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INTERFACE CHARACTERISATION AND BEHAVIOUR

Critical Review of Interface Testing Methods for Composites

M J Lodeiro, S Maudgal, L N McCartney, R Morrell and B Roebuck

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ABSTRACT

This report provides an assessment of the test methods and models suitable for characterising the nature and degree of interface adhesion in composite materials, in order to ascertain the impact on full composite behaviour. All material classes are evaluated: polymer, metal and ceramic matrix composites. Particular emphasis has been placed on the key issues associated with each of the tests and their ability to provide reliable, quantitative data. Mechanical tests used to measure a 'characteristic' interface parameter, such as shear/tensile strength or fracture energy, are identified. The measurement of surface roughness, physical wetting and chemical species contributing to interfacial adhesion have also been considered.

The preliminary review demonstrates the complexity of the effects of specimen preparation, experimental procedures, data reduction models and failure modes associated with the mechanical test methods. Similarly, the assessment of the physico-chemical evaluation techniques shows that these properties are often difficult to quantify and to incorporate into a full interface model for predicting the interfacial properties and subsequently composite performance.

The review highlights several important issues. There is no single ideal or universally applicable interface test technique. No standard test methods exist, although it is recognised that the geometry of the test-pieces and the conditions of the test affect the measured results, contributing to the variability of published data and the difficulty associated with making comparisons between them. There is doubt about which interface property to measure and how best to analyse the test data. In general, the tests are well-documented and researched, but further refinement is necessary. The most satisfactory methods appear to be the fragmentation test for polymer matrix composites and the push-through/push-back test for metal and ceramic matrix composites. Atomic force microscopy, wetting angle, differential scanning calorimetry and dynamic mechanical thermal analysis techniques show the best potential from the physico-chemical measurement methods available.

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CONTENTS

1 INTRODUCTION

'Interface characterisation and behaviour' is one of five projects that comprise the DTI Materials Measurement Programme on Composites Performance and Design (CPD). A review of interface test and analysis methods for polymer, metal and ceramic composite materials has been carried out. This report presents the information and conclusions reached during the course of this initial review stage.

The report is split into five, individually authored, sections:

- Section 3 : Mechanical test methods for the characterisation of the interface in polymer matrix composites - M J Lodeiro
- Section 4 : Chemical and physical test methods for the determination of the nature of fibre surfaces and fibre / matrix interactions - S Maudgal
- Section 5 : Interface strength measurements in metal matrix composites - B Roebuck
- Section 6 : Interface tests for ceramic matrix composites - R Morrell
- Section 7 : Modelling aspects - L N McCartney

The mechanical tests are assessed in terms of their material compatibility, the data generated, ease of use, consistency of data, data reduction requirements and stress uniformity. The 'fitness for purpose' of the physical and chemical methods is evaluated. The models are discussed in terms of their accuracy, simplicity and comprehensiveness in covering all the many parameters affecting the interface in composite materials (not included are methods for assessing interface integrity). Each section is self-contained, including conclusions, tables, diagrams and references (denoted by [ ] in the text) relevant to the section theme. A final section summarises the general conclusions of the document.

2 BACKGROUND

The properties of the interface between a matrix and reinforcing fibre, plays an important role in determining the mechanical properties and behaviour of composites. All unidirectional fibre reinforced composite materials exhibit significantly different behaviour when comparing axial properties to those measured transversely. During loading, the transverse properties are degraded more than axial properties because of interface failure. The development of a strong interface helps to maintain off-axis properties during loading and to reduce the rate of accumulation of damage.

For most polymer and metal matrix composites, the requirement is for a very strong interface so that the properties of the composite become susceptible to microstructural damage formation only at relatively high loading. For brittle (glass or ceramic) matrix composites, interface debonding is preferable, delaying the onset of fibre failure when the composite is loaded axially and leading to increased strength and energy absorption during the fracture process.

The interface is also important in determining long-term property retention for both fatigue and environmental endurance. For polymer matrix composites, the resistance of the interface to moisture or aggressive environments affects the durability of the composite properties. For ceramic and metal matrix composites, the stability of the interface at elevated temperatures is critical to their performance.

3 MECHANICAL TEST METHODS FOR THE CHARACTERISATION OF THE INTERFACE IN POLYMER MATRIX COMPOSITES

Traditionally coupon tests have been used to investigate the interfacial properties of polymer matrix composites (PMCs), indirectly. Those most strongly affected by the interface include: longitudinal compression, short beam shear, longitudinal and transverse flexure, in-plane shear (±45° tension, Iosipescu, etc.), transverse tension and mode I and II fracture toughness.

Although these tests yield qualitative and quantitative information that can be used to determine the relative degree of interfacial adhesion, they do not isolate the mechanical parameters affecting the interface alone from the contributions of the other constituents to the measured strength. They are thus unable to provide a definitive quantitative value for interfacial adhesion [1].

3.1 MICROMECHANICAL INTERFACE TESTS

Several test geometries employing single fibres have been developed in order to provide reliable and reproducible methods of measuring fibre-matrix adhesion. These tests have several benefits:

- eliminating the complicating effects of multiple fibre interactions,
- enabling full control of:
  - specimen preparation,
  - experimental parameters,
- simplifying modelling requirements,
- using small amounts of material (ideal for ensuring uniform environmental exposure and rapid environmental ageing),
- availability of additional *in-situ* techniques to aid test analysis:
  - Raman spectroscopy,
  - scanning electron microscopy,
  - acoustic emission,
  - photoelastic microscopy.

There is, however, a large degree of variability in:

- the detailed nature of the specimen geometry / dimensions employed,
- the specimen fabrication procedure,
- the test equipment and testing procedures used,
- the data recorded during / after the test,
- the data reduction analyses adopted.

3.1.1 Single Fibre Fragmentation Test

This test consists of a single fibre aligned axially in a dogbone resin coupon, loaded in tension. The tensile load applied is transferred to the fibre through shear transfer at the interface. As loading proceeds the tensile forces exerted on the fibre exceed its tensile strength and the fibre breaks, first at its weakest point (largest flaw) and then at successively

weak points (smaller flaws). The embedded fibre fractures into shorter lengths as the test continues and the stress gradients on the fibre ends begin to merge (Figure 3.1). This fragmentation process halts when the shear stress transfer through the interface can no longer build up enough tensile stress within a fragment to cause any further failures. This is termed the saturation point and the corresponding maximum final fragment length of the fibre is termed the critical length (affected by flaw size, density and distribution) [2].

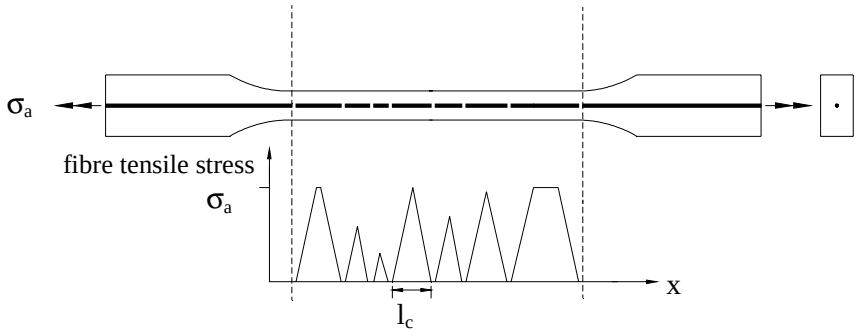

Figure 3.1 - Fragmentation specimen geometry and associated axial fibre tensile stress distribution of fragments.

The number and spacing of fibre breaks are recorded with respect to the load / strain applied to the specimen, either continuously or at intervals, until saturation. The experimental data are presented as plots of cumulative number of fragments with applied strain or as histograms of fragment length [3]. The test is normally accompanied by single fibre strength tests performed at various gauge lengths to ascertain the relationship between fibre length and strength. These data are used to extrapolate to the strengths of the very short fragment lengths achieved at saturation, using statistical methods. The specimen dimensions vary from 43-130 mm long, 10-25 mm wide and 0·2-3 mm thick.

There is a great diversity of reduction methods and models for analysing the experimental data. The most common / simplest analysis is:

$$l_c = \frac{d\,\sigma_f}{2\,\tau_i}$$

τ_i - interfacial shear strength,
σ_f - fibre strength at critical length l_c,

Failure at the interface is mode II dominated superimposed by radial compression caused by the Poisson's contraction of the matrix onto the fibre [4, 5, 6]. For weak interfaces, frictional slip or interfacial cracking occurs. For strong interfaces, matrix cracking may occur in the vicinity of the fibre breaks, in conjunction with yielding for ductile matrices or catastrophic specimen failure for brittle matrices.

Several useful variations to the conventional single fibre fragmentation test geometry and analysis have been developed. These include:

(a) **the coaxial geometry fragmentation test** - extends applicability to brittle resin systems which cannot successfully achieve saturation. A thin coating of the brittle resin of interest is applied to the surface of the fibre prior to being encased in a tough resin dogbone specimen [4, 7];

(b) **the multi-fibre fragmentation test** - consists of several continuous fibres aligned in parallel, at pre-defined inter-fibre spacings, in a dogbone specimen. This is useful for analysing fibre-fibre interactions [8];

(c) **the strand test** - prepared with fibre tows rather than single fibres, aligned parallel or perpendicular to the dogbone specimen axis [9];

(d) **the *in-situ* fibre strength test** - performed using the fragmentation test specimen to provide data on the strength of fibres whilst embedded in the matrix of interest [3].

Advantages	Disadvantages

• Simple specimen handling.
• Large statistical sampling of the interface.
• Replicates the stress transfer characteristics in real composites.
• Critical length is sensitive to and reflects changes in the level of fibre-matrix adhesion.
• Energy and fracture mechanics analysis methods being developed which do not require specimen saturation.
• Variety of methods available for observing / analysing failure processes directly:
  • acoustic emission,
  • photoelasticity,
  • Raman spectroscopy.
• Useful variations on the fragmentation test provide additional / complementary information:
  • coaxial test,
  • multi-fibre test,
  • strand test,
  • *in-situ* fibre strength test.
	• Indirect method of interface loading
	• Time consuming:
	  • specimen preparation,
	  • testing and data collection,
	  • data analysis.
	• Additional fibre strength tests required.
	• Limited material applicability (tough, high strain to failure matrices).
	• Highly complex / non-uniform stress state at the interface:
	  • interfacial shear stress concentration near fragment ends,
	  • neighbouring fractures,
	  • affected by fibre pre-tension,
	  • sensitive to level of interface adhesion.
	• Multiple failure events:
	  • interfacial debonding,
	  • matrix cracking
	  • plastic yielding,
	  • frictional slip.
	• Does not allow determination of the coefficient of friction / interface pressure.
	• Interfacial shear strength value depends on the constituent properties.
	• Relationship between critical fibre length and average fragment length unknown.
	• Extrapolation of Weibull fibre strength data to short fragment strengths is not understood.
	• High radial compression stresses can give rise to overestimated interfacial strengths.
	• Premature fragmentation of hygrothermally aged samples; changes in residual stresses due to environmental conditioning..

3.1.1.1 Supplementary Analytical Techniques

The fragmentation method can be evaluated with the aid of a variety of additional techniques, which are increasingly being used with the other interface tests. These techniques help to assess and distinguish failure events, evaluate stress / strain profiles and allow a greater understanding of the failure processes associated with each test. These include:

(i) **Raman spectroscopy** - a Raman spectrum can be obtained from materials which inelastically scatter light. Using the well-defined relationship between the peak frequency position of a strain sensitive Raman band and the applied strain, the true axial strain distribution in an embedded fibre can be determined at the microscopic level (limited to non-amorphous reinforcement with strong Raman signals and transparent matrices) [10].

(ii) **acoustic emission** - employed for the detection of interfacial debonding events and the detection and location of fibre fracture. This technique permits continuous monitoring of the fragmentation test and extends its application to opaque resins and fibrillar breaks (detection of relevant signal from noise may be difficult) [2, 11].

(iii) **photoelasticity** - used extensively to monitor failure processes in the single fibre fragmentation test (applicable to transparent matrices which are birefringent under the application of external load) [2].

3.1.2 Pull-out Test

This test consists of a fibre embedded in a matrix block of known geometry (Figure 3.2). A steadily increasing force is applied to the free end of the fibre in order to pull it out of the matrix. The fibre may be pulled out of a resin disk, block or droplet [2]. This test shows great variation in geometry and specimen fabrication procedure.

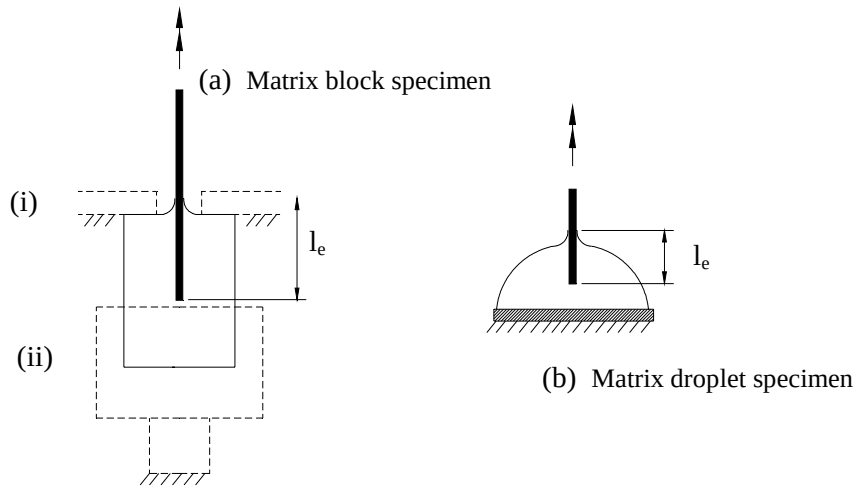

Figure 3.2 - Pull-out specimen geometries showing fibre embedded lengths and different geometries and loading configurations: (a) matrix block sample (i) restrained from above and (ii) restrained from below; and (b) matrix droplet sample.

A test device optimised for stiffness encourages stable crack propagation [12]. The maximum load achieved is usually defined as the debonding force. Data are presented as plots of debonding force versus embedded length. The embedded lengths achievable range from 30-500μm.

In its simplest form, the interface shear strength is calculated using a balance of forces:

$$\tau_i = \frac{F}{\pi d l_e}$$

F - debonding load,
l_e - embedded fibre length,
τ_i - interfacial shear strength,
d - fibre diameter.

A mode I dominated, mixed mode failure is initiated (due in part to the Poisson contraction of the fibre away from the surrounding matrix) [6]. Subsequent interfacial crack propagation may be rapid or stable depending on the system stiffness. After debonding, friction at the interface must be overcome for pull-out to proceed. Strongly bonded interfaces, may fail initially within the resin meniscus, leaving a conical residue on the fibre, followed by interfacial debonding.

Advantages	Disadvantages

• Direct method of loading interface.
• Single force value recorded at failure.
• Applicable to most fibre-matrix combinations.
• Simple test to perform.
• Simple basic analysis.
• Provides information on friction coefficients and shrinkage pressures.
	• Difficult specimen preparation and handling.
	• Variability in specimen geometry and fabrication procedure.
	• Highly complex / non-uniform stress state at interface:
	  • high shear stress concentration near point of fibre entry into matrix,
	  • lower shear stress concentration at embedded fibre end,
	  • affected by elevated meniscus and embedded fibre length.
	• Restrictions on embedded length:
	  • lower bound due to meniscus formation,
	  • upper bound limited by fibre strength.
	• Large scatter obtained.
	• Single data point per test.
	• Local interface measurement.
	• Meniscus failures are invalid.
	• Test device stiffness affects nature of failure and results.
	• Radial stresses on fibre change with thermal and moisture expansion mismatch between fibre, resin and resin holder; fibre strength degradation due to environmental ageing.

3.1.3 Microdrop / Microbond Test

This is a specific type of pull-out test which aims to deal with some of the limitations / problems of the conventional pull-out test. A droplet of cured resin is formed axisymmetrically on a fibre. A shear force is applied to the interface via an adjustable pair of knife edges (flat-ended, bevelled or semi-circular) exerting a downward force on the droplet causing debonding (Figure 3.3) [2, 5].

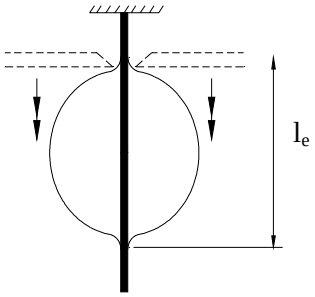

Figure 3.3 - Microdrop specimen geometry showing fibre embedded length.

The data are presented in the form of debonding force (maximum load) versus embedded length plots. The droplet lengths vary from 40 to 200μm. Again, as in the fibre pull-out test, the interfacial shear strength is given by:

$$\tau_i = \frac{F}{\pi d\, l_e}$$

F - debonding load,
l_e - embedded fibre length within droplet,
τ_i - interfacial shear strength,
d - fibre diameter.

As in the conventional pull-out test, the interface fails in a mixed mode with mode I dominating initially (due in part to the Poisson contraction of the fibre away from the surrounding matrix). Once a debond initiates, it propagates rapidly and catastrophic failure

occurs. After this point, some load transfer due to frictional sliding occurs, as the fibre is pulled through the droplet.

Advantages	Disadvantages

• Direct method of loading interface.
• Single force value recorded at failure.
• Applicable to most fibre-matrix combinations.
• Simple specimen preparation.
• Simple test to perform.
• Simple basic analysis.
• Requires very small amounts of material.
• Cohesive or adhesive nature of failure can be ascertained through SEM examination of the fibre surface after failure.
• Reduced meniscus.
	• Difficult specimen handling.
	• Variability in droplet shape and dimensions.
	• Restrictions on embedded length:
	  • lower bound due to droplet formation,
	  • upper bound limited by fibre strength and droplet formation.
	• Highly complex / non-uniform stress state at interface:
	  • high shear stress concentration near point of fibre entry into matrix,
	  • affected by stress concentrations at loading points of contact,
	  • affected by location of loading points on droplet,
	  • affected by shape of loading blades,
	  • affected by droplet shape and size.
	• Large scatter obtained.
	• Single data point per test.
	• Local interface measurement.
	• Meniscus failures are invalid.
	• Droplet mechanical properties vary with size.
	• Resin plasticisation at loading points following hygrothermal exposure; high diffusion rates demand prompt post-conditioning testing.

3.1.4 Micro-indentation Test

In this technique, single fibres perpendicular to a cut and polished surface of a high volume fraction composite are compressively loaded in the axial fibre direction with an indenter to produce debonding or fibre slippage (Figure 3.4) [2]. This test is less commonly used for reinforced polymers than the other interface test methods.

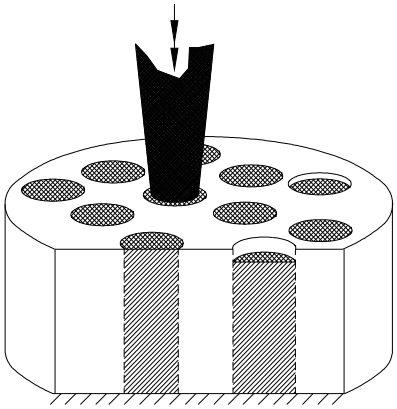

Figure 3.4 - Micro-indentation specimen geometry.

A fibre is manoeuvred into place directly beneath a flat / rounded end indenter. This is loaded via high stiffness apparatus in a cyclic loading-and-observation process or at a constant rate, with the load and depth of indentation monitored continuously. Detection of fibre debonding is recorded as a characteristic change in the load-depth trace, by monitoring acoustic emission events or by unloading and observing the fibre optically. The specimen

dimensions vary in order to accommodate specific indenting equipment, but are approximately 10×10×3-10 mm in size.

As a first analytical approximation the debonding strength of the interface can be calculated from the equation [13]:

$$\tau_i = \frac{nF}{2\pi r^2}$$

τ_i - interfacial shear strength,
F - debonding load,
n - volume fraction and fibre / matrix stiffness parameter,
r - fibre radius.

More commonly, the interfacial shear strength is calculated using finite or boundary element analysis of the stressed area, for each fibre tested, to include the effect of neighbouring fibres located at various distances and distributions [5].

Mode II dominated failure occurs superimposed with compressive stresses (due to the Poisson expansion of the fibre) [6]. It is likely that debonding initiates within the maximum shear stress region, below the contact surface, and quickly spreads over the circumference up to the specimen surface. In some cases, especially for thermoplastics with weak interfaces, the interface is already destroyed by the polishing process.

Advantages	Disadvantages

• Direct method of loading interface.
• Single force value recorded at failure.
• One specimen provides many data points.
• Applicable to most fibre-matrix combinations.
• Simple specimen preparation.
• Simple specimen handling.
• *In-situ* test providing a real environment for assessing the interface.
• Independent of fibre tensile strength.
	• Difficult specimen testing:
	  • high position accuracy required,
	  • difficulty detecting debonding load.
	• Failure criterion is subjective and arbitrary.
	• Highly complex / non-uniform stress state at interface:
	  • high shear stress concentration near fibre contact surface,
	  • stress concentration at loading region of contact,
	  • affected by proximity / configuration of neighbouring fibres,
	  • affected by indenter shape / size / stiffness and position on the fibre cross-section,
	  • affected by the polishing protocol.
	• Fibre damage is common, limiting material applicability.
	• Failure mode and locus cannot be observed.
	• Finite or boundary element analyses required for accurate data reduction.

3.2 CONCLUSIONS

The conclusions from the review of interface tests for polymer matrix composites can be summarised as follows:

- There is no complete and unambiguous interface measurement method.
- There is a lack of consensus and understanding regarding the fundamental failure processes occurring at the interface and the data reduction analyses which best provide meaningful and reliable data.
- Data reduction methods are oversimplified, using constant shear assumptions when non-uniform distributions and mixed mode failures occur in all cases.
- There is generally a high scatter in the data generated due to the intrinsic random nature of the interface, the small areas considered in the tests and the sensitivity to specimen and test details.

- Results differ significantly between tests due to differences in, specimen geometry and loading methods, stress distributions, thermal mismatch and curing stresses, friction and Poisson's effects and stiffness mismatch effects.
- An inherent problem with the tests is the determination of an interfacial property that is independent from the specific test arrangement; this is being addressed by work on fracture mechanics and energy based analysis methods (§ 7).

Despite these problems, it is possible that by tightening the controls on specimen preparation, dimensions and test parameters, and by formalising the analysis methods, that consistency can be achieved. The fragmentation and pull-out techniques are the most favourable techniques for achieving these aims, giving the best balance between testing and modelling aspects. A recent round-robin highlighted the potential for harmonisation of each of the interface test methods, where initial results showed a promising correlation in results for each of the four tests between 12 separate sites, each using their own in-house procedures, specimen geometries and equipment [14].

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4 CHEMICAL AND PHYSICAL TEST METHODS FOR THE DETERMINATION OF THE NATURE OF FIBRE SURFACES AND FIBRE/MATRIX INTERACTIONS

The primary mechanisms of adhesion in polymer matrix composites are:

(i) **Mechanical Adhesion**: related to the degree of roughness of the fibre.

(ii) **Adsorption and Wetting**: the formation of a physical bond resulting from highly localised intermolecular forces. Resins that have surface energies less than that of the fibre will readily wet the fibre surface and yield good bonds.

(iii) **Chemical Bonding**: when a functional grouping on the fibre and a compatible group in the matrix reacts to form a stable linkage.

These mechanisms are discussed further in this section. Several of the techniques which have been developed or are undergoing a process of refinement to study and quantify the above effects, are summarised in Tables 4.1 and 4.2.

4.1 SURFACE ROUGHNESS

Fibre roughness plays an important part in the mechanical bonding at the interface in all composite materials. This form of bonding is purely due to the physical interlocking of the fibre and matrix surfaces in contact, as matrix material penetrates available crevices in the fibre surface. Although the tensile strength can depend upon the presence of re-entrant angles on the fibre surface, shear strength increases significantly with the degree of roughness. Increasing roughness also results in beneficial increase in friction. Further, the roughness of the fibre surface increases the potential for chemical bonding because of the larger surface area available.

Various techniques exist for imaging structural elements at the fine scale required for analysing reinforcement fibres [1, 2, 3]. X-ray diffraction, transmission electron microscopy, scanning electron microscopy and the group of techniques called scanning probe microscopy have been used to study reinforcement fibres.

4.2 WETTING CHARACTERISTICS

For polymer matrix composites, if sufficiently intimate contact is achieved between the fibre and liquid resin, a physical attraction develops between the atoms of the two surfaces which results in the wetting of the fibre by the resin. The extent of this wetting is related to the differences in the surface free energies of the solid, liquid and the interface that is formed by the contact between them. Wetting at the surface may be due to acid-base interactions, weak hydrogen bonding or Van der Waal's forces (dipole-dipole interactions and dispersion forces) depending on the chemical nature of the solids and liquids involved [4].

4.3 CHEMICAL METHODS

Fibre surfaces generally require surface treatment to enhance adhesion between the fibre and matrix, when used as reinforcement in polymer matrix composites. In the case of carbon fibres, this is accomplished by heating in an oxidising atmosphere and treatment with an oxidising reagent such as concentrated nitric acid or, most commonly, anodic oxidation in an aqueous electrolyte. Similarly, glass fibres are treated with silane coupling

agents of the structure $(RO)_3 - Si - (CH_2)_n - Y$ where Y is a functional group designed to react with the matrix functional species. These treatments serve to increase the concentration of oxygen and nitrogen containing functional groups on the fibre surface.

Characterisation methods [5, 6, 7, 8, 9] include: X-ray photoelectron spectroscopy, infrared and Fourier transform infrared spectroscopy, nuclear magnetic resonance spectroscopy, mass spectrometry, gas chromatography, differential scanning calorimetry and modulated differential scanning calorimetry, and dynamic mechanical thermal analysis. All of these methods aim to determine the concentration of surface active sites, such as -COOH, -OH and -NHR, since these functional species are believed to enhance adhesion at the fibre-matrix interface by forming covalent or hydrogen bonds.

4.4 CONCLUSIONS

From a review of the available literature, an effort has been made to highlight the utility of physical and chemical test methods to study the effects of the interface on composite properties (Tables 4.1 and 4.2).

- Atomic force microscopy is considered the most relevant surface roughness technique for composite application. AFM lends itself to analysis of all kinds of fibre surfaces, conductive and non-conductive allowing a comparison of results generated by testing different reinforcements. With careful interpretation, it provides a numerical value of roughness in terms of peak-to-peak and peak-to-trough distances.
- Wetting measurements can be used to determine whether a resin system is compatible with a given reinforcement. This can be useful when modifying fillers and additives in resin formulations.
- The techniques for investigating chemical bonding at the interface have overlapping capabilities, thus several techniques in conjunction may be required for a full analysis of the reactions and functional groups.

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Table 4.1 - Summary of surface roughness and wetting characterisation techniques, applications and limitations

Test	Parameter Measured	Utility	Limitation
(i) MECHANICAL ADHESION			
X-ray Diffraction	Crystal structure	Measurement of dimensions and orientation crystal platelets	• macroscopic measurement • not applicable to topography
Transmission Electron Microscopy	Imaging at higher magnifications than optical microscopy	Scrutiny of local orientation of individual units	• 2-dimensional image only • extensive sample preparation
Scanning Electron Microscopy	3 dimensional imaging with broad range of magnification	Qualitative picture of surface features	• magnification is insufficient to allow surface roughness characterisation
Scanning Tunnelling Microscopy	Tunnelling of electrons between the surface under analysis and the scanning tip	Provides direct topographical information with atomic resolution	• non-conductive fibres cannot be analysed
Atomic Force Microscopy	Inter-atomic repulsion between surface being analysed and the scanning tip	Quantification of surface roughness of small diameter fibres	• interpretation may be difficult • support with SEM data is useful
(ii) WETTING CHARACTERISTICS			
Dynamic Contact Angle Analysis	Force on a fibre as it is immersed and withdrawn from a liquid	Determination of wetting forces and surface energetic properties, comparison of surface treatments	• no direct correlation with mechanical behaviour of composite

Table 4.2 - Summary of interfacial chemical bond analysis techniques, applications and limitations

Test	Parameter Measured	Utility	Limitation
(iii) CHEMICAL ANALYSIS TESTS			
X-ray Photoelectron Spectroscopy	Chemical characterisation of surfaces in terms of atomic concentration	Quantifying matrix and fibre related photoelectrons in a fracture surface and hence the relative amount of cohesive and adhesive failure	• only fracture surfaces can be analysed • thickness of matrix coated on a fibre is beyond the XPS sampling depth
Infrared Spectroscopy and Fourier Transform Infrared Spectroscopy	Characterisation of molecular structure by observation of spectral differences between samples	Qualitative analysis of environmental effects which result in changes in functional groups	• complex instrumentation
Nuclear Magnetic Resonance Spectroscopy	Analysis of chemical environment in which atomic nuclei are found	Effect of surface treatments and chemical reactions at the interphase can be analysed to provide mechanism of interaction	• low sensitivity makes it applicable only in cases where cross-polarisation is known to occur
Mass Spectrometry	Analysis of elemental species to identify type and distribution of functional groups	Determination of chemical composition of surface species as a result of treatment	• requires isotope enrichment to identify surface related functional groups
Inverse Gas Chromatography	Retention time of a liquid through a column using the fibre surface as stationary phase	Determination of interactions between fibre and resin systems	• packing column, typically of glass, requires a degree of skill
Differential Scanning Calorimetry	Heat flow in samples relative to a stable reference as it is subjected to a controlled thermal profile	Comparison of constrained and unconstrained fibres in terms of interaction parameters defined as a ratio of chosen peaks	• peaks must be carefully chosen as being representative of the constraint • only fibres that melt can be analysed
Dynamic Mechanical Analysis	Measures mechanical properties of samples as a function of temperature as they are deformed under periodic stress	The damping coefficient has been found to be directly related to energy dissipation at the interface, a weaker interface has a higher tan δ	• heating rates have been found to affect resultant thermograms • optimum run conditions must be carefully determined to avoid artefacts

5 INTERFACE STRENGTH MEASUREMENTS IN CONTINUOUS FIBRE REINFORCED METAL MATRIX COMPOSITES

In metal matrix composites (MMCs), the overriding requirement for improving composite properties is a need to make the most of the matrix toughness. It has been commented in a number of reviews [1-3] that, to make the most of the matrix plasticity in MMCs the interface strength needs to be as high as possible, and that there is scope for even higher strengths than are now present in state-of-the-art MMCs.

This section is concerned with measurement methods for interface properties relevant to interface / matrix separation failure modes only, as opposed to those in which matrix shear occurs close to the interface.

5.1 TESTING ISSUES

A number of issues must be considered when addressing the need for interface strength measurement methods:

- Interfaces can be complex, with thin layers applied to the reinforcement fibre, possible reaction layers and matrix compositional differences close to the interface.
- Fracture initiation and propagation sites need to be identified.
- Interfacial stresses are present in the as-processed composite due to thermal expansion differences.
- The residual stress (radial) from differential thermal contraction is compressive and must be accounted for in determining interface strength values.
- Energy can also be dissipated by friction of the debonded interfaces through sliding processes.
- For frictional sliding the shear stress, contact area and sliding distance are needed to calculate how much energy is dissipated.
- For axial loading, frictional processes at the interface, for example caused by fibre surface roughness, can give significant increases in toughness in the absence of gross plasticity.
- For transverse loading, toughness increases due to interface debonding compared with plasticity effects may be small, indicating that test methods may not be sufficiently discriminating.
- Crack tips on interfaces generally have mixed mode loading.
- The stress state around a fibre close to a free surface, as is usual in push-out tests for example, is significantly different to that in the main body of the MMC.
- Interface properties at elevated temperatures are likely to be just as important as at room temperature. Suitable test methods must take this into consideration.

5.2 TESTING APPROACHES

In principle two approaches are possible, although neither are easy to apply in simple test systems because of the geometry of the fibre reinforcement. These are:

(i) Measurement of interface **strength**:

- (a) applied tractions acting normal to the interface,
- (b) applied tractions acting in the interface plane,
- (c) combinations of (a) and (b).

(ii) Measurement of interface **toughness** [3,4]:

- (a) by opening mode I (tension) - G_{IC},
- (b) by sliding mode II (shear) - G_{IIC},
- (c) by combinations of mode I and II.

In summary, three methods have been reported for testing interface strengths in MMCs:

(i) Push-out tests on thin slices, for which there are three varieties:

- (a) with parallel-sided thin slices,
- (b) with tapered (wedge) slices to vary the contribution of debonding and frictional processes,
- (c) with additional in-plane stresses on uniform slices to study mixed mode loading stresses.

(ii) Transverse tensile tests, on multiple or single-ply (cruciform) test-pieces.

(ii) Fibre fragmentation tests on single-ply or single-fibre / matrix test-pieces.

5.2.1 Pull-out Tests

- Test is not practicable for MMCs because of the difficulties of handling the brittle fibre.

5.2.2 Push-out tests

- These tests are used to measure critical stress levels for interface debonding or sliding [1-3, 5].
- Mode I crack propagation cannot be developed in its simplest form.
- It is necessary to impose in-plane radial tension to investigate the effects of residual stresses and mixed mode loading.
- The use of fractography is vital to evaluate crack initiation and propagation modes before applying data reduction analyses.
- A force balance equation is generally used to analyse the load-displacement curves:

$$\tau_i = P \big/ 2\pi r t$$

P - applied load
r - fibre radius
t - thickness of the test-piece
τ_i - interfacial shear stress

- The analysis does not account for residual stresses or effects of localised interface failure and growth.

- Fracture mechanics analyses are being developed to measure interface toughness, G_C, values [3, 4], but these may be dependent on the fracture morphology.
- Wedge-shaped (tapered) test-pieces can provide information on whether debonding or frictional sliding requires a larger load.

5.2.3 Transverse Tests

- This test [1-3] enables debonding to be monitored through changes in the stress / strain curve, acoustic signals and / or in Poisson's ratio, ν.
- Analyses have yet to be developed which allow progressive crack development to be separated from matrix plasticity to give interface toughness values.
- Cruciform test-pieces (Figure 5.1) minimise cracks propagating from the free surfaces which otherwise make interpretation difficult.

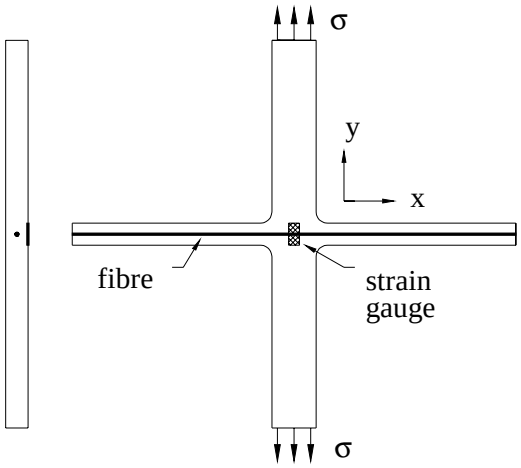

Figure 5.1 - Schematic diagram of a cruciform, transverse single-fibre specimen.

- Single-ply transverse tests [6] are easier to interpret than multiple-ply test-pieces, although the matrix is more constrained in the latter giving a markedly different stress / strain response.
- In transverse tests, the applied stress cannot be directly associated with the interface debonding stress because of stress concentration and residual stress effects, hence stress analyses must be performed [3,6].

5.2.4 Fibre Fragmentation Tests

- In this test [1-3, 6, 7], fragmentation lengths are determined either by polishing, selective matrix etching or by acoustic emission.
- A cruciform-shaped single-ply test-piece has been developed in order to reduce edge effects [8].

- A shear lag analysis [3] is used to calculate an interface friction stress, τ_i:

$$\tau_i = \sigma_F r / L_c$$

σ_F - fibre strength
r - fibre radius
l_c -fibre fragment length at saturation

- Saturation may be difficult to ascertain (where saturation is defined as a characteristic density of fibre fractures).
- The interface friction stresses are generally higher than those obtained from push-out tests.

5.2.5 Interface Strength and Toughness Measurements

Studies [3] have shown that results obtained from a wide range of methods are difficult to compare, because of the lack of rigour in specification of interfacial parameters, and because of differences in composition and geometry. Also where fractographic observations are not included in the test, the significance of the results is difficult to assess. There is also a lack of toughness compared to strength data available. This is highlighted in Table 5.1, which summarises interface test results from several authors.

5.3 CONCLUSIONS

The conclusions of the MMC interface review can be summarised as follows:

- Both conventional push-out and transverse tests are affected by edge preparation procedures and edge stress transfer effects, however:
    - crack initiation is possible where the fibres intersect the specimen surface; thus the tests are of questionable relevance to interface separation in larger components,
    - fractography is essential to confirm that interface cracks are formed away from these surfaces.
- Cruciform test-pieces containing a single ply or a single fibre within the matrix of interest would seem to be the most amenable to interpretation in terms of either strength or toughness, however:
    - test-pieces are costly,
    - a complex stress analysis is needed to interpret the measurements,
    - the matrix compliance is not the same as in a multiple ply set-up representative of components.

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Table 5.1 - Room temperature interface properties

Material		Strength, MPa			Interfacial Toughness J/m²	Test (SF - single fibre)	Ref
Matrix	Fibre*	Interfacial Debonding Shear Stress	Frictional Shear Stress	Tensile Strength			
Ti-6Al-4V	SCS-0		290			Push-out	8
Ti-6Al-4V	SCS-6		180			Push-out	8
Ti-6Al-4V	SCS-0		900			Fragmentation SF	8
Ti-6Al-4V	SCS-6		420			Fragmentation SF	8
Ti-6Al-4V	Sigma SM1240		390			Fragmentation SF	8
Ti-6Al-4V	SCS-0			380		Cruciform transverse SF	3, 7, 8
Ti-6Al-4V	SCS-6			115		Cruciform transverse SF	3, 6, 7, 8
Ti-6Al-4V	SCS-0	305				Push-out	7
Ti-6Al-4V	SCS-6	175				Push-out	7
Ti-6Al-4V	Sigma SM1240	210				Push-out + in-plane tension	2
Ti-6Al-4V	Sigma SM1240	150				Push-out + in-plane tension	2
Ti metal-21S	SCS-6	220				Push-out	5
Ti metal-21S	SCS-6		160			Push-out	5
Al	Al_2O_3	77	70			Push-out	8
Al	SiC	131	124			Push-out	8
Ti-15-3	SCS-6				50	Push-out	4
Ti-6Al-4V	SCS-6				52✌5	Push-out	4
Ti metal-21S	SCS-6				54	Push-out	4

* - SCS-0, SCS-6 and Sigma SM1240 are all SiC fibres, >100 μm diameter.

6 INTERFACE TESTS FOR CERAMIC MATRIX COMPOSITES

In ceramic matrix composite (CMC) materials, the interface design is a crucial controlling factor in determining the properties and performance. There is considerable evidence that parameters such as high interfacial shear stress and interface friction correlate with brittle behaviour. Degrading fibre interfaces in CMCs tends to result in the development of a strong bond, which is deleterious to crack deflection. In addition to the factors involved in brittle/brittle systems at temperatures close to ambient, CMCs are primarily intended to operate at elevated temperatures, so the long-term stability of the interface, and hence of interface-related properties, becomes a major factor. Since processing usually requires high temperatures, reactions between the fibre and interface can occur during manufacture, and the variety of fibre and matrix expansion coefficients means that interfaces can be in compression or tension when cooled to room temperature.

A variety of PMC-type tests have been tried, but CMCs present some difficulties:

- large diameter (>100 μm) fibres cause considerable gripping and alignment problems,
- it is almost impossible to manufacture the special test-pieces with a representative interface.

Tests tend therefore to be limited to those that require bulk composite test-pieces (Figure 6.1). The various test methods available are: (i) single fibre pull-out, (ii) single fibre push-down, (iii) single fibre push-through, (iv) single-fibre push-through and push-back, (v) fibre bundle push-though and (vi) slice compression tests.

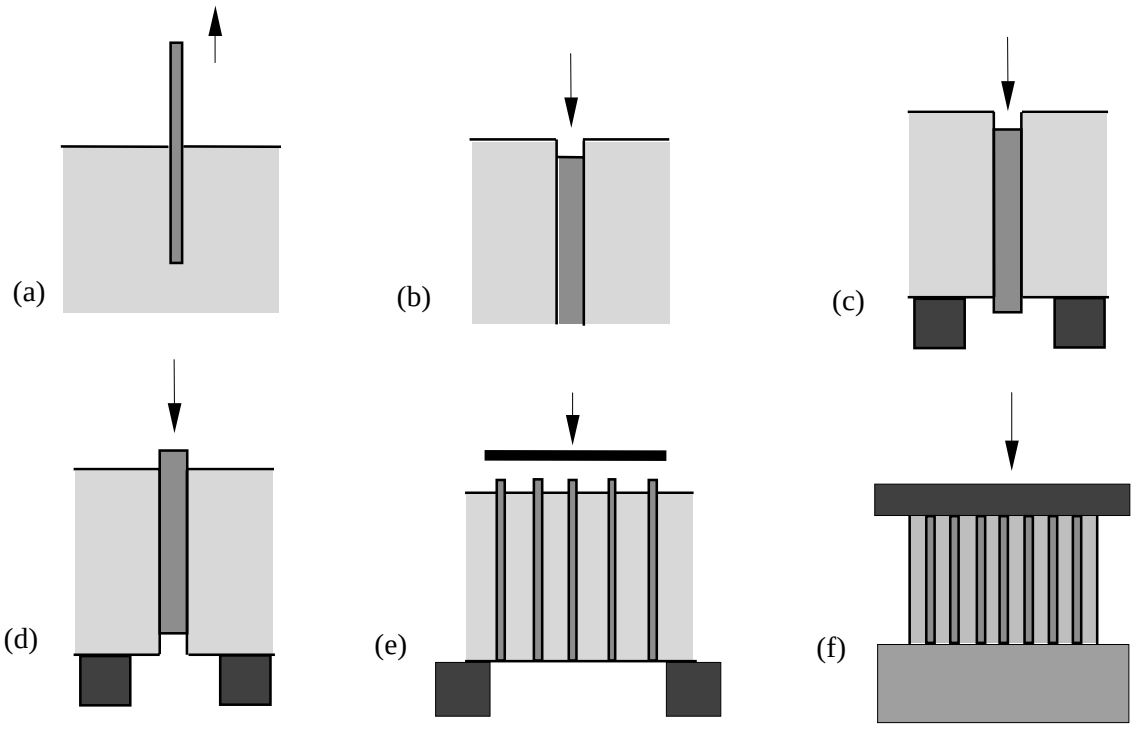

Figure 6.1 - Schematic diagrams of the CMC interface test configurations: (a) Fibre pull-out, (b) Fibre push-down, (c) Fibre push-through, (d) Fibre push-back, (e) Fibre bundle push-through and (f) Slice compression.

This section discusses the various test methods, together with some relevant analyses, and criticisms or limitations of the techniques.

6.1 INTERFACIAL TEST TECHNIQUES

6.1.1 Single-fibre Pull-out Test

In this test, a single fibre protruding from a surrounding block of matrix is gripped and pulled-out [1,2].

- The force which corresponds to ultimate failure of the whole interface area is recorded.
- Smooth sliding and stick-slip behaviours can be seen depending on the fibre surface treatment.
- Interfacial shear strengths are essentially independent of the embedded length.
- It is important to get good alignment.
- It is an ideal way of representing tensile failure characteristics.
- The test is feasible only for large diameter fibres which can be readily handled.
- Suitable only for simple systems (e.g. glass matrix) which can be readily fabricated around single fibres:
    - for most practical ceramic systems the usual fabrication route cannot be modified to use single fibres,
    - the alternative process of bulk material matrix dissolution is difficult and risks destroying the interface.

6.1.2 Indentation Tests

This family of tests all require composite samples whose cross-section is normal to the fibre axis and a degree of surface preparation (usually polishing or lapping). The indenters used to apply the compressive load are preferably flat-ended, to reduce fibre damage and local stress concentrations. It is also important that accurate positioning systems are employed with the test devices to locate individual fibres, especially for small diameter fibres.

6.1.2.1 Single-fibre Push-down Test

This test [3, 4] usually employs a nano-indenter (or similar device) to load an individual fibre in a bulk composite specimen whilst recording the indenter load and displacement. The test examines the debonding process which progressively moves down the chosen fibre as the load and displacement increase.

- This method works fairly well on large diameter fibres, but is much more difficult with fine fibres.
- Some test devices can be used in conjunction with a scanning electron microscope to locate fine fibres accurately and monitor the test.
- Loading and unloading cycles can be used to identify the depth of indentation into the end of the fibre.
- A simple shear-lag model is commonly used to analyse data to provide a value for the interface strength.
- Results can vary between individual fibres, indicating a need to undertake such measurements statistically.

- It is possible to extract interfacial parameters from the loading / unloading curves.

6.1.2.2 Single-fibre Push-through Test

In this test [5, 6], a thin slice of composite material is used (0·5-2 mm). A single fibre, positioned over a hole in the supporting base, is loaded with suitable indentation devices until the fibre slides through the slice.

- This method yields the sliding friction coefficient, using values for radial clamping stresses estimated from thermal mismatch data.
- The analysis of this test is simpler than the push-down test on a thick section. Models are based on:
    - shear-lag,
    - progressive debonding,
    - thermal expansion mismatch analyses.
- For transparent matrices (e.g. glasses), fibres which are suitable for indentation can be selected via optical transmission examination and stress birefringence measurement, thus avoiding those with interfaces damaged as a result of the thin slice preparation process.
- The shapes and slopes of the load / displacement curves can be used to interpret the state of the interface and the effects of roughness (Figure 6.2), which although negligible during debonding, cannot be ignored during steady-state push-out.

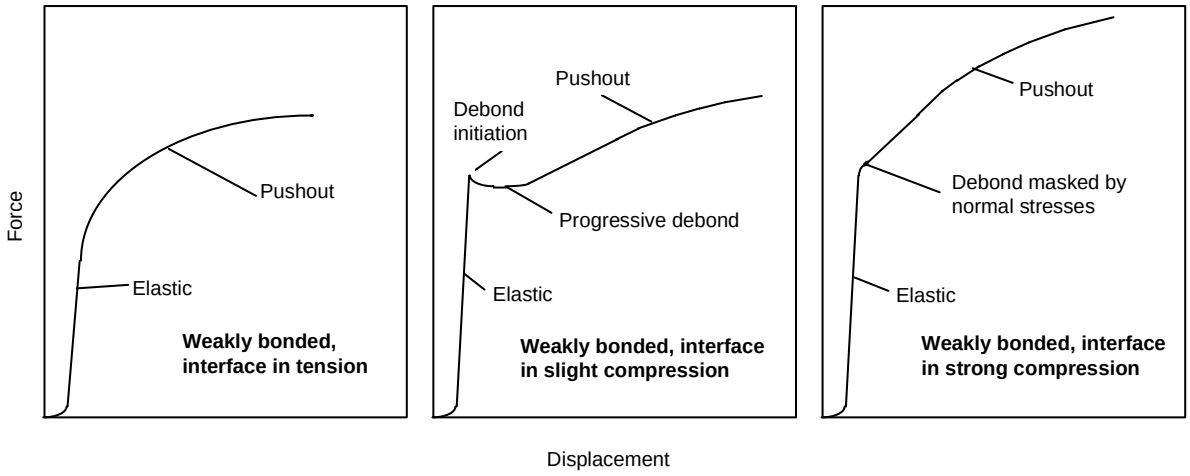

Figure 6.2 - Schematic behaviour of large fibres in the thin-foil push-out test. The stress condition and the roughness influence the shapes of the curves.

- Low loads are generally required for testing, resulting in minimal indentation damage on the fibre and minimal effects of Poisson expansion due to fibre axial compression.

6.1.2.3 Single-fibre Push-through and Push-back Test

This test [7, 8] consists of a single fibre push-through test, which is repeated after turning the test-piece over. The pushed-out fibre is located and reloaded, reversing the movement of the fibre, until it returns to its original position.

- Primarily to study frictional and interface roughness effects.

- Provides an additional measure of friction coefficient.
- The frictional sliding resistance on reversal is similar to the initial stage.
- A distinct drop in force is noted during push-back when the fibre 're-seats' itself, such that its surface roughness matches that of the matrix.
- There are indications from the test that the initial stages of fibre movement are easier than steady state sliding, probably due to the increasing influence of roughness on movement.
- There is a risk that the interface may wear in the process, although for short sliding distances the effect is likely to be small.

6.1.2.4 Fibre Bundle Push-through Test

Similar to the single fibre push-through, but a bundle of fibres (20-30) are loaded by a flat-ended pushrod [9, 10]. The matrix is removed to a suitable distance (10 μm) from the prepared surface of a bulk specimen (1 mm thick) by chemical dissolution, to leave bare fibre ends.

- This technique is applicable only to particular classes of materials where matrix dissolution is possible without compromising fibre or interface integrity.
- An extensometer rod, placed in contact with the lower ends of the fibres to be indented, can be used to measure the fibre displacement directly.
- It should be able to provide an averaging of the push-out forces required to simultaneously move a number of fibres, whilst retaining the appropriate lateral constraint.
- The distance of movement is controlled by the amount of surface removed in preparing the specimen, which can clearly be rather more than the micrometre or two movement possible when indenting from a flat surface.
- The test suffers from matrix cracking and fibre breakage, leading to overestimates of the normal stress.
- Yields greater scatter than the single fibre tests.
- The interfacial properties determined are less in accordance with inferences from bulk mechanical properties than for single fibre tests.
- Blocks of matrix within the indented fibre bundle can fracture and slide with the fibres, reducing the number of sliding interfaces and underestimating frictional effects.
- The unsupported protruding fibre ends can become chipped, especially around the periphery of the push-rod, and consequently they may not behave as intact fibres.
- The radial constraint on one of the fibres within the bundle being pushed is influenced by the fact that neighbours are also being pushed, thus a different data reduction model is required compared to single fibre tests.

6.1.2.5 Slice Compression Test

This test compresses a thin slice of the test material between one hard and one soft anvil, over an area defined by a rigid push-rod [11, 12]. It is assumed that the differential stiffness of the matrix and fibres leads to a differential movement, resulting in the more compliant matrix being compressed more than the fibres which debond and protrude from the surface.

The fibres make indentations in the soft anvil, the depth of which is measured after the test using a scanning electron microscope.

- The principle of the test assumes that the top surface of the test piece is uniform, therefore a good starting surface is required.
- The maximum depression of the matrix relative to the fibres at the peak load is replicated in the soft metal anvil and is measured by a suitable surface profilometry method and compared with permanent changes to the specimen surface.
- A simple model is used to analyse the results, assuming that the shear stress at the debonded interface is constant along the interface length:

$$\tau_i = \frac{R(1-V_f)\sigma_a^2(E_f - E_m)^2}{4\delta_0 E_m E_f E_c}$$

R - fibre radius
V_f - fibre volume fraction
σ_a - applied pressure
E - Young's modulus of matrix (m), fibre (f) and composite (c)
δ_0 - fibre/matrix displacement

- The test requires a very thin slice (a few tens of micrometres thick) of composite.
- High forces are experienced during testing which may influence the lateral elastic constraint, with the unknown influence of spreading of the soft anvil.
- Only the result at peak load is recorded and not the progression of the process.
- The Poisson constraint is lower than in other tests, thus the frictional interface stress may be underestimated.

6.2 CONCLUSIONS

The pull-out test is fraught with a large number of practical difficulties and is seldom used. Consequently, it is necessary to employ compression-type testing to investigate the interface in CMCs. From the various ceramic matrix composite interface tests reviewed, several conclusions can be drawn:

- It is clear that each of the compression test methods imposes different stress conditions on the test sample which differ from those experienced in conventional mechanical testing or in use.
- Differences in the loads required to perform the tests result in different Poisson expansion effects in the fibre, and hence different contributions from radial stresses.
- The thickness of the material, notably the aspect ratio of the fibre being pushed, also plays a role with the effect being worse for multiple rather than single fibre tests.
- The analysis of each test has to be tailored to the explicit test geometry in order to derive appropriate and accurate parameters, ensuring the inclusion of factors such as:
    - test-piece thickness,
    - fibre Poisson effects,
    - lateral composite stiffness,
    - residual thermal stresses,
    - appropriate debond geometry,
    - interface roughness,
    - the load regime in which the tests are performed.
- Results are thus very sensitive to the model used to derive the interface properties.

- Many of the multitude of models available are not valid generally, or have simplifying assumptions associated with the character of the interface.

The test most likely to give consistent results seems to be the thin-section push-through test. Parameters derived from these tests on thin sections could be useful in screening interface behaviour before fabricating large areas of expensive material for mechanical testing.

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7 MODELLING ASPECTS

The properties of the fibre/matrix interface in any fibre reinforced composite material are crucial with regard to the resultant properties of the composite. Various methods of measuring these properties have been developed most of which require the use of a micromechanical model to interpret the results of the experiments in terms of 'interface properties'.

Some of the interface test methods are more suitable for quality control purposes and data obtained from these tests cannot be used for the reliable prediction of composite properties taking account of interface behaviour. The principal reason for this is that, when interpreting the results of measurements to estimate the interfacial 'strength', gross simplifications are made regarding the stress state arising in the interface. In order to achieve the reliable prediction of composite properties from fibre, matrix and interface properties, it is clear that reliable models of interface behaviour must be identified, and that test methods must be used that determine interface properties using these models.

This section will concentrate on identifying suitable models and their association with the various test methods discussed elsewhere in this review.

7.1 DEFINING INTERFACE PROPERTIES

A key problem is defining exactly what is meant by the term 'interface failure'. The following concepts need to be addressed: interface strength, interface fracture energy and surface roughness.

7.1.1 Interface Strength

This is often regarded as being a key parameter characterising the failure of an interface. The term 'strength' implies a critical value of a particular component of stress, but which one?

(i) For a simple composite having a single fibre perfectly bonded to a surrounding hollow cylinder of matrix; increasing the temperature leads to a uniform tensile radial stress being induced at the fibre/matrix interface until, at some temperature, the interface fails. The interfacial normal stress at this point can be regarded as the interface strength in tension.

(ii) In many interfacial tests the loading of the interface is dominated by shear deformation. It is often assumed that, at the point of shear dominated interfacial failure a critical stress known as the interfacial shear strength has been reached. Frequently it is assumed that the shear stress on the interface is uniform, enabling the easy calculation of an 'apparent' interfacial shear strength. For all experimental configurations of interface tests the shear stress will not be uniform, so that an assumption of uniformity will introduce errors and lead to interfacial strengths that may not be relevant in other loading situations.

What happens if the interface is subject to combined tensile and shear loading? This is often the case in interfacial test method configurations and for which there are no easy solutions using strength concepts.

7.1.2 Interfacial Fracture Energy

Interfacial failure can also be characterised by an interfacial fracture energy that quantifies for degree of bonding between fibre and matrix [1]. This concept is ideal for the consideration of the propagation of a debonded zone in the interface due to progressive axial loading, such as occurs in fibre pull-out tests, and fibre fragmentation tests. This approach balances the energy available from the fibre and matrix, accounting for:

- potential energy introduced by the applied loads,
- energy dissipation due to friction at the interface in the debonded region,
- energy required to overcome cohesive interface forces during the creation of new areas of debonded interface.

This technique solves the problem of taking account of the effects of combined tensile and shear loading of the interface. The difficulty is that it is not clear how the initiation of debonding can be considered. One viable approach is to assume that the interface in a composite either has a distribution of small defects in the form of local debonds, or that a stress singularity promotes fibre / matrix debonding near the singularity at very low loads. Such singularities occur, for example, where fibres enter the matrix in pull-out tests, or where fibre fractures occur in fragmentation tests. The initiation of the growth of the defects is governed by fracture mechanics principles. The initial stages of debond growth may be highly unstable so that a significant portion of the interface suddenly debonds followed by arrest. Subsequent debond growth then requires additional loading. Another difficulty is that debond growth involves mixed mode fracture and frictional effects. For mixed mode fracture, the correct fracture criterion to use is unresolved, most are simply interpolations between the limiting cases of pure mode I and mode II without physical justification.

7.1.3 Fibre Surface Roughness

When an interface debonds so that the fibre and matrix remain in contact, there will clearly be frictional shear tractions acting over the debonded part of the interface. The Coulomb friction law has often been used to model such frictional behaviour [2,3], as well as assuming that the interfacial shear stress has a constant value [4]. Particularly for large diameter fibres where interfaces have been tested using push-through tests, the effects of surface roughness have been shown to be important [5].

7.2 DISCUSSION OF TEST CONFIGURATIONS

7.2.1 Microdrop / Microbond Test

The microdrop test (§ 3.1.3) leads to significant difficulties when interpreting the experimental results.

- It is often assumed that the shear stress is uniform along the entire length of the fibre / matrix interface, even when the interface is perfectly bonded.
- The shear stress is actually highly non-uniform and involves a singularity (infinite value) at the point of fibre entry into the microdrop, close to the loading edges.
- The normal stress can be tensile and infinite at the singularity leading to the development of localised interface debonding at low loads.

- Thermal residual stresses may be sufficient to promote debonding at the singularity prior to testing.
- The application of load using knife edges results in a shear stress which is not only non-uniform along the fibre, but also exhibits an angular variation.
- Complex 3-D stress analysis is needed therefore to interpret the results of the test reliably.
- This problem could be overcome by the use of an axisymmetric loading mechanism.
- The separation of the knife edges and the size and shape of the droplet will vary for each test leading to differing stress distributions, and to different failure stresses.

7.2.2 Fragmentation Tests

The geometry (§ 3.1.1, § 5.2.4) and mode of loading are such that the models used to interpret the results can be assumed to be axisymmetric, due to the large volume of matrix used whose external boundary is remote from the interface itself. A great deal of modelling has been devoted to analysing the stress distribution associated with fibre fractures.

- If the interface is very strong and remains perfectly bonded after the fibre has fractured, the interface shear and normal stresses tend to infinity as the fibre fracture is approached.
- The normal stress, very near the fibre fracture, is again often tensile, promoting the debonding of the interface even for very small applied loads.
- As the axial load is increased, the region of debonding increases as the tip of the debond propagates along the fibre / matrix interface away from the fibre fracture.

The major problem encountered with the technique is the uncertainty regarding the nature of contact between fibre and matrix after the interface has debonded.

(i) It is often assumed that the interfacial shear stress in the debonded region is uniform, having a value that can be regarded as a material constant. This does not make sense, as the value of the shear stress is highly likely to be dependent upon the local normal stresses as would occur for frictional effects. The thermal residual stresses can in some situations increase the value of the normal stress acting on the interface because of clamping effects arising from thermal expansion mismatch between fibre and matrix. Another problem is the non-zero interfacial shear stress value predicted at the fibre fracture position, which is physically impossible when it must have a zero value at the broken fibre surface. This situation can only be modelled if a suitable type of singularity is allowed to exist in the interface at the point of fibre fracture. Such an approach does not seem to have been used in the modelling field.

(ii) An alternative modelling method assumes that stress transfer in the debonded region is governed by a friction law that is sometimes modified to take account of surface roughness effects. The advantage of this approach is that it allows the interfacial shear stress to tend to zero as the fibre fracture is approached (so that the fibre fracture surface is stress free as required) and leads to a non-uniform interfacial shear stress that is affected by the degree of fibre clamping arising from thermal expansion mismatch effects. The Coulomb friction law is often assumed, introducing a friction coefficient that characterises interface behaviour. This, however, leads to the problem of determining its value from the results of fragmentation experiments.

As fragmentation tests proceed, the fibre successively fractures at points which are well removed from each other. As the crack density increases the cracks begin to interact, leading to a situation where it becomes more difficult for further fibre fractures to occur (saturation).

Very few models are capable of accurately accounting for such interaction effects. The popular constant interfacial shear stress model is often used to predict the onset of saturation, allowing the interfacial shear strength to be determined from the fracture spacings. The problem with this approach is that the model of stress transfer on which it is based is far too approximate for there to be confidence in the methodology.

7.2.2.1 Fibre Fracture

The fragmentation test is the only interface test that involves fibre fracture. It is useful to consider in more detail the modelling of fibre fracture and its effect on the behaviour of interface debonding. The strength of fibres can be subject to significant scatter (see NPL Report CMMT(A) 95 for review of statistical methods).

Consider an idealised fragmentation test where a single fibre is embedded in matrix material which is assumed to have the form of a cylindrical tube surrounding the fibre which is perfectly bonded to the matrix at all points on the interface. The concentric cylinder system is loaded axially and can be subject to thermal residual stresses. There are two distinct ways of approaching the prediction of the initiation of a fibre fracture which lead to differing estimates for the stress at which a fibre fracture will initiate.

(i) One approach assumes that the fibre/matrix interface remains intact during axial loading, thus at the point of fibre failure the tractions and displacements are continuous at all points on the interface. Immediately following fibre fracture the system responds elastically and an equilibrium stress field arises that can be predicted by solving the elasticity problem. The fibre fracture event is controlled by an energy balance where the energy required to form the crack in the fibre (characterised by a suitable fracture energy) is balanced by an availability of energy arising from a change in the elastic stored energy and the work done by the applied tractions. Using a multiple cylinder representation for the fibre and matrix it is possible to solve the elasticity problem very accurately [6]. A characteristic of the stress distribution is that the stress field is singular at the location of the intersection of the fibre break and the fibre/matrix interface. The radial stress is usually tensile and this will lead directly to the initiation of debonding along the interface, and eventually an equilibrium configuration will be achieved.

(ii) The second approach considers that fibre fracture and interface debonding occur simultaneously, thus the energy availability is calculated from the energy difference between the state of the system just before fracture with the equilibrium state that arises following fibre fracture including the fibre/matrix debonding. When performing such calculations account must be taken of the energy required to debond the fibre/matrix interface and the energy dissipated by friction within the debond zone. The final equilibrium state may be difficult to determine as dynamic effects in the form of stress waves may lead to more than one fibre fracture at a given load, and to stick/slip friction effects occurring in the debonded zones of the interface.

7.2.2.2 Laser Raman Test

The laser Raman testing technique offers an opportunity of measuring the axial strain distributions in fibres having non-uniform axial stress distributions directly. It can distinguish between the perfectly bonded and debonded regions of an interface, and can be used to validate stress transfer models. It has often been used to infer values for the axial stress in the fibre and the shear stress in the interface. The problem with this inference is that the axial stress distribution is calculated from the measured axial strain by making use

of the stress-strain relation for the axial strain in terms of stress. This relation involves transverse stresses, which are not measured by the technique. These terms are usually ignored which is equivalent to assuming that the Poisson's ratios for the fibre are zero. Using this inaccurate value for the axial stress in the fibre, the force balance equation is then used to infer the corresponding interfacial shear stress distribution. These stress distributions are thus subject to unquantified error. A more reliable interpretation technique needs to be developed, based on a more accurate method of analysis.

7.2.3 Pull-out Tests

The fibre pull-out test (§ 3.1.2, § 5.2.1, § 6.1.1) shares most of the features described for fragmentation tests. The same type of stress transfer models are used and the only major difference concerns the nature of the boundary conditions that must be applied. There are, however, two additional problems that arise from the use of the pull-out test leading to difficulties when attempting to interpret the results using an accurate stress transfer model:

- the variability that arises because the fibre direction is not normal to the loading direction leading to bending effects and angular variations in the interfacial shear stress distribution,
- the geometry of the specimen (especially the shape and size of the matrix into which the fibre end is placed, and the embedded length of fibre) will affect the results.

7.2.4 Indentation Tests

This technique (§ 3.1.4, § 5.2.2, § 6.1.2) is a variant of the pull-out test and the same stress transfer models can be used to interpret the experimental measurements.

- It is important that the tip of the indenter is located at the centre of the fibre at the point of first contact so that an axisymmetric stress field results.
- A difficulty is that the sharp tip of the indenter leads to local stress concentrations that will have an effect on the interfacial stress distribution near the point of indentation.
- The prediction of the shear stress distribution requires the use of finite element analysis as the nature of such stress concentrations is beyond the capabilities of analytical modelling.
- Stress concentrations will arise if flat-ended cylindrical punches are used, unless the diameter corresponds to that of the fibre.
- Some indentation tests have been carried out on thin slices of unidirectional composites, having a relatively large fibre diameter (especially for metal and ceramic composites), where the proximity of the free surfaces of the slices leads to a modified stress field.

7.3 CONCLUSIONS

Several important conclusions can be drawn from the review of interface modelling:

- Most methods currently used to interpret the results of interface tests are based on very simplistic stress analysis models that can at best lead only to nominal values for the interface properties. Such interface data cannot be expected to show consistency when comparing results from different types of test. In addition, such data cannot be expected

to be useful when attempting to predict the behaviour of the fibre / matrix interfaces within a typical composite material.

- The fragmentation test is the only technique that involves fibre fracture. The energy released during such fracture processes is such that dynamic effects may be important where stress waves may initiate more than one fracture at a given load, and lead to stick-slip processes within the interface debonding zone.
- Interface performance is best characterised by the consideration of interfacial fracture energies rather than interfacial strength. The issue of mixed mode loading arises and requires thorough investigation as currently used criteria for predicting mixed mode interface failure are empirical.
- Modelling stress transfer during interfacial failure needs to be accurate if the results of the tests are to have potential for use in predictions of behaviour in other situations, e.g. in a composite, or when analysing the results of different types of interface test method. Accurate analysis is best achieved by using axisymmetric uncomplicated test geometries.
- Interfacial behaviour is significantly affected by frictional contact between fibre and matrix in the region of debonding. Currently, there appears to be a problem of determining the correct friction coefficient when using models that are known to be accurate. Surface roughness effects need to be considered when developing models of frictional stress transfer.

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8 GENERAL CONCLUSIONS

To summarise the conclusions reached:

(i) It is clear that when considering the range of materials covered by polymer, metal and ceramic matrix composites, there is no one test method that is ideal nor universally applicable.

(ii) At present no standard test methods exist and published values for interface properties are quite variable. There is even some considerable doubt as to which interface property to measure.

(iii) In all the test methods, it is recognised that the geometry of the test-piece, the testing devices, mode of loading, thermal stresses, etc. can affect the value of the measured interface property. Clearly, this is of special concern in the development of standardised test methods to measure interfacial properties that can be used in predictive models, rather than for quality control purposes. Many of these factors are ignored in the simple analyses usually adopted.

(iv) For PMCs the simple micro-drop test method would appear to be useful only for quality control purposes. The fragmentation test is perhaps the best way forward and this is supported by current international testing activity that is being carried out under the auspices of VAMAS.

(v) For metal and ceramic matrix composites, the fibre push-through / push-back tests appear to be the most satisfactory.

(vi) The analysis methods for interpreting the results of both fragmentation tests and push-down / push-back tests are well documented and researched, although some refinement is still necessary.

(vii) Atomic force microscopy is the most promising method for the investigation of roughness on small, highly curved surfaces such as fibres, with wetting measurements providing basic information on resin / fibre compatibility. DSC and DMTA are the simplest methods for studying chemical interactions at the interface, requiring only small amounts of as-fabricated composite.

The approach to be adopted will concentrate on evaluating and optimising the effects of specimen preparation, specimen geometry, test conditions, data reduction analyses and models for the mechanical, thermal, physico-chemical and modelling aspects of interfaces in composites.

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