

Project CPD3 - Report 5
INTERFACE CHARACTERISATION AND BEHAVIOUR

**Investigation of PMC Interface Properties Using the
Single-Fibre Fragmentation Technique**

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ABSTRACT

This report 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. Step-by-step procedures are provided to enable test-pieces to be manufactured from constituent materials, with particular emphasis on the precautions required to achieve consistency and repeatability. Similarly, detailed 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 aggressive environments. The role of photoelastic microscopy to supplement the test method and provide complementary qualitative assessments of the interfacial adhesion and failure process was evaluated.

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1 INTRODUCTION

This report describes an experimental evaluation of the single fibre fragmentation test for polymer matrix composites (PMCs). An assessment of the sensitivity of the technique in detecting changes in interface properties due to applied surface preparations and hot/wet environment conditioning is also presented. Details of the specimen manufacture, test equipment, test procedure and monitoring, failure processes, results and analyses are included. The research presented in this report was carried out as part of the DTI funded project "Composites Performance and Design (CPD3) – Interface Characterisation and Behaviour". The project comprised the development and validation of test methods and predictive models for characterising the behaviour of the interfacial region in composite materials.

The report is divided into eight sections (including Section 1, the Introduction). Sections 2 and 3 cover the background of interface testing and the fragmentation test in particular. Section 4 describes in detail the procedure for the reproducible preparation of test-pieces. Section 5 outlines the test equipment and specifies the test protocol employed, including photoelastic monitoring techniques. Sections 6 and 7 report on the findings from the test programme carried out and recommendations based on the experimental work. These sections also consider the materials used, the data generated (both quantitative and qualitative), failure modes and the results of analysis and comparisons. Section 8 summarises the principal conclusions from this work. Additional information of interest is presented in Appendices A to J.

More details on the fragmentation test analysis and interface modelling/prediction for PMCs, carried out at NPL as part of the programme, are also available [1, 2].

2 BACKGROUND

2.1 COMPOSITE INTERFACES

The interface between a matrix and reinforcing fibre plays an important role in determining the mechanical properties and behaviour of composites. A strong interface helps to ensure good off-axis composite properties which are maintained during loading, delays the onset of microstructural damage formation to relatively high loading levels and reduces the rate of damage accumulation. Weaker interfaces promote interfacial debonding, delay the onset of fibre failure and lead to increased energy absorption in the composite during the fracture process. 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 degrading agents affects the composite durability.

The interface between a fibre and matrix is generally defined as the surface common to both constituents and for analysis purposes is considered to be of zero thickness. In reality, various factors contribute to altering the local properties of both surfaces making it more meaningful to refer to the interphase, defined as the region existing between bulk fibre and bulk matrix.

2.2 MICROMECHANICAL INTERFACE TESTS

Traditional PMC coupon tests, such as longitudinal compression, flexure and transverse tension that are most strongly affected by the interface, have been 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 to the measured strengths.

In order to overcome this limitation, several test geometries employing single fibres have been 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 specimen preparation and interface loading,
- availability of in-situ monitoring techniques.

Following an initial review of the micromechanical interface test methods [3], several conclusions were drawn:

- (i) none of the methods provides an accurate and unambiguous interface property measurement,
- (ii) there is very high scatter in the published data,
- (iii) there are large differences between tests due to different specimen geometries, load introduction, stress distributions, thermal and stiffness mismatch, Poisson's and friction effects, cure shrinkage and mixed failure modes,
- (iv) data reduction methods are heavily oversimplified.

In the studies carried out previously and published in the literature, the review also found that for each method there was large variability in:

- (i) details of the specimen geometry/dimensions and manufacture,
- (ii) test equipment and procedure used,
- (iii) parameters monitored/recorded,
- (iv) data reduction/analysis methods.

The fragmentation test was eventually chosen for further study in this programme. The reason for this was primarily one of practicality, which is an important consideration if the method is to be adopted for use in an industrial environment. The fragmentation test allows for a large statistical sampling of the interface due to the long embedded fibre lengths used. The specimen dimensions allow for easy handling and sample manufacture is relatively

straightforward. The tests can be carried out using a standard tensile test machine with minimal ancillary equipment. An additional benefit is that the method replicates the stress transfer characteristics which occur in real composites providing, in theory, useful information on the behaviour of the interface in a manufactured composite component.

The test was investigated in two stages. The first stage was to evaluate several features of the test method, assessing the effects of specimen geometry/manufacture and test procedure on the results obtained. The second stage was to demonstrate the validity of the method to determine changes in interfacial properties for a variety of applied surface coatings and for moisture-conditioned specimens. A VAMAS (The Versailles Project on Advanced Materials and Standards) round-robin on this method is also currently underway which will provide much needed reliability and repeatability information.

3 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, 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 (affected by flaw size, density and distribution) [4].

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.

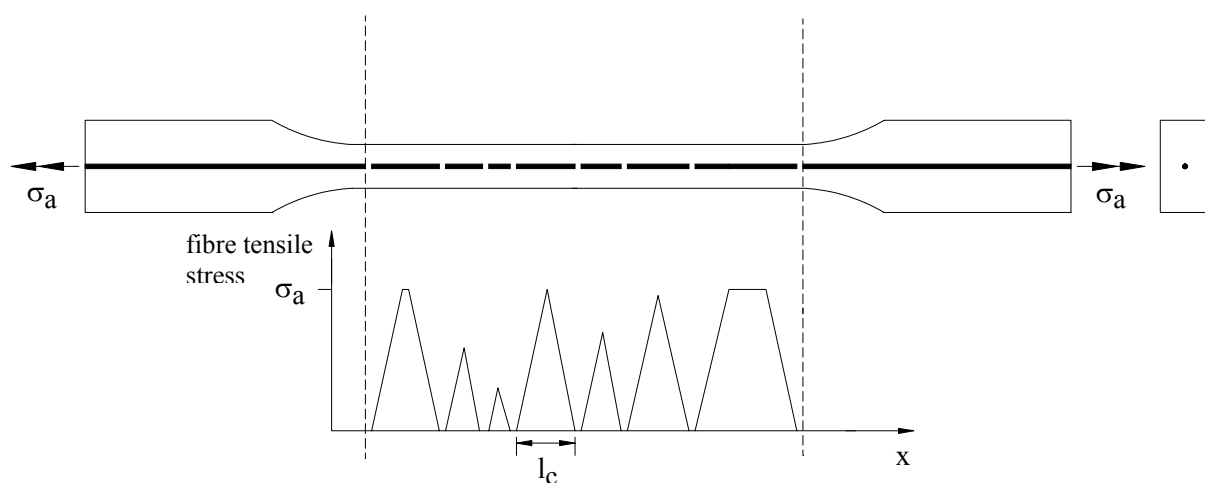


Figure 1 - Fragmentation specimen geometry and associated axial fibre tensile stress distribution of fragments.

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 [5]. All of these factors would need to be included in an accurate, rigorous scheme. However, simple analyses tend to be used which require only the critical fibre fragment length and the strength of the fibre at the critical length. The critical fibre length, l_c , can then be correlated to the interfacial shear strength, τ_i , by the equation:

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

where σ_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 (as final fragments lengths of between $l_c/2$ and l_c can be expected with an average of $3/4 l_c$). 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 experimental fragment length 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 experimentally recorded data are usually presented as plots of cumulative number of fragments with applied stress/strain or as histograms of saturation fragment length distributions. The specimen dimensions used in previous studies vary widely from 43-130 mm long (with gauge-lengths between 15 and 50 mm), 10-25 mm wide (with gauge-widths of between 2 and 10 mm) and 0.2-3 mm thick.

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, using statistical methods.

The single fibres for these additional tests are randomly selected from a tow and contain flaws of all sizes. However, this may not necessarily 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 under-predicted 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.

Instead, the fragmentation test itself may be used to obtain an *in-situ* assessment of embedded fibre strengths at short lengths without the need for extrapolation [6, 7]. However, there is no consensus on the accuracy or effectiveness of this technique.

A variety of failure events are possible when performing the fragmentation test since depending on the strength and integrity of interfacial adhesion and the properties of the fibre and matrix, the balance of competing failure processes may change. These failure modes, described below and shown in Figure 2, are effectively observed and differentiated using photoelastic microscopy:

- for weak interfaces: a crack travels down the interface extending along the fragments on either side of a fibre break,
- for strong interfaces: elliptical matrix cracking in the region surrounding a fibre break occurs which may form at 45° or 90° to the fibre,
 - for ductile matrices: matrix yielding may occur at the interface extending along the fragments on either side of a fibre break with continued loading,
 - for brittle matrices: first fibre fracture may result in catastrophic failure of the specimen.

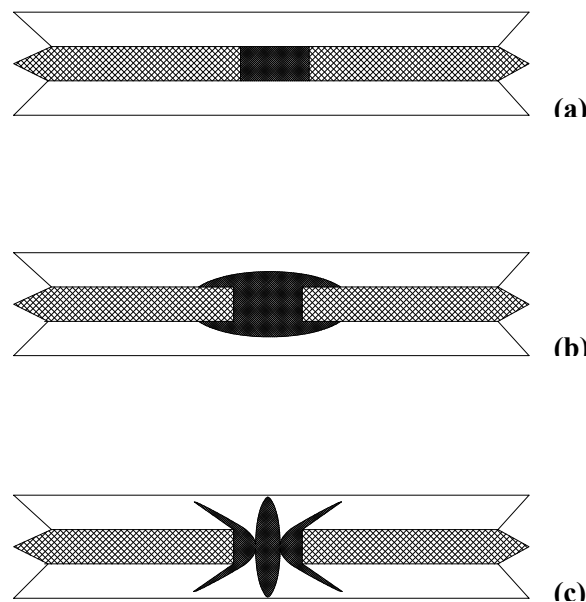


Figure 2 – Schematic diagram of typical fragmentation failure modes associated with fibre fracture in thermoset matrices; (a) frictional debonding, (b) interfacial crack growth and (c) matrix crack growth.

Experimentally observed examples of these principal failure modes are presented in Appendix A.

The strengths and weaknesses of the fragmentation test method are summarised in Table 1.

Table 1 – Summary of the Fragmentation Test Characteristics

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, • <i>in-situ</i> 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.

4 SPECIMEN MANUFACTURE

This is a critical stage in the successful execution of single fibre fragmentation tests. The most important aspect of the specimen preparation is consistency. The test is likely to be sensitive to minor differences in sample fabrication due to the inherent difficulty associated with producing identical samples, contributing to the observed experimental scatter. In order to ensure that differences observed between different batches of specimens were due entirely to the test method or geometry adopted, a tightly controlled specimen fabrication procedure was developed which minimised the handling of the fibres and guaranteed repeatable cure profiles for each set of specimens. The test-piece preparation process is comprised of several steps:

- (i) production of the silicone dogbone moulds,
- (ii) fibre separation and lay-up,
- (iii) resin casting and curing,
- (iv) specimen polishing.

4.1 DIMENSIONS

The basic geometry of the single fibre fragmentation dog-bone specimen employed in this investigation is shown in Figure 3. The variations on this geometry which were investigated can be seen in Appendix B.

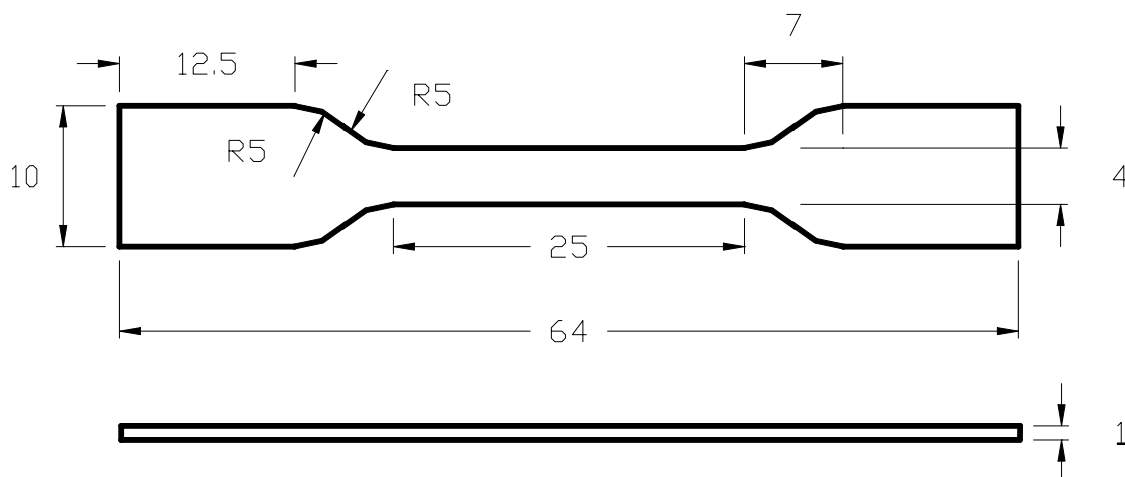


Figure 3 – Dimensions of reference fragmentation specimen, both top view (upper) and side view (lower). All dimensions in mm.

4.2 FABRICATION OF MOULDS

For each of the specimen geometries investigated, a set of five templates of the required dimensions were machined from stainless steel and all faces and edges were polished with a 6 micron diamond polishing compound and a soft cloth. These were subsequently cleaned thoroughly with acetone.

A 5 mm length of 0.3 mm diameter boron fibre was bonded onto the centre point of both end faces of each of the specimen templates, which had been marked with a centre-punch. The prepared templates can be seen in Figure 4. These templates, and surrounding square-section boundary dams for the mould, were bonded onto a flat PTFE sheet using cyanoacrylate adhesive and activator. (NB. A design for an improved multi-specimen steel template is given in Appendix C). The completed assembly was subsequently degassed in a vacuum oven at 100 °C.

The silicone moulding compound (RS medium viscosity, two-part colourless liquid silicone elastomer) was mixed in the ratio of 10 parts base to 1 part of curing agent by weight (approx. 100 g mixture prepared for a single mould). The catalysed mixture was degassed in a disposable 500 ml polypropylene beaker. The compound was then poured slowly into the central part of the prepared mould and degassed again to remove any entrapped air thus ensuring a smooth, defect-free impression of the template surfaces. This was cured, on a levelled surface, at 100 °C for 1 hour and allowed to cool naturally to room temperature.

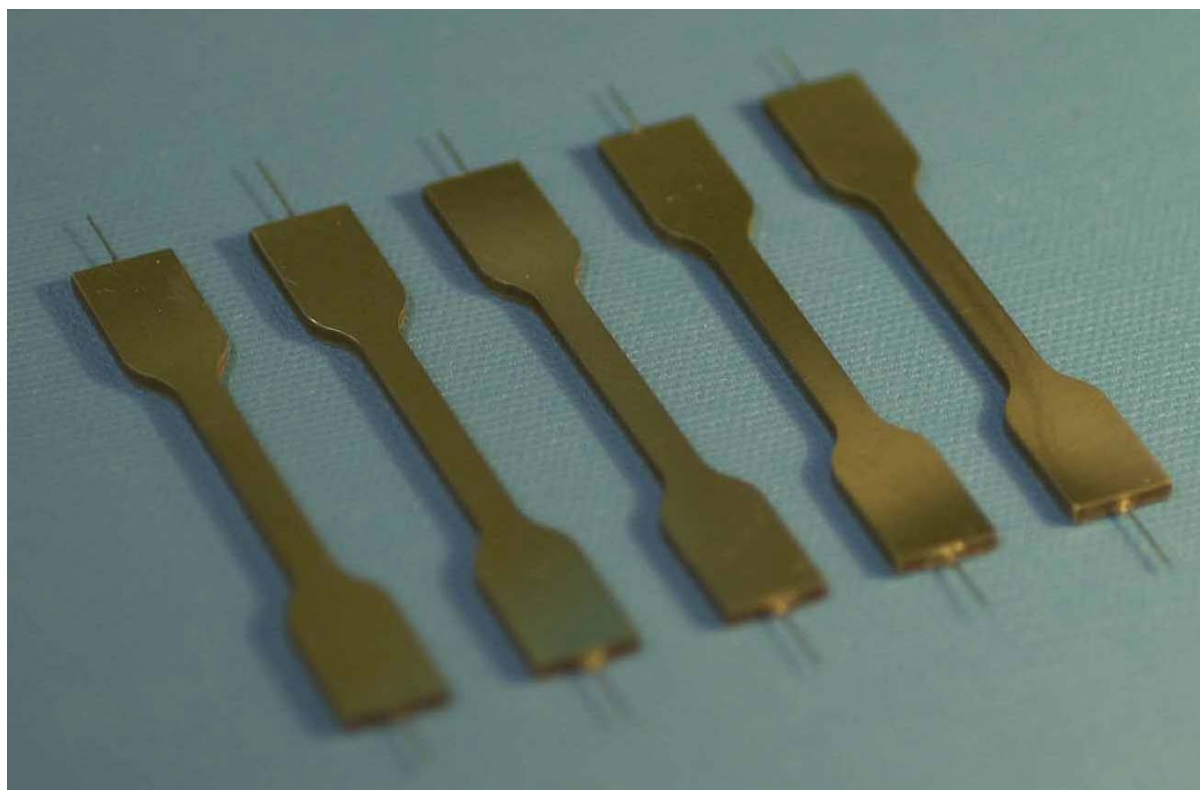


Figure 4 – Metal specimen templates showing details of boron fibre guides.

Once cooled, the silicone mould was carefully peeled away from the templates and the attached boron fibres were removed from the mould. This was achieved by cutting a V-shaped slice through the silicone from the upper side of the mould down to the fibre surface, extending from the cavity to the end, as illustrated in Figure 5. This formed a mid-thickness/mid-width guide along which to lay the test fibres during the next stage. The edges of the moulds were trimmed to remove any residual meniscus and these were then post-cured at 150 °C for 30 minutes. Figure 6 shows an example of the moulds thus produced.

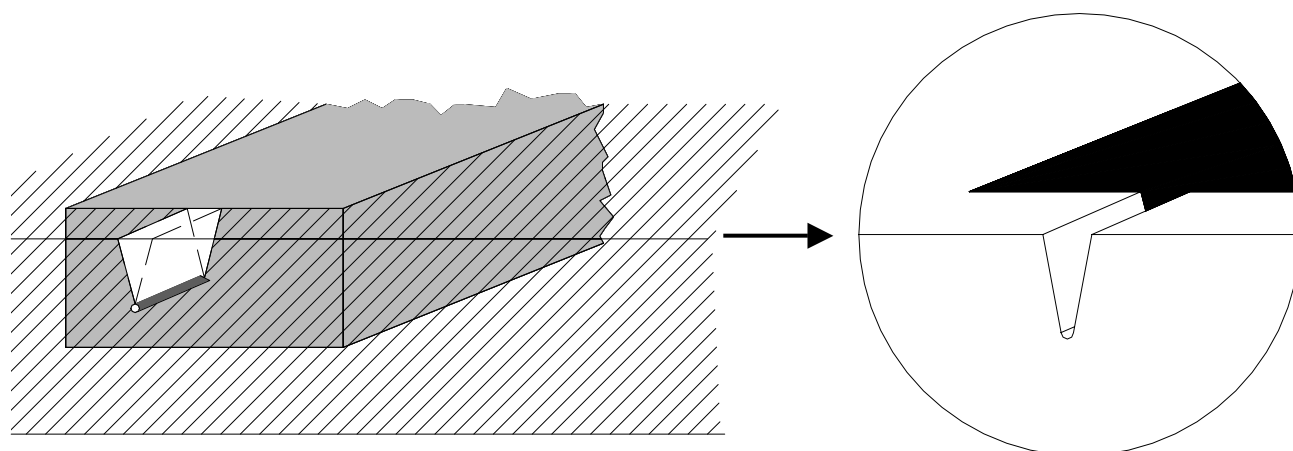


Figure 5 – Formation of fibre guides in silicone moulds showing the cut region (left) and detailed view of the final geometry (right).

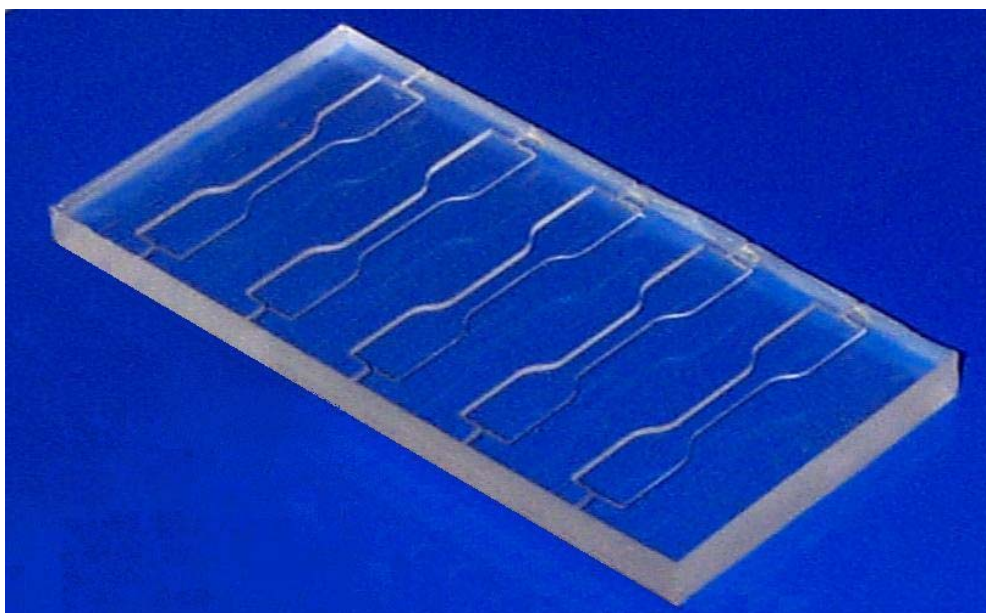


Figure 6 – Silicone mould for fragmentation specimen manufacture.

4.3 FIBRE LAY-UP

The fibres were prepared in a draught-free room with a strong light source. A 150 mm length of fibre tow was laid onto a thick, smooth, clean plate of solid PTFE which minimised fibre surface damage and maximised fibre visibility allowing for easier fibre removal (sufficient contrast was naturally available for carbon fibres; for glass fibres, both ends of the fibre tow were painted using a brightly coloured permanent marker to aid fibre location and separation). This section of tow was repeatedly subdivided into thinner bundles. The ends of these thinned bundles were then spread into “fans” to facilitate fibre separation.

Single fibres were identified and removed by gently pulling between thumb and forefinger whilst holding the other end of the bundle still. Care was taken not to touch the fibres anywhere other than at the ends so as to not to contaminate the fibre surface and adversely influence results. Individual, disentangled fibres were then laid onto another clean plate of PTFE until required. Cleanliness was maintained by securing broken or unwanted fibres/bundles with adhesive tape.

Only fibres which were successfully removed at full-length were used to manufacture specimens. Despite this precaution, fundamental difficulties exist when trying to achieve a representative fibre length since many weaker fibres fail either during the separation or pre-loading stages causing an unavoidable skew in results.

The silicone moulds (moulds were cleaned between uses by rinsing thoroughly with acetone) were placed upon another PTFE plate. A drop of fast setting 2-part epoxy adhesive (Permabond “Double Bubble”) was placed at one end of each of the specimen moulds. The fibre was positioned in the mould, using the V-shaped fibre guide slots for alignment, and one end was immersed in the adhesive drop using fine tipped tweezers to secure it. Care was taken to avoid fibre debris settling in the moulds.

This process was repeated for each of the five specimens in the moulds until they all contained a single intact fibre length. Once the adhesive had cured, pre-tensioning loads were applied. In order to achieve fibre pre-stressing, a split-weight of known mass was clipped onto a small piece of folded paper. This was bonded onto the free end of each fibre using a drop of cyanoacrylate. This construction is shown in Figure 7.

Attaching the pre-loads in this manner reduced fibre failure at this stage by allowing load to be applied over a longer fibre length and avoiding direct clamping of the split-weight onto the fibre. The application of a pre-load also helped to keep the fibre aligned and correctly positioned (away from the mould surfaces). Excess fibre lengths were trimmed and the fibres were checked for straightness and integrity. Where necessary, fibres were teased away from the bottom/sides of the mould.

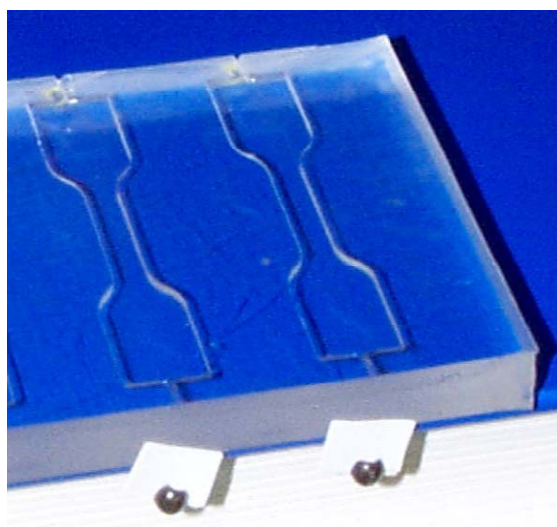


Figure 7 – Detail of the laying-up process; fibres bonded onto mould at one end and pre-loads attached to paper carrier bonded onto fibre at the other.

4.4 RESIN PREPARATION AND CURING

All specimen batches were prepared identically using specified heating and cooling rates and defined hold times to eliminate this potential source of variation.

Whilst the matrix resin was prepared, the fibre/mould assemblies were pre-heated on a levelled shelf in a programmable oven at 70-75 °C using dry air circulation. A high strain to failure resin was chosen to ensure fragment saturation was achieved for all the specimens. Shell Epikote 828 (diglycidyl ether of bisphenol-A epoxy, DGEBA) and a meta phenylene diamine (mPDA) hardener were selected for this purpose (this selection maintained consistency with the VAMAS round-robin study). Due to the toxic nature of the reagents, safety requirements included: goggles, gloves, laboratory coat (preferably chemical resistant), face mask (low organic removal) and fume extraction unit.

The epoxy resin (approx. 100 g prepared per batch) and 14.5 weight% of mPDA crystals were added to separate, clean, disposable 500 ml polypropylene beakers. These reagents were melted at 70 °C whilst being stirred periodically. Once this had been achieved, the epoxy resin was stirred into the liquid mPDA. The contents of this beaker were transferred back to

the resin beaker and again blended thoroughly to achieve homogeneity. This mixture was then placed into a vacuum oven pre-heated to 70 °C for degassing. Debubbling was continued until bubbles at the surface of the mixture had dissipated, typically no more than 10 minutes. At this point the chamber was vented and the mixture removed.

The hot mixture was pipetted into the pre-heated moulds, starting at one end-tab region and moving down the gauge length to the other end-tab, keeping the tip of the pipette close to the mould. This minimised fibre disturbance and air entrapment during the casting process. The resin was subsequently allowed to settle and spread to fill the mould for a couple of minutes. Extra resin was added as required to yield a fill level just higher than the top surface of the mould.

After all the moulds had been filled in this manner, the cure cycle programme was run. The temperature of the oven was ramped at 3 °C/min to 75 °C and held for 2 hours. This was followed by a second ramp of 3 °C/min to 125 °C and another 2-hour dwell at this temperature. At the conclusion of the cure cycle, the specimens were cooled to 20 °C at 1 °C/min. All temperatures were maintained to a tolerance of ± 1 °C to ensure repeatability of the curing process and the final cured resin properties. A typical cure temperature profile is given in Appendix D.

Specimens were carefully removed from the moulds by bending the mould parallel to the fibre axis in order to avoid fibre breakage. These were checked for visible defects caused by poor mould filling and also under a microscope for fibre misalignment and fractures. Any damaged/defective specimens were discarded. Specimens were then stored with desiccant until needed.

4.5 SPECIMEN POLISHING

Finally, the specimens were flattened and polished using a 3-step process to obtain uniform rectangular cross-sections. All operations were performed without water lubrication. A large composite block was coated in a thin layer of silicone rubber and cured. This was used for holding specimens firmly in place during the sanding/polishing process without the need for adhesive tape. Two strips of hardened steel of the required final specimen thickness were used as low abrasion guides at either end of the silicone-coated support block. Specimens were placed in batches on the support block, between the steel strips, with the meniscus side up.

The first sanding stage was performed using 600 grit silicon carbide paper. Specimens were abraded until the meniscus had been removed. This process was performed intermittently to avoid excessive heat build-up in the specimens. The second stage was to smooth the specimens using 1200 grit silicon carbide paper. The specimen edges along the gauge-length were similarly smoothed to remove nicks which could induce premature failure. Lastly, specimens were polished on both sides using 1-micron diamond/oil suspension spray (Buehler Metadi diamond compound) on a medium nap polishing cloth attached to a rotating wheel (150 rpm). Once completed, specimens were wiped with a clean, lint-free tissue dampened with acetone. Specimens were individually labelled and stored with desiccant.

5 TEST EQUIPMENT AND PROCEDURE

5.1 FIBRE DIAMETER MEASUREMENT

Prior to testing, the fibre diameter was measured for each specimen. An Olympus BH reflected light microscope equipped with a 20x magnification objective lens, a 7.5x high power photo lens and a JVC 3-CCD colour camera was used to capture a double resolution, bright field image of the fibre at the centre of the gauge length of the specimen. The specimens were placed on a mirrored surface to maximise the image contrast. Typical examples are shown in Figure 8.

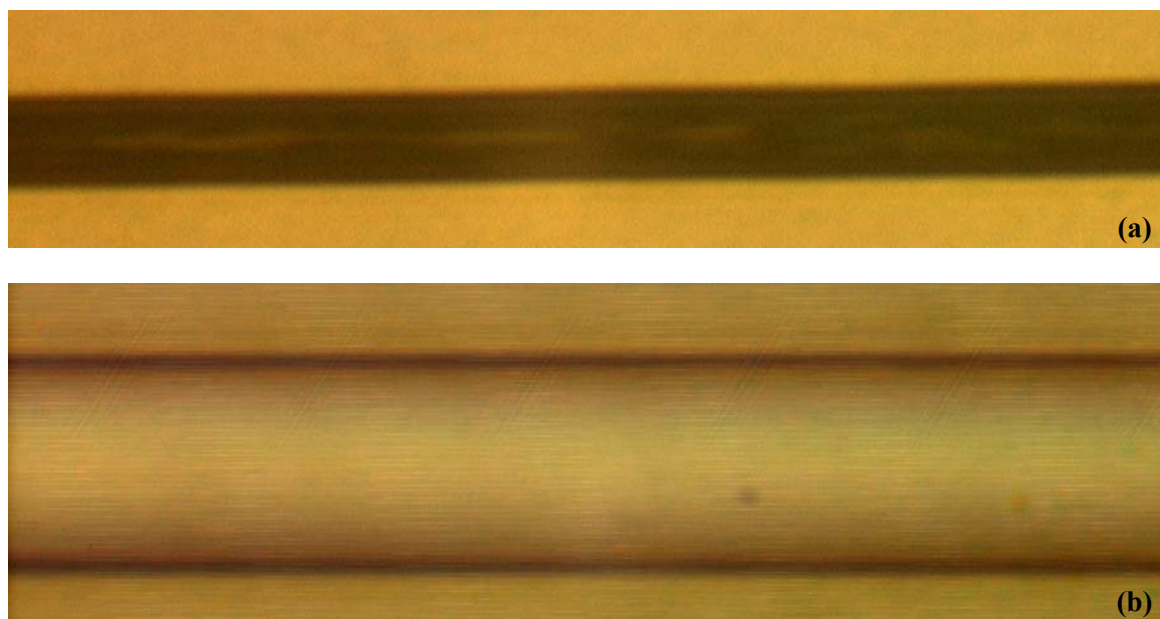


Figure 8 – Typical fibre images (a) HTA carbon ($\sim \varnothing 7 \mu\text{m}$) and (b) E-glass ($\sim \varnothing 17 \mu\text{m}$).

Image Pro Analysis software was subsequently used to determine the fibre diameter from these images. In order to calibrate the system, the same microscope set-up was used to capture an image of an aluminised 0.8 micron diffraction grating (accurate to within 2%) which was placed on the stage at a slight incline to provide off-axis illumination. This calibration image is shown in Figure 9.

75 periodic spacings of the diffraction grating image were marked on the monitor and set to 60 microns in length using the image analysis software measurement calibration function. This set the pixel resolution to 0.062 micron. The uncertainty in the calibration was estimated by making 15 separate measurements on the diffraction grating image over a distance of 10 periodic spacings, i.e. 8 microns. This process gave an average value of 8.013 microns with a standard deviation of 0.042 micron.

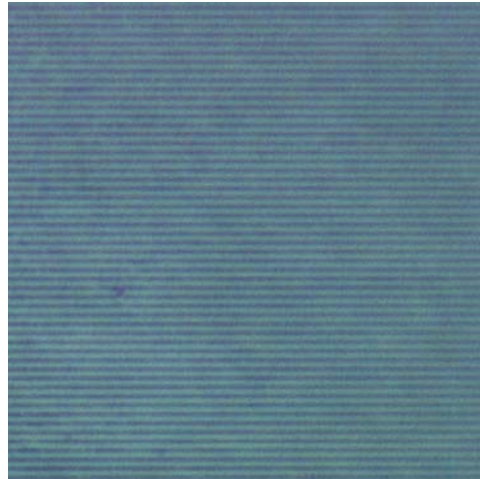


Figure 9 – Calibration image of 0.8 micron diffraction grating.

For each of the sample images taken, the diameter was measured at 3 positions and an average value was calculated. Despite this accurate and careful dimensioning, in practice the difficulties associated with achieving a sharp image of the fibre through light-diffusing resin, diffraction at the fibre edges and the quality of the polishing can be expected to have a large impact on the measurement uncertainty.

5.2 TESTING APPARATUS

A Minimat (Rheometric Scientific) screw-driven miniature test machine, controlled using PC software, was used to apply and monitor load and extension during the tests. This was mounted on the X-Y stage of a microscope (Leica DMLM) operated in transmitted light mode enabling the loading frame to be precisely manipulated beneath the objective lens via the stage controls. In this way, the entire gauge length could be scanned during the test and the fracture process observed *in-situ*. The assembly used in these studies is shown in Figures 10 and 11.

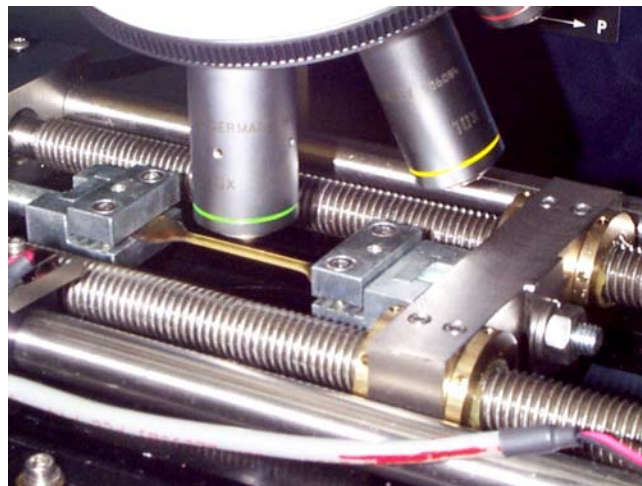


Figure 10 – Detailed view of specimen loading arrangement.

The microscope was configured with a polariser and full wave (λ) plate positioned below the specimen and a 180° rotatable analyser located above it. Objective lenses of 5 - 20x magnification and 10x magnification eyepieces were used to observe the specimen throughout the test.

A digital indicator (Mitutoyo) with 1 micron resolution monitored 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.

A 1 kN load beam was used with a resolution of 1 N and grip displacement was measurable to 1 micron. A spring of known elastic constant (determined from tests conducted using calibrated Instron load cell/machine) was used to calibrate the load beam on the Minimat and a calibrated static mass was subsequently used for further verification. The accuracy of the digital indicator and its alignment with respect to the loading frame was confirmed by measurement of a 1 mm slide micrometer with 0.01 mm divisions. This was measured repeatedly to an accuracy of ± 1 micron.



Figure 11 – Single fibre fragmentation test facility – global view.

The grip design is shown in Figure 12. The grip faces need to minimise both specimen slipping and stress concentrations which can induce premature failure, thus these may have an effect on the results of the experiment. In this instance, the grip faces are square notched to a depth of 0.5 mm. The ridges are 1.5 mm wide with a 1.5 mm spacing. The top plate is tightened onto the specimen with two M3 socket head screws spaced 15 mm apart. The gripped length is approximately 11 mm. Slippage was minimised by ensuring a balanced gripping load was applied to both sides of an end-tab and at both end-tabs of a specimen.

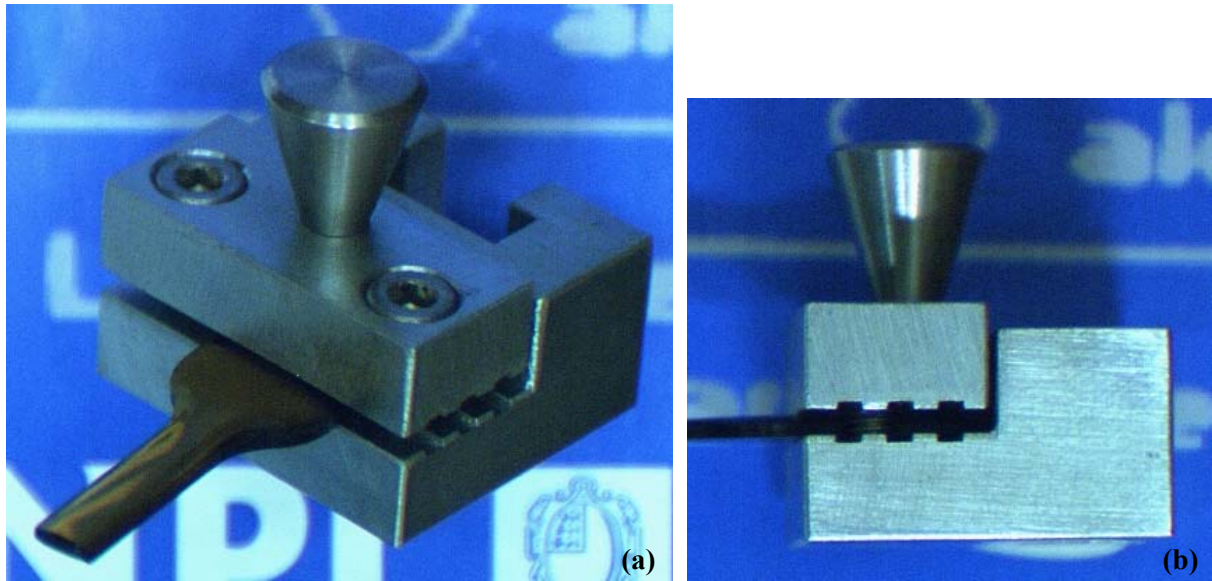


Figure 12 – Grip design: (a) global view, and (b) side view showing detail of gripping surfaces.

5.3 TEST PROTOCOL

All specimens were inserted into the rig in the same orientation and fragments were always measured from the same edge to enable checks to be carried out post-test if necessary. Five specimens were tested from each batch to provide a strong statistical base for subsequent calculations.

5.3.1 Measurements Before Testing

The thickness and width of the specimens was measured at three points within the gauge-length using a calibrated digital micrometer (resolution 1 micron). Averages were calculated and recorded.

5.3.1.1 Gauge-Length and Strain Measurement

Two parallel lines, approximately 12 mm apart were marked onto the central region of the specimen gauge-length using a fine, permanent felt tip pen. This was used as a frame of reference to define the monitored gauge-length, since one end of the sample is held fixed and the other end is pulled to introduce load causing the gauge-length to shift as well as stretch during the experiment. Thus only breaks occurring in this area were considered during the test.

Specimen strain was measured by monitoring the position of the two markers at each step of the experiment and recording the distance between them. Direct strain measurements are necessary since grip displacements do not adequately characterise strain in the sample especially if the specimen slips during the test. Changing the focus on the microscope from the marks to the embedded fibre enabled both strain to be measured and breaks occurring within these limits during fragmentation to be monitored.

5.3.1.2 Zero Load/Stress and Zero Strain Measurement

Specimens were placed within the loosened grips and aligned to the axis of loading by orienting the fibre length under the microscope. This ensures minimal error due to off-axis measurements for strain or fragment length. The distance between the two markers was measured and recorded. The specimen was carefully clamped by tightening the two M3 socket head screws, avoiding disturbance of the specimen or unbalanced clamping. Excess load was removed from the specimen, as monitored by the load cell, and the monitored gauge-length measured again. Similar readings at these two stages ensure no additional stress/strain has been introduced into the specimen. The colour of the matrix, as observed under polarised light, was monitored as independent confirmation of load removal.

5.3.2 Loading Regimes and Measurements

Principally, there are two methods for testing/monitoring fragmentation specimens: incremental step loading and continuous constant rate loading.

5.3.2.1 Step Loading

In this procedure, the specimen was loaded to 0.2 % strain within a couple of seconds and held at this position for 10 minutes. After 8 minutes, the monitored gauge-length was scanned and the number of breaks measured. Also at this point, the separation of the two markers was measured and the actual strain step was calculated and recorded. This process is shown diagrammatically in Figure 13. 10 minutes after the initial strain step, the process was repeated at 0.2% strain increments. Saturation was defined as the point at which during 3 or more successive cycles (i.e. totalling a minimum of 0.6% strain) no additional fibre breaks within the monitored gauge-length were observed. Once saturation was achieved the fragment lengths within the monitored gauge-length were measured before load was removed.

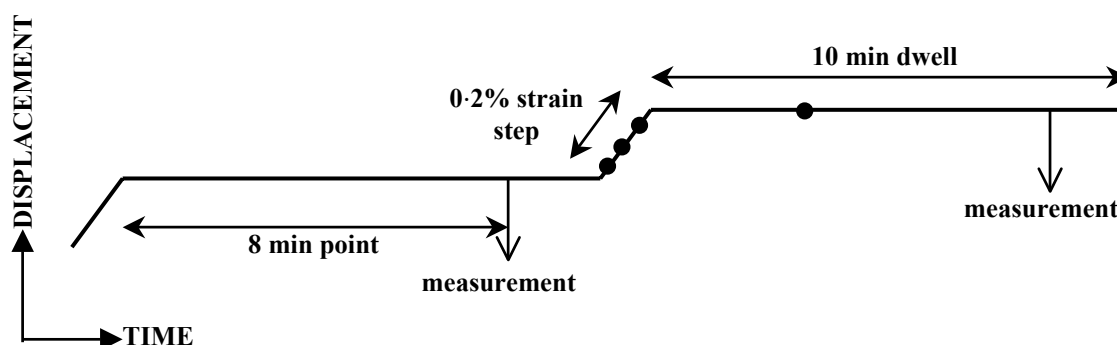


Figure 13 – Diagram showing the step loading regime (dots represent fibre breaks).

The control software was programmed to run multiple two-stage tests. The first stage was displacement rate controlled at 3 mm/min to a pre-specified displacement chosen between 0.03 and 0.15 mm. The actual strain achieved in each step was used to adjust the subsequent step to be as close to 0.2% as possible. Invariably the first step needed to be 30% higher on average than later steps due to slack being taken up in the system. The second stage was set to hold the displacement constant for 10 minutes. Most fractures occur in rapid succession within the straining step at the start of each cycle and are difficult to monitor individually; creep during the dwell time causes a small number of additional fractures. The load and displacement in the specimen were monitored throughout and these data were stored for each loading cycle. Typical test time was 3 hours plus fragment characterisation time.

5.3.2.2 Continuous Loading

In this procedure, the specimen was loaded at a constant displacement rate (nominal strain rate of 0.2%/min). After suitable load intervals (every 5 - 10 N), the gauge-length was scanned and the separation of the two markers was measured and recorded for the determination of true strain. This process is shown schematically in Figure 14. Once fragmentation began, each fracture was recorded as a function of test time (actual strain was no longer recorded after this point but crosshead displacement enabled global strain of the specimen to be determined throughout the test). This enabled the load at each fibre break to be determined. Saturation was defined as the point at which during 3 or more minutes (i.e. equivalent 0.6% strain increment as for the step-wise process) that no additional fibre breaks were observed. Once saturation was achieved the fragment lengths within the monitored gauge-length were measured before load was removed.

The control software was programmed to run a single one-stage test, displacement rate controlled at 0.1 mm/min. Fractures occur continually from fracture onset to saturation, allowing the progression of fractures to be monitored individually. The load and displacement in the specimen were monitored throughout each test and these data were stored. Typical test time was 0.3 hours plus fragment characterisation time.

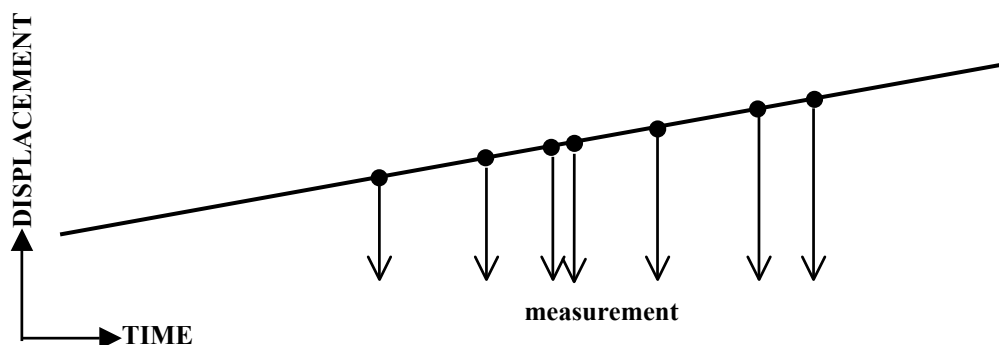


Figure 14 – Diagram showing the continuous loading regime (dots represent fibre breaks).

5.3.3 Determining Location of Fibre Breaks

The fibre breaks were determined by microscopic inspection at magnifications of 200x using polarised light.

In glass fibres the breaks are clearly defined with a dark region marking the location of a fibre fracture. This is shown in Figure 15. This is accompanied by an associated photoelastic stress pattern when viewed under polarised light.

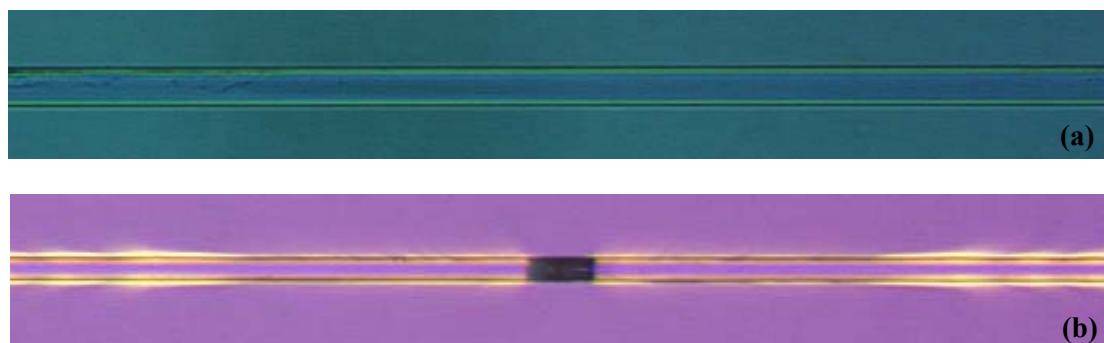


Figure 15 – Glass fibre images using polarised light showing (a) intact fibre and (b) distinctive dark band characteristic of a fibre fracture.

For carbon fibres it is sometimes difficult to locate breaks without the aid of the photoelastic stress patterns they create. However, bright spots (pinholes of light) often accompany a break, indicating a gap between the two fragments of fibre. This is confirmed by the growth of the bright spots which form bright lines at the position of the break with continued loading. This phenomenon is shown in Figure 16.

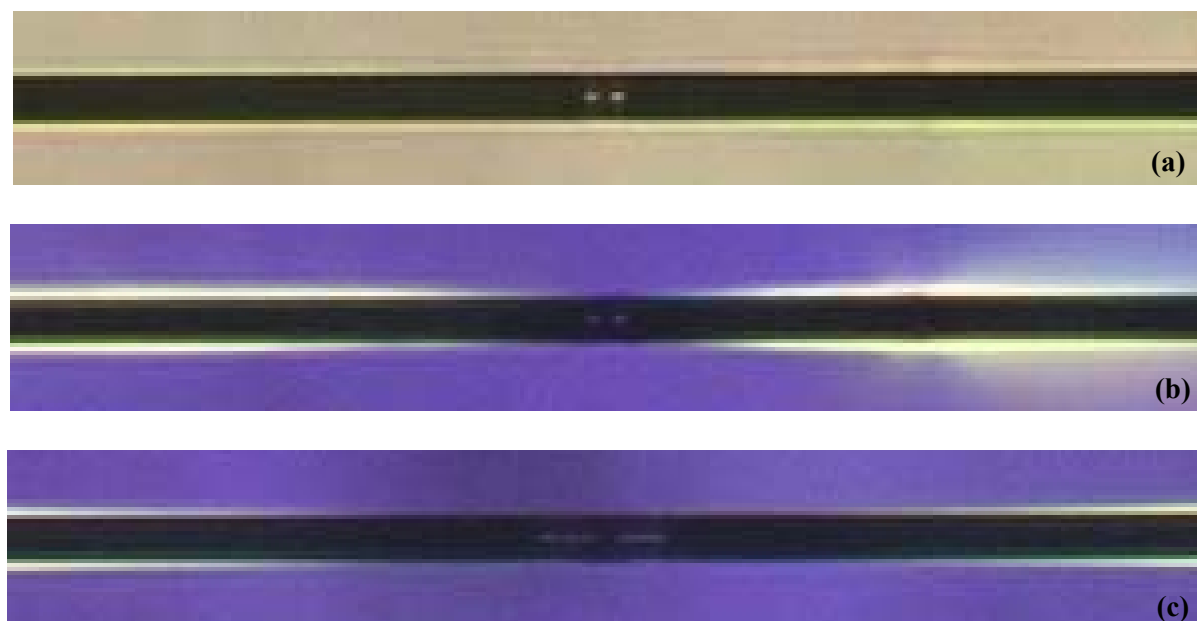


Figure 16 – Carbon fibre images showing (a) fibre break, (b) the same fibre break viewed under polarised light and (c) after further loading.

5.3.4 Fragment Length Measurements

At saturation, the fragment lengths were measured. A digital indicator was used to perform the measurements in preference to an eyepiece graticule. This provided an accurate fragment length measurement, which was independent of the microscope magnification used to observe the specimen. This also ensured there was no limit to the magnification required to make the measurements since the whole fragment did not need to be within the objective's field of view.

The stage X-Y controls were used to position the first fibre break within the monitored gauge-length (starting from the left-hand marker) under the eyepiece crosshair. The digital indicator was zeroed at this position. The adjacent break was then positioned under the crosshair and the indicator reading was recorded. This was repeated for each break until the second marker was reached. Gaps present between the two fibre ends at a break, caused by the relaxation of stress on the fibre as debond growth occurs, were ignored. Thus measurements were made from the original break positions, not from the ends of the fibre fragments.

In the same way, the position of fibre breaks during the test was monitored to study the progression of fragmentation with increasing stress/strain for some specimens. This also enabled lengths to be determined from the relative positions of breaks. The adjacent breaks were positioned under the crosshair, but the indicator was not zeroed between them, such that an absolute position was recorded for each break from one edge of the monitored region.

5.3.5 Test Automation

Both the test processes have potential for automation. The stepped process could be run using control software on a loop, with timed triggers used to capture information at the specified points during the dwell stage. The continuous method would be simpler to set-up with load or displacement triggered capture events. A motorised microscope stage and image capture/analysis system could be used to record the progression of fragmentation over the monitored length and then analysed post-test. Alternatively, an acoustic emission method can be employed which identifies and locates fibre breaks as a function of test time, without the need for operator intervention during the test. This allows fragment lengths to be evaluated and saturation to be defined as an extended period of acoustic inactivity. However, this method has problems with external noise, and event definition as well as access to the specimen surface.

5.4 PHOTOELASTICITY

Many polymeric matrices when subjected to stress become birefringent and if sufficiently transparent can be studied with polarised light. Three regions can be identified: the fibre break, interface debond propagation zone and regions of elastic stress transfer between resin and fibre. Initially, colour cycling is observed in the matrix which disappears at high strain levels. The high strains present at the fibre ends after a fracture produce bright white ellipsoidal regions, changes in which are used to elucidate the nature of the deformation process.

The detection of fractures and disbonds can be enhanced by viewing the specimen through polarisers which are close to extinction. A full wave plate also assists in the observation process. Due to the birefringence of the matrix a photoelastic stress pattern will form around the fibre failure. This stress pattern can be used as an aid in distinguishing and evaluating fibre-matrix deformation processes *in-situ* as the test proceeds and offering insight to fibre-matrix interactions as the stress pattern grows along the fibre fragment away from the point of fracture with increasing load. The photoelastic pattern can also assist in the assessment of whether saturation has been achieved (see Appendix E).

A colour digital camera (JVC 3-CCD) was mounted onto the microscope with a 0.5 - 1x magnification C-mount. Images of the breaks and photoelastic stress distributions were taken to document representative failures at multiple intervals during the test. A typical example of this can be seen in Figure 17.

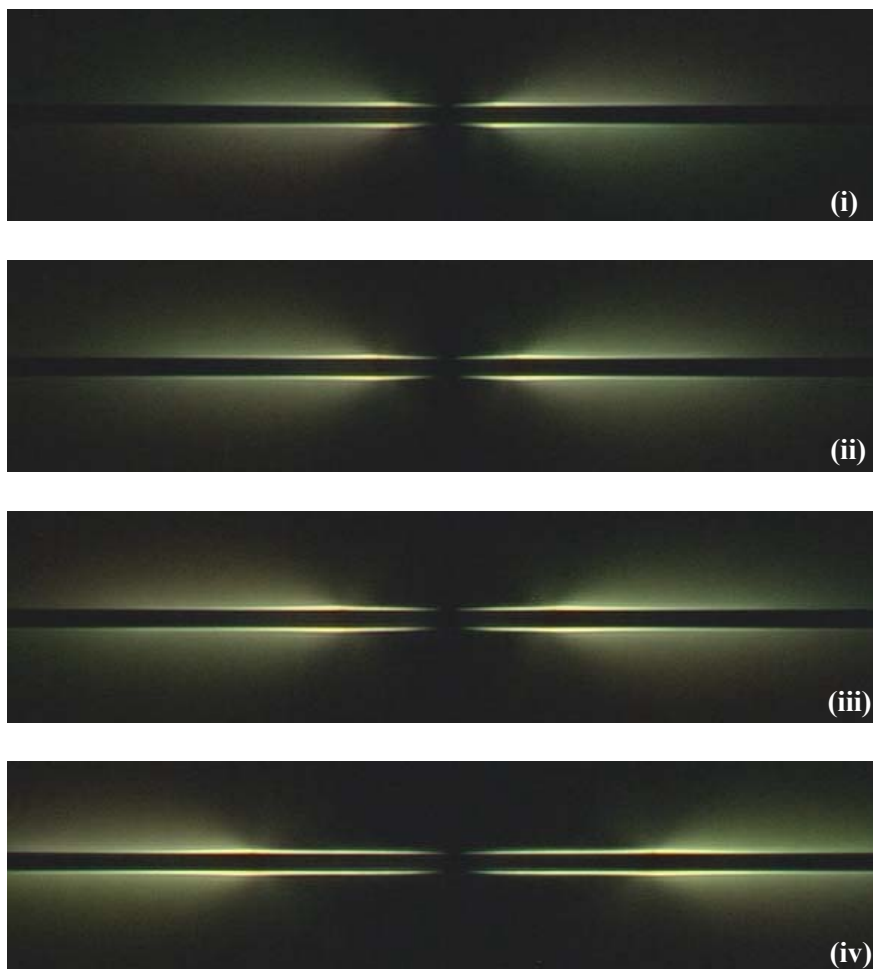


Figure 17 – Images detailing damage progression in a single carbon break under full extinction with increasing strain; (i) ~1.5%, (ii) ~1.7%, (iii) ~1.9% and (iv) ~2.2%.

Qualitative differences can be observed in the photoelastic stress pattern in the region immediately adjacent to a fibre break, resulting from changes in the mechanism of stress transfer or failure at the interface. Indicators of common failure modes are given in Figure 18.

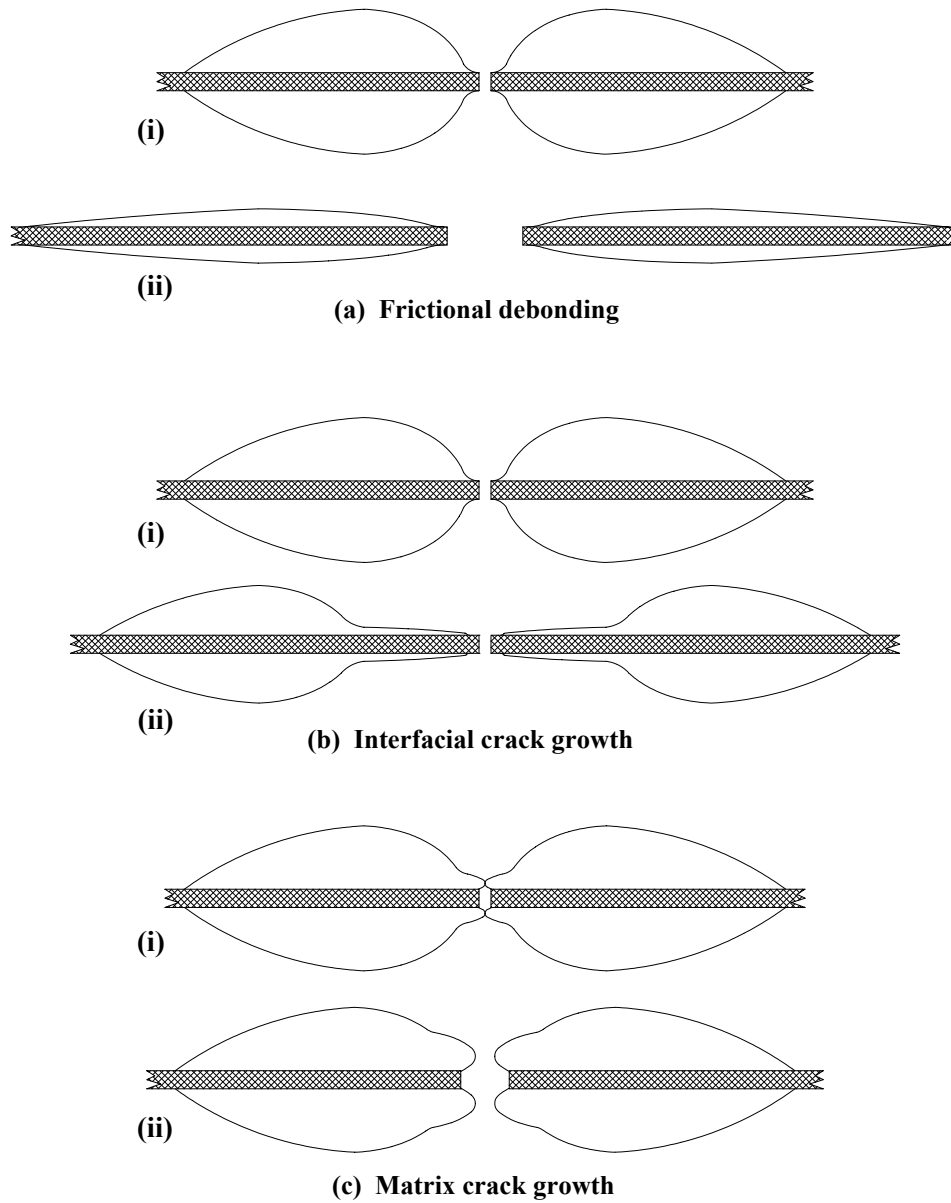


Figure 18 – Schematic diagrams of photoelastic patterns characteristic of different failure modes following fibre fracture; (i) initial intensity distribution and (ii) intensity distribution with continued loading.

6 EVALUATION STAGE

An extensive screening study was carried out, aimed at optimising both geometry and test method for the fragmentation technique, which 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.

6.1 MATERIALS AND TESTS

All specimens were made from Tenax HTA 5131 carbon fibre and Epikote 828 epoxy (Shell Chemicals Europe) with mPDA hardener (Fluka, >99% purity in white crystalline form stored in dark and under desiccant) using the procedure described in Section 4. Details of the fragmentation tests performed are given in Table 2 below.

Table 2 – Summary of the Experimental Parameters used in the Screening Design

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

Specimen widths between 2 and 5 mm, specimen gauge-lengths between 15 and 45 mm and thicknesses of 1 and 2 mm were investigated (see also Appendix B). A principal monitored length of 12 mm was used for strain, break counts and fragment length measurements. In addition, where possible, on the longer gauge-length specimens monitored lengths of 24 and

36 mm were used for fragment length measurements at saturation. Fibre pre-loads of either 0.2 or 0.8 g were employed during specimen manufacture.

Both continuous and step loading tests were conducted by varying the step size (0.1 and 0.2% strain steps), the dwell time (2.5, 5 and 10 minutes) and the crosshead displacement rate (0.01 and 0.1 mm/min). For the different gauge-length specimens, the test rates were varied to give a nominally constant strain rate of 0.2% /min as shown in Table 3. The initial grip separation required to accommodate the different specimens is also presented here.

Table 3 – Additional Experimental Parameters used in the Screening Design.

Specimen Type	Gauge Length (mm)	Test Protocol	Nominal Strain Rate (%/min)	Initial Grip Separation (mm)
2mm Thick	25	0.2% ϵ step, 10 min dwell	0.02	45
1mm Thick	25	0.2% ϵ step, 10 min dwell	0.02	45
Half Step	25	0.1% ϵ step, 5 min dwell	0.02	45
Quarter Step	25	0.1% ϵ step, 2.5 min dwell	0.04	45
0.01mm/min	25	Continuous @ 0.01 mm/min	0.02	45
0.1mm/min	25	Continuous @ 0.1 mm/min	0.2	45
15mm High Load	15	Continuous @ 0.06 mm/min	0.2	35
15mm Low Load	15	Continuous @ 0.06 mm/min	0.2	35
35mm Long	35	Continuous @ 0.14 mm/min	0.2	55
45mm High Load	45	Continuous @ 0.18 mm/min	0.2	65
45mm Low Load	45	Continuous @ 0.18 mm/min	0.2	65
2mm Wide	25	Continuous @ 0.1 mm/min	0.2	45
3mm Wide	25	Continuous @ 0.1 mm/min	0.2	45
5mm Wide	25	Continuous @ 0.1 mm/min	0.2	45

6.2 RESULTS AND DISCUSSION

The results from the screening stage were evaluated for consistent global and fragmentation behaviour which are detailed in the following.

6.2.1 Comparison of Specimen Stress/Strain Behaviour

For specimens tested using the continuous method, the global/apparent strain of the specimen was recorded throughout the test (i.e. determined from the specimen extension as measured by the crosshead displacement), but the true strain (as measured directly from the specimen markers) was only measured prior to the first break. For specimens tested in the step loading manner, the strain was measured directly from the specimen throughout the test duration (inclusive of creep experienced by specimen during dwell periods).

The graphs in Figure 19 (a) – (e) examine the bulk stress/strain (global or true) behaviour of all the specimens tested. These show the responses were comparable and that the controlled cure procedure was effective in reducing the resin property variation from batch to batch. This is an important factor since the test is dependent on the properties of the constituents

which affect how load is transferred to the interface and change the measured values, even where interfacial adhesion is nominally identical.

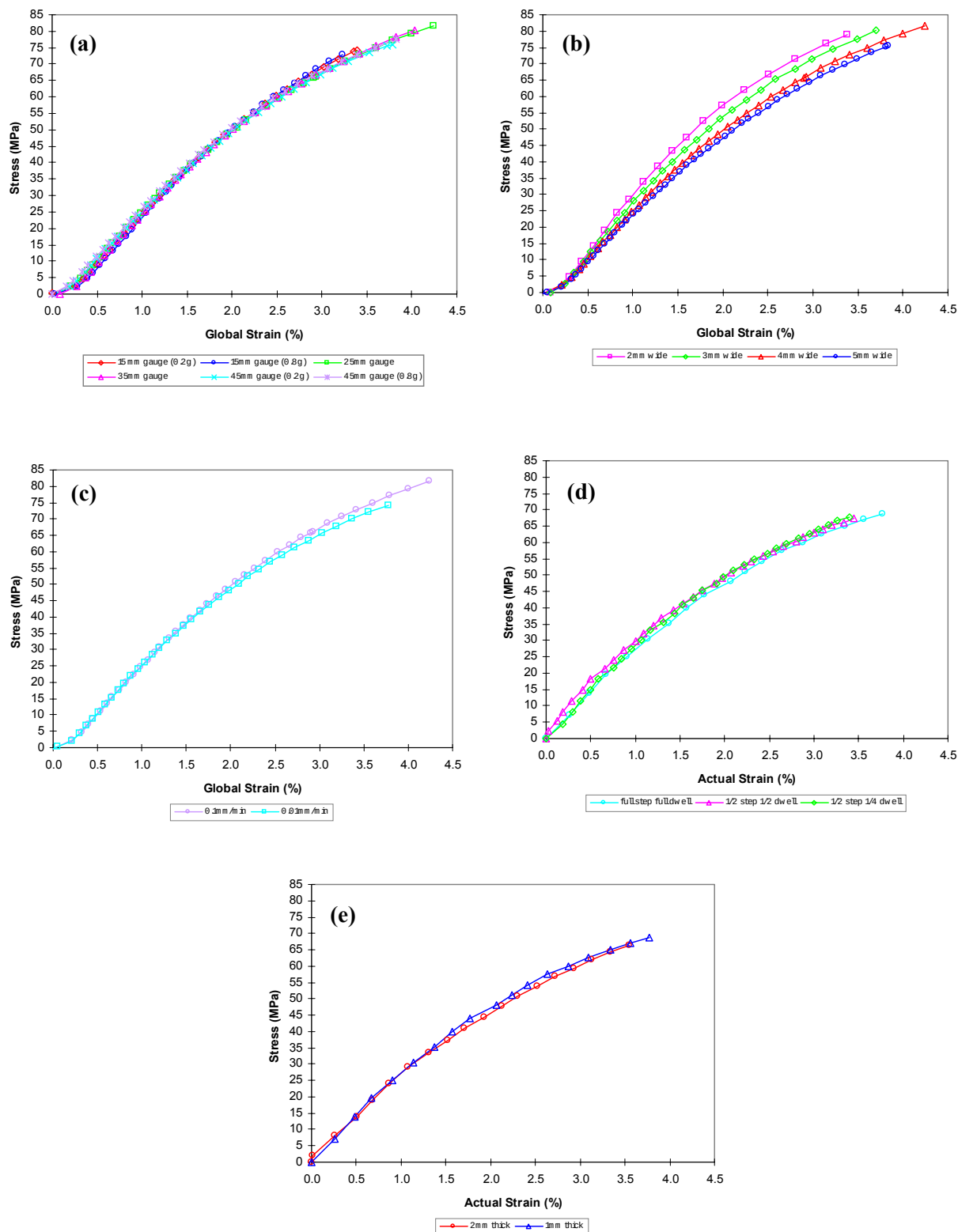


Figure 19 – Typical plots of stress/strain data: (a) change of gauge-length and fibre pre-load, (b) change of width, (c) change of strain rate, (d) change of step-size and dwell time and (e) change of specimen thickness. Graphs (a) – (c) show apparent strain and (d) – (e) show true strain.

These graphs also show the intrinsic consistency in the global behaviour of the specimen of the different geometries, strain rates and step sizes/dwells. The apparent exception is the progressive increase in the bulk stiffness of the specimens with decreasing width seen in Figure 19 (b). This is due to the partial contribution of the end-tabs within the gripped region to the crosshead load/displacement measurements, the proportion of stiffening increasing with decreasing width. This is evident in the graph in Figure 20 which shows the typical stress/true strain behaviour of all the specimens where all curves superimpose. Similarly, this graph shows the two different test techniques, step-wise and continuous, produce nominally equivalent specimen behaviour.

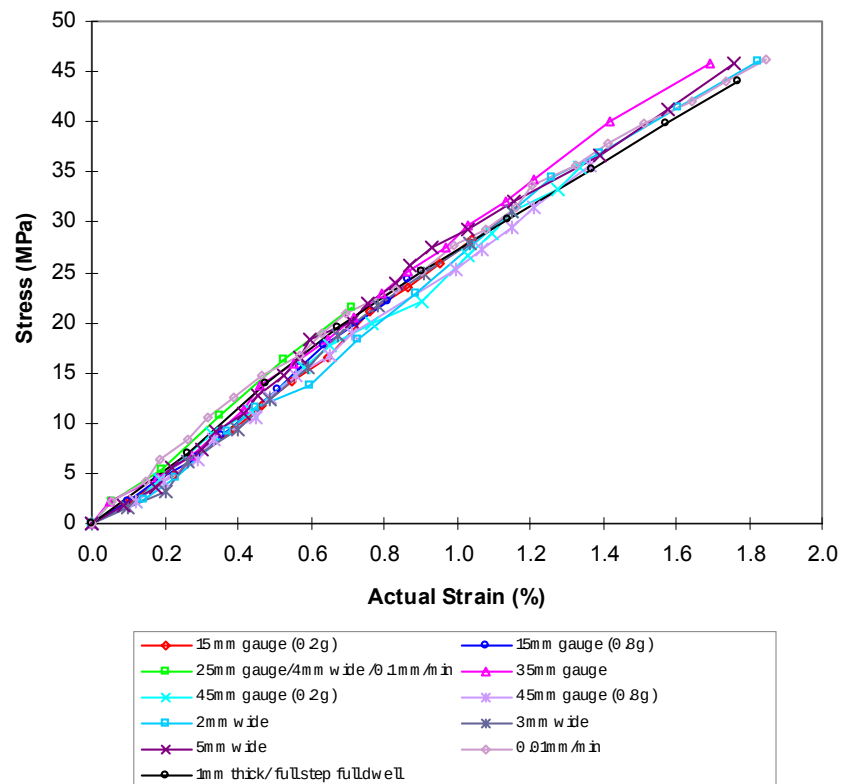


Figure 20 – Graph of stress/true strain data for all continuous test specimens compared with 1 mm thick step loaded test specimen.

6.2.2 Failure Mode Associated with Fibre Breaks

The typical failure process for the interface in this fibre/matrix combination was interface cracking combined with some limited matrix cracking at the point of fibre fracture. This can be seen in Figure 21.

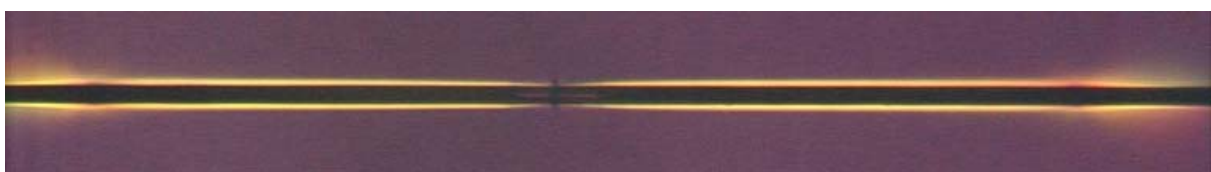


Figure 21 – Image showing typical carbon fibre fracture viewed under polarised light.

6.2.3 Representation of Fragmentation Data

Figure 22 (a) and (b) show typical fragmentation plots of total number of breaks within the monitored length against the specimen strain (either global or actual depending on whether the stepped or continuous test method was employed) or stress. Figure 22 (c) and (d) show the same data with the frequency values normalised to the total number of breaks at fragmentation. These show very consistent specimen fragmentation behaviour within a batch during the test.

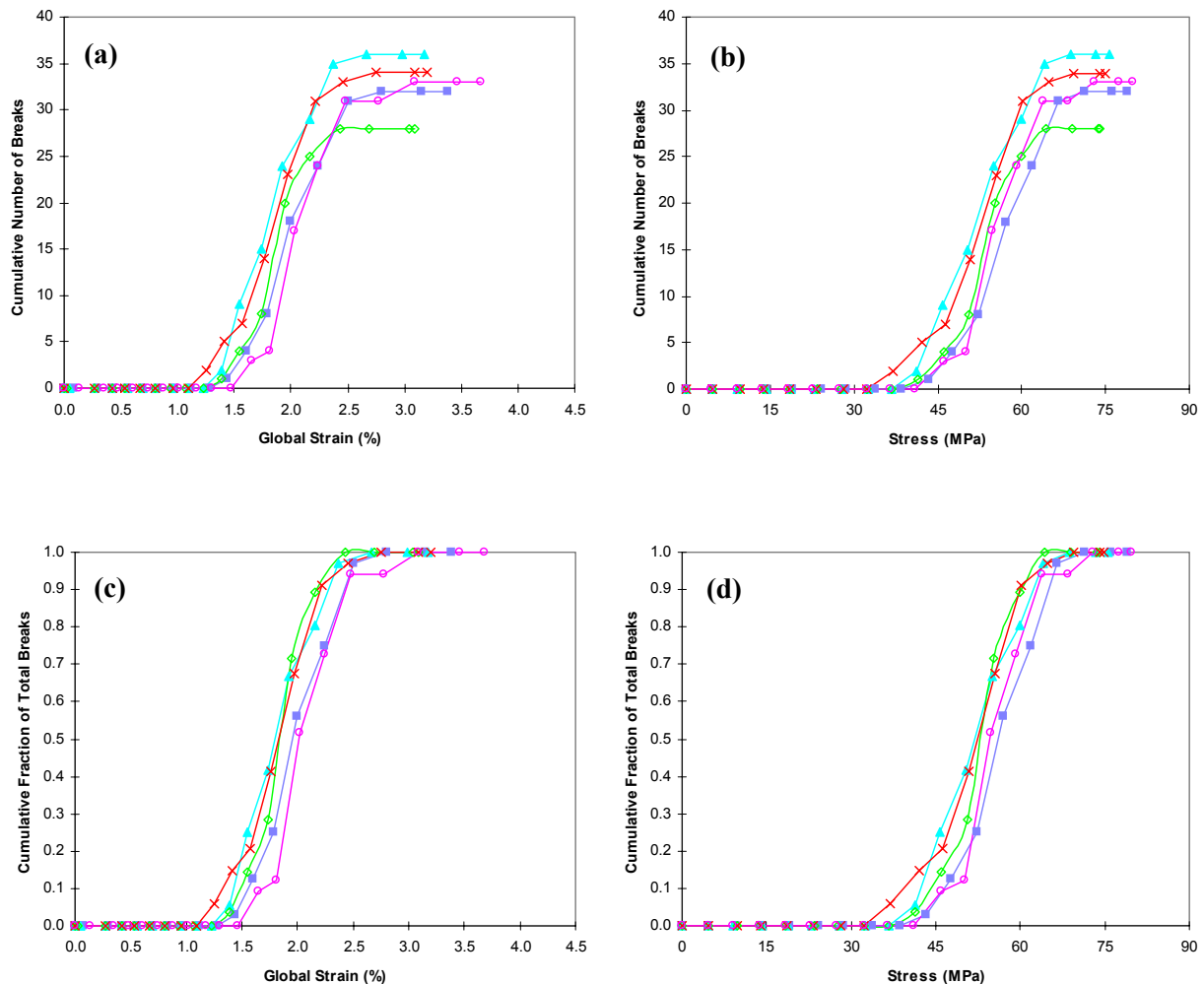


Figure 22 – Typical plots of fragmentation data (2 mm wide specimens in these examples); (a) and (b) cumulative fibre breaks against specimen stress and strain respectively, (c) and (d) data normalised to breaks at saturation.

Figure 23 (a) and (b) show specific details of individual breaks and fragmentation progression throughout a test for continuous tests and step-wise tests respectively, providing additional information for quantitative analysis or qualitative comparison purposes. For continuous tests, the specimen load at each individual break is recorded during the test and allows detailed fragmentation progression to be examined. For incrementally stepped tests, the position of breaks with respect to a reference (one end of the monitored length) are recorded at the end of a dwell period and allow the distribution of fragments with position and strain to be investigated.

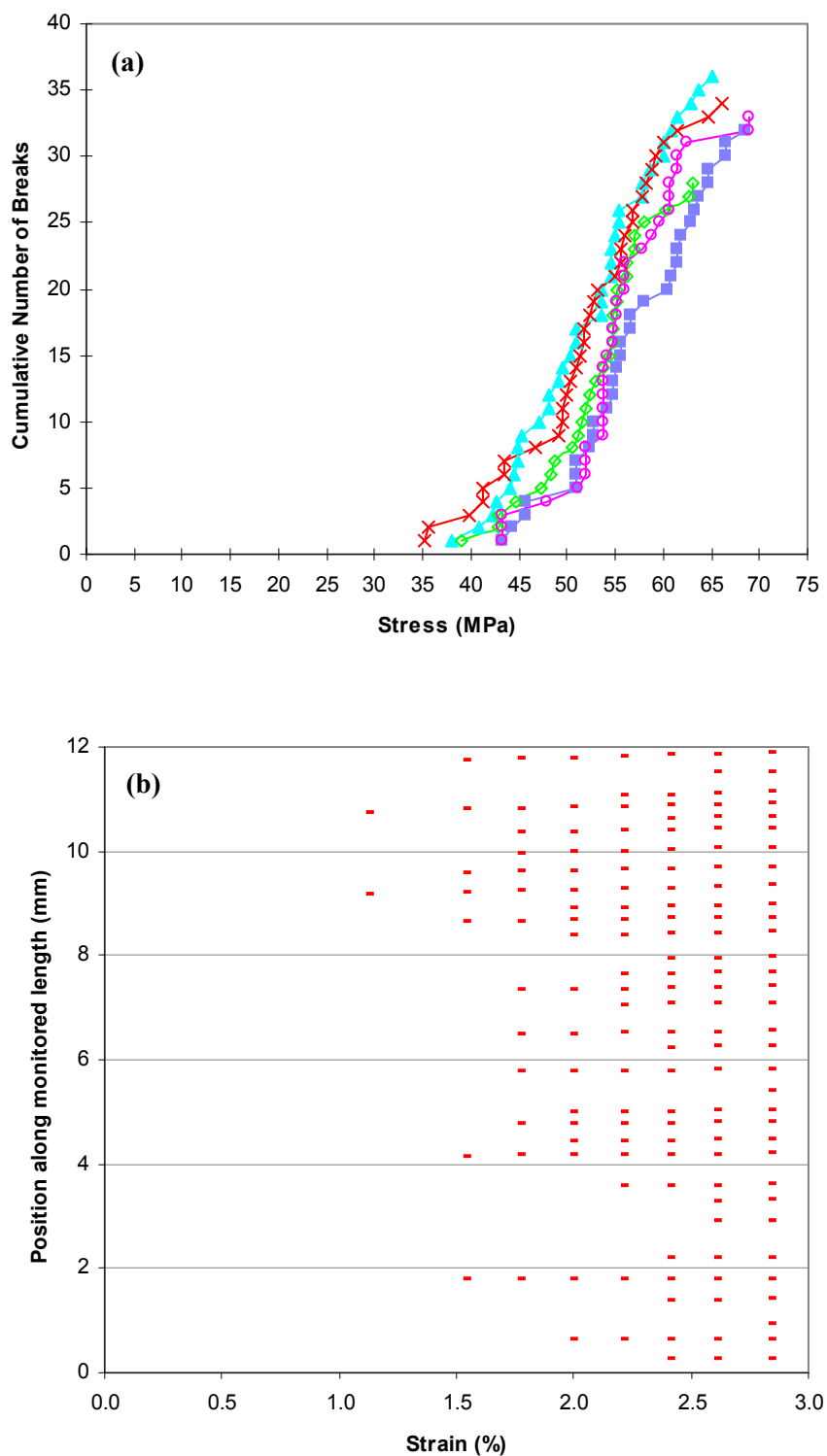


Figure 23 – Detailed fragmentation plots (a) failure stresses for individual breaks in continuous tests (2 mm wide specimens in this example) and (b) position and distribution of breaks within monitored length as a function of increasing strain in step-wise tests (1 mm thick specimens in this example).

Figure 24 (a) and (b) show typical fragment length histograms for individual specimens and a whole batch, respectively. These show consistent and reproducible fragment length distributions for all specimens within a batch.

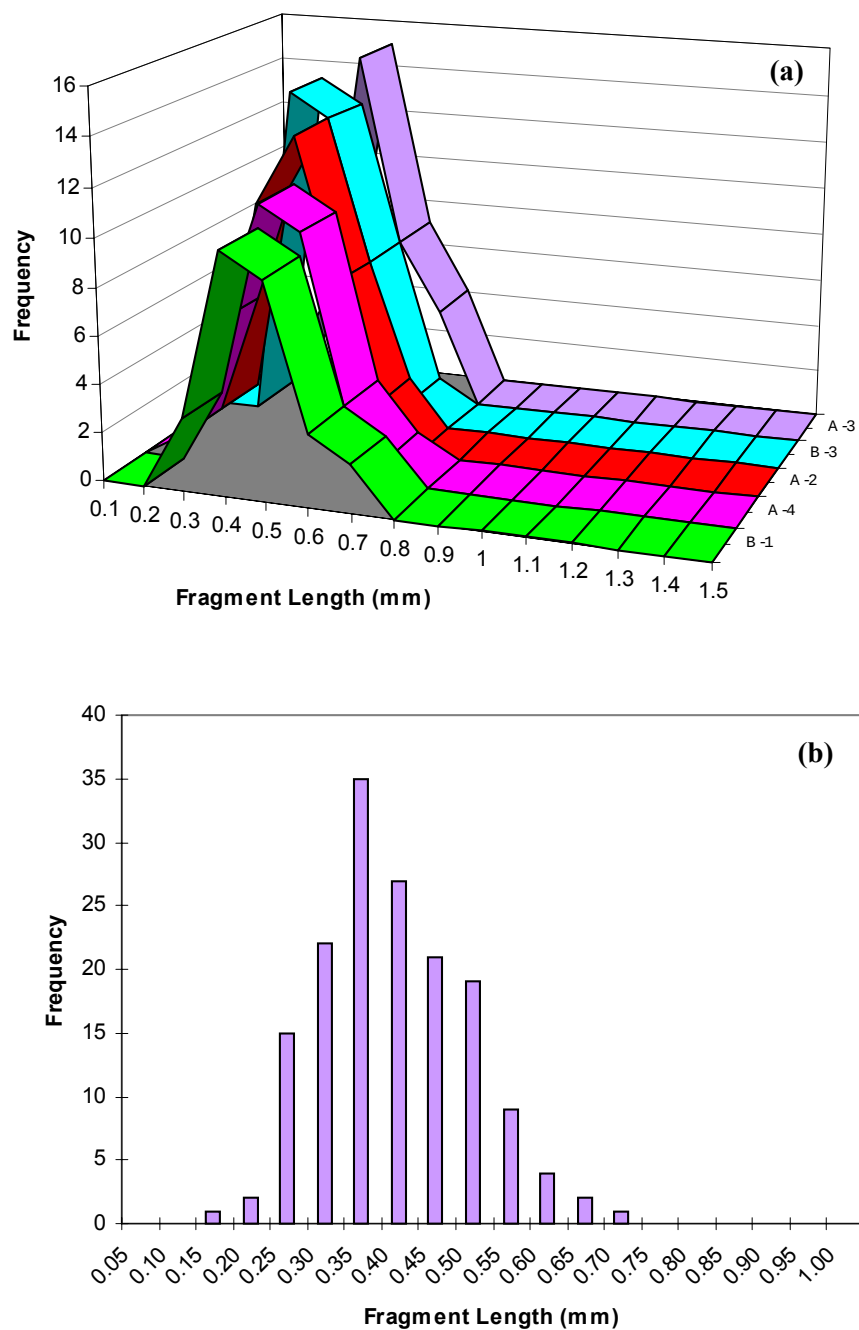


Figure 24 – Typical histograms of fragment length distributions at saturation (2 mm wide specimens in this example) (a) fragment length distributions from individual specimens, (b) global fragment length distribution using all specimens within a batch.

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

$$y = y_0 + Ae^{-\frac{[\ln(x/x_c)]^2}{2w^2}}$$

An example of this is shown in Figure 25; in this case $y_0 = -0.149$, $x_c = 0.389$, $w = 0.251$ and $A = 63.266$. It is important to note that the choice of bin size affects both the shape of the distribution and the best fit log-normal equation.

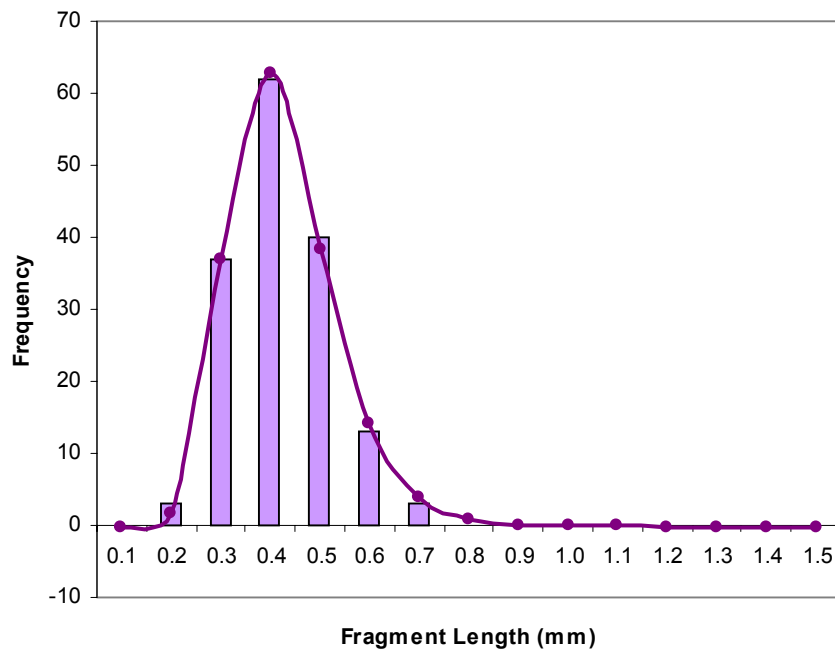


Figure 25 – Example of log-normal distribution fit to histogram of fragment length distribution at saturation for 2 mm wide specimens.

6.2.4 Comparison of Specimen Fragment Length Distributions

The effect of the screening parameters on the fragment length distribution at saturation is examined below. It can be seen from Figure 26 (a) – (e) that in general there are no large differences in the shape or peak values of the distributions, even in cases where a secondary peak is evident, such as 45 mm gauge-length/0.2 g pre-load and 5 mm wide specimens among others. This may be as a result of fibre alignment issues in wide or long specimens. There also seems to be a secondary peak in most of the low strain rates or long dwell time step experiments, which is not seen in their counterparts or in the thicker step loaded specimen.

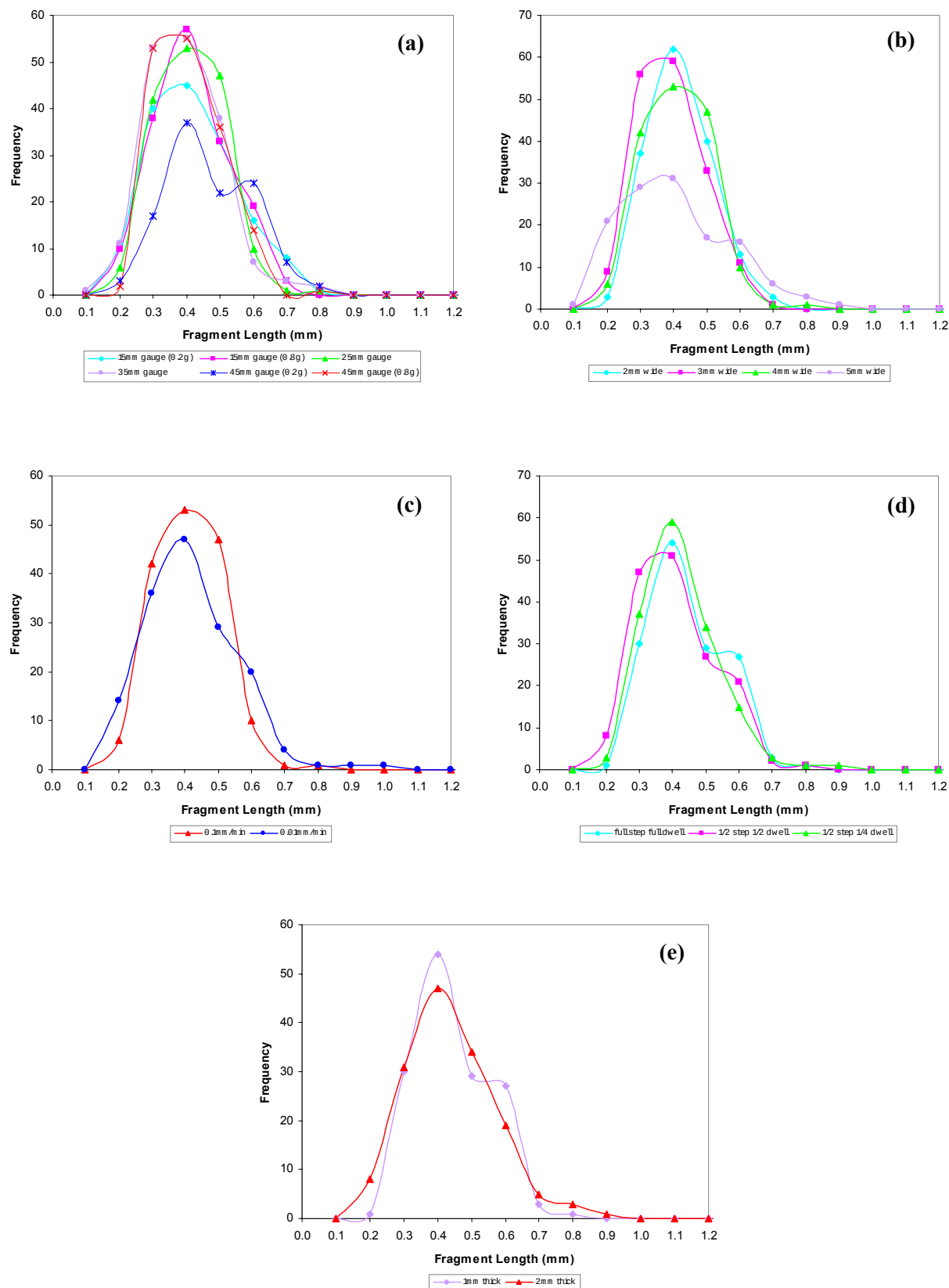


Figure 26 – Fragment length histograms for specimen batches: (a) change of gauge-length and fibre pre-load, (b) change of width, (c) change of strain rate, (d) change of step-size and dwell time and (e) change of specimen thickness.

The graphs in Figure 27 (a) – (c) show the effect of measuring the fragment length distribution at saturation over different monitored lengths. In this investigation, nominal lengths of 12, 24 and 36 mm were used where the specimen gauge-length permitted. Although changing the monitored length increases the statistical population of fragments measured, improving both the accuracy and interface sampling, it has no significant impact on the final distribution. In all cases, it can be seen that the fragment length distribution remains fundamentally unchanged in shape but exhibits an increase in frequency amplitude at the peak value. This is highlighted in Figure 28 (a) which shows the distributions in Figure 27 (c) where the frequency is normalised with respect to the total fragment population. Figure 28 (b) shows the distribution in Figure 26 (a) similarly normalised. Both show close agreement between different distributions.

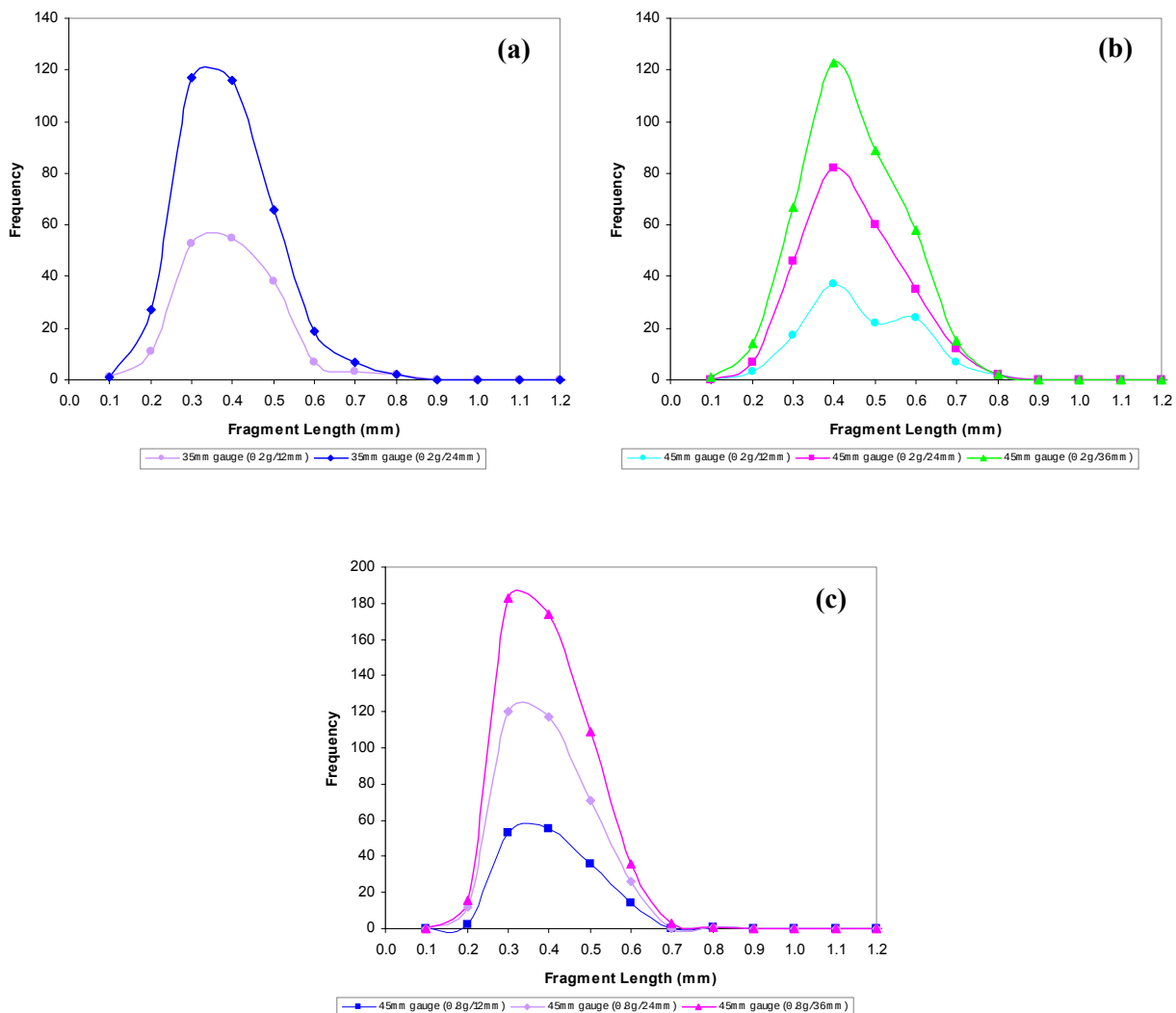


Figure 27 – Graphs comparing the fragment length distributions at saturation for the longer gauge-length specimens with nominal monitored lengths of 12, 24 and 36 mm (a) 35 mm gauge-length, (b) 45 mm gauge-length (0.2 g fibre pre-load) and (c) 45 mm gauge-length (0.8 g fibre pre-load).

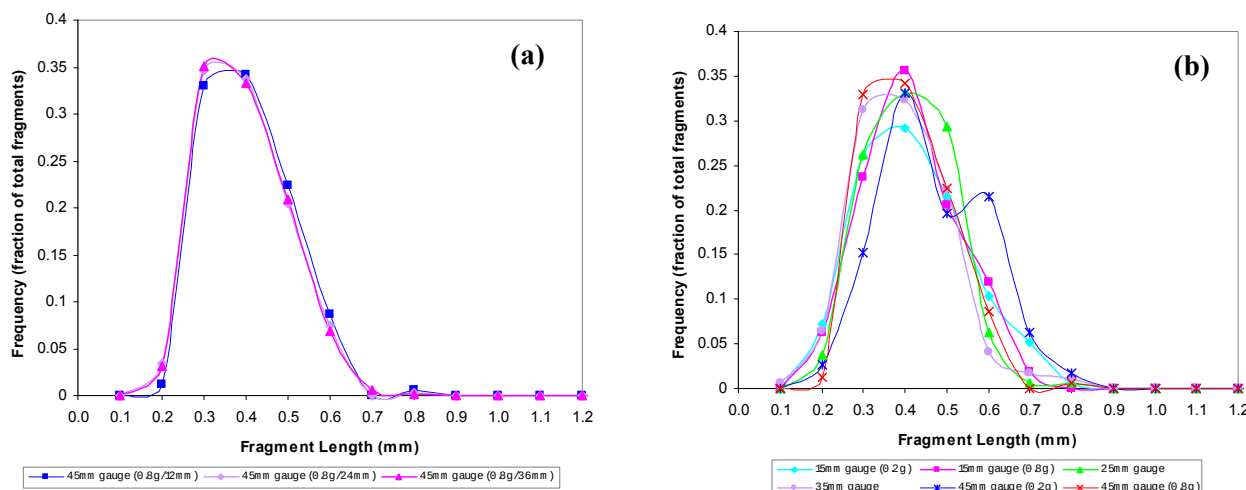


Figure 28 – Graphs of normalised fragment length distributions at saturation; (a) 45 mm gauge-length (0.8 g fibre pre-load) with three different monitored lengths and (b) different specimen gauge-lengths and pre-loads.

6.2.5 Comparison of Specimen Fragmentation Data

Tables 4 and 5 summarise all the characteristic fragmentation data from the screening experiments. The data were evaluated for the influence of the test geometry and procedure on the average fragment lengths, the onset of fragmentation and the stress needed to reach saturation. In general, the number of fragments at saturation was very consistent throughout all the screening tests and there was no noticeable effect of changing from a stepped to a continuous loading test method.

Table 4 – Summary of the Screening Stage Fragmentation Test Results: Stresses

Specimen Type	Number of Tests	Stress (MPa)	
		@ Fragmentation Onset	@ Fragment Saturation
2mm Thick	5	37.3 ± 1.0	59.4 ± 4.1
1mm Thick	5	35.7 ± 4.8	60.8 ± 2.3
Half Step	5	34.9 ± 3.6	59.9 ± 3.5
Quarter Step	5	37.1 ± 7.1	62.5 ± 4.2
0.01mm/min	5	35.0 ± 5.7	60.3 ± 3.2
0.1mm/min	5	39.4 ± 6.2	67.9 ± 2.7
15mm High Load	5	30.3 ± 1.1	63.5 ± 1.2
15mm Low Load	5	32.1 ± 7.1	67.8 ± 1.9
35mm Long	5	41.6 ± 5.5	69.8 ± 1.2
45mm High Load	5	39.1 ± 4.6	64.9 ± 1.3
45mm Low Load	4	39.9 ± 5.7	65.6 ± 3.2
2mm Wide	5	39.8 ± 3.5	66.3 ± 2.4
3mm Wide	5	36.4 ± 5.8	66.2 ± 1.2
5mm Wide	4	37.2 ± 9.2	65.5 ± 5.6

Table 5 – Summary of the Screening Stage Fragmentation Test Results: Fragment Lengths

Specimen Type	Average Fragment Length (mm)		Average Number of Fragments	Average Maximum Fragment Length (mm)
	Median	Arithmetic		
2mm Thick	0.398 ± 0.073	0.387 ± 0.074	30 ± 5	0.680 ± 0.125
1mm Thick	0.397 ± 0.021	0.385 ± 0.020	29 ± 2	0.626 ± 0.056
Half Step	0.364 ± 0.008	0.356 ± 0.004	31 ± 1	0.616 ± 0.075
Quarter Step	0.384 ± 0.070	0.374 ± 0.077	31 ± 6	0.604 ± 0.142
0.01mm/min	0.378 ± 0.061	0.371 ± 0.058	30 ± 6	0.688 ± 0.178
0.1mm/min	0.362 ± 0.016	0.347 ± 0.014	32 ± 1	0.590 ± 0.092
15mm High Load	0.364 ± 0.010	0.348 ± 0.012	32 ± 1	0.583 ± 0.033
15mm Low Load	0.376 ± 0.036	0.363 ± 0.036	31 ± 3	0.642 ± 0.047
35mm Long	0.349 ± 0.050 0.344 ± 0.052^{24}	0.334 ± 0.050	34 ± 6 71 ± 12^{24}	0.572 ± 0.113 0.587 ± 0.123^{24}
45mm High Load	0.361 ± 0.043 0.349 ± 0.035^{24} 0.350 ± 0.031^{36}	0.348 ± 0.042	32 ± 4 69 ± 7^{24} 104 ± 9^{36}	0.571 ± 0.082 0.579 ± 0.080^{24} 0.608 ± 0.059^{36}
45mm Low Load	0.421 ± 0.061 0.399 ± 0.017^{24} 0.397 ± 0.024^{36}	0.404 ± 0.058	28 ± 6 61 ± 3^{24} 92 ± 6^{36}	0.708 ± 0.178 0.708 ± 0.033^{24} 0.708 ± 0.033^{36}
2mm Wide	0.370 ± 0.030	0.354 ± 0.031	32 ± 3	0.578 ± 0.064
3mm Wide	0.347 ± 0.034	0.331 ± 0.031	34 ± 3	0.554 ± 0.062
5mm Wide	0.394 ± 0.124	0.388 ± 0.110	31 ± 10	0.666 ± 0.112

²⁴, ³⁶ – indicate values measured over a nominal 24 or 36 mm monitored length, respectively, instead of the standard 12 mm.

The differences between the median and arithmetic average fragment lengths at saturation can be mostly attributed to the difference in specimen strain for the two calculations. The arithmetic average is based on the original monitored length as measured prior to testing, whilst the median value is calculated from measurements made with the specimen under considerable strain at saturation. If this increased strain is taken into account the values are much closer as shown in Table 6. This correction also decreases the scatter in the median values by taking into account the differences in strain experienced by each individual specimen at saturation.

Table 6 – Effect of Correcting Final Fragment Lengths for Specimen Strain

Specimen Type	Median Average Fragment Length (mm)		Arithmetic Average Fragment Length (mm)
	Measured	Corrected	
2mm Thick	0.398 ± 0.073	0.384 ± 0.070	0.387 ± 0.074
1mm Thick	0.397 ± 0.021	0.383 ± 0.020	0.385 ± 0.020
Half Step	0.364 ± 0.008	0.352 ± 0.008	0.356 ± 0.004
Quarter Step	0.384 ± 0.070	0.371 ± 0.068	0.374 ± 0.077

There are no obvious dynamic effects where breaks are initiated by stress waves from nearby breaks. This might have been expected in the longer and wider specimens (higher strain energies) and in the high rate tests (no stress relaxation). This effect would have resulted in shorter average fragment lengths.

The parameters varied in the screening study had no detectable influence on the mean fragment length, which is approximately 0.376 (median) and 0.364 (arithmetic). No influence of thickness or width is apparent. From the Monte-Carlo analysis implemented (refer to Section 6.3), the dimension that would be expected to change the axial stress response in the fibre is $\leq rE_f/E_m \approx 350$ microns in this case; where r is the fibre radius and E_f and E_m are the moduli of the fibre and matrix respectively. Since all specimen dimensions employed in this study were well above this limiting value, no effect was evident. This lower limit is unlikely to be met due to the need for practicality and specimen handling. However, wider and longer specimens showed a greater tendency to fail prior to saturation, reducing the number of successful test completions. Similarly, the final fragment lengths of incrementally strained specimens might be expected to be longer than those that are monotonically loaded, since a highly stressed interface can creep affecting the stress redistribution process during the test. The rate required to see this effect may be beyond the levels applied in this study.

The high rate continuous tests (carried out at 0.1 mm/min, refer to Table 2) reach saturation at higher stresses than the low rate continuous or step loaded tests. This is most probably because of creep effects in the latter two cases. The 15 mm gauge-length specimens exhibit lower fragmentation onset stresses compared to all the other tests. This may be due to edge effects because a larger proportion of the gauge-length is included in the monitored length and early breaks occurring near to stress concentrations (in the radius of curvature of the test-piece) are being recorded.

The effect of pre-load is not marked and a larger pre-load would be required to see this effect more clearly. This was difficult to achieve because of the bending to which the brittle fibre was subjected during pre-load attachment, limiting the loads which could be applied. Ideally, a pure axial pre-tensioning would enable much higher loads to be introduced. The inclusion of a pre-load at the preparation stage is expected to shift the onset of fragmentation to lower stresses by counteracting a proportion of the cure shrinkage and thermal expansion mismatch.

6.3 ANALYSIS

The data were analysed using a strength-based approach derived from a simple, closed-form, modified shear-lag model. This was used to describe the axial stress in the fibre and the interfacial shear stress. A simulation of the fragmentation process employing this model was implemented using a Monte-Carlo method [1]. This simulation, although basic, proved consistent with experimental results when plotted in the form: $\log_e$ average fragment length against $\log_e$ specimen strain. These plots, an example of which is given in Figure 29, do not superpose and each test/specimen is different to every other nominally identical test/specimen. Similar plots are observed using the simulation.

This simulation was then used to mimic the specimen behaviour and thus allow characteristic Weibull parameters to be estimated and the internal stress level to be predicted (the model parameters were varied to obtain a best fit to experimental data). The simulation was also used to assess the effect of gauge-length on the fragmentation data and confirmed the experimental findings that there was no effect within the range used in this study.

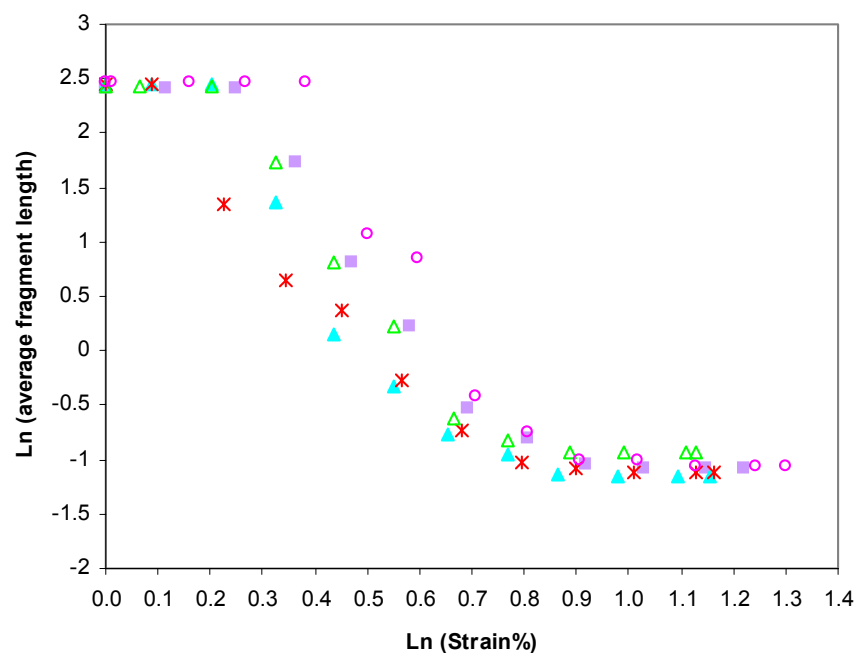


Figure 29 – Typical plot from experimental fragmentation data (2 mm wide specimens in this example) showing different slopes and plateau levels for each specimen tested within a batch.

6.4 RECOMMENDATIONS

From the initial experimental screening programme, the optimum parameters for use in a single fibre fragmentation test are:

- maximum gauge-length possible/practical to ensure fragment lengths are a statistically representative population, but restricted by a higher incidence of premature failure for longer gauge-lengths;
- thickness and width can be any value (but greater than rE_f/E_m) to improve handling and/or accommodate equipment limitations, but restricted by a higher incidence of premature failure for larger values;
- pre-load as high as practically possible to reduce scatter in results;
- constant strain rate experiments to significantly reduce test time, increase accuracy of measurements of the fragmentation process and remove creep effects;
- maximum monitored lengths possible/practical situated at least 5 mm away from either end of the gauge-length to minimise effects of local stress concentrations within the observed region.

7 APPLICATION/VALIDATION STAGE

The optimised single fibre fragmentation test was subsequently used to assess several systems with different factors known to affect the nature of the interface:

- fibre type,
- sizing,
- environmental conditioning.

7.1 MATERIALS AND TESTS

To extend the applicability of the method to low-strain-to-failure resins, it has been shown that a 1-10 micron layer of a brittle resin coating can be formed on the fibre which is cured as necessary and then encased in a tough resin dog-bone to produce a coaxial fragmentation specimen [8]. Attempts made in this programme to create a brittle resin coating were unsuccessful due to surface tension effects causing droplet formation rather than a smooth, uniform coating. The issue of achieving saturation with brittle resins will be effectively eliminated when a local analysis, based on the first fibre break, is established. As a result, Epikote 828 epoxy resin with an mPDA hardener was used as the matrix for all specimens.

The continuous testing procedure was used for all tests in the validation stage. This enabled stresses at each break to be determined and reduced test times to one fifth of their step-wise counterparts. Thus all specimens were tested at a continuous displacement of 0.1 mm/min which is approximately equivalent to a strain rate over the gauge-length of 0.002 /min.

Longer gauge-lengths and wider/thicker specimens, whilst preferable for statistical and handling purposes respectively, suffer from an increase in premature failure during testing. This is probably due to the larger mould surface and matrix volume in these specimens which results in a higher incidence of edge defects being created during mould filling and processing. Thus the specimen geometry selected for the application stage was a width of

4 mm, a gauge-length of 25 mm and a thickness of 2 mm (see Figure 3). High pre-loads are difficult to apply using the current method due to the fibre bending induced and resultant high preparation breakage rates. The application of a uniaxial load with a novel tensioning device, which can remain in place during the cure process, will make it possible to achieve higher pre-loads. For this investigation lower pre-loads were applied, as listed in Table 7.

Batches of carbon fibre specimens were made using Tenax HTA 5131 (as used in the screening study) with different interfacial treatments:

- (i) Standard carbon was produced with as-received fibres.
- (ii) Release agent coated carbon was made with standard fibres coated with release agent (Tygavac release agent SP441 containing aliphatic hydrocarbons). These were prepared by isolating individual fibres in the usual manner and then passing these through a pool of the liquid coating. Each fibre was coated in this manner 3 times and allowed to dry between each coating pass. Once laid into the moulds, the coating on the fibres was cured at 125 °C for 10 mins and allowed to cool to 70 °C, prior to performing the normal fabrication procedure.
- (iii) Conditioned carbon was made with as-received fibres. After fabrication these were subjected to an aggressive steam environment. Exposure was carried out at 100 °C for 24 hrs using a programmable autoclave (Priorclave Ltd). These conditioning parameters were chosen to minimise the number of specimen failures during exposure whilst ensuring significant moisture absorption. The small amounts of material involved allowed rapid and uniform environmental ageing; globally, specimens absorbed on average 2.4 wt% of water and displayed residual swelling strains of 0.46%.

Batches of glass fibre specimens were manufactured using Vetrotex 480 tex, 17 microns E-glass (fibre sections taken from internal unwindings) with different as-received sizing treatments. Details of these sizing treatments are specified in Table 7.

Table 7 – Summary of the Material/Test Parameters used in the Application/Validation Experimental Design

Specimen Type	Width (mm)	Thickness (mm)	Gauge Length (mm)	Test Protocol	Pre-load (g)	Monitored Length (mm)
Standard Carbon	4	2	25	Continuous @ 0.1mm/min	0.2	12
Release Agent Coated Carbon	4	2	25	Continuous @ 0.1mm/min	0.2	12
Conditioned Carbon	4	2	25	Continuous @ 0.1mm/min	0.2	12
Glass-162 (complete sizing for polyester and epoxy)	4	2	25	Continuous @ 0.1mm/min	1.6	12
Glass-163 (water sizing)	4	2	25	Continuous @ 0.1mm/min	1.6	12
Glass-164 (water + silane sizing for epoxy)	4	2	25	Continuous @ 0.1mm/min	1.6	12
Glass-165 (water + silane sizing for polyester)	4	2	25	Continuous @ 0.1mm/min	1.6	12

7.2 RESULTS AND DISCUSSION

The E-glass 164 specimens all failed prematurely, prior to achieving saturation, thus no data from this batch are presented in the following summary of the validation stage test results. Photoelastic images during monitoring of damage progression in typical specimens from fibre breaks for each of the batches are given in Appendix F and G.

7.2.1 Specimen Stress/Strain Behaviour

Figure 30 (a) and (b) show the typical stress-strain response of one specimen from each batch tested. As was seen in the screening stage experiments, the overall behaviour of the specimens are very consistent suggesting reproducible resin properties between batches. The conditioned specimens show a similar initial modulus, but due to the plasticising effects of moisture absorption on the resin matrix, exhibit a different non-linear response during loading compared to the dry resin.

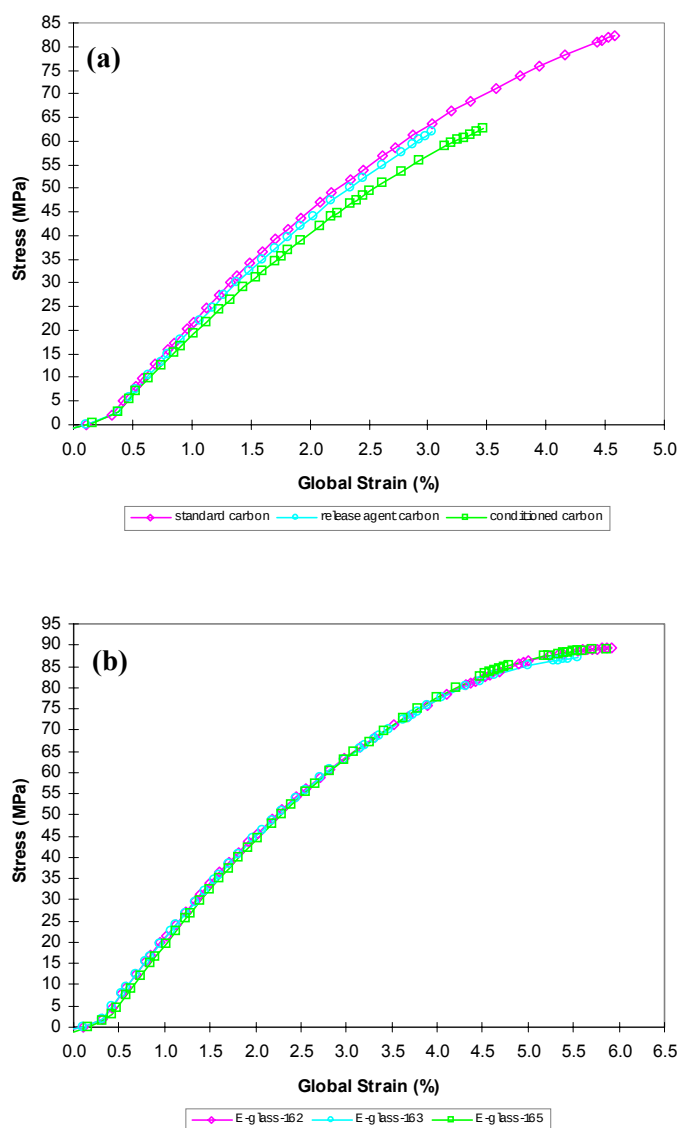


Figure 30 – Typical plots of stress/strain data: (a) carbon fibre specimens and (b) glass fibre specimens. All the graphs are plotted with respect to global strain.

7.2.2 Specimen Fragmentation Data

Tables 8 and 9 summarise all the characteristic fragmentation data from the validation experiments. The data were evaluated for the influence of the different surface or conditioning treatments on the average fragment lengths, the onset of fragmentation and the stress needed to reach saturation. Analysis was also carried out to determine and compare the interfacial shear strengths (see Section 7.3). As the resin and fibre properties and the specimen preparation and test details are nominally identical for the carbon and glass specimens, the fragmentation data from within each fibre type can initially be compared directly without need for further analysis to account for these factors.

Table 8 – Summary of the Validation Stage Fragmentation Test Results: Fragment Lengths

Specimen Type	Average Fragment Length (mm)		Average Number of Fragments
	Median	Arithmetic	
Standard Carbon	0.374 ± 0.042	0.359 ± 0.039	31 ± 4
Release Agent Coated Carbon	0.782 ± 0.290	0.745 ± 0.267	16 ± 6
Conditioned Carbon	0.401 ± 0.043	0.394 ± 0.040	28 ± 3
Glass-162 (complete sizing for polyester and epoxy)	0.882 ± 0.246	0.816 ± 0.223	14 ± 5
Glass-163 (water sizing)	0.498 ± 0.093	0.461 ± 0.085	25 ± 5
Glass-165 (water + silane sizing for polyester)	0.741 ± 0.149	0.688 ± 0.123	17 ± 4

The carbon data show a larger average fragment length in the conditioned than the standard carbon specimens which suggests a marginally weaker interface, even though the number of fragments at saturation in both are similar. The coated carbon specimens have far fewer fragments at saturation and very long average fragment lengths indicating a very poor interfacial adhesion.

The glass data show a larger average fragment length in the glass-162 specimens compared to the glass-165 specimens suggesting a weaker interface in the former, despite similar final fragment numbers in both systems. The glass-163 surface treatment gives the strongest interface, of the three presented here, supported by the significantly shorter average fragment lengths and greater number of saturation fragments. The glass-164 specimens failed prematurely due to the interface being so strong that very limited interfacial debonding/cracking occurred around breaks. Instead, fibre fractures resulted in large matrix cracks which continued to grow with increased loading. Thus glass-164 specimens exhibit the strongest interface of all the glass surface treatments investigated in this study.

Table 9 – Summary of the Validation Stage Fragmentation Test Results: Stresses

Specimen Type	Number of Tests	Stress (MPa)	
		@ Fragmentation Onset	@ Fragment Saturation
Standard Carbon	5	37.8 ± 3.8	68.2 ± 4.3
Release Agent Coated Carbon	7	38.1 ± 4.4	57.9 ± 5.2
Conditioned Carbon	5	23.8 ± 1.9	48.5 ± 3.6
Glass-162 (complete sizing for polyester and epoxy)	6	67.3 ± 11.6	84.9 ± 2.1
Glass-163 (water sizing)	4	52.4 ± 12.2	86.1 ± 2.3
Glass-165 (water + silane sizing for polyester)	4	75.2 ± 7.5	86.8 ± 0.7

From the carbon data: the stress for the onset of fragmentation is significantly lower in the conditioned carbon than the standard or coated carbon, both of which experience the first fibre break at the same stress. This suggests an enhanced stress transfer mechanism in the conditioned specimens presumably due to higher radial compression stresses induced by the resin swelling after moisture exposure. The lower saturation stresses for the coated and conditioned carbon specimens suggest weaker interfacial strengths, although the much lower value for the conditioned material may be caused by the local matrix plasticisation (fibres act as conduits for greater moisture uptake in the vicinity of the interface) resulting in local resin yielding or creep at lower stresses.

From the glass data: the different fibre treatments all saturate at the same stress and so indicate the fragmentation process in these materials is limited by local matrix yielding. The onset stresses confirm the fragment length data conclusions that the glass-163 surface treatment gives the strongest interface since the lower onset stress signifies a more efficient shear stress transfer through the interface to the fibre.

7.2.3 Fragment Length Distributions

The fragment length distributions from specimens tested in the validation stage of the study are presented in Figure 31 (a) and (b). The carbon fibre fragment length distributions in Figure 31 (a) exhibit a large degree of similarity in shape and peak value between the conditioned and standard specimens. The release agent coated carbon fibre specimens, however, have a very distinctive distribution with a very flat, broad spread and gradual taper into long fragment lengths. The glass fibre fragment length distributions in Figure 31 (b) all show bimodal characteristics, with two peaks of approximately equal amplitude. Glass-162 and glass-165 show very similar low, broad forms with the former being displaced slightly towards longer fragment lengths and therefore weaker interfacial strength. Glass-163 has a much narrower band distribution.

The width of the distribution may be partly caused by irregularity in the surface treatment, either from one fibre to the next or within a single fibre, especially in the case of the release agent coated carbon fibre specimens which were treated individually. A repeatable, uniform

coating along the fibre length was difficult to produce as well as maintaining a constant coating thickness from fibre to fibre within the batch.

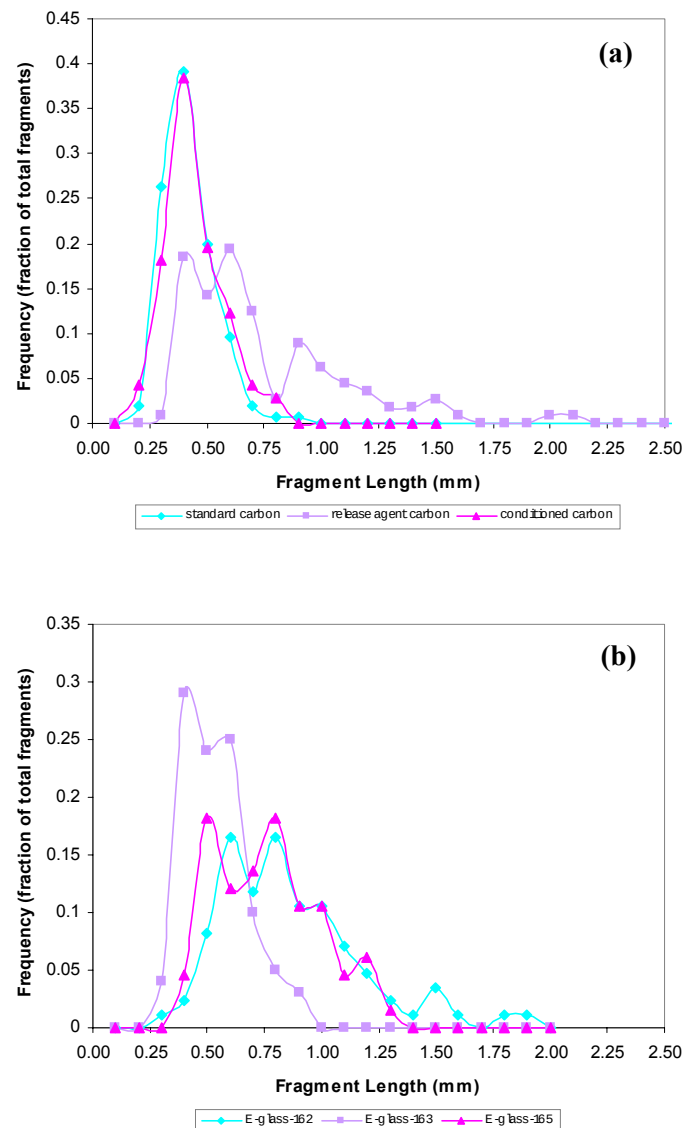


Figure 31 – Typical histogram plots of fragment length distributions: (a) carbon fibre specimens and (b) glass fibre specimens.

7.2.4 Failure Modes Associated with Fibre Breaks

The standard and conditioned carbon fibre specimens exhibited interface cracking combined with some limited matrix cracking at the point of fibre fracture, as seen in the screening experiments. The release agent coated carbon fibre specimens showed no matrix cracking but instead exhibited extensive frictional debonding (shown in Appendix F).

Glass-162, 163 and 165 specimens show interfacial crack growth and debonding (shown in Appendix G). Images of representative fibre breaks are given in Appendix H. In particular, Glass-162 exhibits a very rough debonded surface. Glass-164 specimens experienced premature failure during testing and no specimens achieved saturation. Figure 32 shows a

fracture surface from one of these test-pieces in which the cause of the failure is the catastrophic growth of the matrix crack extending out radially from the fibre break.

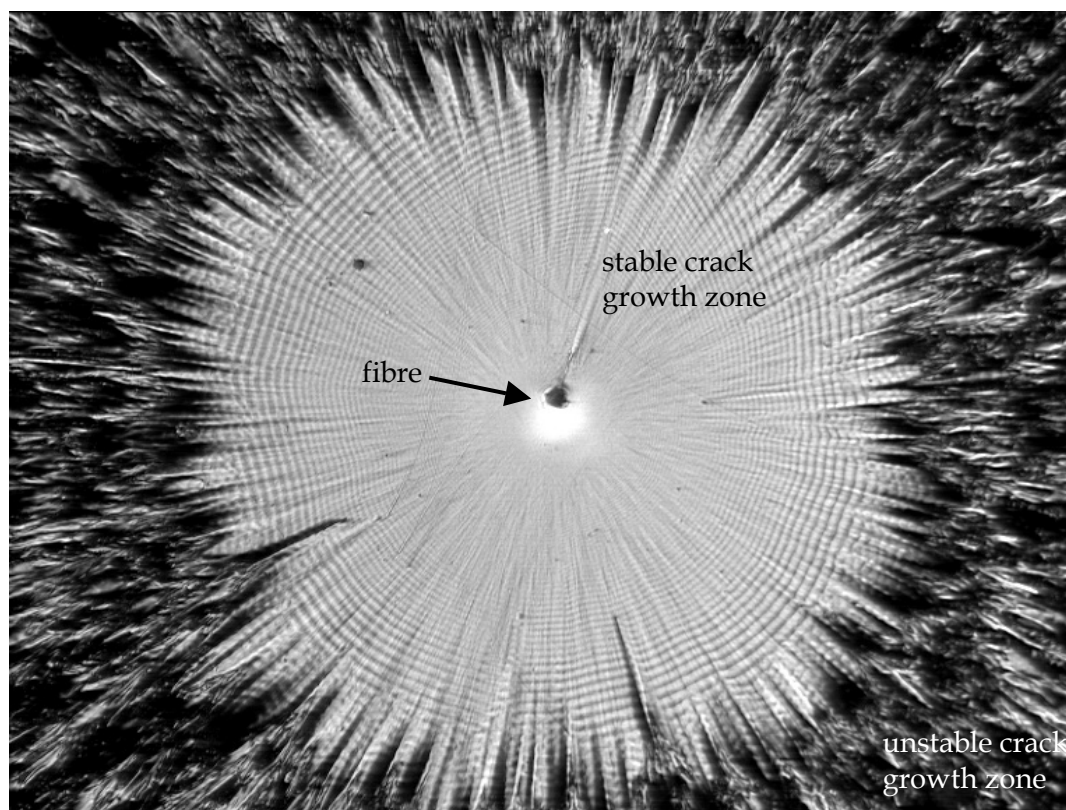


Figure 32 – Image of typical Glass-164 specimen fracture surface showing fibre (centre) and regions of stable and unstable matrix crack growth.

7.3 ANALYSIS

A simplified local analysis was adopted to analyse the fragmentation data, in order to determine interfacial shear strengths. The longest fragment length from each test, subject to the greatest shear stress, was analysed using the load corresponding to the onset of fragmentation. Axial internal stresses due to thermal expansion mismatch and cure shrinkage were taken into account in the analysis, but radial stresses were ignored. Debonding at the interface, affecting the stress transfer ability, was also included with friction over this zone considered. Debonded lengths were measured on the unloaded specimens under polarised light using an optical microscope (typical photoelastic images of unloaded specimens and visible debonds are presented in Appendix J). These are often difficult to see clearly and thus measure accurately suggesting a need for a better means of determining the debonded length of a fragment.

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. This pre-test viscoelasticity was corrected for but the matrix was assumed linear-elastic for the purposes of in-test analysis, ignoring viscoelastic effects during loading. The data used in the interfacial strength analysis are summarised in Table 10 and the resulting interfacial shear strengths are depicted in Figure 33 (a) and (b).

**Table 10 – Summary of the Validation Stage Fragmentation Test Results:
Analysis Input Data**

Specimen Type	Average Fibre Diameter (μm)	Average Debond Length of Longest Fragment (mm)	Average Maximum Fragment Length (mm)
Standard Carbon	7.2 ± 0.1	0.149 ± 0.028	0.659 ± 0.151
Release Agent Coated Carbon	7.1 ± 0.4	0.421 ± 0.076	1.321 ± 0.415
Conditioned Carbon	7.0 ± 0.3	0.167 ± 0.024	0.684 ± 0.077
Glass-162 (complete sizing for polyester and epoxy)	15.1 ± 1.8	0.324 ± 0.021	1.347 ± 0.366
Glass-163 (water sizing)	16.5 ± 1.4	0.209 ± 0.052	0.7418 ± 0.096
Glass-165 (water + silane sizing for polyester)	15.2 ± 0.7	0.208 ± 0.026	1.034 ± 0.193

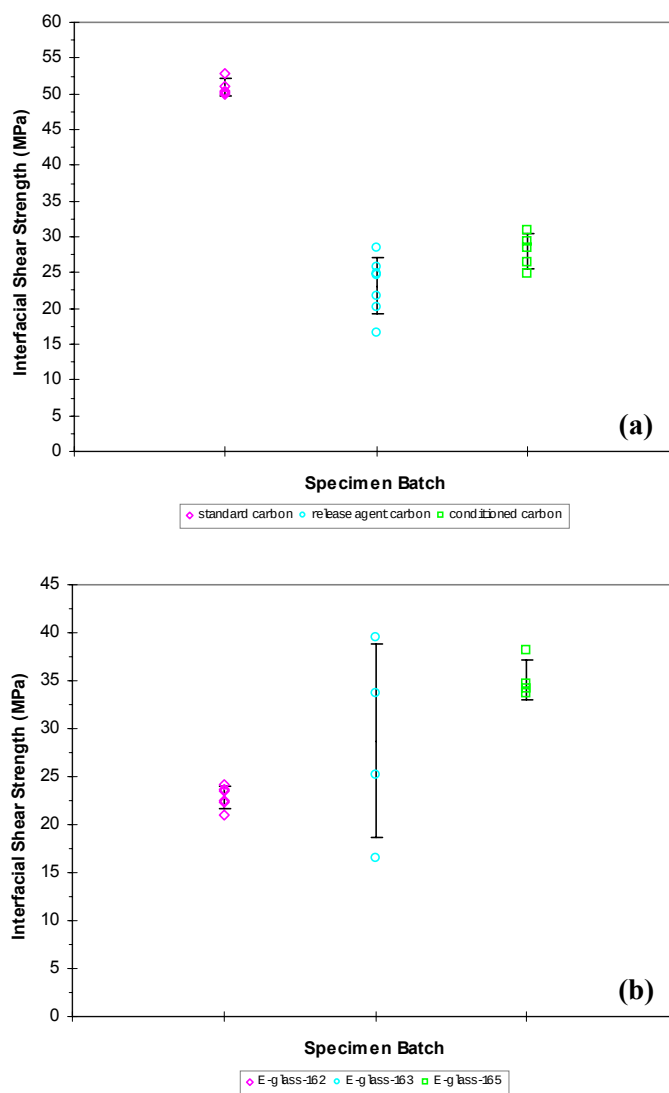


Figure 33 – Plots of interfacial shear strength: (a) carbon fibre specimens and (b) glass fibre specimens.

Figure 33 (a) shows that the level of interfacial adhesion in the conditioned and coated carbon fibres has decreased by a factor of 2. As the values for both of these are close it is likely that most of the covalent bonds at the interface have been either prevented from forming or destroyed and the resultant strength is characteristic of the mechanical interlocking associated with the surface roughness of the carbon fibre.

Many more specimens need to be tested for low strength interfaces, such as the release agent coated carbon fibres, to achieve comparable 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, which would then need to be dealt with in the analysis of results.

Figure 33 (b) shows the ability of the fragmentation test to distinguish between the closely matched interfacial strengths of the three glass fibre systems, providing a qualitative ranking. The scatter in results may also provide some indication of the homogeneity of the surface coating or treatment.

In all cases, it is possible that the longest 'intact' length would be better used in the analysis since this would be expected to be subject to the highest shear stress. Thus all fragments and their associated debond lengths would need to be measured and the maximum bonded fragment length determined and used in the analysis.

8 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. The method is not suitable for all material systems until a practical energy-based analysis, applicable in the small strain regime, is devised.

Interfacial strengths determined using the fragmentation technique are usually high because of the nature of the test; a fragmentation only occurs if a minimum bond strength is achieved. The stress state in the specimen is also a contributing factor. High stress concentrations exist in the interfacial shear stress near the fragment ends; responsible for plastic deformation in the matrix or interfacial crack growth. These stresses diminish more slowly than for other micromechanical tests. In addition, the applied tensile load induces high intensity compressive radial stresses such that further stress transfer can occur at the interface after debonding, through frictional stresses.

The scatter seen in fragmentation results is due, in part, to the random local nature of the interface and fibre surface. Other contributory factors such as specimen fabrication, preparation and loading were eliminated in this study by introducing tight controls on sample manufacture and testing. The accuracy of fibre diameter measurements and fragment lengths also affect the commonly observed scatter in results. In order to reduce this yet further, a number of recommendations on specimen geometry and testing are made.

It has been shown that it is possible to continuously monitor the fragmentation process. This is an improvement as it prevents the stress relaxation associated with long dwell times in

step loaded tests which complicate analysis and fragment lengths in the test-piece were unaffected by this change. Photoelastic analysis is also an important part of the test technique enabling failure modes to be determined and confirming the ranking order for comparative assessments of interfacial strength.

The following recommendations should improve consistency, repeatability, practicality and reduce scatter in fragmentation tests:

- use the maximum gauge-length possible;
- use any thickness and width $\geq rE_f/E_m$;
- use the highest pre-load possible;
- use constant strain rate experiments;
- use the maximum monitored lengths possible.

For the specimen preparation stage:

- use polished steel templates;
- use only unbroken fibres;
- do not handle the central region of the fibre;
- use a pre-load on the fibre;
- use a pipette to carefully fill the mould with resin;
- use a tightly controlled resin cure cycle;
- remove specimens from mould by bending parallel to fibre axis;
- avoid water lubrication during polishing.

ACKNOWLEDGEMENTS

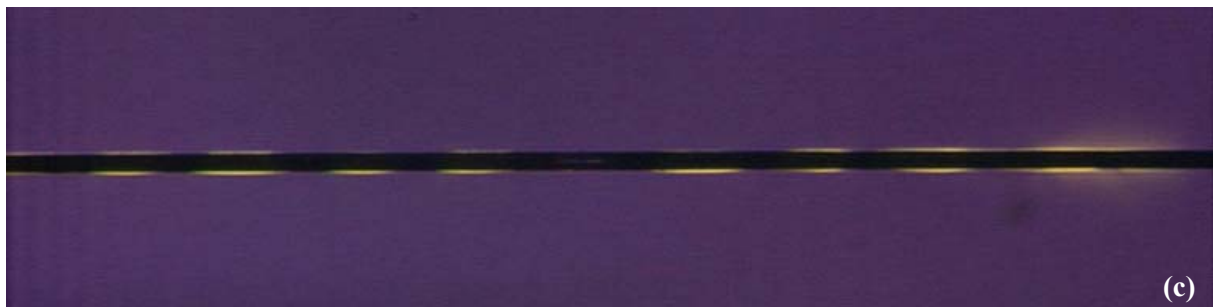
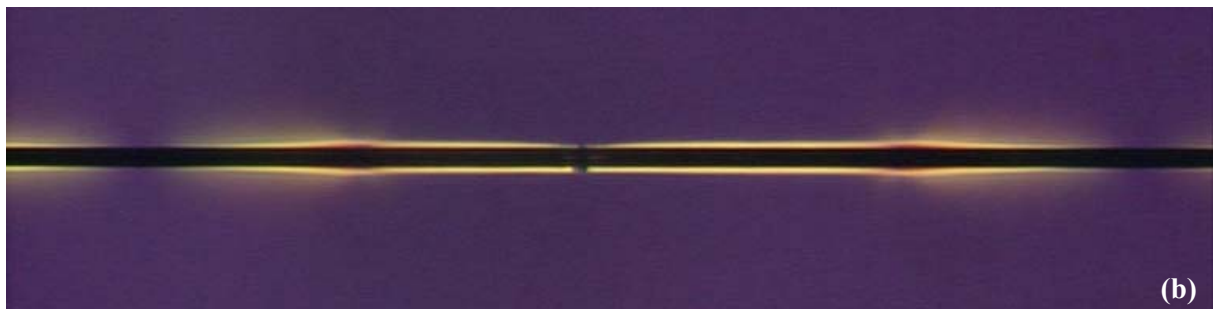
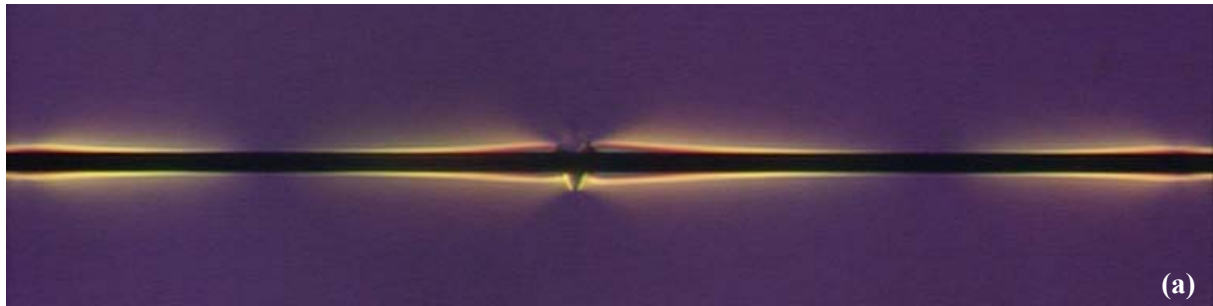
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APPENDIX A

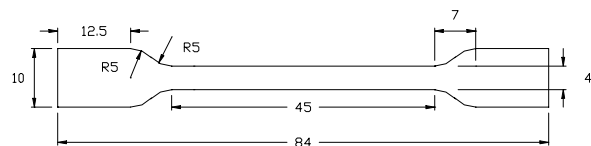
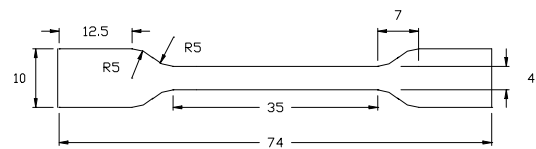
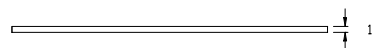
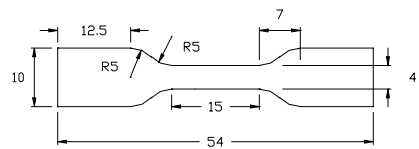
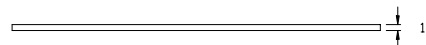
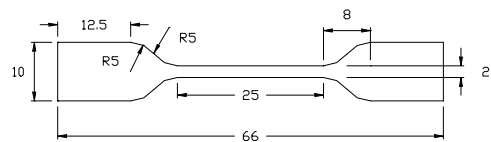
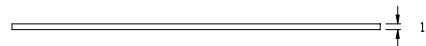
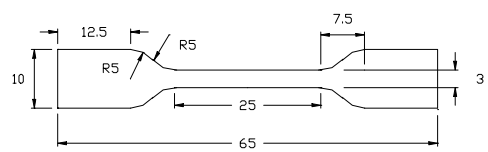
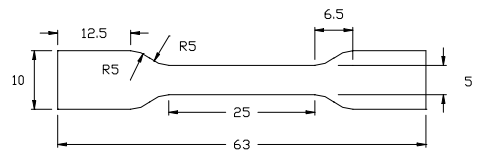
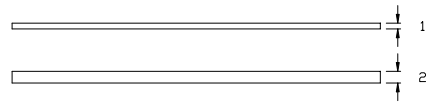
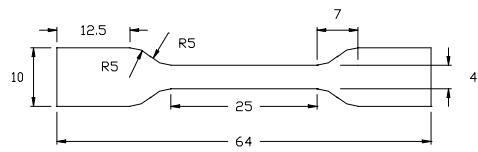
Photoelastic Evidence of Common Failure Modes



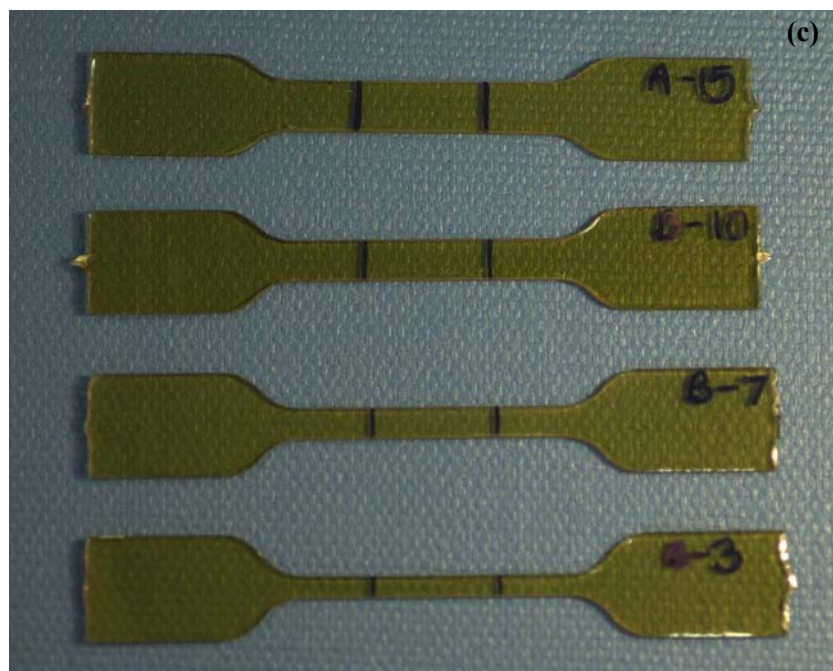
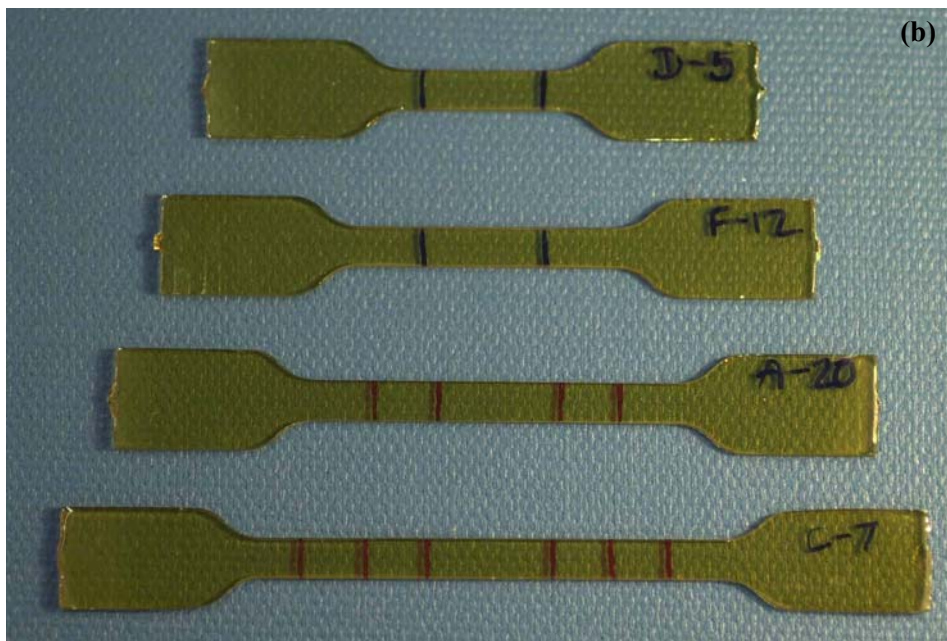
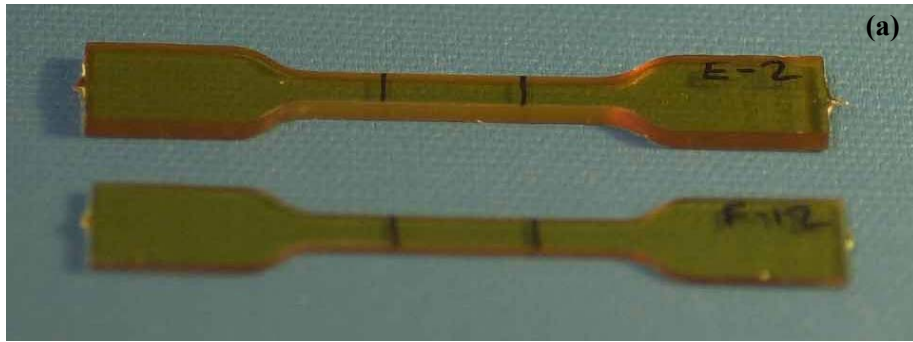
Typical failure processes associated with carbon fibre breaks viewed under polarised light; (a) a strong interface with elliptical resin cracking (matrix yielding for ductile matrices), (b) a weak interface with debonding and (c) a very weak interface showing frictional debonding.

APPENDIX B

Specimen Geometries



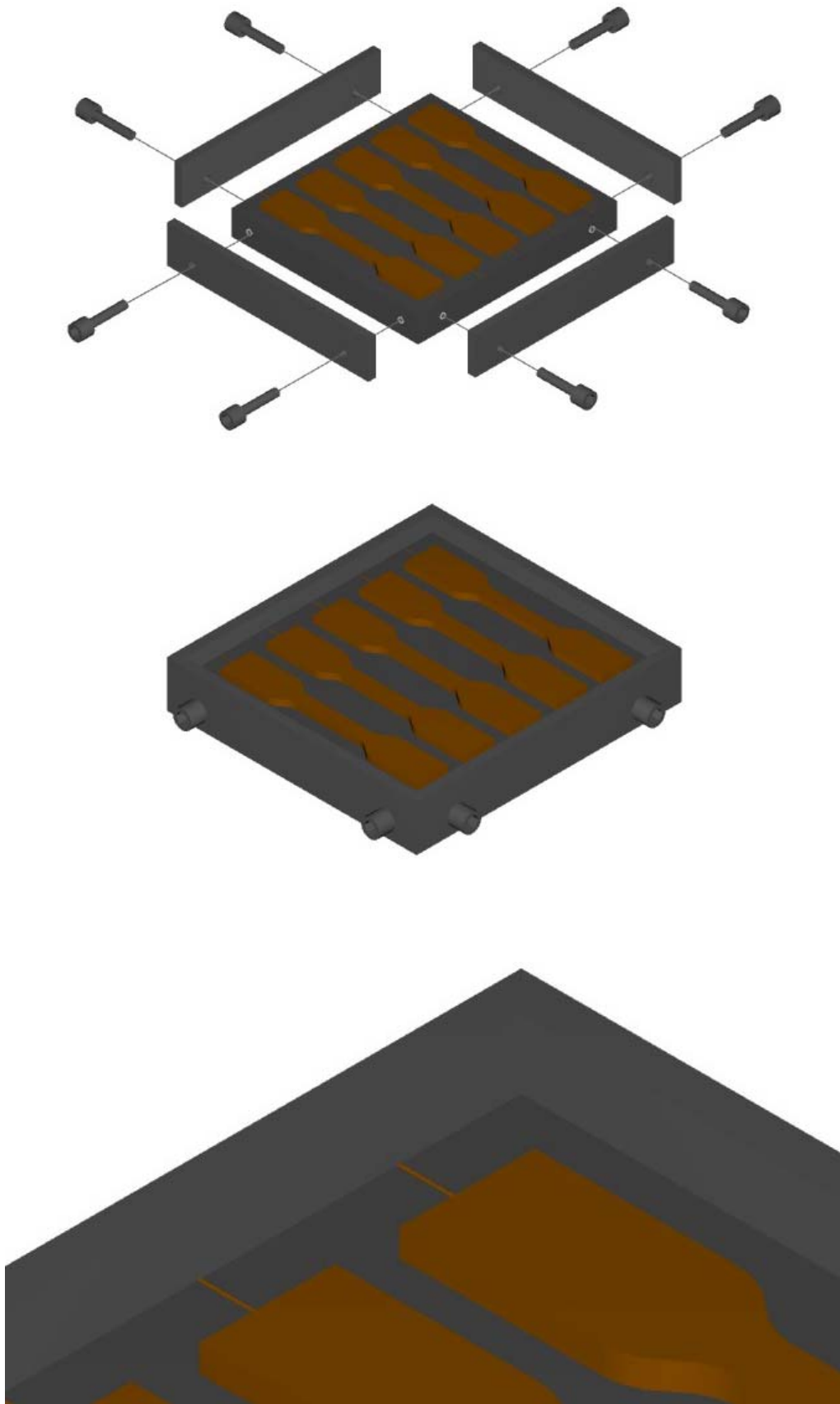
All dimensions in mm. All geometries based on reference specimen (top). Study of thickness effect (top), study of width effect (middle) and study of gauge-length effect (bottom).



Typical specimens: (a) 1 and 2 mm thick; (b) 15, 25, 35 and 45 mm gauge-lengths and (c) 5, 4, 3, and 2 mm widths.

APPENDIX C

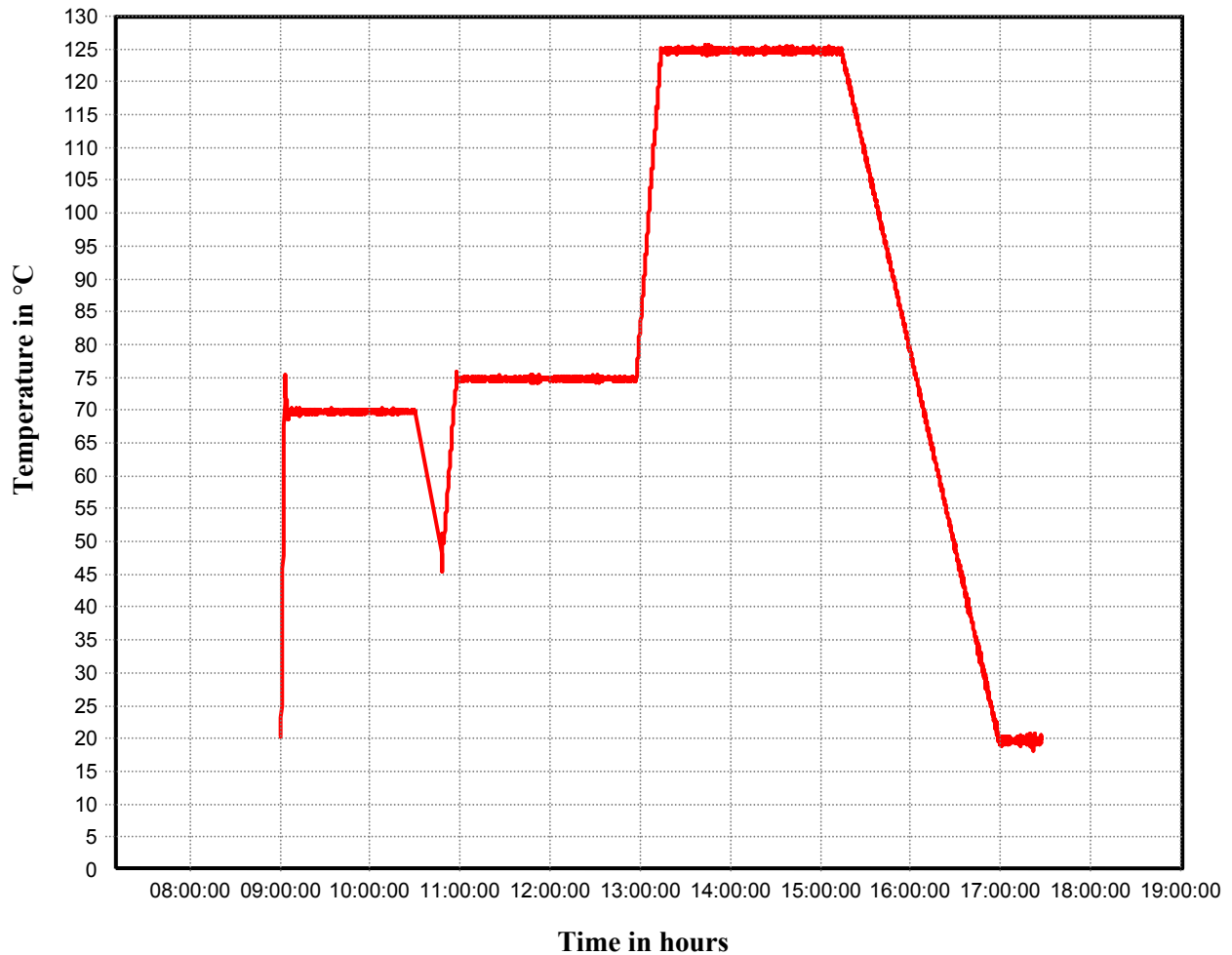
Improved Mould Template Design



Polished multi-specimen steel template with edge dams built-in, enabling simple preparation and removal of silicone moulds. Exploded view (top) and close-up (bottom).

APPENDIX D

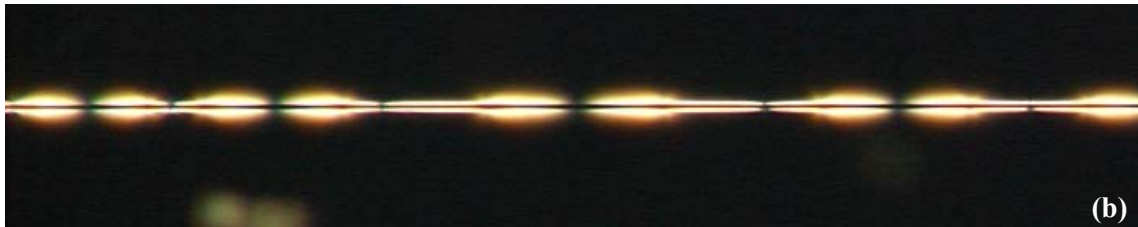
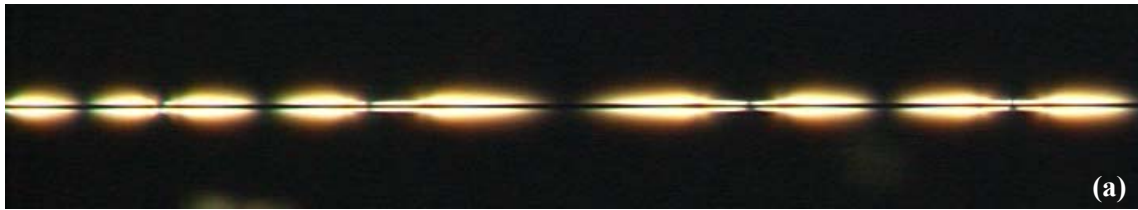
Typical Cure Schedule



Actual cure profile showing initial pre-heating stage, large drop as moulds filled and subsequent ramped cure schedule with 2 hr dwells at 75°C and 125°C and controlled cooling period to room temperature.

APPENDIX E

Photoelastic Evidence of Saturation



The definition and identification of saturation can be aided by photoelastic patterns around fibre breaks: (a) unsaturated breaks in standard carbon specimens showing tapering diffuse clouds extending away from the fracture, (b) the same region at saturation showing rounder, sharper edges and greater intensity to the diffuse clouds with a distinct dark band between them; (c) saturated breaks in Glass-162 showing similar features.

APPENDIX F

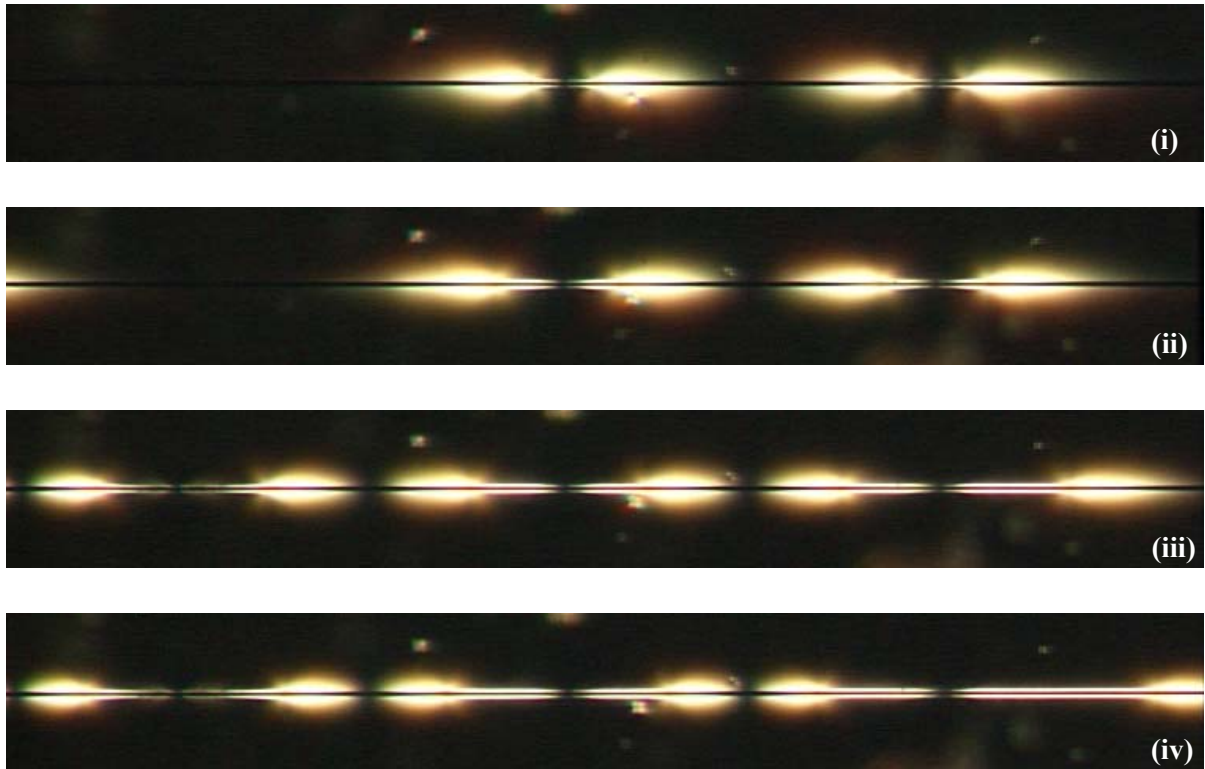
Damage Progression in Carbon Fibre Specimens



Appendix F1 – Images detailing damage progression in a single break in a standard carbon specimen under full extinction with increasing stress; (i) ~45MPa, (ii) ~52MPa, (iii) ~57MPa, (iv) ~60MPa and (v) ~66MPa.



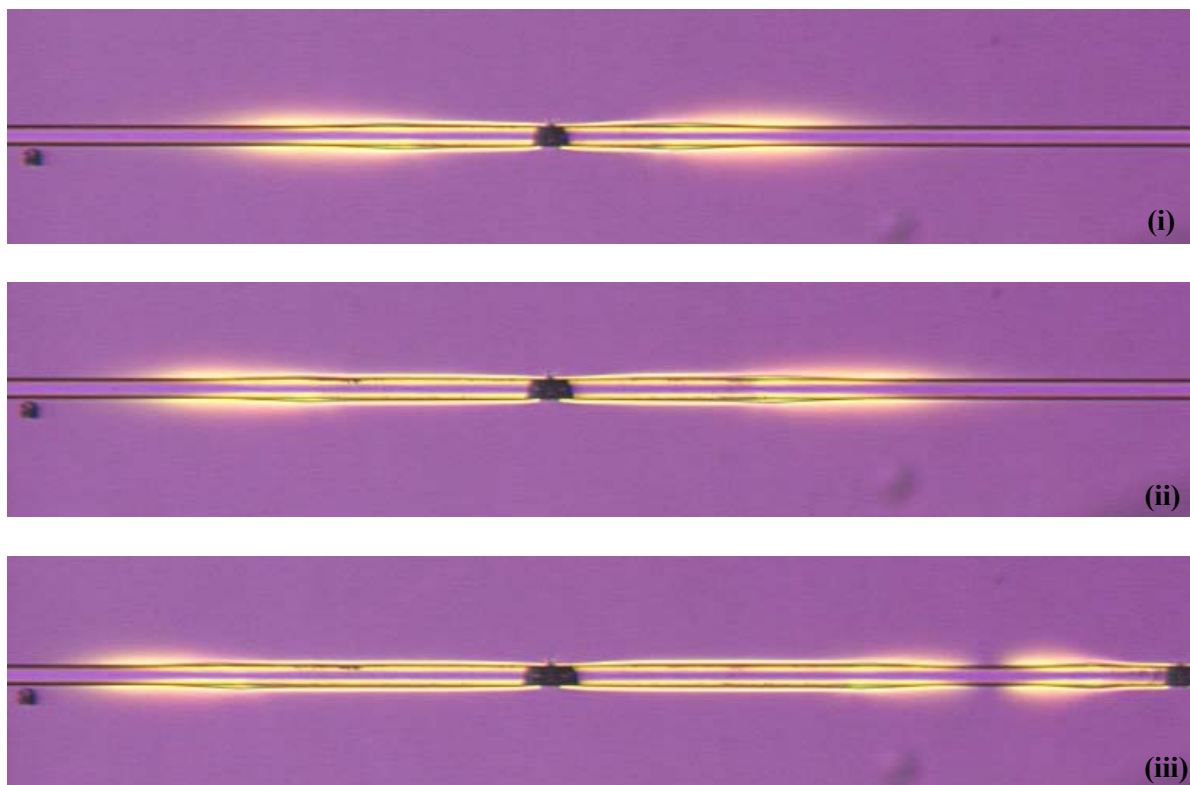
Appendix F2 – Images detailing damage progression in a single break in a release agent coated carbon specimen under polarised light with increasing stress; (i) ~45MPa, (ii) ~49MPa and (iii) ~55MPa.



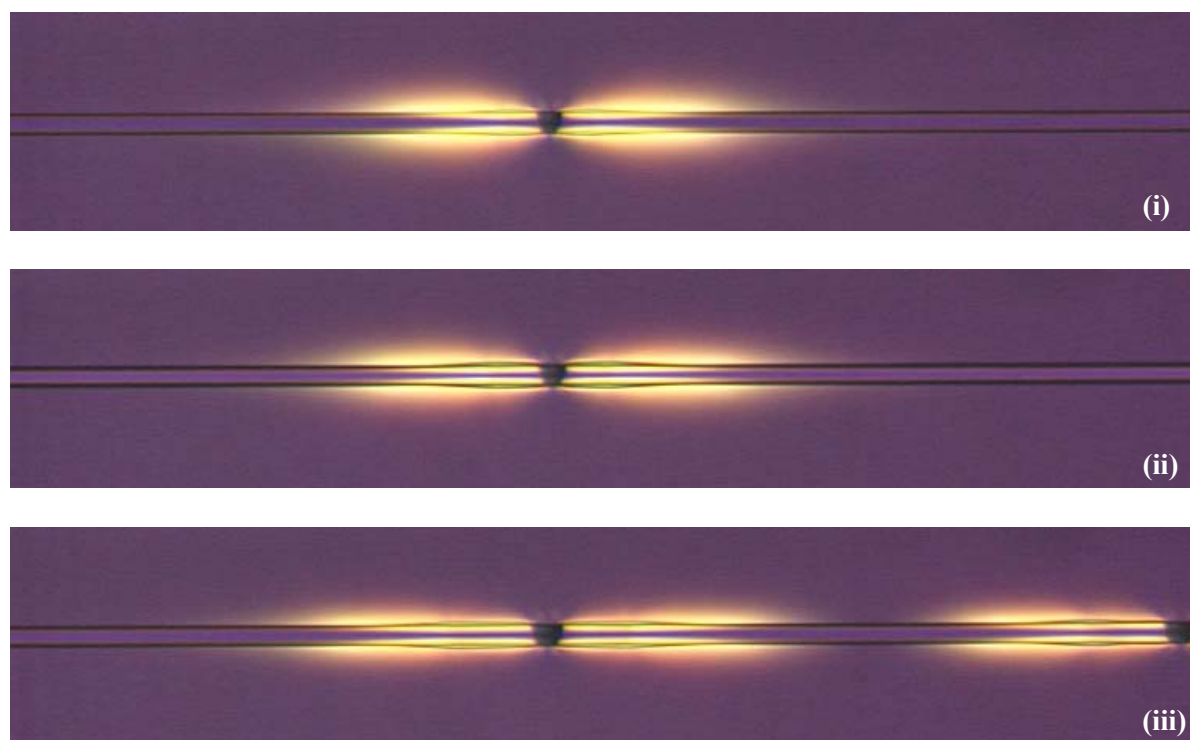
Appendix F3 – Images detailing damage progression in a single break in an environmentally conditioned carbon specimen under full extinction with increasing stress; (i) ~35MPa, (ii) ~41MPa, (iii) ~45MPa and (iv) ~51MPa. Speckling visible in matrix is due to moisture damage.

APPENDIX G

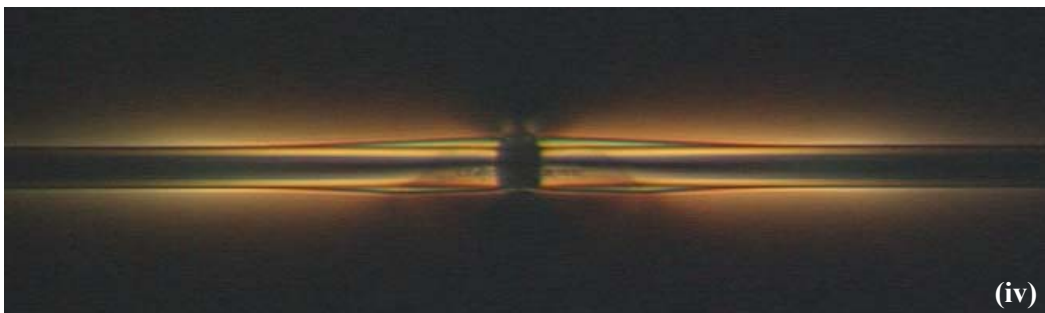
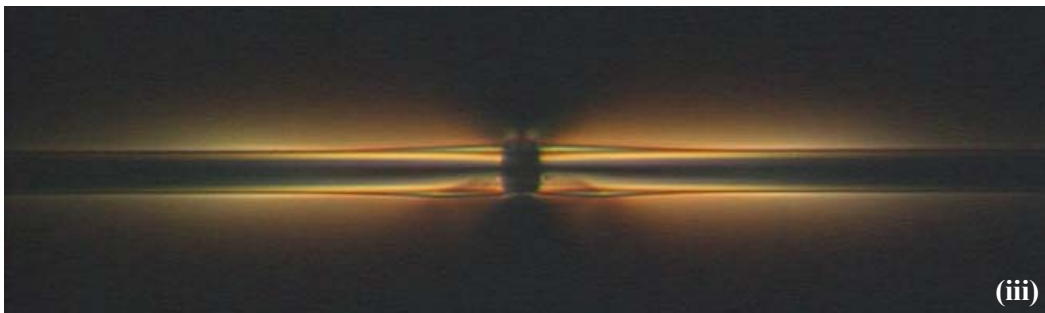
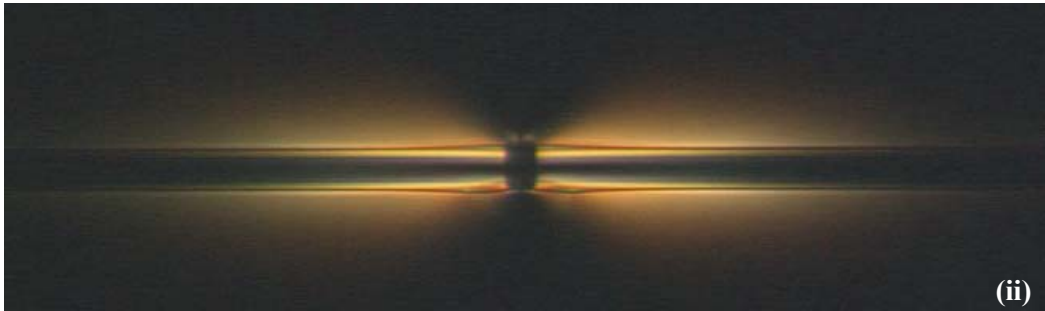
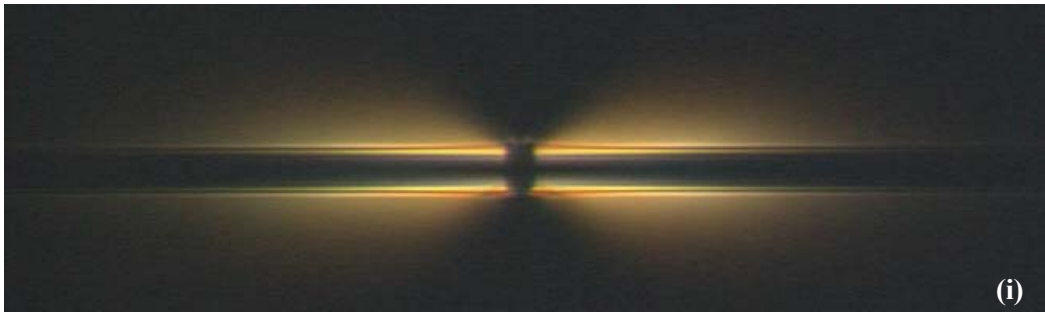
Damage Progression in Glass Fibre Specimens



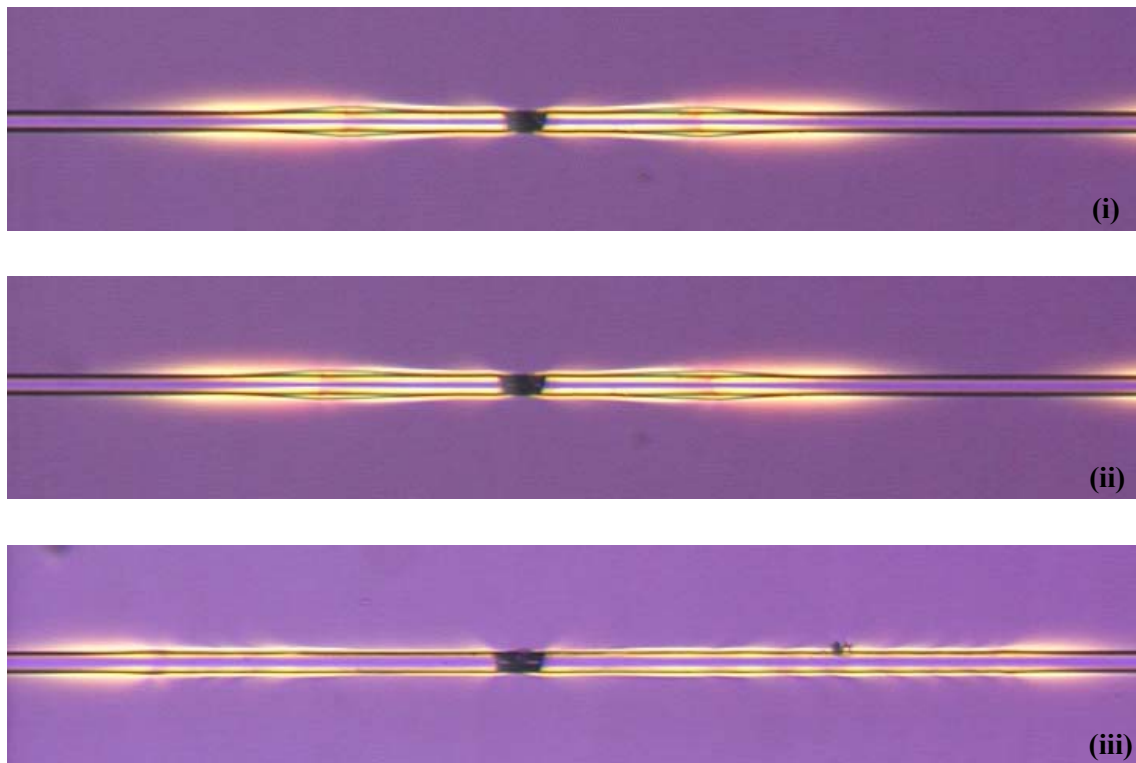
Appendix G1 – Images detailing damage progression in a single break in an E-glass-162 specimen with a water and epoxy compatible sizing under polarised light with increasing stress; (i) ~66MPa, (ii) ~71MPa and (iii) ~76MPa.



Appendix G2 – Images detailing damage progression in a single break in an E-glass-163 specimen with a water and epoxy compatible sizing under polarised light with increasing stress; (i) ~64MPa, (ii) ~71MPa and (iii) ~79MPa.



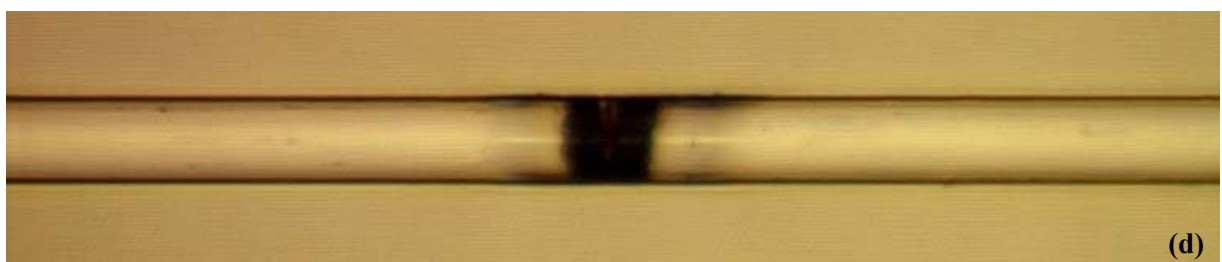
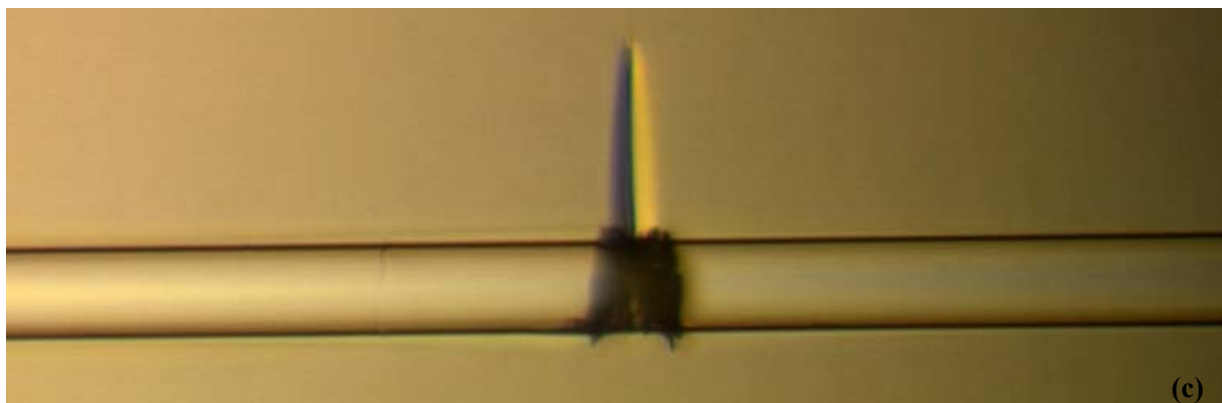
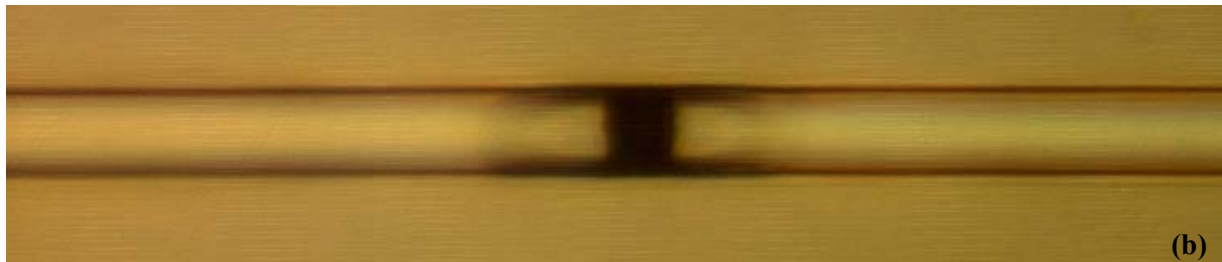
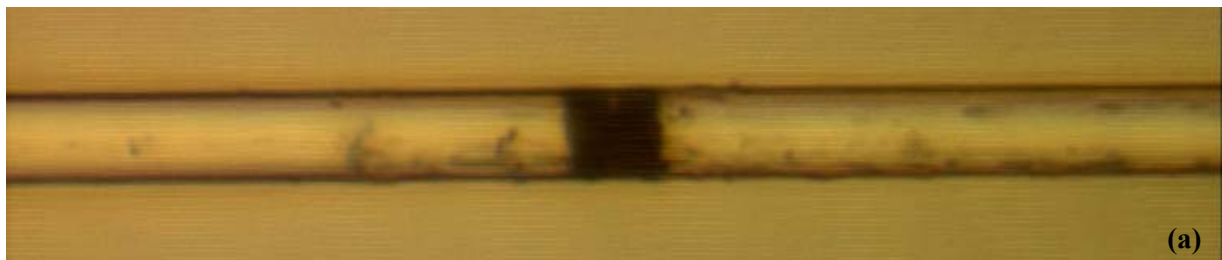
Appendix G3 – Images detailing damage progression in a single break in an E-glass-164 specimen with a water and epoxy compatible sizing under full extinction with increasing stress; (i) ~38MPa, (ii) ~54MPa, (iii) ~62MPa and (iv) ~70MPa.



Appendix G4 – Images detailing damage progression in a single break in an E-glass-165 specimen with a water sizing treatment under polarised light with increasing stress; (i) ~85MPa, (ii) ~86MPa and (iii) ~88MPa.

APPENDIX H

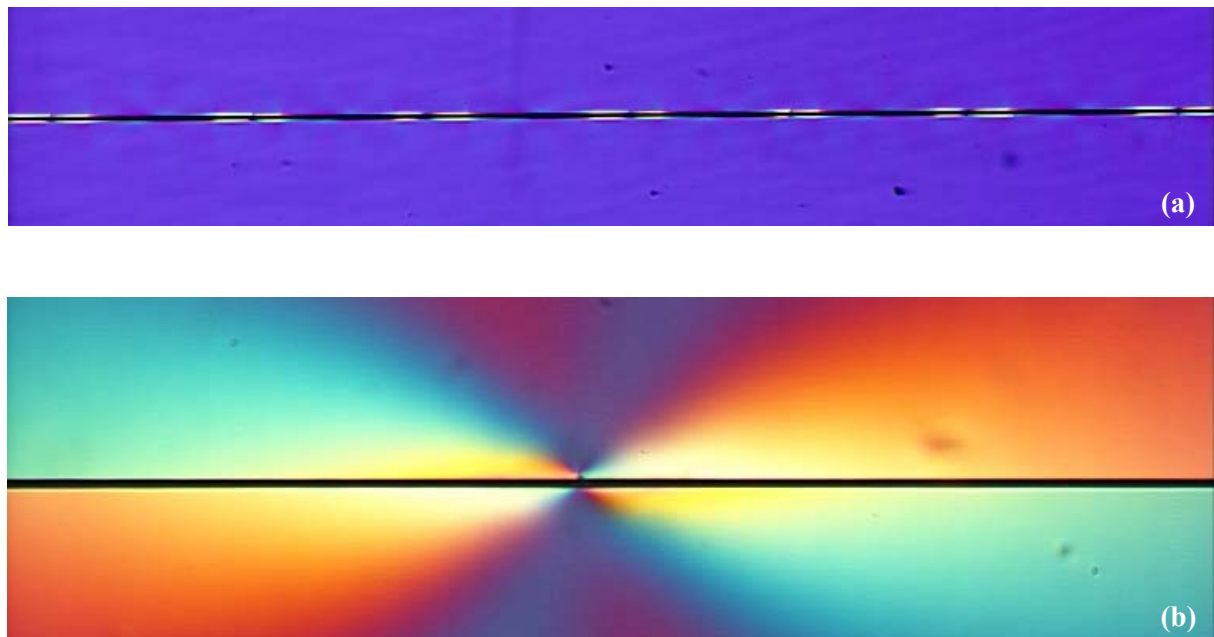
Typical Fibre Breaks in Glass Fibre Specimens



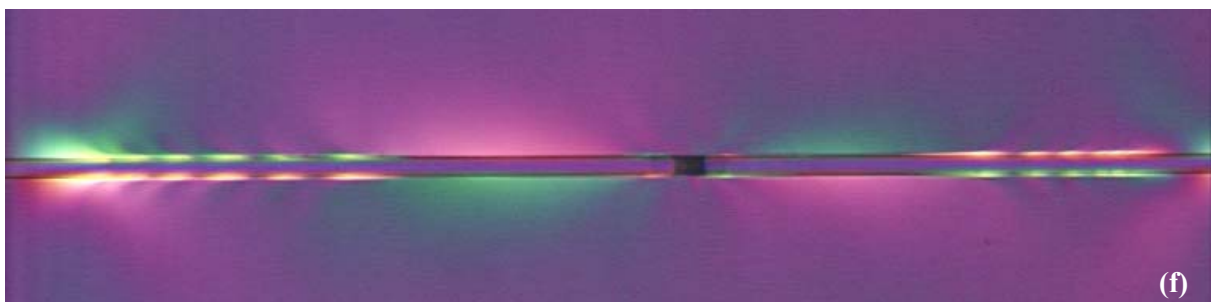
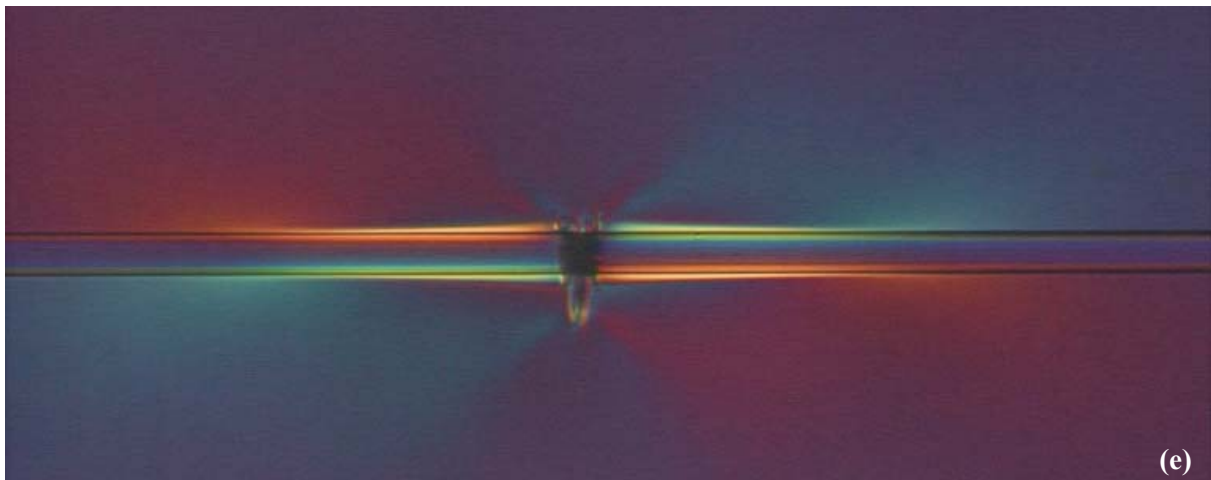
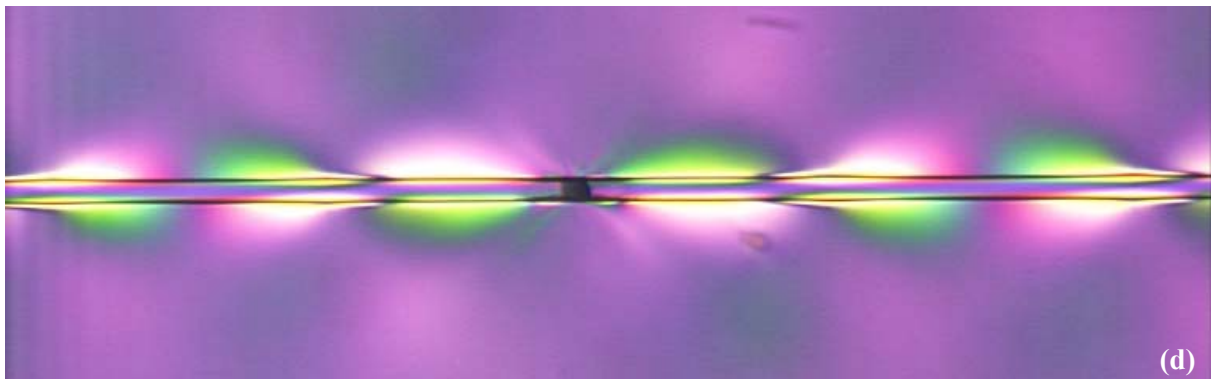
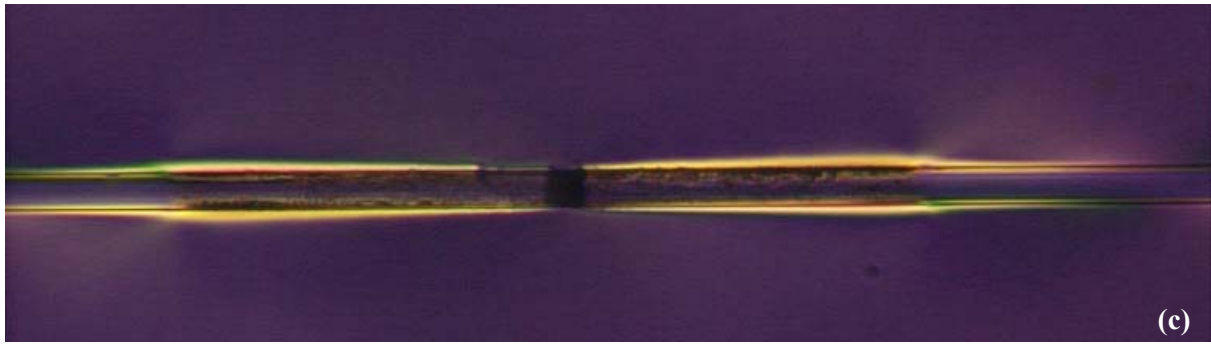
Typical glass fibre breaks viewed under reflected light; (a) Glass-162, (b) Glass-163, (c) Glass-164 and (d) Glass-165. Glass-162 exhibits a distinctive rough debonded fibre surface, Glass 163 and 165 show some interfacial debonding and Glass-164 exhibits extensive matrix cracking with no debonding.

APPENDIX J

Residual Photoelastic Distributions around Breaks in Unloaded Specimens



Typical residual photoelastic patterns in unloaded carbon fibre breaks; (a) standard carbon showing debonded lengths, (b) pre-test break in conditioned carbon specimen caused by thermal and moisture expansion stresses.



Typical residual photoelastic patterns in unloaded glass fibre breaks; (c) Glass-162 showing debonded length, (d) Glass-163 showing debonded lengths and friction induced periodic intensity distribution, (e) Glass-164 showing high matrix stresses around break and (f) Glass-165 showing debonded length and low levels of friction induced periodic intensity distribution.