Single-Fibre Fragmentation Test for the Characterisation of Interfacial Phenomena in PMCs

The interface between a matrix and reinforcing fibre is defined as the surface common to both constituents, and plays an important role in determining the mechanical behaviour of composites. A strong interface helps to ensure good off-axis properties, delays the onset of microstructural damage formation and reduces the rate of damage accumulation. Weaker interfaces promote interfacial debonding, delay the onset of fibre failure and lead to increased energy absorption. The interface is also pivotal in determining the long-term property retention in aggressive environments, where the resistance of the interface to moisture or other reagents affects composite durability.

This Measurement Note provides an evaluation of the single-fibre fragmentation test for assessing the interface in polymer matrix composites (PMCs). All aspects of the technique are covered, from specimen preparation through testing procedures and test measurements to data handling/analysis. Particular emphasis is placed on the precautions required to achieve consistency and repeatability in both specimen preparation and test results. Similarly, information on the apparatus, measurement requirements and test procedure are covered.

An extensive experimental screening study was carried out and recommendations are made for the optimum specimen parameters/geometry and test protocol. The optimised fragmentation method was subsequently shown to be applicable to the determination of interfacial shear strengths in both glass and carbon fibre systems with different surface treatments and subjected to a hot/wet environment. The role of photoelastic microscopy to supplement the test method and provide complementary qualitative assessments of the interfacial adhesion and failure process was also evaluated.

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INTRODUCTION

Coupon tests that are strongly affected by the interface, e.g. longitudinal compression, flexure and transverse tension, were traditionally used to investigate interfacial properties indirectly. These methods are unable to provide a definitive quantitative value for interfacial adhesion since they do not isolate the mechanical parameters which affect the interface alone from the contributions of the other constituents (fibres and matrix) to the measured strengths.

In order to overcome this limitation, several test geometries employing single fibres were developed. The most common of these methods are:

- single-fibre fragmentation test,
- pull-out test,
- microbond/microdrop test,
- micro-indentation test.

These tests have several benefits compared to PMC coupon tests, including:

- minimised fibre-fibre interactions,
- simplified stress states and modelling,
- minimal material requirements,
- full control of interface loading,
- in-situ monitoring techniques.

Following an initial review of the micromechanical interface test methods [1], several conclusions were drawn:

- no method provides accurate/unequivocal interface property measurement,
- very high scatter in the published data,
- widely differing results between different test methods,
- data reduction methods are heavily oversimplified.

The review also found that, for each method, there was large variability in:

- details of the specimen geometry/dimensions and manufacture,
- test equipment and procedure used,
- parameters monitored/recorded,
- data reduction/analysis methods.

Experimental work concentrated on the single-fibre fragmentation test, which is currently the subject of a VAMAS (the Versailles Project on Advanced Materials and Standards) international round-robin and is summarised in Table 1.

SINGLE-FIBRE FRAGMENTATION

This test consists of a single fibre aligned axially in a dog-bone 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, shown schematically in Figure 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 [2].

The number and/or spacing of fibre breaks are monitored with respect to the load/strain applied to the specimen, either continuously or at intervals, until saturation. At saturation, the positions of fibre breaks or the length of the fragments formed are measured and recorded. A matrix possessing a failure strain three times that of the fibre ensures that saturation is successfully achieved.

![Figure 1 - Axial tensile stress distribution in single-fibre fragments.](image-url)
Analysis of the fragmentation test is complicated because of the different modes of stress transfer and rupture mechanisms operating at the interface such as elastic shear loading, debonding, friction, brittle failure and plastic yielding [3]. However, simple analyses tend to be used which require only the critical fibre fragment length and the strength of the fibre at that length. The critical fibre length, \( l_c \), can then be correlated to the interfacial shear strength, \( \tau_i \), by the equation:

\[
l_c = \frac{d \sigma_f}{2 \tau_i}
\]

where \( \sigma_f \) is the fibre strength at the critical length and \( d \) is the fibre diameter.

The critical length is defined as either the longest length from the experimental distribution of fragments or 4/3 of the average fragment length. In turn, the average fragment length can be defined as the arithmetic mean (monitored length divided by the number of breaks observed within that length) or the median (average value of the individually measured fragment lengths) of the distribution. All other factors being equivalent (fibre and surface treatment, resin, specimen geometry, test method etc), longer fragment lengths and fewer fragments are indicative of a weaker interface.

The test is normally accompanied by bare single fibre tensile strength tests to enable the strength of the fibre at the critical length to be determined. These are performed on long fibre lengths at several gauge-lengths to ascertain the relationship between fibre length and strength. These data are then used to extrapolate to the strengths of the very short fragment lengths achieved at saturation in the fragmentation test, using statistical methods (e.g. Weibull). These may not correspond to an equivalent length of fibre resulting statistically from the fragmentation process. As the fibre in a fragmentation specimen fractures at large flaws and is subsequently repeatedly broken at smaller flaws, the fragments formed comprise non-random material and the associated fragment strength actually increases. As a result, the tensile strength of the critical length is commonly underestimated using this method. In addition, the fibre properties in air may differ to those of the fibre when embedded in resin and uniformly supported along its length.

A variety of failure events are possible when performing the fragmentation test since the balance of competing failure processes may change, depending on the strength and integrity of interfacial adhesion and the properties of the fibre and matrix. These failure modes are:

- **for weak interfaces**: a crack travels down the interface of the fragments on either side of a fibre break,
- **for strong interfaces**: elliptical matrix cracking in the region surrounding a fibre break occurs,
- **for ductile matrices**: matrix yielding may occur at the interface,
- **for brittle matrices**: a fibre fracture may result in catastrophic failure of the specimen.

A photoelastic stress pattern forms around the fibre failure which effectively differentiates failure modes and fibre-matrix interactions in-situ, as shown in Figure 2.

![Figure 2](image_url)
Specimen Geometry and Preparation

The test is likely to be sensitive to minor differences in specimen fabrication thus a detailed, tightly controlled procedure was developed [4] comprising several steps:

(i) producing silicone dog-bone moulds,
(ii) fibre separation and lay-up,
(iii) resin casting and curing,
(iv) specimen polishing.

The silicone moulds are prepared using templates of the same geometry as the specimens required, shown in Figure 3.

Fibres are selected and laid into the moulds, bonded in place at one end. Pre-tensioning is achieved by attaching known weights to paper and bonding this assembly onto the free fibre end, as pictured in Figure 4.

Resin is prepared, poured into the dog-bone moulds to just over fill level and cured. Specimens are then polished using a 3 stage process: 600 grit (meniscus removal), 1200 grit (smoothing) and 1 micron (polish).

<table>
<thead>
<tr>
<th>Table 1 – Single-Fibre Fragmentation Test Characteristics</th>
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<tbody>
<tr>
<td><strong>Advantages</strong></td>
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<tr>
<td>- Simple specimen handling.</td>
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<td>- Large statistical sampling of the interface.</td>
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<td>- Replicates the stress transfer characteristics in real composites.</td>
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<td>- Critical length is sensitive to and reflects changes in the level of fibre-matrix adhesion.</td>
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<tr>
<td>- Energy and fracture mechanics analysis methods being developed which do not require specimen saturation.</td>
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<tr>
<td>- Variety of methods available for observing/analysing failure processes directly:</td>
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<tr>
<td>- acoustic emission,</td>
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<td>- photoelastic microscopy,</td>
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<tr>
<td>- Raman spectroscopy.</td>
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<tr>
<td>- Useful variations on the fragmentation test</td>
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<tr>
<td>provide additional/complementary information:</td>
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<tr>
<td>- coaxial test,</td>
</tr>
<tr>
<td>- multi-fibre test,</td>
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<tr>
<td>- strand test,</td>
</tr>
<tr>
<td>- <em>in-situ</em> fibre strength test.</td>
</tr>
<tr>
<td>- Variety of methods available for observing/analysing failure processes directly:</td>
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<tr>
<td>- Does not allow determination of the coefficient of friction/interface pressure.</td>
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<tr>
<td>- Interfacial shear strength value depends on the</td>
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<tr>
<td>constituent properties.</td>
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<tr>
<td>- Relationship between critical fibre length and average fragment length unknown.</td>
</tr>
</tbody>
</table>

Figure 3 – Dimensions, in mm, of reference fragmentation specimen.
Precautions for specimen preparation:

- Use highly polished mould templates to reduce polishing time and surface defects.
- De-gas silicone rubber mixture to ensure a smooth impression of the templates.
- Handle fibres exclusively at the ends to avoid contaminating the fibre surface and adversely influencing results.
- Only use fibres which are removed intact at full-length to achieve representative fibre lengths and avoid skewing results.
- Apply a pre-load to keep the fibre aligned/correctly positioned and give repeatable fibre tensioning.
- Carefully fill the moulds with resin using a pipette to minimise fibre disturbance and air bubble formation.
- Use a tightly controlled, programmed resin cure cycle with specified heating/cooling rates and dwell times to ensure repeatable matrix properties.
- Remove specimens from mould by bending parallel to fibre axis to minimise fibre fractures.
- Avoid water lubrication during polishing process to reduce moisture contamination of the interface or resin, similarly store specimens in a desiccator.

Test Equipment and Procedure

A screw-driven miniature test machine, controlled using PC software, applies and monitors both load and extension during the tests. This is mounted on the X-Y stage of a microscope enabling the loading frame to be precisely manipulated beneath the objective lens via the stage controls, seen in Figure 5. In this way, the entire gauge-length can be scanned during the test and the fracture process observed in-situ. The microscope is operated in transmitted light mode and configured for photoelastic microscopy with a polariser, analyser and full wave plate. A digital indicator with 1 micron resolution monitors the exact position of the loading frame, enabling lengths and positions within the gauge-length to be determined with the aid of a cross-hair graticule situated in the microscope eyepiece.

Pre-Test Measurements

Once the specimen dimensions are measured, two parallel lines approximately 10-15mm apart are marked onto the central region of the specimen gauge-length using a fine, permanent marker. This is used as a frame of reference to define the monitored gauge-length so only breaks occurring in this area are considered during the test.

The fibre diameter is imaged at three positions within the gauge-length for each specimen. Image analysis software is subsequently used to determine the fibre diameter from these images. The system is calibrated using a reference image e.g. an aluminised 0.8 micron diffraction grating.

Test Protocol

Principally, there are two methods for testing/monitoring fragmentation specimens: incremental step loading and continuous...
constant rate loading, as in Figure 6. In each case, the load/strain and number of fibre breaks, within the monitored gauge-length, are measured periodically. Specimen strain is measured directly by monitoring the two marker lines during the test and recording the distance between them. The test is completed when saturation is reached which is defined as a specified period of extended fragmentation inactivity. Once saturation is achieved the fragment lengths within the monitored gauge-length are measured prior to load removal. Typical test times are: 3 and 0.3 hours for incremental and constant loading, respectively, plus fragment characterisation time.

**Recommendations for testing:**
- Use accurate diameter measurements and measure each fibre/specimen individually to minimise this important source of error.
- Grip faces must minimise both specimen slipping and stress concentrations which can induce premature failure.
- Minimise slippage in the grips by ensuring a balanced gripping load is applied to both sides of an end-tab and at both ends of a specimen.
- Align specimens to the axis of loading by orienting the fibre under the microscope.
- Ensure residual load after tightening is removed and that the zero load monitored length is measured.
- Use photoelastic microscopy to aid in the detection of fibre fractures, disbands, saturation point and the nature of the deformation process.
- Test sufficient specimens to provide a strong statistical base for subsequent calculations, improving both accuracy and interface sampling.

**Evaluation Stage**

An extensive screening study, detailed in Table 2, was carried out. This was aimed at optimising both geometry and test method for the fragmentation technique and considered the effects of the following factors:
- specimen width,
- specimen thickness,
- specimen gauge-length,
- monitored length,
- fibre pre-load level,
- step size and dwell time,
- monotonic loading,
- strain rate.

All specimens were made from Tenax HTA 5131 carbon fibre. A high strain to failure resin, Shell Epikote 828 epoxy (diglycidyl ether of bisphenol-A, DGEBA) with a metaphenylenediamine (mPDA) hardener, was selected to ensure fragment saturation was achieved for all the specimens.

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**Figure 6 – Schematics of incremental (upper) and continuous constant rate (lower) test regimes.**
For the different gauge-length specimens, the test speeds were varied to maintain a nominally constant strain rate. On the longer gauge-length specimens, monitored lengths of 12, 24 and 36 mm were used for fragment length measurements at saturation.

Results and Representation of Data

The data were initially analysed with respect to the stresses at fragmentation onset and saturation, the average (both arithmetic and median) and maximum fragment lengths, and the number of fragments at saturation.

These data showed generally consistent specimen behaviour with few artefacts due to geometry or test method.

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Width (mm)</th>
<th>Thickness (mm)</th>
<th>Gauge Length (mm)</th>
<th>Test Protocol</th>
<th>Pre-load (g)</th>
<th>Monitored Length (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2mm Thick</td>
<td>4</td>
<td>2</td>
<td>25</td>
<td>0.2%ε step, 10 min dwell</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>1mm Thick</td>
<td>4</td>
<td>1</td>
<td>25</td>
<td>0.2%ε step, 10 min dwell</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>Half Step</td>
<td>4</td>
<td>1</td>
<td>25</td>
<td>0.1%ε step, 5 min dwell</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>Quarter Step</td>
<td>4</td>
<td>1</td>
<td>25</td>
<td>0.1%ε step, 2.5 min dwell</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>0.01mm/min</td>
<td>4</td>
<td>1</td>
<td>25</td>
<td>Continuous @ 0.01 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>0.1mm/min</td>
<td>4</td>
<td>1</td>
<td>25</td>
<td>Continuous @ 0.1 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>15mm High Load</td>
<td>4</td>
<td>1</td>
<td>15</td>
<td>Continuous @ 0.06 mm/min</td>
<td>0.8</td>
<td>12</td>
</tr>
<tr>
<td>15mm Low Load</td>
<td>4</td>
<td>1</td>
<td>15</td>
<td>Continuous @ 0.06 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>35mm Long</td>
<td>4</td>
<td>1</td>
<td>35</td>
<td>Continuous @ 0.14 mm/min</td>
<td>0.2</td>
<td>12 &amp; 24</td>
</tr>
<tr>
<td>45mm High Load</td>
<td>4</td>
<td>1</td>
<td>45</td>
<td>Continuous @ 0.18 mm/min</td>
<td>0.8</td>
<td>12, 24 &amp; 36</td>
</tr>
<tr>
<td>45mm Low Load</td>
<td>4</td>
<td>1</td>
<td>45</td>
<td>Continuous @ 0.18 mm/min</td>
<td>0.2</td>
<td>12, 24 &amp; 36</td>
</tr>
<tr>
<td>2mm Wide</td>
<td>2</td>
<td>1</td>
<td>25</td>
<td>Continuous @ 0.1 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>3mm Wide</td>
<td>3</td>
<td>1</td>
<td>25</td>
<td>Continuous @ 0.1 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
<tr>
<td>5mm Wide</td>
<td>5</td>
<td>1</td>
<td>25</td>
<td>Continuous @ 0.1 mm/min</td>
<td>0.2</td>
<td>12</td>
</tr>
</tbody>
</table>

The experimentally recorded data are usually presented as plots of cumulative number of fragments with applied stress/strain or histograms of saturation fragment length distributions, examples of which are shown in Figures 7 and 8.

Figure 7 – Typical fragmentation plot (2mm wide specimens).

Figure 8 – Typical fragment length distribution histograms for individual specimens (2mm wide specimens).

The distributions of fragment lengths can be approximated by a log-normal statistical distribution of the form:

$$y = y_a + Ae^{-\frac{[\ln(x/x_c)]^2}{2w^2}}$$

This is highlighted in Figure 9.
The parameters varied in the screening study had no detectable influence on the mean fragment length, which was approximately 0.376 mm (median) and 0.364 mm (arithmetic). The difference in these two values is mainly due to the difference in specimen strain for the two measurements which can be corrected, as shown in Table 3.

**Table 3 – Effect of Strain Correction on Mean Fragment Lengths**

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Median (mm)</th>
<th>Arithmetic (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measured</td>
<td>Corrected</td>
</tr>
<tr>
<td>2mm Thick</td>
<td>0.398 ± 0.073</td>
<td>0.384 ± 0.070</td>
</tr>
<tr>
<td>1mm Thick</td>
<td>0.397 ± 0.021</td>
<td>0.383 ± 0.020</td>
</tr>
<tr>
<td>Half Step</td>
<td>0.364 ± 0.008</td>
<td>0.352 ± 0.008</td>
</tr>
<tr>
<td>Quarter Step</td>
<td>0.384 ± 0.070</td>
<td>0.371 ± 0.068</td>
</tr>
</tbody>
</table>

From this work several conclusions were drawn for the optimum parameters to be used in a single fibre fragmentation test.

**Recommendations for sample geometry:**

- Use the maximum gauge-length possible to ensure fragment lengths are a statistically representative population (some restrictions due to the greater incidence of premature failure with longer gauge-lengths).
- Thickness and width can be any value greater than \( r E_f / E_m \), where \( r \) is the fibre radius and \( E_f, E_m \) are the fibre and matrix moduli respectively, in order to facilitate handling or accommodate equipment limitations (again some restrictions due to higher incidence of premature failure for thicker or wider specimens).
- Use the greatest fibre pre-load practically possible in order to reduce scatter in the fragmentation results.

**NB.** – Larger volume specimens tend to possess more fabrication defects (i.e. longer, wider and thicker specimens).

**Recommendations for test technique:**

- Conduct constant strain rate experiments to significantly reduce test time, increase accuracy of fragmentation process measurements and eliminate creep effects.
- Use the maximum monitored length possible, situated at least 5 mm away from either end of the gauge-length to minimise effects of local stress concentrations within the observed region, in order to ensure fragments are a statistically representative population.

**Application Stage**

A subsequent study was carried out to demonstrate the validity of the optimised method to determine changes in interfacial properties for a variety of different factors known to affect the nature of the interface:

- fibre type,
- sizing,
- environmental conditioning.

The matrix for all specimens was DGEBA with an mPDA hardener. Specimens were made with Tenax HTA 5131 carbon fibre and Vetrotex E-glass. The fibres/surface treatments investigated were:

- HTA carbon,
- HTA carbon with release agent coating,
- HTA carbon subsequently hot/wet conditioned,
- E-glass with a complete epoxy and polyester compatible sizing (G-162),
- E-glass with a water sizing (G-163),
- E-glass with a water and epoxy compatible silane sizing (G-164),
- E-glass with a water and polyester compatible silane sizing (G-165).
The release agent coating was produced using Tygavac release agent SP441 containing aliphatic hydrocarbons. The post-fabrication conditioning was carried out in steam at 100°C for 24hrs using an autoclave. The small amounts of material involved allowed rapid and uniform environmental ageing; globally, specimens absorbed on average 2-4 wt% of water and exhibited residual swelling strains of 0-46%.

The reference specimen geometry was employed for these tests, Figure 3, with a thickness of 2 mm and a monitored length of 12 mm. The fibre pre-loads were 0.2 and 1.6 g for carbon and glass, respectively.

**Results and Analysis**

All G-164 (E-glass with a water and epoxy compatible silane sizing) specimens failed prematurely, prior to achieving saturation, due to the interface being of sufficient strength that very limited interfacial debonding/cracking occurred. Instead, fibre fractures resulted in large matrix cracks which continued to grow radially with increased loading, eventually causing catastrophic failure of the specimen.

Specimens showed good consistency except the hot/wet conditioned specimens, Figure 10. Here, the matrix plasticising effect of moisture absorption results in a different non-linear response after continued loading.

The fragment length distributions in Figures 11 and 12 reinforce the conclusions from the numerical data. The displacement of the curve into the longer fragment lengths suggests lower interfacial strength, and wider distributions signify irregularity in the surface treatment, either from one fibre to the next or along a single fibre.
Analysis and Interfacial Shear Strengths

A simplified local analysis was adopted to analyse the fragmentation data [5], for the determination of interfacial shear strengths. The longest fragment length from each test, subject to the greatest shear stress, was considered using the load corresponding to the onset of fragmentation. Axial internal stresses due to thermal expansion mismatch and cure shrinkage were taken into account, but radial stresses were ignored. Debonding at the interface, affecting the stress transfer ability, was also included with associated friction over this zone. Debonded lengths were measured from unloaded specimens, under polarised light, using an optical microscope, quoted in Table 6. The ageing/
post-curing of the resin was also included as an important factor, since this would affect both the resin properties and the internal stresses with time. The interfacial strengths thus determined are presented in Figures 15 and 16.

Figure 15 – Interfacial shear strengths for HTA carbon specimens.

Figure 16 – Interfacial shear strengths for E-glass specimens.

Table 6 – Results: Analysis Input Data

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Average Fibre Ø (µm)</th>
<th>Longest Fragment Averages</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Debond (mm)</td>
</tr>
<tr>
<td>HTA</td>
<td>7.2 ± 0.1</td>
<td>0.149 ± 0.028</td>
</tr>
<tr>
<td>HTA + release coating</td>
<td>7.1 ± 0.4</td>
<td>0.421 ± 0.076</td>
</tr>
<tr>
<td>HTA + hot/wet</td>
<td>7.0 ± 0.3</td>
<td>0.167 ± 0.024</td>
</tr>
<tr>
<td>G-162</td>
<td>15.1 ± 1.8</td>
<td>0.324 ± 0.021</td>
</tr>
<tr>
<td>G-163</td>
<td>16.5 ± 1.4</td>
<td>0.209 ± 0.052</td>
</tr>
<tr>
<td>G-165</td>
<td>15.2 ± 0.7</td>
<td>0.208 ± 0.026</td>
</tr>
</tbody>
</table>

indication of the homogeneity of the surface coating or treatment.

Notes:

- Many more specimens need to be tested for low strength interfaces to achieve comparable/sufficient fragment numbers for accurate statistical representation.
- An additional problem can occur when conditioning specimens since there is a tendency for premature fibre fractures to occur during exposure to high temperatures and moisture contents. This complication would need to be dealt with in the analysis of results.

CONCLUDING REMARKS

Following a detailed and comprehensive study into the single-fibre fragmentation test method, it was shown that the method could be successfully employed to discriminate between different levels of interface adhesion. It enables detailed inspection of the failure mode giving a clearer interpretation of interface quality and consistency/homogeneity.

The method is good for quality control and comparative purposes, but is less effective at providing accurate quantitative data for design or predictive models [6].

The method is not suitable for all material systems until a practical energy–based analysis, applicable in the small strain regime, is devised. Currently, high strain to failure resins must be used to guarantee saturation is achieved.
References


Acknowledgements

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