A technique for the measurement of reinforcement fibre tensile strength at sub-millimetre gauge lengths

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Abstract

The strength of composite reinforcement fibres is normally measured on samples of much greater length than the actual residual fibre lengths found in many composite materials. This is due to a number of limitations of the standard techniques which are employed. We present a description of a technique which enables values for the tensile strength of composite reinforcement fibres at short gauge lengths to be obtained. The technique is based on an adaptation of a micro-mechanical test apparatus for fibre pullout measurements. Data is presented which was obtained at gauge lengths of 180-380 μm on E-glass and S-2 glass® fibres taken from different chopped reinforcement products. The technique can be used at gauge lengths as short as 20 μm. The data indicates that the values of average fibre strength in these products are significantly below the pristine glass strength values.

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Introduction

Fibre reinforced thermoplastic (FRTP) such as polypropylene, polybutyleneterephthalate and the polyamides are excellent composite materials in terms of their performance-processibility-profitability ratios. By far the largest proportion of the market for FRTP's utilizes glass fibres as the reinforcement due to their excellent performance-cost ratio. The mechanical properties of thermoplastic composites containing glass fibres has been the subject of much attention. These properties result from a combination of the fibre and matrix properties and the ability to transfer stresses across the fibre-matrix interface. Variables such as the fibre content, diameter, orientation and the interfacial strength are of prime importance to the final balance of properties exhibited by such thermoplastic composites[1-6]. Considering the fibre contribution to composite performance, a 'good' glass fibre product delivers the correct (as required by the end-use application) balance of stiffness, strength, aspect ratio (length/diameter) and adhesion[7-15]. The initial stiffness and strength of a glass fibre is a function of the glass composition and the fibre processing conditions. During the glass fibre manufacturing process the structure of the fibre is 'frozen' in a non-equilibrium state. Consequently, although the stiffness of glass fibre is often considered a constant, fibre stiffness can actually be changed significantly by annealing effects during composite processing[16]. Fibre strength may also change (be reduced) significantly after fibre formation by damage caused during both the fibre and composite production processes[17]. Although there is little available published data on the direct measurement of the residual strength of fibres in a moulded composite part, there is a growing body of indirect evidence indicating that the strength of glass fibres has been significantly reduced by the time that they actually become the load bearing component of a composites[3-7].

The strength of fibres is normally measured by tensile testing at gauge lengths of 6 mm upward. One of the reasons for utilizing tensile testing is the sensitivity of brittle reinforcement fibres to surface damage. Flaws of many types can be introduced either through damage by fibre-fibre contact or contact with the sample preparation and mounting materials. Care must also be taken to avoid any chemical contamination through contact with human skin. Because of these considerations, measurement of fibre tensile strength becomes over-proportionally difficult and time consuming as the gauge length is reduced below the centimeter range. However, if we are to fully understand the contribution of fibre strength to composite performance then we must be able to measure strength at gauge lengths representative of the reinforcement length in the composite. Thomason et al have recently demonstrated the wide range of fibre strength to be found in fibres used in Glass Mat Thermoplastic (GMT). Differences in fibre strength were attributed to different levels of processing damage and fibre sizing protection efficiency. They reported values for average E-glass fibre strength as low as 1.1 MPa in fibres extracted from commercial GMT's. This value is less than one third of the accepted value of 3.5 GPa for pristine E-glass. In this case it was possible to use a gauge length of 10 mm due to the long fibre lengths present in GMT materials. However, this example clearly shows how important it is to have representative values for actual fibre strength when using models to predict composite strength.

One of the largest applications of fibre reinforcement in thermoplastics is as injection moulding materials, traditionally in "short" fibre reinforced extruded compounds, and more recently there has been a surge of interest in "long" fibre compounds prepared by processes other than extrusion compounding. In many cases the average fibre length of both the input fibres and the fibres in the final composite part are well below the current

"normal" lengths for tensile testing. Figure 1 shows an example of how the weight average fibre length of glass fibre changes during processing, from manufacture of the chopped glass through extrusion compounding and finally injection moulding of a glass fibre reinforced polyamide 6,6 test bar. In this case it would not be possible, using standard methods, to determine the effect of any of these processes on the average fibre strength since the fibres are already too short when they are chopped, after which the fibre length only reduces further. Notwithstanding this problem, there have been a number of analyses of the properties of these composites which have shown indirectly that the fibre strength in these materials is also well below the 3.5 GPa often quoted for glass fibre strength. There is an urgent need for the development of tools to measure the effects of processing (such as chopping and moulding) on the properties of reinforcement fibres. Furthermore, if we are ever going to be able to fully understand the structure-property relationships in composites then we must be able to measure the properties of the composite constituent materials in the same state and under the same conditions in which they must perform (e.g. the strength of moulded fibres of sub-millimeter length). Figure 2 shows theoretical data based on the Kelly-Tyson model showing the tensile strength of 33% w/w glass-fibre-reinforced polyamide 6,6 injection moulded composites as a function of the residual fibre strength and length in the composite part. This figure emphasises the need to maintain both the length and strength of the glass fibres through the various processing steps. In particular, we need to better understand the degradation of fibre strength through the processes of fibre production and handling, and composite processing, to the final composite part, if we are to continue improving composite performance. From a fundamental view it is also desirable to be able to measure fibre strength at short gauge lengths for verification of micro-mechanical models of composite properties and for use in "adhesion" measurement techniques such as the fibre fragmentation test. There have been some attempts to go to shorter gauge lengths by using flexural test methods [18]. However, the fact that the test gauge length of the fibre must be contacted at the point of bending makes flexural testing values suspect due to the sensitivity of brittle fibres to surface damage by any contact. For many of the same reasons described above there has recently been a great deal of activity in the development of test methods for measuring a value representative of the fibre-matrix adhesion[19]. The fibre pullout test is a well known example which has recently undergone development to a micro-scale test with very short embedded lengths and free fibre lengths[20-22]. It is an interesting phenomenon of this test that a combination of long embedded lengths, high levels of adhesion, and low fibre strength will lead to tensile failure of the fibre before debonding and pullout occurs. In adhesion measurement terms this result is seen as an unsuccessful test and the sample and data are often discarded. We have been investigating the adaptation of the micro-pullout test to the measurement of fibre strength at very short gauge lengths. In this paper we describe the development of a method capable of measuring fibre tensile strength at gauge lengths down to 20μm and present some initial data comparing different glass fibres.

Experimental

Test equipment

The test apparatus shown in Figure 3 was originally designed to determine the amount of energy dissipated during a single fibre pull-out test [20]. For this purpose, it was designed with all components having a high stiffness. Since the most compliant part of the pull-out test system is the free fibre length, the apparatus had already been optimized for handling very short free fibre lengths (gauge lengths). In the case of fibre strength determination the same arrangement was used with the exception that the polymer droplet was replaced by a second 'paddle' and the fibre was glued at both sides. This enabled measurements to be made at gauge lengths in the range from 3mm down to 20μm.

The basic arrangement for mounting a single fibre test specimen is shown in Figure 4. One part of the fibre is glued to a small aluminum block (sample holder). Cyanoacrylate glue was used because of its fast curing. After mounting this block to the actuator side of the test apparatus, the other side of the fibre is fixed on a 'paddle'. The advantages of mounting the fibre successively in two steps include
- Once one side is fixed the fibre is easier to transport and handle
- No further contact to the fibre is required after fixing one side
- The gauge length of the fibre can be controlled accurately
- The one-side fixed fibre can be adjusted precisely in the force direction when it is mounted to the test apparatus

Typically the dimensions of sample mounting are: 1 mm glued fibre length at each end, gauge length of the fibre: 0.02 to 3 mm, we chose a standard gauge length of 0.3 mm as being representative of the fibre length in 'short' fibre injection moulded composites. The result is a test specimen which is connected to a piezo actuator and a force transducer, as shown in Figure 4. A problem experienced by many researchers in preparing single fibre test samples of small gauge lengths is the wicking of the glue along the fibre length, effectively sealing flaws on the fibre surface and enhancing fibre strength. In order to avoid such problems at these short gauge lengths we used a fast curing glue (Pattex Blitzgel from Henkel) with a relatively high viscosity (similar to honey) which did not exhibit these wicking problems. Figure 5 shows an SEM micrograph of a glued sample – there is no glue observed on the test length of the fibre. The actuator is able to travel 180μm which is enough for gauge lengths up to 3 mm assuming a maximum elongation-to-break of 5%. For longer gauge lengths or for fibres having a higher elongation-to-break, a miniature tensile tester may be used (e.g. 'Minimat' from Polymer Labs Inc.). The piezo actuator has an integrated elongation sensor. It is a part of a closed loop controller system which ensures a long-time stabilized position of the actuator and avoids an electrical hysteresis when changing the position. The force transducer is made of a piezo ceramic which has the advantage of an extraordinary high stiffness. When connected to an charge amplifier, the resolution is better than 1 mN. However, one disadvantage is a small intrinsic charge leakage which causes a drift in the force signal. In case of fibre strength testing, we found that this drift is not a problem, because the actual force at the fibre break can be taken as the height of the drop to the base line (see Figure 6). The force transducer was calibrated regularly with a known weight.

Test procedure

In order to minimize potential damage during the selection of test samples, fibres were selected from a bundle by using a polished wooden needle. Due to the viscous nature of the fibre coating (sizing), the fibres stuck to the needle and could be extracted from the bundle. Fibres were selected from different areas of the sample and only fibres which could be removed from a bundle without bending, appeared undamaged under optical microscopy, and had a uniform sizing thickness were used for the tests. The selected fibres were stored without contacting each other on a sheet of black paper. One side of each fibre was then glued to a small aluminum block (sample holder) to enable ease of handling, as described above. To mount the sample in the test machine and perform a strength test, the small block with the fibre was clamped using an adjustable grip and the actuator was moved to its maximal displacement. Prior to clamping, the sample holder was adjusted in order to align the fibre precisely in the force direction. The position of the paddle, to which the free end of the fibre is fixed, could be varied in three dimensions to enable accurate alignment in of the fibre axis to the force direction as well as adjustment to the desired gauge length. After the alignment procedure, the free end of the fibre was glued to the support. Only a thin film of glue was used in order to ensure complete hardening through the film thickness. After the glue hardened the actuator was slowly moved with a velocity proportional to the gauge length. For the standard gauge length of 300µm, a velocity of 0.5 µm/s was used. The ramp was generated by 12-bit DA converter connected to the controlling computer. Simultaneously the force was recorded and stored in a data file. When the fibre breaks the force drops to the zero baseline and the test is stopped. At the end of each test the sample was examined using an optical microscope to determine whether the break occurred inside the gauge length. Only the tests where the sample broke along the gauge length were used for further data processing. The fibre strength was then calculated from the load at break and the cross section calculated from individual diameter determinations by optical microscopy.

Results and Discussion

Individual fibre strength results for the three glass fibre products are shown as a scatter plot in Figure 7. Samples E1 and E2 were taken from 4 mm chopped E-glass products coated with similar sizings giving compatibility with polyamide matrices. Sample S was taken from a 4 mm chopped S-2 glass® product sized to give compatibility with 'high temperature' thermoplastics. The average fibre diameter and standard deviation of these samples are shown in Table 1. The average diameter of these samples are close enough that the small differences are not expected to be a major contributor to the experimental differences that we discuss below. It can be seen in Figure 7 that the *S2-glass* sample clearly has a significantly higher strength than the two E-glass based samples. The level of scatter in the *S2-glass* sample is similar to the E2 E-glass sample, whereas the E1 E-glass sample has a much greater level of scatter. This may well be a reflection of the time-scale involved in the manufacturing process. Both S and E2 were manufactured by a process where the sizing has a much greater time to interact with the fibre surface before the product is chopped. Chopping of glass fibre is a high speed operation which has a high potential for causing strength reducing flaws in the fibres [23, 24]. It is known that sizings in general and the silane coupling agent, which is almost always a component of glass fibre sizing, play an important role in fibre protection, fibre strength retention and flaw healing mechanisms. The fact that sample E1 is chopped within milliseconds of drawing and sizing could well mean that the fibre protection properties of the sizing are not optimised by the time the fibres are chopped. Figure 8 shows the average fibre strengths and 95% confidence limits for the data from all three samples. This Figure

5

clearly illustrates the higher strength and low scatter in the *S2-glass* data. However, the results for the two E-glass samples are not significantly different at the 95% confidence level. Using the values of 3500 MPa and 4850 MPa for the pristine strength of E-glass and *S2-glass* we calculate a residual fibre strength in the 53-60% range for the E-glass samples and 75% for the *S2-glass* sample. So not only does the *S2-glass* have a higher absolute strength but also has a relatively higher apparent level of strength retention than the E-glass samples.

It is well known that the results from any fibre strength test will depend on the gauge length at which that fibre was tested. Some of the observed scatter in our data may be due to this phenomenon since the measurements presented above were made over a range of gauge lengths (180-380 μm). The fibre strength results are presented as function of the gauge length in Figure 9. Linear regression analysis was applied on samples S and E1 since they were measured over a wider range of gauge length, and the resultant lines are also presented in Figure 9. Both regression analysis show a similar trend for increasing fibre strength at shorter gauge lengths. However, it should be realised that the number of data points is probably too low in this case to place any further significance on these results. It is interesting to note that the effect of gauge length on measured fibre strength does not fully explain the observed scatter in the data. Furthermore, it is clear that sample E1 still has considerably more scatter about the trend line than sample S. In terms of the expected performance of composite made with these materials we note that, although the average strength of E1 and E2 are not significantly different, sample E1 contains some fibre with a very low apparent strength (of the order of 1000 MPa). If one accepts a weakest link hypothesis for initiation of damage and/or failure in composite materials then we might expect that E1 would in fact deliver composites with a lower performance level.

In Figure 10 we summarise some of the fibre strength dependent mechanical properties of injection moulded composites made with E1 and E2 in polyamide 6,6. The data is the average of four separate compounding and moulding trials in which these two glass products were compared. The data for each property have been normalised to the value obtained with E1, this allows the difference between the performance of the two products to be easily compared. The error bars show the 95% confidence limits for each sample. For three of the four properties shown in Figure 9 the composites made with E2 glass have significantly better performance at the 95% confidence level. This result would appear to correlate well with the data on fibre strength present above. Of course the glass fibres in the moulded composite have undergone two significant periods of mechanical and thermal exposure during the compounding and moulding of the composite. For a deeper understanding of the structure-performance relationships in these materials we should ideally compare the residual strengths of fibres in the composites. This requires the development of a fibre extraction technique which does not seriously damage or affect the fibres to be tested. Typical fibre lengths in these materials are in the 0.2-0.4 mm range which up to now has restricted the interest in the development of such a technique, since 'normal' fibre strength testing requires much longer samples. We have now shown that measurement at these short gauge lengths is possible, we will discuss the application of the test method in a future paper.

Conclusions

We have presented a description of a technique which enables values for the tensile strength of composite reinforcement fibres at short gauge lengths to be obtained. The technique is based on an adaptation of a micro-mechanical test apparatus for fibre pullout measurements. Data has been presented which was obtained at gauge lengths of 180-380 µm on glass fibres taken from different chopped reinforcement products. The technique can be used at gauge lengths as short as 20 µm. The data indicates that the values of average fibre strength in these products are significantly below the pristine glass strength values.

Glass	Average Diameter (um)	Standard Deviation (%)
E1	9.5	10.7
E2	9.4	10.1
S	9.2	5.4

Table 1. Number average fibre diameters

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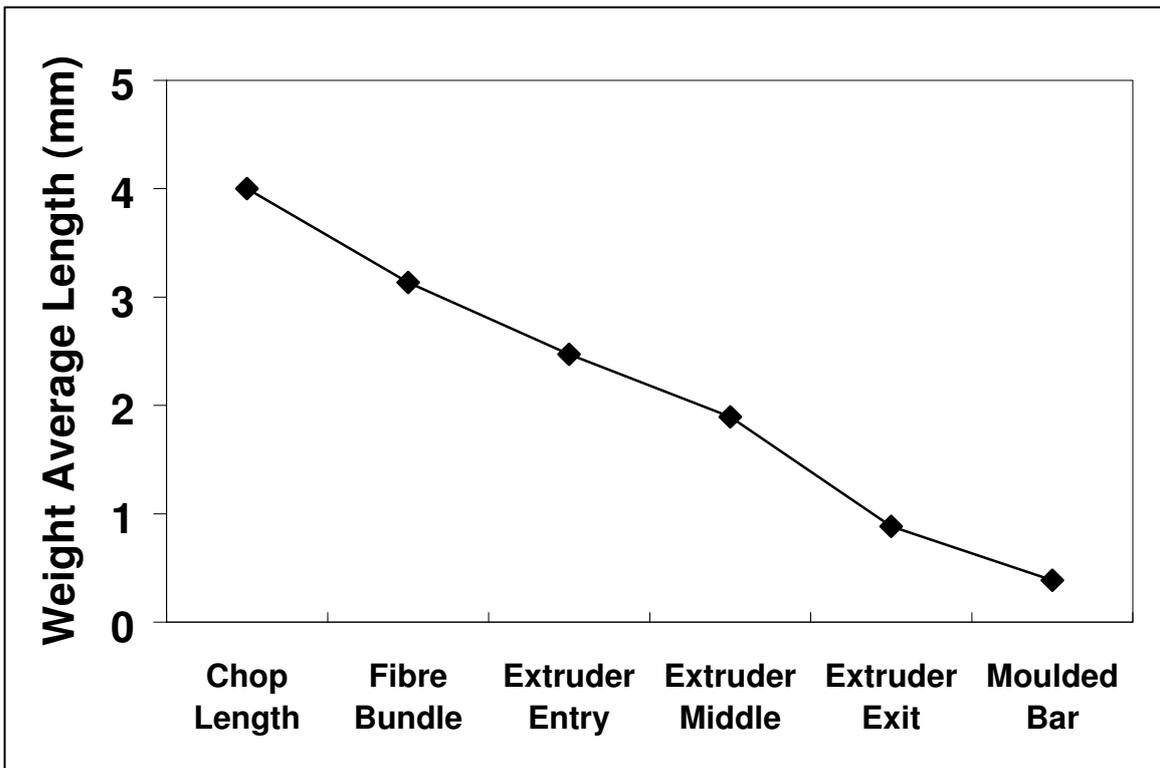
Figure 1: Effect of fibre and composite processing on weight average fibre length
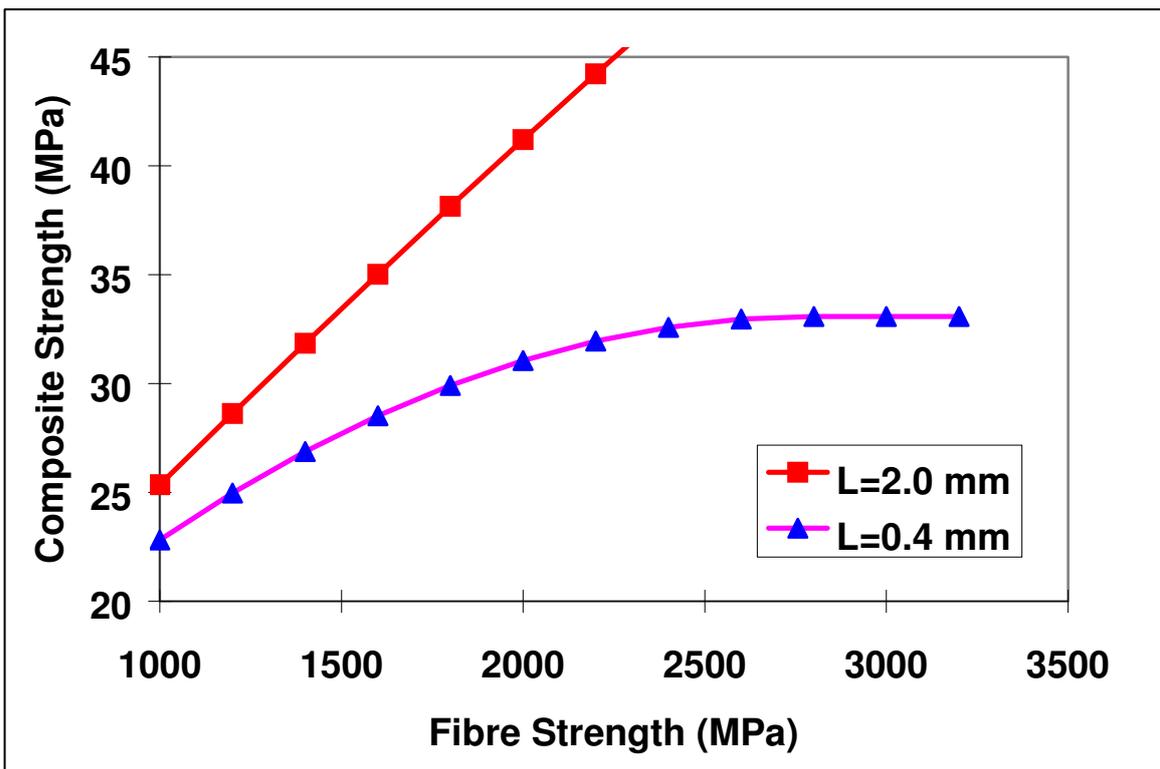

Figure 2: Predicted influence of fibre length and strength on tensile strength of composite
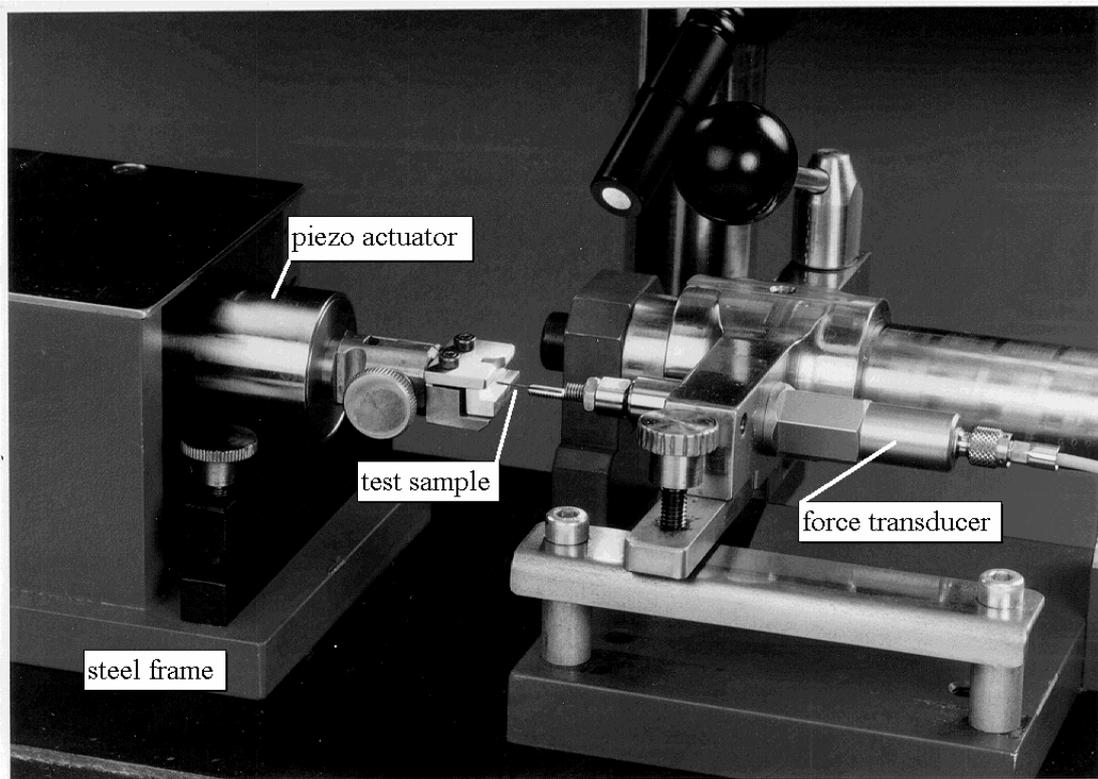
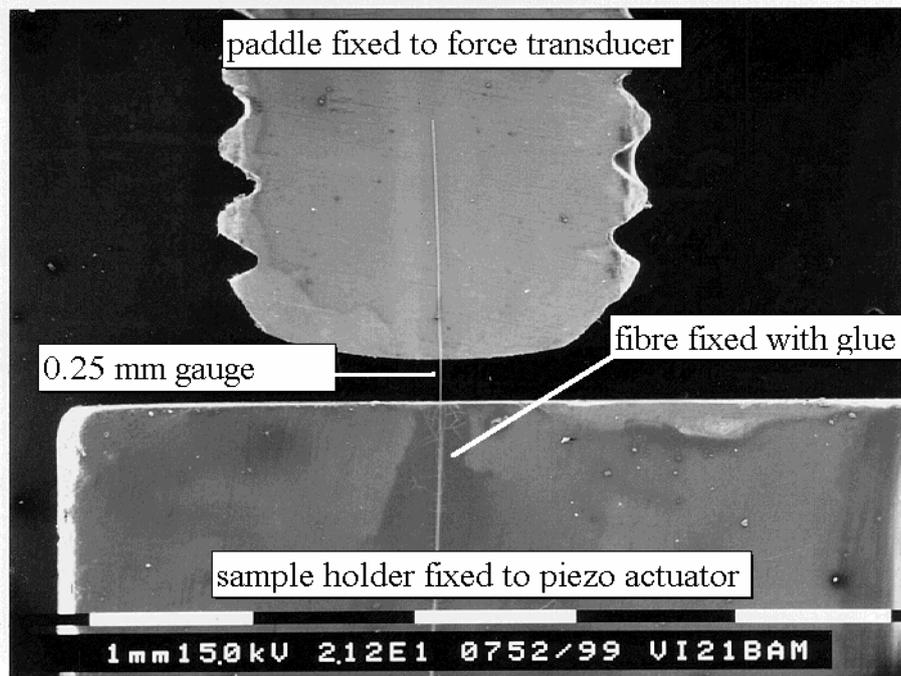Figure 3: Photograph of the test apparatus

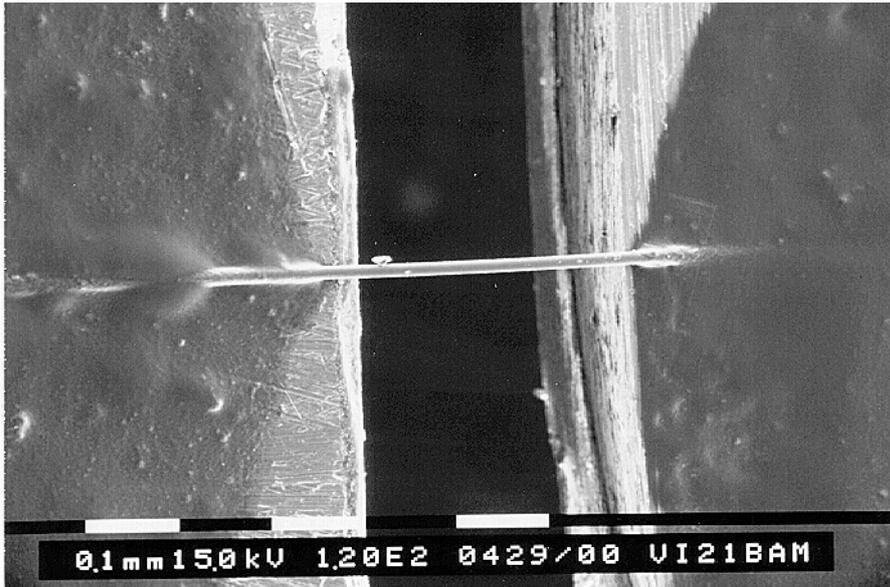Figure 4: Photograph of basic test arrangement for fibre strength measurement

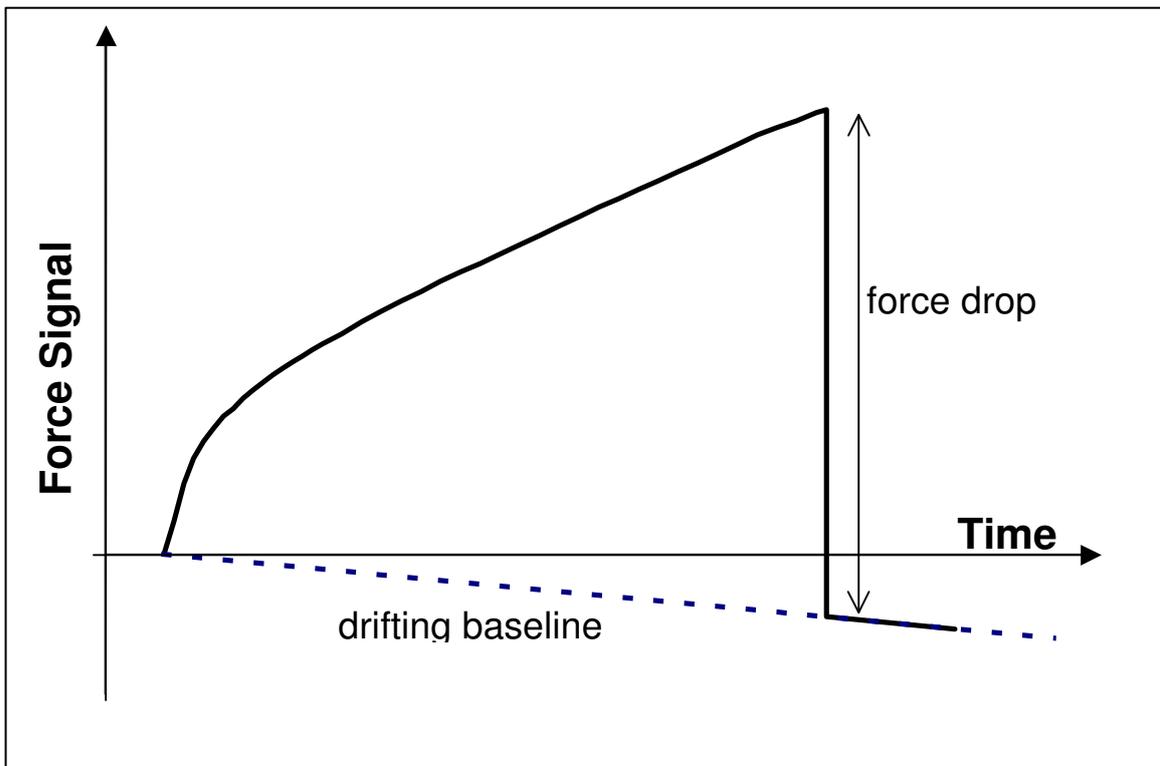Figure 5: SEM showing glue meniscus on fibre

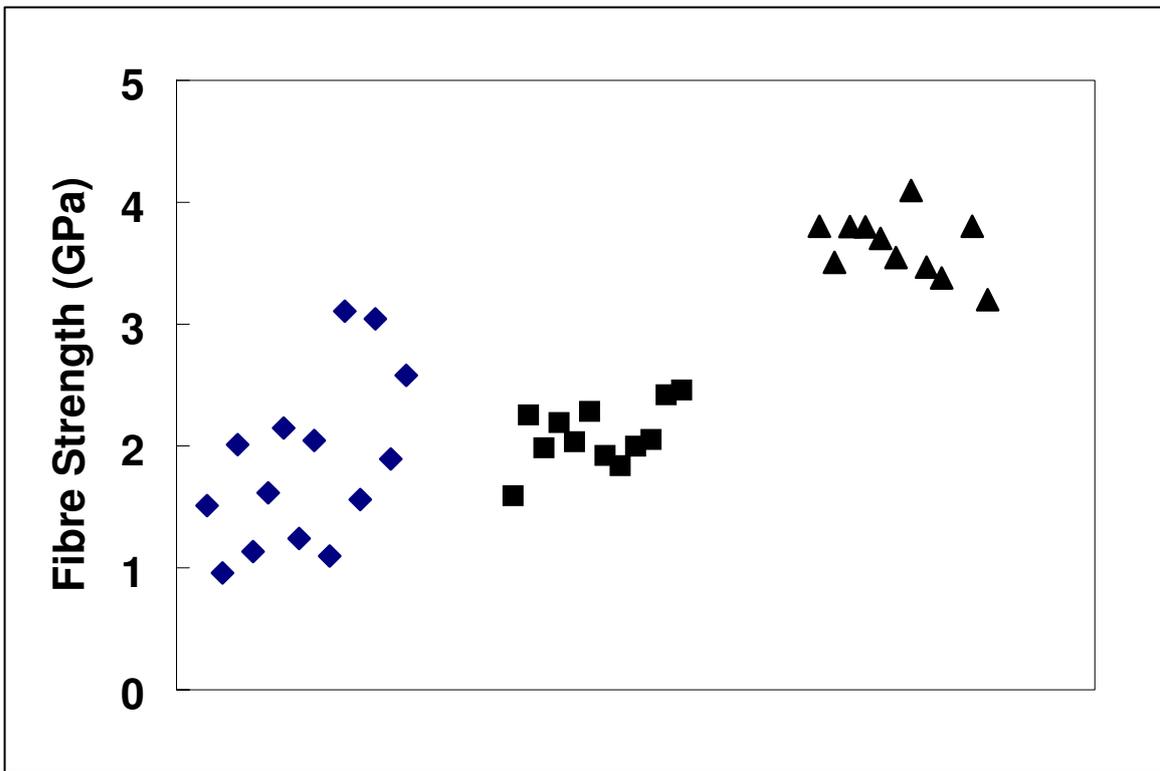Figure 6: Diagram of Force-Displacement curve showing baseline drift
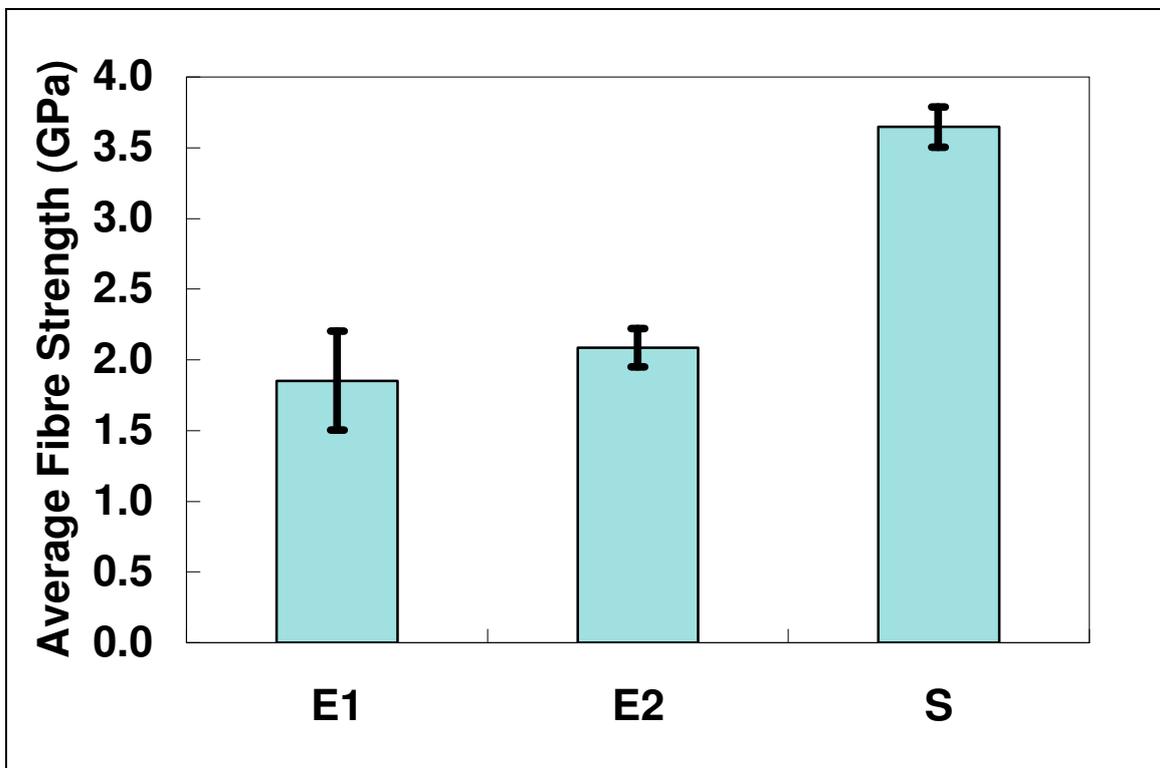
Figure 7: Scatter plot of fibre strength results (υ E1, ν E2, σ S)

Figure 8: Average fibre strengths and 95% confidence limits

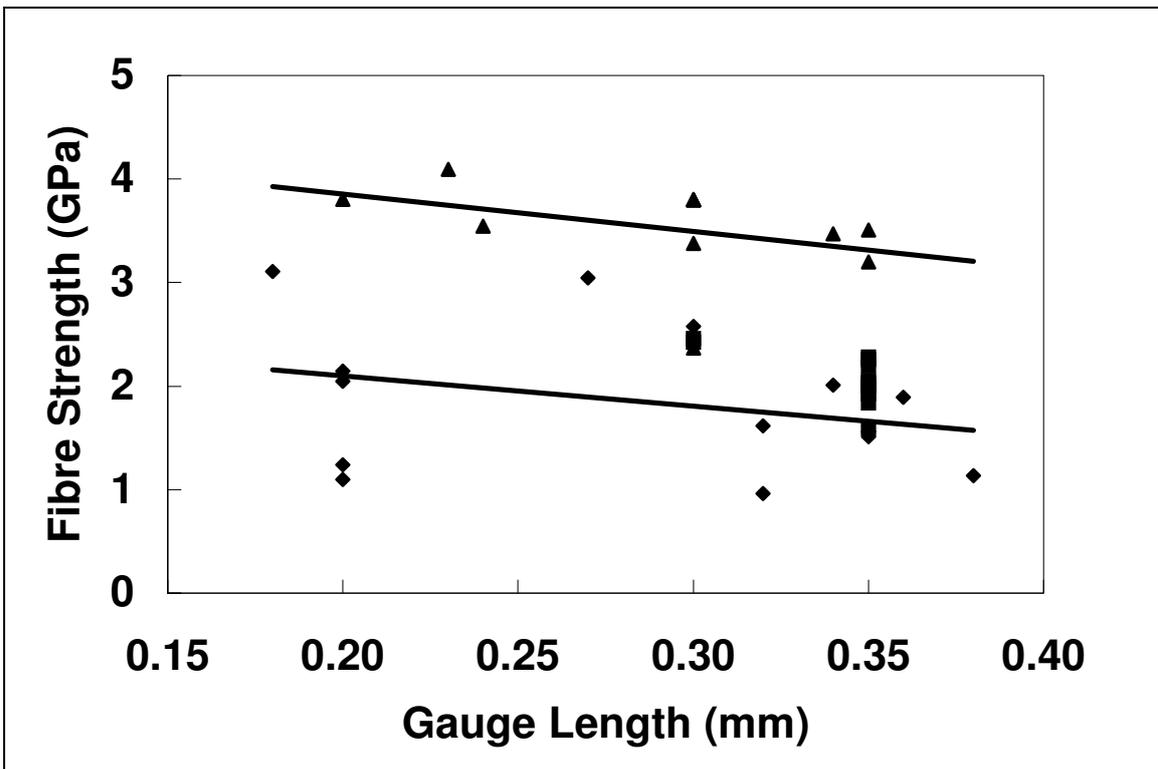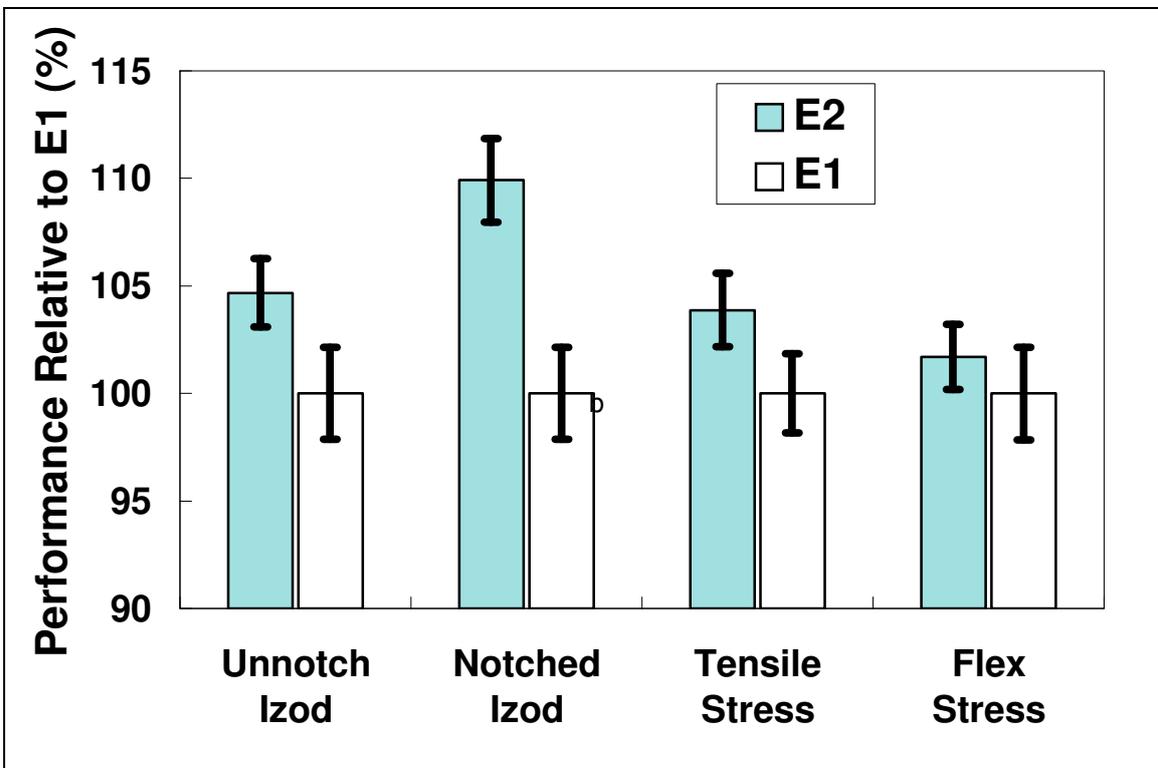Figure 9: Fibre strength versus gauge length (υ E1, ν E2, σ S)

Figure 10: Normalised composite properties