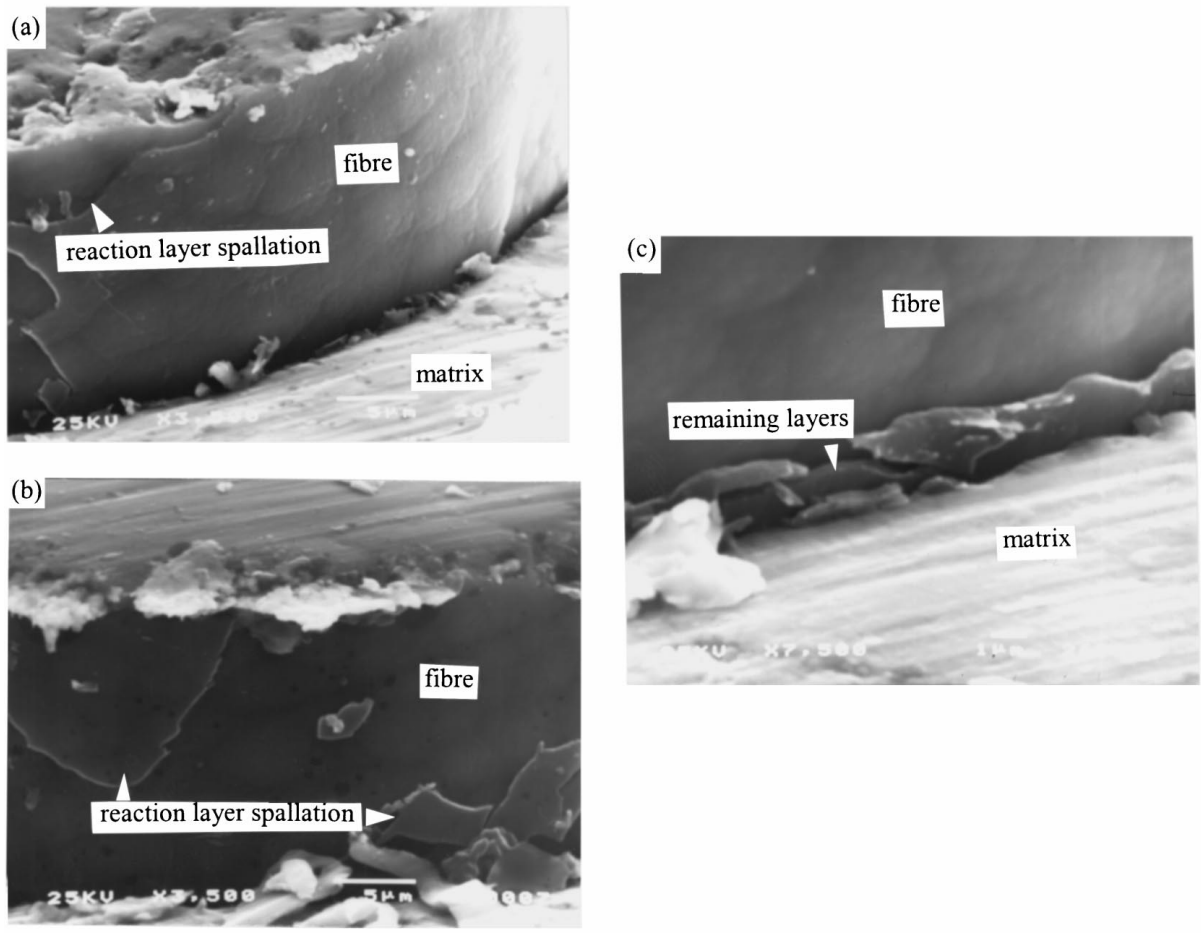

Figure 4 Micrographs from sample 3: (a) Secondary electron image of pushed-out fibre showing areas where reaction layer has spalled off and (b) Secondary electron image of pushed-out fibre showing areas where reaction layer has spalled off; (c) Secondary electron image showing some layers still adhering to the matrix.

increase of 146%), and that of the as received fibres also increased to 313 ± 72 MPa (an increase of 14%). The increase in both cases is clearly due to fibre/matrix reactions increasing the interfacial bond strength. It has been reported that for Sigma 1240 fibres heat treatment results in an increased resistance to sliding of the fibre [18]. For fibres with an aspect ratio of 0.3, and after heat treatment for 50 h at 815 °C, an increase in τ from 80 to 211 MPa was reported. Thus an increase in for this work was also expected. The interfacial thermal stability of the FG coating is not fully understood. A layer between 0.5 and 1 μm thick was observed on the fibres from sample 3 which adhered well to the fibres in some places and not in others. Under backscattered electron analysis the layer is clearly visible and appears lighter than the SiC, shown in Fig. 3. This suggests the presence of Ti in the reaction layer with the possible formation of either a titanium silicide or a ternary Ti/Si/C compound. The layer was observed to be brittle in nature due areas on the fibre surface where it had spalled off, shown in Fig. 4a and b. In addition other layers were once again observed at the base of the protruding fibre still adhering to the matrix, shown in Fig. 4c. It is not clear whether these layers are remnants of the coating or other reaction products. However, electron probe microanalysis (EPMA) on the above system within bulk composites has shown extensive interdiffusion of all three elements but mainly Si after heat-treatment, with the likely formation of a ternary compound adjacent to the fibre and a titanium silicide adjacent to the matrix [22]. The as received fibres also displayed a lighter layer around the protruding fibres under backscattered analysis indicating the possible presence of titanium. However in this case, the fibre/matrix reaction was observed to affect the interfacial shear strength relatively little resulting in an increase in shear strength of only 14%.

4. Conclusions

Prior to heat treatment the carbon layer within the graded system may be weakly bonded to the fibre allowing easy de-bonding compared to the as-received fibres.

After heat treatment, a 146% increase in interfacial shear strength was observed for FG coated fibres. This may be due to the formation of a brittle titanium silicide or a ternary compound at the SiC/C interface, leading to better bonding and thus an increase in interfacial shear strength. However, the value for shear strength after heat treatment for FG coated fibres is still lower than that for as-received fibres. Therefore the coating can be said to improve the fracture toughness of the composite before and after heat treatment.

For the FG coated fibres, a reaction layer was observed to have adhered to the fibre during pushout after heat treatment. Other layers were observed still adhering to the matrix, either a titanium silicide or a ternary Ti/Si/C compound.

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