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Interfaces with controlled toughness as mechanical fuses to isolate fibres from damage

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A source of inadequate performance of metal matrix composites has been the loss of strength due to the reaction layers between the fibre and the surrounding metal matrix. Here, we propose that the traditional diffusion barrier coatings on the fibre can be utilized to serve as mechanical fuses to isolate the impinging reaction zone cracks by interface delamination. Requirements on the interface strength and toughness for the specific tailoring of the fibre–coating interface are given. Special problems associated with the graphite–aluminium system are identified. A double cantilever beam experiment has been developed to measure the work of separation of thin coatings (0.1 to 0.3 μm) from bulk substrates. This test has been successfully applied to measure the work of fracture of the interface between a planar pyrolytic graphite substrate with the same chemistry and closely related microstructure as that of the 10 μm Pitch-55 graphite fibre and SiC coatings on them. A value of 60 J m^{-2} was obtained for the critical energy release rate for the PG–SiC interface. Additional measurements of energy release rates in thin layers of glue used to model the aluminium matrix and in PG itself, have given values which account for the high toughness of the main interfaces through the accompanying inelastic deformation work in the glue and the PG while the crack travels along the interface.

1. Introduction

Composite materials are tailored combinations of materials constructed to exploit the desirable properties of the component parts, while minimizing their weaknesses. The points of particular interest are the attainment of high stiffness and strength under both monotonic and cyclic loading, and other improvements in associated properties, such as desired levels of thermal expansion, damping capacity, resistance to environmental attack; all at minimum weight and acceptable cost.

Since composites combine a multiplicity of component material parts, they contain a high volume density of interfaces. The proper performance of composites in service puts extreme and often conflicting demands on these interfaces. In all of our considerations here, we will view composites as heterogeneous media having a relatively coarse microstructural scale, for which the elastic or plastic properties of the heterogeneities are governed by representative volume elements on a much finer scale. This implies that individual phases can be considered as continua, and that they interact with each other only through their interfaces. Thus, the consideration of choice is micromechanics, from which other macroproperties are assumed to be obtainable.

In normal service, interfaces are required to transmit full traction to assure that reinforcing phases such as fibres are fully load bearing to enhance the stiffness of the composite and to promote redundant deformations in a ductile matrix that can markedly increase the overall deformation resistance in it. The difference in the deformation resistance between fibres and matrix results in the development of stress concentrations across the interfaces. These concentrations can initiate cavitation and incipient fracture phenomena, which sooner or later become unstable and result in the premature termination of service by overall fracture. When such fracture is inevitable, it is desirable that as much dissipative work be associated with it as possible to result in tough and energy absorbing structures. Whether that toughness has the clear characteristics of being associated with the propagation of a single crack, and carrying conventional implications of fracture toughness, or is more diffuse and widespread over the entire structure is important in design, but of secondary consideration here. In all of these terminal phenomena, overall instability can be delayed by the systematic decoupling of the reinforcing elements that are on the verge of becoming critically stressed. To the extent that this can be successfully accomplished, subcritical damage can be spread over large volume elements, to result in a quasi-plastic response of the entire composite on the large scale. This desired systematic decoupling of the reinforcing fibres or other heterogeneities can be accomplished by controlling the terminal properties of interfaces, to have them act as mechanical fuses at appropriate predetermined levels of critical tractions. Such fine tuning or "tailoring" of interface properties is in principle possible, but requires a high degree of understanding of both the micromechanics of interfaces and the factors that affect their cohesive strength and overall fracture toughness.

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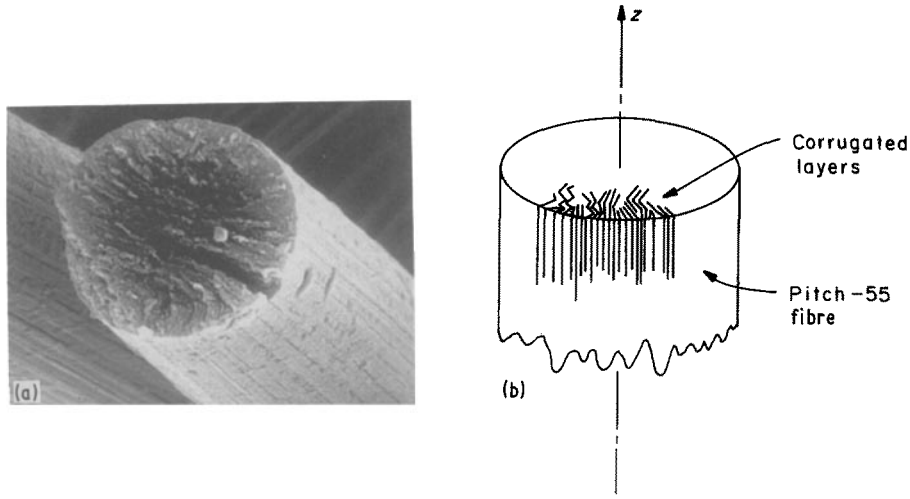

Figure 1 (a) Morphology of a 10 μm diameter Pitch-55 fibre revealed on a transverse fracture surface, (b) idealized fibre morphology as a set of corrugated parallel planes running parallel to the fibre axis. The fibre yield strength is expected to be low in longitudinal shear.

In metal matrix composites, where chemical reactions are also possible between the fibres and the metal matrix, forming reaction product layers along interfaces, the positive material misfit that often accompanies such reactions can initiate cracks in the fibres. To protect the fibres from such reaction damage, it is customary to provide them with non-reactive coatings. Since such coatings are introduced on the fibres under carefully controlled conditions, they can also offer the opportunity for tailoring the strength and toughness of their interfaces with the fibre, to achieve the mechanical fuse action discussed above to isolate damage. Such damage may have originated either among neighbouring fibres, and is transmitted across the matrix to the fibre, or it may have been generated by reaction products forming on the outer interface between the matrix and the protective coating.

We report here the results of a combined experimental and theoretical study on such key interfaces between the fibres and their protective coatings. It should be emphasized that the concept of using such interfaces as mechanical fuses is a very general one, and in principle, could be applied to any composite system. However, in this study, we focus on its application to metal matrix composites.

2. Interface mechanics

2.1. Metal matrix composite systems

For metal matrix composites, a large number of fibre and matrix systems have been explored. For structural applications at intermediate temperatures, the light metals of aluminium, titanium, and magnesium have been under primary consideration as matrices. While beryllium has most attractive properties in its own right, even without any reinforcement, it has not received much attention because of the difficulties associated with its processing. Of these, aluminium has been the matrix most widely considered. As a fcc metal, it exhibits exceptional ductility, and has a large number of well developed alloys with impressive properties. Of the various fibres used to reinforce aluminium, such as, boron, Al_2O_3, SiC, and carbon, the one that will be of primary interest to this investigation is the system of meso-phase pitch base fibres obtained from spun polymer precursors by a sequence of carbonization (1000 to 2000° C) or graphitization (2000 to 3000° C) treatments. Such pitch base fibres have axial fibre Young's moduli that range from 100 to 500 GPa with increasing molecular orientation, increasing long-period of crystalline domains, and increasing density (1.9 to 2.2 g cm^{-3}). In graphitized form, their tensile strengths are of the order of 2 GPa and relatively independent of their stiffness [1]. Fig. 1a gives the microstructure of a Pitch-55 (axial modulus 55 $\times$ 10^6 psi = 385 GPa) fibre, as viewed in a cross section across the axis. The fibres are typically of 10 μm in diameter, and are predominantly available in 500 to 10 000 filament yarns. The long axial grooves along the external cylindrical surface and the microstructure of the transverse section indicate that the principal morphology of the fibre is in the form of randomly corrugated and densely packed lamellae, oriented parallel to the fibre axis. This suggests an idealized fibre morphology, shown in Fig. 1b, with "planes of weakness" terminating roughly at right angles in the external surface. The consequence of this fibre morphology is a very low transverse modulus (14 GPa for the Pitch-55 fibre), and correspondingly low transverse tensile strength as well, estimated to be around 75 MPa on the basis of the usual correlation between modulus and strength. The particular morphology of the fibre also results in low shear stiffness and shear strength in longitudinal shear response to stresses σ_{zr} and $\sigma_{z\theta}$. Although this has no important consequence in the normal service of the composite; it can have important beneficial effects in the spreading of damage. Pitch-55 fibres have a density of 2.0 g cm^{-3}. The major advantage of Pitch-55 fibres is their chemical inertness due to high carbon content, high density, and high crystallinity. In the case of metal matrix composites for space structures, the attractions of this are the combination of properties which promote thermal dimensional stability, i.e., high Young's modulus, low and negative coefficient of thermal expansion, and high thermal conductivity.

The fibres are usually provided with a SiC coating of roughly 0.2 μm thickness, applied by a plasma-

assisted chemical vapour deposition process (PACVD) to isolate them from the matrix. The properties of such SiC coatings and means for their placement have been investigated by Landis *et al.*, and will be reported by them elsewhere [2]. Such coatings can be under substantial biaxial misfit stresses, dependent upon their structure and levels of entrapped hydrogen. These misfit stresses are compressive in the asdeposited form of the coatings, but can be relieved and even turned into tensile stresses upon thermal treatment. They are usually either maintained at zero or slightly compressive levels. The coatings are generally amorphous and have isotropic Young's moduli of about 300 GPa.

As discussed earlier, the overall mechanical properties of the composite are critically dependent on the properties of the key interfaces between the fibre and coating. The general mechanics, and in particular, the problems associated with Gr–Al systems involving the fibre-coating interactions are of a generic nature, and are potentially applicable to many other composite systems as well.

2.2. Fibre–matrix interaction

In composites, certain conflicting requirements must be satisfied by the interfaces between fibre and matrix. First, in a successful composite, it is important to decouple the fibre from the matrix during fracture, so as to prevent planar, low energy absorbing fractures. In fact, fibre fracture at random levels, followed by fibre pull out is desirable. This demands that the interfacial strength should not be too high. On the other hand, adequate interfacial strength is required to provide good transverse properties to the composite. Hence, in order to tailor the interfaces to desired properties, for them to act as mechanical fuses, it is necessary to bound the interface strength and toughness.

2.3. Transverse behaviour and lower bound to the interface strength

The interface strength is probed directly when the composite is stressed in the transverse direction. Hence, the interface strength should be high enough, so that transverse service stresses can be transferred to the fibre. The complexity of the stress field at the interfaces in a bundle of fibres has required that the transverse loading problem be approached chiefly with numerical techniques. The interfacial stresses developed under transverse tensile loading in composites of Pitch-55 fibres in pure aluminium matrices has been studied to a considerable extent by Zywicz [3]. Fig. 2, relating to his work, shows the geometry of a typical close-packed array of Pitch-55 fibres surrounded by an aluminium matrix. The relevant results for our consideration are given in Table I, where the stress concentrations in purely elastic and elastic–plastic behaviour of the aluminium matrix are summarized as a function of volume fraction of fibres for two different modes of tensile loading in the transverse direction, i.e., $\sigma_{xx\infty}$, in the close-packed direction and $\sigma_{yy\infty}$, mid-way between the close-packed directions.

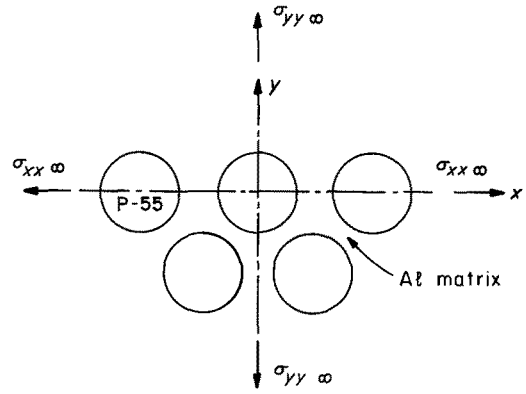

Figure 2 A hexagonal packing of aligned fibres in a metal matrix. The transverse plastic resistance differs in the y direction from that of the x direction.

The table shows that since the transverse modulus of the fibres is less than that of the aluminium, the fibres have actually a deficiency in load carrying capacity in purely elastic behaviour of fibres and matrix, resulting in a stress concentration in the matrix. This, however, is changed when the matrix can undergo plastic deformation, and can continue to load the fibres by plastic drag. Thus, for an equivalent plastic strain of even as small as 0.01, interface stress concentrations ranging from 1.14 to 2.40 appear on the cylindrical fibre surface, depending on fibre volume fraction and are governed by the equivalent plastic resistance of the matrix. Based upon what the equivalent plastic resistance of the matrix is, i.e., whether the matrix is pure aluminium or an aluminium alloy, the results in Table I provide a lower bound for the required interface strength, i.e.,

$$\sigma^*_{i(\text{lower bound})} > k\sigma_{T\infty}. \quad (1)$$

where k is the maximum stress concentration factor, and $\sigma_{T\infty}$ is the desired transverse strength of the composite.

2.4. Controlled delamination of coatings and upper bound to the interface strength

The nature of the interface cracking problem is of particular interest, as depicted in Fig. 3, which shows a schematic view of a crack in a SiC coating, terminating on the interface between the coating and the Pitch-55 fibre. The crack could alternatively have resulted from a matrix strain concentration produced by fractures in the surrounding fibres, a surface notch, or as shown in the figure, by a reaction product produced

TABLE I Transverse stress concentration for Pitch-55 fibres in an aluminium matrix*

Elastic stress concentration factors k				
V_f	0.4	0.6	0.8	
$\sigma_{rr_{max}}/\sigma_{xx\infty}$	0.44	0.56	0.74	
$\sigma_{rr_{max}}/\sigma_{yy\infty}$	0.60	0.82	1.04	
Elastic–plastic ($\bar{\varepsilon} = 0.01$) stress concentration factors $k^\dagger$				
$\sigma_{rr_{max}}/\bar{\sigma}$	1.14	1.55	2.40	($\sigma_{xx\infty}$)
$\sigma_{rr_{max}}/\bar{\sigma}$	1.14	1.14	1.33	($\sigma_{yy\infty}$)

*Zywicz [3].
†$\bar{\sigma}$ is the Mises equivalent stress.

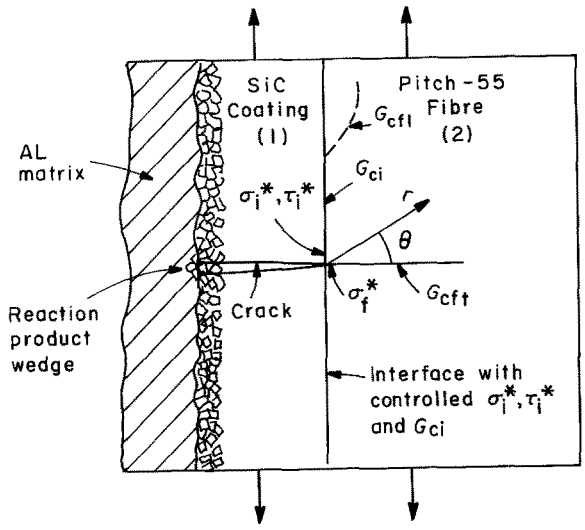

Figure 3 Sketch showing a potential form of mechanical probing of a fibre by a crack in the coating pried open by a misfit wedge produced by a coating–matrix reaction. To protect the fibre, controlled delamination at the interface is desired.

misfit wedge propping the flanks of the crack open. In all these cases, the principal concern is to protect the fibre from fracturing by penetration of the main crack into the fibre. This is to be achieved by decoupling the fibre at the interface between the coating and the fibre by producing either normal or shear failure at the interface using the concentrated interface stresses $\sigma_{\theta\theta}(\pi/2)$ or $\sigma_{r\theta}(\pi/2)$. The crack deflection process along the interface requires the following conditions to be satisfied by the stress field near the tip of the crack:

(a) The ratio of the fibre tensile strength σ_f^* to the interface cohesive strength σ_i^* should be greater than the ratio of the elastic crack tip stresses probing the plane across the fibre to the stress probing the interface to separate it in tension, i.e., the ratio of $\sigma_{\theta\theta}$ (at $\theta = 0$) to $\sigma_{\theta\theta}$ (at $\theta = \pi/2$). This will lead to the delamination of the interface in tension, provided that such tensile stress can be achieved. This condition leads to an upper bound for the interface cohesive strength.

(b) The ratio of the fibre tensile strength to the interface shear strength τ_i^* should be greater than the ratio of $\sigma_{\theta\theta}$ (at $\theta = 0$) to $\sigma_{r\theta}$ at the interface (i.e., at $\theta = \pi/2$). This will ensure the separation of the interface in shear, and bounds the interface shear strength. When the ratio of the interface cohesive strength to the interface shear strength is less than the ratio of $\sigma_{\theta\theta}/\sigma_{r\theta}$ at the interface, tensile separation will be preferred. Alternatively, for two possible directions of growth of the crack, i.e., across the fibre as opposed to along the interface, if the ratio of the energy release rate for growth across the fibre, to growth along the interface is less than the ratio of the work of fracture G_{cft} of transversely across the fibre to work of separation of the interface G_{ci}, then the fracture will follow the interface.

(c) The work of separation of the interface G_{ci} in any appropriate combination of separation across $\sigma_{\theta\theta}$ and $\sigma_{r\theta}$ along the interface should be less than the work of fracture G_{cfl} of the fibre in the longitudinal direction for the crack to continue to travel along the interface.

Thus, it is of interest to determine the stress $\sigma_{\theta\theta}$ and $\sigma_{r\theta}$ acting across the interface for a crack terminating at right angles on the interface, and establish the energy release rates that result when the crack branches into the interface to relieve these stresses. Furthermore, it is of interest to compare these energy release rates with the release rate for a crack going into the fibre.

Upon the initial successful diversion of the delamination crack along the interface, continued preferential delamination, as opposed to the crack entering the fibre, requires in addition, knowledge of the stress intensity associated with cracks lying on the interface, to satisfy the condition under (c) above.

Although knowledge of these stresses and energy release rates for cracks terminating on the interface or lying along the interface in these bi-material problems should be quite useful for purely elastic behaviour, it will be clear that additional considerations will be necessary to understand the interface behaviour, when plastic deformation in the fibre or in the matrix outside the coating accompanies the propagation of the delamination crack.

For isotropic bi-material media with a crack terminating at right angles on the interface, a very useful solution has been provided by Swenson and Rau [4] for the Mode I loading of such a crack. As these authors show, such cracks have a singularity which differs fundamentaly from that of cracks in homogeneous media, with singularity exponents either larger or smaller than 0.5, depending upon whether the crack is in the stiffer or the more compliant medium. The principal result of their analysis for the changes in stress intensification around the crack tip in a bi-material with semi-infinite extent is reproduced in Fig. 4 for plane strain, Mode I loading, as a function of the shear modulus ratio for a pair of materials having the same Poisson's ratios of 0.3. It is to be noted that, when the crack is in the stiffer medium, as would be the case of interest in this study with a crack in the SiC coating, the intensification of delaminating tensile stress $\sigma_{\theta\theta}(\pi/2)$ decreases, while the delaminating shear stress $\sigma_{r\theta}(\pi/2)$ increases with increasing shear modulus ratio μ_1/μ_2. In fact, when μ_1/μ_2 exceeds 10, the opening mode stress $\sigma_{\theta\theta}(\pi/2)$ across the interface drops to zero, while the magnitude of $\sigma_{r\theta}(\pi/2)$ has doubled. It is to be noted that in the case of interest here, the ratio between the modulus of the SiC and the axial modulus of the fibre is 1.17, and thus differs only marginally from the isotropic case. This implies that the ratio of the opening mode stress $\sigma_{\theta\theta}(\pi/2)$ across the interface to that in the fibre, i.e., $\sigma_{\theta\theta}(\pi/2)/\sigma_{\theta\theta}(0)$ is roughly 0.35 (as is the ratio $\sigma_{r\theta}(\pi/2)/\sigma_{\theta\theta}(0)$). We note, however, that the Pitch-55 fibre is intensely anisotropic and has a transverse modulus that is only 4% of the axial modulus. Thus, it can be expected that the fibre can readily flex in the transverse direction to release the transverse normal stress $\sigma_{\theta\theta}(\pi/2)$; perhaps completely. This indicates that decoupling the fibre from the coating may prove to be quite difficult, particularly if the interface has no means of responding plastically in shear. Clearly, a full solution of this bi-material problem with the anisotropy of the fibre fully taken into account is necessary.

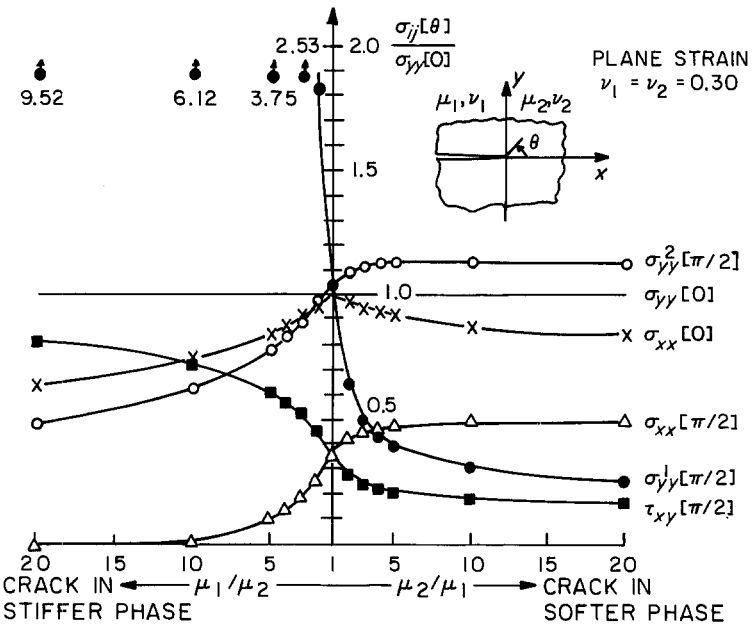

Figure 4 The effect of shear modulus ratios on crack tip stresses for a crack in one medium ending perpendicularly on the interface, under plane strain loading (from Swenson and Rau [4], courtesy of Pergamon Press).

The associated bi-material problem of a crack lying in the interface of two elastic media that would be required to follow the path of the delamination crack has been considered by a number of investigators [5, 6], and reliable solutions are now available. Incorporation of plastic response of one or both of the two materials is also presently being explored [7]. Solutions of this type, however, will be necessary not only for the problem of continued propagation of delamination cracks, but also in the final interpretation of experimental measures of interface toughness, as we describe below.

3. Control and measurement of toughness of interfaces

3.1. General strategy

To use the interface between the fibre and its coating as a reliable mechanical fuse to protect the fibre from damage, requires both control of interface strength and toughness in processing, as well as reliable methods of measuring such strength and toughness. The process of producing high quality coatings of SiC on Pitch-55 fibres and other substrates with the desired properties of interfaces by plasma-assisted chemical vapour deposition has been discussed by Landis *et al.* [2]. The actual tailoring of the properties of these interfaces between coating and substrate to place them between the required lower and upper bounds discussed above, is still in progress, and will be reported in the future. Here, we will discuss primarily the procedures developed for the measurement of interface properties. Of these, the interface cohesive strength and shear strength are difficult to measure, since they will be very sensitive to imperfections and are not likely to be of great value in governing the mode of the delamination of the interface to protect the fibre. For this latter purpose, the work of separation across the interface will be of greater value. From fundamental considerations, it is expected that the actual fracture work of an interface could be dependent on the mixture of the modes that are forcing it apart, i.e., the mixture of the applied Mode I and Mode II, to which the interface crack is subjected. However, since the nature of the singularity related to interface cracks separating two dissimilar materials with some plastic response, under any mode of loading is presently not available, we have concentrated attention on the development of reliable and reproducible methods of measurement of the fracture work of an interface by the most convenient means possible. In this quest, however, two limiting approaches have been distinguished. The interfaces which are of interest are expected to be relatively sharply defined along a steep material gradient between the coating and the fibre. The decohesion should then follow along a smooth surface, and have the appearance of a typical cleavage fracture. Thus, the actual intrinsic toughness G_{co}, i.e., the energy release rate necessary for the decohesion is expected to be in the range of only 3 to 5 $J m^{-2}$, which is typical for a cleavage-like fracture in hard inorganic solid. On the other hand, the energy release rate G_c for an actual interface delamination in a composite is likely to be very much larger, because of the presence of accompanying inelastic deformation in the fibre and in the matrix surrounding the fibres. In such fractures where the eventual separation process is of a cleavage type, the additional energy release rate G_p that is associated with the surrounding inelastic dissipations will be scaled by the intrinsic toughness of the interface G_{co} [8], i.e.

$$G_c = G_{co} + G_p = G_{co}(1 + K) \quad (2)$$

where

$$K = G_p/G_{co} \quad (3)$$

Therefore, in spite of the much larger dissipations associated with delaminations of interfaces in composites, the fundamental interface property to be controlled is the interface toughness G_{co}, which is expected to be related directly to the reversible work of separation of the interface by the well known relation

$$G_{co} = \chi_{SiC} + \chi_C - \chi_i \quad (4)$$

where χ_{SiC} and χ_C are the surface free energies of SiC and carbon and χ_i is the interface energy of SiC and carbon. Hence, in the control of the fuse-like action of the interface between coating and fibre, the reliable measurement of G_{co} and how it may be affected by the processing conditions is of vital importance. Nevertheless, of almost equal importance is the measurement of the interface toughness in the actual composite itself.

In what follows, we will discuss the methods developed to measure the overall toughness G_c of the interface in systems very similar to those of the actual composite. The special methods necessary to measure the intrinsic toughness of the interface are discussed in a separate communication [9].

3.2. Measurement of critical energy release rates G_c of interfaces in composites

Measurement of interface fracture work is not a new problem in thin film and coating technology. The literature is replete with practical techniques for the measurement of some average properties of thin film or coating interfaces, recently reviewed by Mittal [10]. Here, we shall develop some special considerations and procedures necessary for the accurate measurement of interface fracture work.

The preferred test for the determination of interfacial properties should measure the state of adhesion directly using simple fundamental procedures of mechanics relatively free of artifacts relying on complex models, based on unverifiable assumptions. For example, in the scratch test [11], the process of scratch formation is complex and cannot be readily explained in terms of pure models. Furthermore, there is no preferential failure at the film–substrate interface, and the size and shape of the stylus can influence the mode of failure in an undeterminable manner. In the periodic cracking technique utilized by Chow, Liu and Penwell [12] and by Davutoglu and Askay [13], the complex state of stress at the edge of the film in contact with the substrate is not taken into account. More detailed stress field modelling, such as that carried out by Yang and Freund [14] using sliding stress intensity factors, are necessary. The indentation technique of Chiang, Marshall, and Evans [15], requiring the presence of the interface in the vicinity of a plastic zone, is also based on a fairly complicated model, and does not measure the interface work of fracture directly. On the other hand, the double cantilever beam test provides a simple and direct method to measure the total adhesive fracture work, and has been used earlier by Gilman [16] quite effectively to measure the work of cleavage fracture of bulk materials. However, we have modified it to measure the work of fracture of thin film interfaces in systems structurally and chemically resembling those in the metal matrix composite itself.

4. Interfaces in model composite systems

4.1. The pyrolytic graphite SiC interface

An important prerequisite in the control of interface properties, such as tensile cohesive strength and the work of fracture, is that these properties should be measurable and the effectiveness of the crack deflection process should be demonstrable.

It would be best to perform experiments on actual interfaces in composites but is very difficult because of the very small diameter of the fibre (c.a. 10 μm). Therefore, it is desirable that the experiments be performed on similar planar interfaces on a larger scale. This requires finding material available in bulk, having the same chemical and morphological characteristics as that of the fibre, which can then be adopted for macroscopic testing procedures.

For this purpose, pyrolytic graphite was chosen as a first approximation to the Pitch-55 fibres. Pyrolytic graphite (PG) is obtained by vapour phase deposition of pure carbon in a temperature range of 1900 to 2500° C. It has a density of 2.2 g cm^{-3}, which is approaching the theoretical density of graphitic carbon, i.e., 2.28 g cm^{-3}. It grows in polycrystalline, nearly planar spherulitic forms in a layer-like manner, but with a great degree of anisotropy in the growth direction across which it is very compliant and weak. Its isotropic Young's modulus in the growth planes is 28.5 GPa, and in the growth direction across the layers is only 7 GPa. Fig. 5 shows the basis of modelling of the fibre surface with PG. The surface of PG parallel to the *c* axis resembles the surface of the Pitch-55 fibre. It has the same chemistry as Pitch-55, a somewhat higher density, and rather similar anisotropic morphology that resembles that of the Pitch-55 fibres, as viewed in the circumferential direction. However, the analogy is quantitatively quite different. The Pitch-55 fibres have a stiff direction modulus 13.5 times that of PG, but a compliant direction modulus that is only 2 times that of PG. Thus, the fibres are both far more

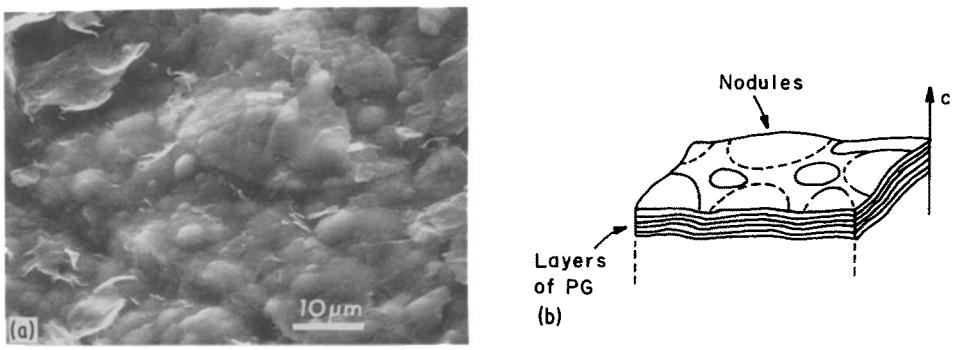

Figure 5 (a) Scanning electron micrograph of the morphology of a smooth layer plane perpendicular to the "*c*" axis in pyrolitic graphite showing nodules, (b) idealized rendering of the layer planes in PG.

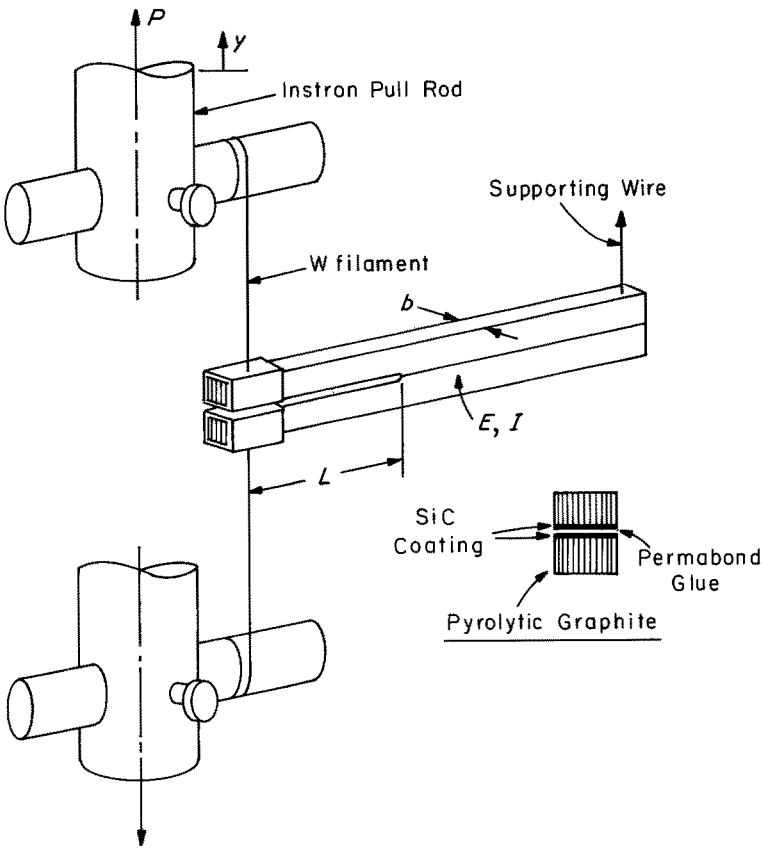

Figure 6 The double cantilever beam experiment for the measurement of interface toughness.

anisotropic and far stiffer than the PG. Nevertheless, experimentation of relative interface properties and general response of coating on carbon substrates are more readily investigated on PG than on individual Pitch-55 fibres, and permit simulation of coating-substrate interactions in bulk.

4.2. The double-cantilever beam experiment

This experiment was designed to measure the work of separation of a SiC coating of 0.1 to 0.5 μm thickness, deposited on a planar PG substrate. The specimen consists of strips of PG of 6.3 mm width, 3.2 mm thickness, and 150 mm length with the layer planes oriented perpendicular to the surface, as shown in Fig. 6. The surfaces of these strips with the given orientation of the planes were metalographically polished in a Syntron vibratory polisher. These polished surfaces were then coated with SiC of near stoichiometric composition, using a plasma-assisted chemical vapour deposition process. The coated strips were annealed at 600° C in a vacuum oven in order to relieve the residual stresses inside the films. Two of these coated strips were then bonded together using a very tough Permabond glue (ESP 109 and 110) over 75 mm of their length, so as to create an initial crack of 75 mm between the two strips. This laminate assembly was heated to 200° C to cure the glue. As will become clear below, the glue layer between the two strips has mechanical properties close to those of pure aluminium.

A schematic diagram of the test apparatus is shown in Fig. 6. The free ends of the graphite strips were connected to tightly fitting metal cages, which were then connected to the Instron machine load train, as shown in the figure. Load was transferred to the free ends of the cantilever strips by 200 μm thick tungsten wire. This wire was made taut prior to the testing, using a pin and a set screw arrangement, shown in the figure. The double cantilevers were pulled apart with a cross-head speed of 0.5 mm min^{-1}. A plot of load against load point displacement was obtained. By initial selection of components, stable testing conditions were obtained, requiring that the unloading stiffness of the specimen is substantially less than the stiffness of the testing machine.

Treating the arms of the crack as cantilever beams and using simple beam theory, the bending force F at the time of the crack propagation can be related to the fracture work of the interface G_c, through the Griffith criterion by means of the formula

$$G_c = F^2L^2/EIb \quad (5)$$

where L is the initial crack length, I the moment of inertia, b the width, and E the modulus of elasticity of the PG strips.

4.3. Double cantilever experimental results

Since the glue and the interface between the glue and the SiC coating is tougher than the interface between PG and SiC coating, the crack preferentially propagates along the latter interface. In some instances, the crack was found to initiate in the glue layer, but jumped to the less tough interface between SiC and PG. In such cases, the portion of the force–crack length curve pertaining to this type of propagation was not considered. Scanning electron microscopy, such as Fig. 7 of the fracture surface clearly shows the plane of the crack and the regions where the crack deviated from the interface, dipped inside the glue, and came back to the PG–SiC interface. Additional stereo-images of the fracture surface helped to establish better these

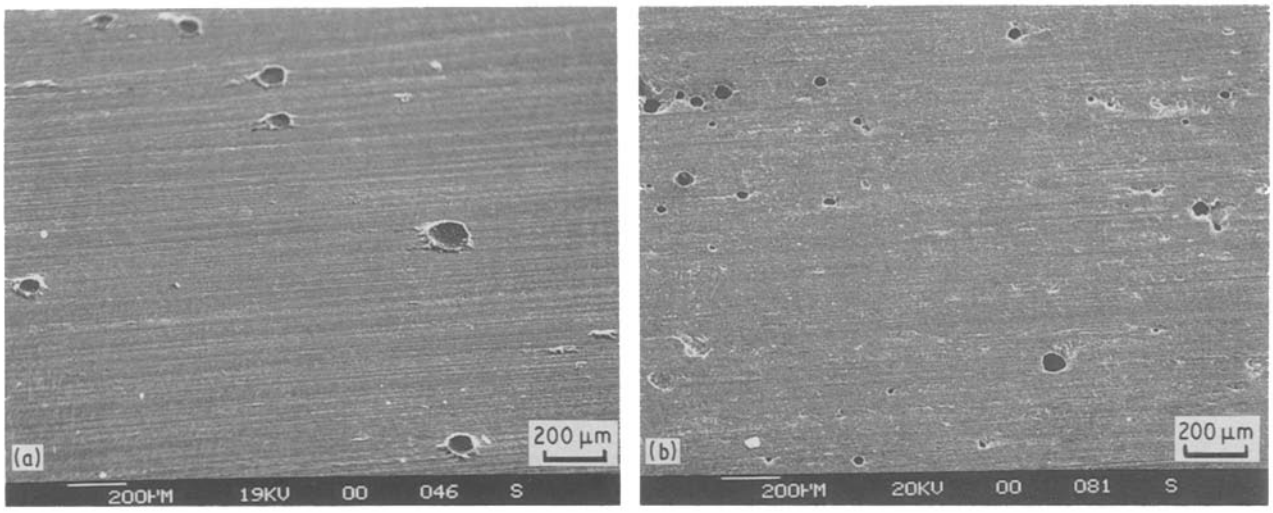

Figure 7 A SEM micrograph of the fracture surface between a SiC coating and a PG substrate having its surface parallel to the *c* axis of the PG. The crack propagation direction was parallel to the surface markings: (a) fracture surface viewed toward PG, (b) viewed toward SiC and glue layer.

occasional deviations of the crack path. Fig. 8 shows an example of such a stereo pair. These observations established that the area of the fracture surface through the glue is only a small fraction (about 0.1) of the total fractured area along the interface. To make corrections for such deviations, to obtain the actual energy relase rate along the PG/SiC interface, it is necessary to measure the fracture toughness of a comparably thin glue layer. This was accomplished by bonding two high strength steel strips together with the same glue of the same thickness, and prying the strips apart in a double-cantilever beam experiment. This gave a critical energy release rate $G_c = 244\,J\,m^{-2}$ for the glue layer, which was used in making the desired corrections.

Fig. 9 shows a typical load extension curve for a crack running along the planar interface between SiC and PG. Three drops in the curve indicate three stable jumps ahead of the crack. The average value of the fracture work G_c (the critical energy release rate) for the interface determined from such experiments, and incorporating corrections for deviations of the path through the glue gave a value of $60 \mp 6.85\,J\,m^{-2}$.

The quoted value of the critical energy release rate G_c along the interface obtained from the double cantilever beam experiment is quite high when compared to an expected level of 3 to $5\,J\,m^{-2}$, based on interface energy for hard inorganic solids. These high values must incorporate additional inelastic dissipation when the crack runs along the interface between SiC and PG. Thus, the crack also probes the PG and the glue on both sides, and forces them to undergo plastic relaxation. It is known that the tensile yield strength of the glue is 64 MPa, while the intra-laminar shear strength of the PG is 52 MPa, which translates into a Mises tensile yield strength of 90 MPa. As a crude first approximation we consider the interface cracking to occur in a conventional homogeneous material under small scale yielding conditions, thus we can estimate the inelastically affected layer depth h of material near the fracture surfaces from the well known linear elastic fracture mechanics relation of

$$h = \frac{1}{2\pi}\frac{G_c}{(1-\nu^2)Y\varepsilon_y}. \quad (6)$$

where $G_c = 60\,J\,m^{-2}$ is the measured work of fracture,

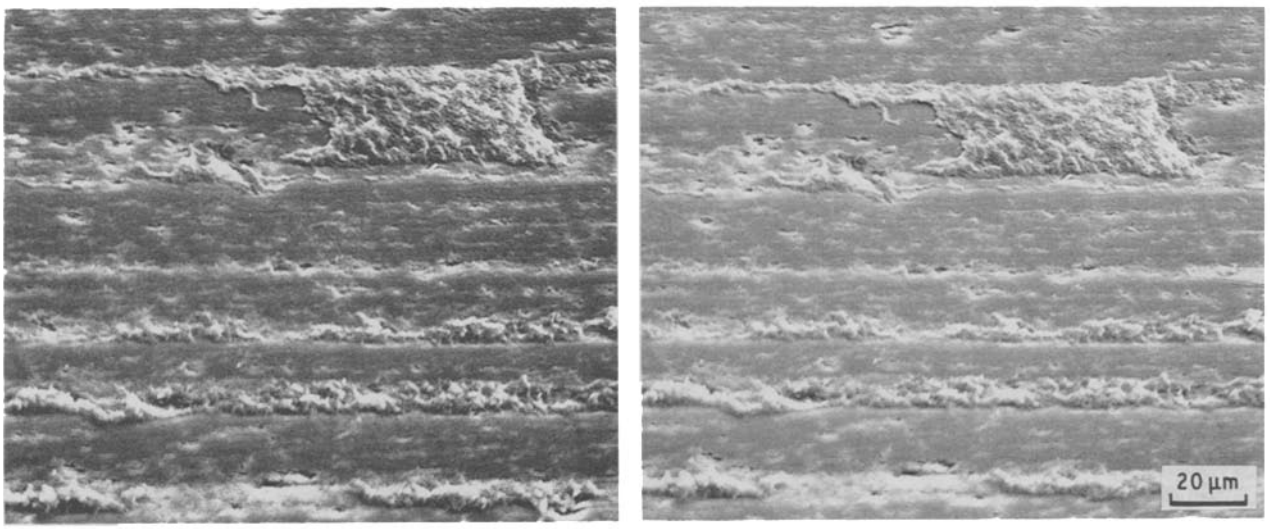

Figure 8 A stereo-pair of SEM micrographs of the fracture surface between a SiC coating and PG substrate. The crack propagation direction was parallel to the markings.

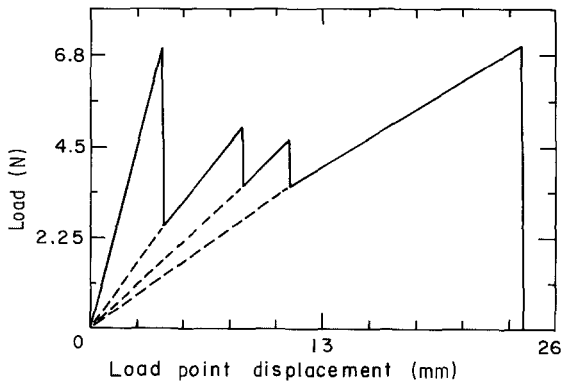

Figure 9 A typical load (displacement) curve recorded in a double cantilever beam experiment, showing three quasi-stable extensions of the crack before it finally ran unstably. Calculated G_c = 64 J m^{-2}.

Y the tensile yield strength of the equivalent homogeneous material (take Y = 71 MPa as the average for the glue and the PG), ε_y is the elastic strain at yield ($\sim$0.02 for the glue and $\sim$0.013 for the PG, take 0.015), and ν Poisson's ratio ($\approx$0.3). Evaluation of this expression gives a layer depth of about 10 μm. As a similar first approximation, the plastic work per unit area of fracture, locked-up in the crack surface layers of thickness h, should be of the order of $2\beta Y\varepsilon_y h$, where β, which is a constant of integration involving the plastic strain gradient into the surface in the deformed layers, is expected to be in the range of 2 to 3 (take 2.5). With the above choices for Y, ε_y and h, we estimate the dissipative component G_p of the energy release rate to be 53 J m^{-2}, or quite close to what was measured.

In order to gain more confidence in the above explanations, the critical energy release rate for fracture of the PG in the same orientation as in the above tests (i.e. with its weak planes oriented perpendicular to the fracture surface and running parallel to the crack growth direction) was also measured using the double cantilever beam experiment. The measured average value of G_c = 136 J m^{-2} is about twice as large as the critical energy release rates measured for cracks propagating along the PG–SiC interface. Scanning electron microscopy of the fracture surfaces of the PG, as shown in Fig. 10, revealed the layered structure of PG and indicated considerable roughness in the forms of steps between lamina. The measured difference in

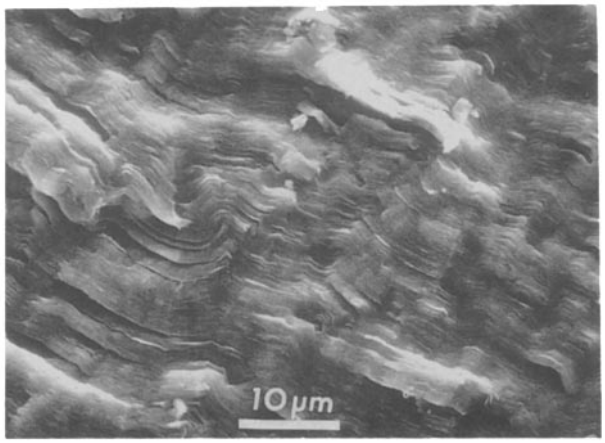

Figure 10 A SEM micrograph of the fracture surface in PG running transverse to the smooth layer planes.

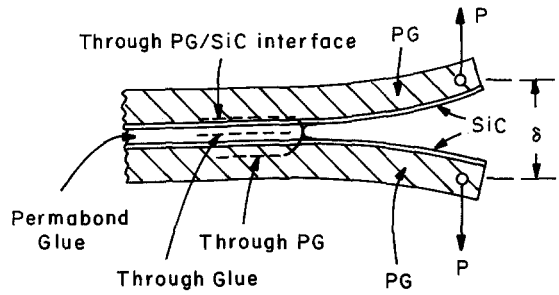

Figure 11 Sketch showing the three possible paths of a crack running parallel to an interface between SiC and PG.

fracture work between the PG–SiC interface and the PG substrate itself is quite large, indicating that the preferred delamination path should indeed be along the PG–SiC interface, as was observed.

The toughness of the glue layer is worth more consideration. As mentioned above, the thin glue layer exhibited a critical energy release rate at fracture of the order of 244 J m^{-2}, and has a flow stress equal to 64 MPa. This is close to the yield strength of unalloyed aluminium, which, however, in bulk form, exhibits energy release rates in plane strain fracture nearly two orders of magnitude higher, with deformation zones being correspondingly thicker. However, when tested in confined spaces in thin layers, the fracture work of aluminium is likely to be no larger than what was found for the glue. This indicates that the glue in many respects acts as a good model for the aluminium matrix in the prototype composite. Therefore, we conclude that the double-cantilever beam experiment for the fracture work of the PG–SiC interface, being surrounded by a glue layer and a PG substrate, mimicks the behaviour of the actual composite of SiC coated Pitch-55 fibres in an unalloyed aluminium matrix. Table II summarizes all the measured overall critical energy release rates for fracturing along the interface and in the PG as well as in the glue along paths shown in Fig. 11. From these measurements of the properties of the PG–SiC interfaces, we conclude that the overall energy release rates G_c are indeed made up of an intrinsic component G_{co} and a much larger plastic dissipation component G_p in the surrounding material, as discussed in connection with Equation 2, but that G_{co} is still the fundamental quantity which sets the scale of the overall fracture work.

Therefore, for the purpose of monitoring the toughness properties of the tailored interface, it is essential to find reliable means of measuring the intrinsic toughness level G_{co} alone. In a separate communication [9], we discuss how this intrinsic work of separation of the coatings from its substrate can be measured under nearly ideal conditions of quasi-static delamination of residually stressed coatings under the driving force of the locked-in strain energy of the

TABLE II Total specific fracture work or critical energy release rate

Fracture path	G_c(J m^{-2})
PG–SiC interface	62.4 $\pm$ 6.8
Permabond glue	244
Pyrolytic graphite	136

coating, which produces only negligible inelastic effects in the substrate.

5. Discussion

The delamination, or fracture toughness of interfaces play a key role in all composites, but particularly so in metal matrix composites, where unwanted reactions between the matrix and the fibre can produce reaction products with material misfit that can severely damage the fibre. This was observed in elegant experiments of Metcalf [17] in the boron fibre/aluminium system. Since during the production of composites processing histories that give proper wetting of fibres by the molten matrix and good adhesion are often in conflict with conditions to limit reaction damage, the proposal has been made here to separate functions of proper wetting from careful control of interface mechanical properties. This can be accomplished by tailoring the desired mechanical properties of the interface between the protective coating and the reinforcing fibre, while accepting some wetting related reaction damage between the matrix and the outer surface of the protective coating of the fibre. When properly controlled, this permits the pedigreed key interface between the coating and the fibre to act as a mechanical fuse to decouple the fibre from its surroundings by initiating delamination along it.

In this communication, we have discussed techniques for the measurement of the fracture work of such interfaces in simulated conditions of local environments representative of those in the composite itself. The double cantilever beam experiment that was used to measure the fracture work in symmetrical sandwiches of PG–SiC–glue–SiC–PG showed that very substantial inelastic deformation in the PG and the thin glue layer accompanies the apparent cleavage-like separation of the interface between the PG and the SiC. Since the PG has qualitatively similar inelastic behaviour and morphology to that of the Pitch-55 fibres, and the glue has similar yield behaviour to that of a pure aluminium matrix in the composite, the toughness measurements reflect behaviour that can be expected from the composite itself. Direct meaningful measurement of fracture work of interfaces between SiC coatings and Pitch-55 fibres in prototype composites have so far not been carried out.

Clearly, additional developments are necessary both for the measurement of intrinsic toughness of interfaces, and for the analysis of interface cracks with accompanying inelastic behaviour in the surrounding matrix and fibres. In a separate communication, we present new developments on the measurement of intrinsic toughness of interfaces [9].

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