

Micro-mechanical investigation of glass fibre/resin interface failure in mode I and mode II

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Abstract. Fibre reinforced composites are experiencing a period of consistent and significant growth which is predicted to continue, due to the sustained global increase in demand across a number of industrial sectors. One of the critical elements in the success of such composites in both the short and longer term is the interface/interphase between reinforcement fibres and matrix. Poor adhesion will generally lead to composites with insufficient mechanical performance. Degradation of the interface/interphase region during service life will lead to a decrease in performance, eventually to a point where the part can no longer fulfil its intended function. Micro-mechanical test methods can be an advantageous approach to analysing the fibre-matrix interface as they reduce material consumption and simplify the analysis compared with composites. In this paper some of the disadvantages of the commonly used microbond test are discussed, particularly difficulties related to testing of vinyl esters. A new micro-mechanical interface test is introduced which induces failure at the interface in mode I as opposed to the shear failure that occurs with microbond and several other contemporary interface tests.

1. Introduction

The global use of fibre reinforced composite materials continues to grow year on year, across numerous industries. This is driven by their universally attractive properties such as low density, high strength and stiffness and resistance to corrosion, as well as a wide applicability due to the range of reinforcement fibres and matrix materials on the market. Fibre reinforced polymers (FRPs) form a large percentage of the composites market and are relatively mature from a technological viewpoint. A huge array of matrix materials, both thermosets and thermoplastics, are available depending on the requirements of the application. The most widely used reinforcement fibre is glass fibre (GF) due to its high specific properties and low cost. Other important fibres include carbon, basalt, aramid and some polymer fibres. In addition to these generally uniform manufactured fibres there is also a growing market for non-uniform plant-derived natural fibres.

The interface between matrix and reinforcement fibre is one of the most important factors in the success of any composite and significant research effort has been made to understand its role [1–4]. The vast majority of fibres require surface modification to improve compatibility with matrix materials and to produce a strong interface. The precise nature of the interface, or interphase region more generally, is challenging to define and so its discussion is often simplified to one of adhesion. Over the past several decades, a number of micro-mechanical tests have been created and subsequently improved to measure adhesion. In polymer composites research the microbond test has been applied relatively widely, as have



the single fibre fragmentation, pull out and push out tests. Advantages of such tests include provision of direct data about interfacial adhesion and relatively small material consumption compared with composite macro-tests.

All of the interface tests listed above share some important features; for example, test specimens are considered symmetric around the longitudinal fibre axis and the failure induced at the interface is assumed to be of a mode II shear type. The most common data output from these tests is a value of apparent interfacial shear strength (IFSS); although more complex versions have been formulated to obtain parameters such as interfacial fracture energy and shear sliding stress [5]. In the literature, however, some critiques have been levelled at all of these micro-mechanical tests [6, 7]. These include that the centro-symmetric form of failure is unrealistic and that results may be significantly influenced by factors such as fibre surface roughness and cure shrinkage during sample manufacture.

Another challenging aspect of these tests can be the effect of the relatively small scale on sample preparation. The microbond test, for example, has been applied relatively widely to different matrix materials [3, 8, 9] but it is not unusual to encounter effects that must be mitigated against to obtain reliable test data. For example, the apparent adhesion varies due to degradation of thermoplastics [10], with relatively small changes in testing temperature [11, 12] or due to thermoset mixing ratios away from stoichiometry [13, 14].

In this paper, specific challenges related to the micro-mechanical adhesion testing of a vinyl ester (VE) resins system are presented and discussed. The procedure developed to successfully perform adhesion testing between VEs and reinforcement glass fibre is described. Following this, a new micro-scale interface test is introduced which addresses some of the criticism that has been made of the most commonly used micro-scale tests for measurement of adhesion. Some early results from this new single fibre peel test are presented.

2. Experimental

2.1. Materials

Two types of glass fibre were utilised in the work described in the paper. A commercially available Advantex E-glass fibre with 17 μm nominal diameter was provided as a single end continuous roving. The fibre was sized at point of manufacture by a contact roller then dried for 24h as a 20kg package. The size applied was for general compatibility with thermoset resins (including vinyl esters) and constituted approximately 0.6 wt% of the total product weight. It is designated GF_{com} hereafter. A smaller 10 kg roving was supplied by Owens-Corning; it was cooled by water spray following fibre drawing but had no surface size applied. The fibres are, therefore, referred to as being bare or unsized, GF_{us} .

A sample of lab-scale manufactured glass fibre was provided by RWTH Aachen Institute of Textile Technology, produced using a small experimental bushing. Fibres were of E-glass formulation; they were not coated with any size but were cooled by water spray before being wound onto cardboard tubes. 203 individual filaments were provided with continuous length of approximately 50 m. The fibre thickness was about 35 μm ; these unsized fibres are referred to as GF_{ITA} .

A commercially available vinyl ester resin (VE_{vac}) designed for vacuum infusion with approximate styrene content of 50 wt% was supplied. Curing was achieved using the peroxide based catalyst PBC-21 from United Initiators GmbH by mixing at a ratio of 97.5 : 2.5 by weight respectively. The cure and post-cure procedures and their effect on results form part of the research presented in this paper; the manufacturer recommends a cure cycle of 24 h at room temperature followed by 24 h at 60 °C.

Epoxy resin DER 332 and triethylenetetramine were sourced from Sigma Aldrich and mixed at the stoichiometric ratio. The resin was mixed and degassed under vacuum for approximately 10 mins before application. The cure cycle utilised was heating to 60 °C for one hour followed by a further two hour isothermal at 120 °C, with intermediate heating rates of 2 °C/min.

2.2. The microbond test

The adhesion between reinforcement fibres and VE/epoxy resin was measured using an in-house designed microbond rig. The experimental setup is based on the original conception of Miller et al. [15] and has been described in detail in the literature [16]. A schematic of the system used is shown in Figure 1.

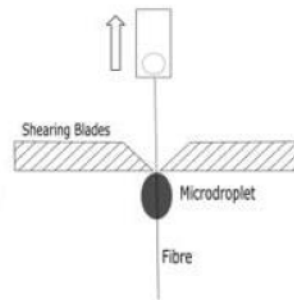


Figure 1: Image and diagram showing parts of the microbond rig used

Tests were carried out at a constant extension rate of 0.1 mm/min and only droplets within a diameter range of approximately 70-120 μm were selected. The apparent interfacial adhesion of each sample was calculated using (1)

$$\tau = \frac{F_{max}}{\pi d_f l_e} \quad (1)$$

In this equation F_{max} is the peak of the load deformation plot during debonding, d_f the fibre diameter and l_e the embedded length of the fibre within the droplet. At least 20 individual tests were carried out for each condition to obtain an average apparent interfacial shear stress (IFSS).

The sample preparation process comprises several steps which are of particular importance in this work due to the influence they can have on the value of IFSS that is obtained. First, individual fibres are isolated and affixed to card frames with an adhesive so that resin can later be applied in a controlled manner. The standard adhesive used for this purpose is a cyanoacrylate gel superglue which is given 24 hours to fully dry. Next, a VE or epoxy resin was mixed following the relevant directions and applied to fibres using a thin piece of steel wire to produce droplets in the preferred size range. The desired cure cycle was then initiated immediately.

3. Microbond testing of vinyl ester resin

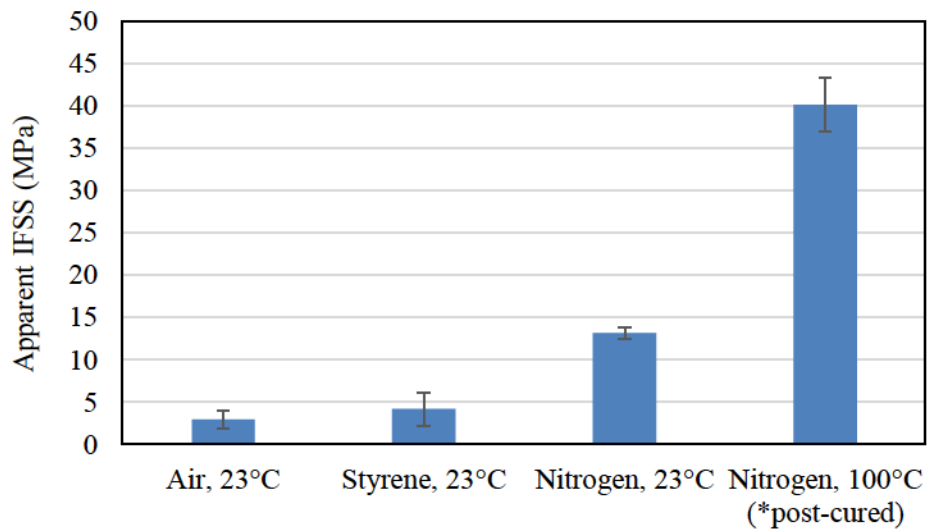
A significant volume of data regarding the microbond testing of various fibres with epoxy resins and with many different thermoplastic polymers can be found in the literature. Research on polyesters and vinyl esters, in particular, are less prevalent although some can be found [9, 17].

The following sub-sections discuss results acquired using the fibre GF_{com} and resin VE_{vac} . Several factors were found to exert a significant influence on the apparent IFSS values obtained, all of which must be controlled in order that meaningful results are generated, therefore allowing comparisons to be made between fibre sizes and resin formulations, for example.

3.1. Proper cure of samples: atmospheric control and post-cure

Vinyl ester resins are capable of achieving similar mechanical performance as some epoxies, but often with more straightforward processing requirements. The expected apparent IFSS for glass fibres with

many epoxy resins is in the range 40-50 MPa [18] when proper cure is achieved. Values obtained for commercially available GF and vacuum infusion VE resin are given in Figure 2.



Cure atmosphere, maximum cure temperature

Figure 2: Apparent IFSS between GF_{com} and VE_{vac} dependent on cure atmosphere and maximum post-cure temperature

Very low values of apparent IFSS were obtained when initial curing for 24 h was carried out under air or a styrene-rich atmosphere. In reality droplets do not cure and solidify sufficiently to bear load, so there is no measurable interface by this method. Performing the initial 24 h cure under inert nitrogen allowed sufficient curing of droplets such that they could bear load during the microbond test, yielding a value of 13.2 MPa. Addition of a post-cure procedure at elevated temperature significantly increased the IFSS to around 40 MPa. The post-cure utilised was adapted from the manufacturers recommendation for vacuum infused panels: following 24 h at room temperature (under nitrogen) samples were post-cured in a convection oven at 60 °C for 24 h, 80 °C for 3 h and finally 100 °C for 1 hour.

The most critical step in the preparation of VE test specimens for microbond is initial curing under an inert atmosphere such as nitrogen. In the data presented in Figure 2 this was achieved using a vacuum oven to replace the air with dry nitrogen from a cylinder. Doing so significantly reduces the oxygen inhibition effect [19] which acts against the network forming polymerisation reaction.

The post-cure at significantly elevated temperature is required in order to increase the degree of cure of droplets. A standard DSC temperature ramp program was used to investigate the excess enthalpy of samples of VE resin which experienced different post-cure temperatures. Examples of these results are shown in Figure 3. The samples used were not taken directly from microbond specimens; they were prepared alongside them from the same batch of resin and distributed on release film to produce droplets with mass of 10-15 mg.

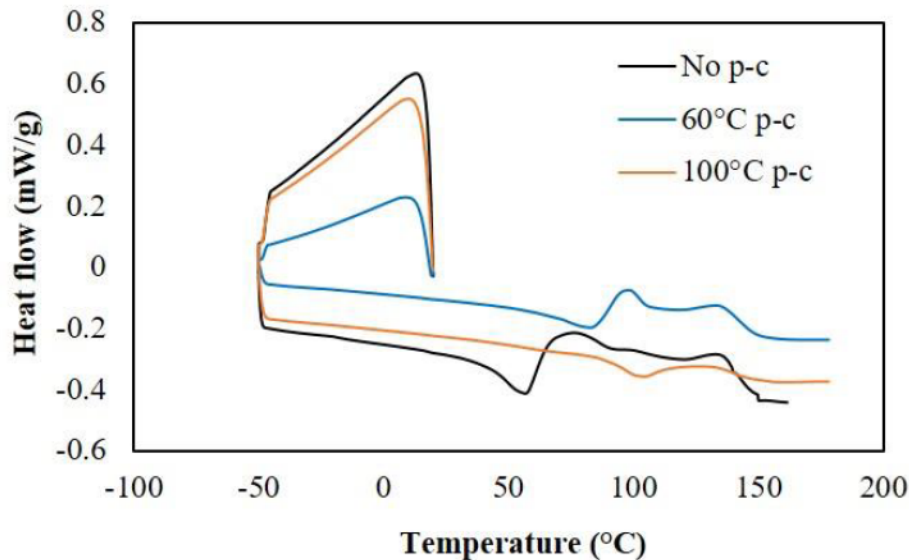


Figure 3: DSC curves (first temperature ramp) of VEvac samples cured at room temperature for 24 h under nitrogen, followed by (a) no post-cure (b) 60 °C for 24 h (c) 60 °C for 24 h, 80 °C for 3 h, 100 °C for 1 h

Elevated temperature following the initial room temperature cure induces further cross-linking within the VE network and this exothermic reaction can be observed in the data in Figure 3. Application of post-curing increases the initiation temperature for the exotherm and reduces the enthalpy of the reaction as expected. Without post-cure the exotherm initiated at 58 °C and excess enthalpy was 70.2 J/g. When a maximum post cure temperature of 60 °C or 100 °C was applied this shifted to enthalpy 31.2 J/g at 85.6 °C and 7.87 J/g at 105.6 °C respectively.

Testing of interfacial adhesion between glass, or other reinforcement, fibres and vinyl ester resins is possible using the microbond test. However, care must be taken to ensure that specimens are properly cured and failure to do so may lead to misleading results that underestimate the level of adhesion. In addition to initial curing under an inert atmosphere, it is necessary to post-cure samples to maximise degree of cure. The conditions required to achieve sufficient cure can be found from straightforward thermal analysis of the resin. The scale at which this analysis is carried out should, ideally, be as close to that of the specimens themselves; in the case of microbond this is very challenging as droplets have mass of the order of micro-grams.

3.2. Effect of fibre adhesive

The role of the fibre adhesive in microbond testing is only to restrain the individual filaments on a frame (usually made of card) so that droplets of the resin under study can be applied in a controlled manner. It is not considered that it should have any influence on the test, because it is applied and allowed to fully dry before the resin is introduced with which adhesion is to be measured. The fibre adhesive is always used in sufficient volume that the fibre will not slip during testing; the embedded length of fibre within this adhesive is many times larger than the embedded length within the resin being tested. Results shown in Figure 4, however, somewhat contradict this prevailing view.

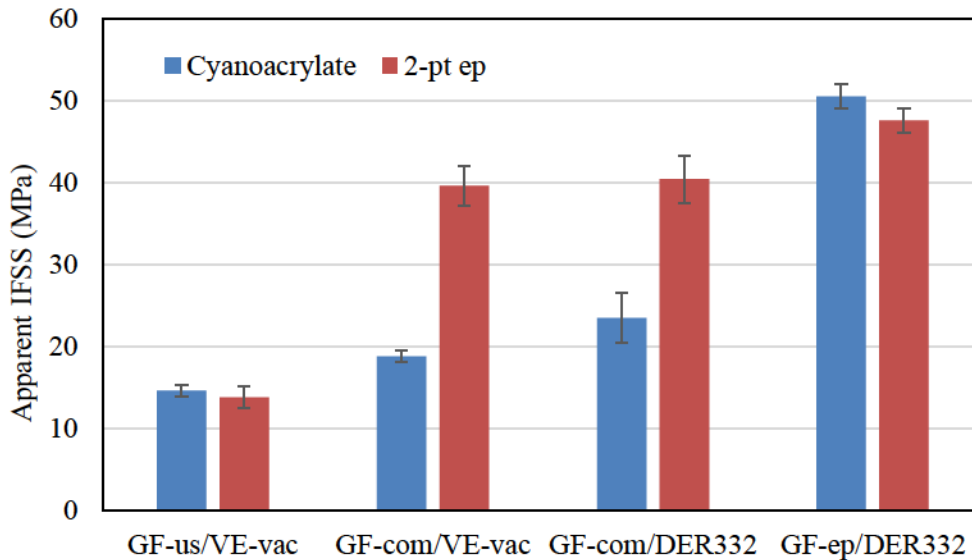


Figure 4: Apparent interfacial shear strength of various glass fibre/resin combinations from microbond testing. Fibre 'GF-ep' is a commercially sized product specifically designed for compatibility with epoxy resins

When using unsized GFs the adhesion obtained is very low between 12-14 MPa. Relatively low adhesion is expected as there is no silane or size present to enhance chemical bonding between the fibre and matrix; instead it is likely a combination of forces from cure shrinkage of the resin and friction. No difference in apparent adhesion was found by changing the fibre fixing adhesive. Similarly, microbond data for an epoxy-compatible GF (GF_{ep}) with a simple epoxy resin were unaffected by the fibre adhesive. The value of approximately 50 MPa is typical for DER332 epoxy resin on fully sized glass fibres.

A very clear effect, however, was found when microbond testing of the multi-resin compatible fibre GF_{com} was carried out. The two central pairs of columns in Figure 4 show that low values of apparent IFSS were obtained when fibres were fixed in place with cyanoacrylate 'superglue' adhesive; which has been the standard selection for microbond testing of resins and thermoplastics. By changing only the fibre adhesive, to a 2-part room temperature cure epoxy, significantly higher values of IFSS were measured. The data strongly suggest that this effect is due to some interaction with the fibre size of GF_{com} : it was not observed for different sizing formulation on GF_{ep} or for unsized fibre GF_{us} .

A satisfactory explanation for this effect has yet to be found; in many ways the result is a curiosity as the use of cyanoacrylate based superglues does not appear to present similar problems when microbond testing with other reinforcement fibres. Currently, it is believed to be a peculiarity of the fibre GF_{com} ; therefore, it may be the result of a reaction with an element or elements within this glass fibre size formulation. It is known that application of cyanoacrylate adhesive produces some vapours which can preferentially deposit [20] and initial experiments suggest that this is being deposited on the surface of the GFs. It is not yet understood, however, why this only effects the particular size on GF_{com} fibres.

4. Single fibre peel test (SFPT) – a mode I interface test

4.1. Introduction

As outlined in the introduction, some criticisms have been made of how representative the microbond, and other mode II micro-mechanical interface, tests are in relation to the mechanisms of failure within real polymer composites. A new interface test could, therefore, be of significant benefit to the study of adhesion in composites. In fact, such a method was proposed and demonstrated in a limited capacity

within the literature over 20 years ago. A peel test based on a single fibre was described and results presented using an epoxy resin and several fibre types including glass, carbon and aramid [21, 22].

Peel testing is commonplace for the study of numerous materials at the macro-scale, such as adhesive joints and pressure-sensitive tapes, and testing is governed by numerous standards. Common methods include 90°, 180°, T-peel and climbing drum [23–26]. It has also found some utility in the study of internal interfaces within natural fibres [27]. At a basic level peel testing is simple: an average debonding force and peel length (normal to the direction of peel) must be measured, and from this the energy of fracture may be calculated.

4.2. Test specimens

In previous work, small capsule of resin were allowed to gel for several hours; then fibres were ‘floated’ on top to ensure they did not sink too deeply in the resin and become embedded by more than 50 % of their circumference [22]. To reduce sample preparation time, an alternative method has been developed. Approximately 1 ml of mixed resin was placed at the centre of a glass or PMMA microscope slide and immediately spun using a Laurell WS-650-23 Spin Coater. Spinning at 9000 rpm for 30 s produced a thin and consistent layer of VE or epoxy resin in the central portion of the slide. A single glass fibre was then manually placed on to the resin, parallel to the slide width.

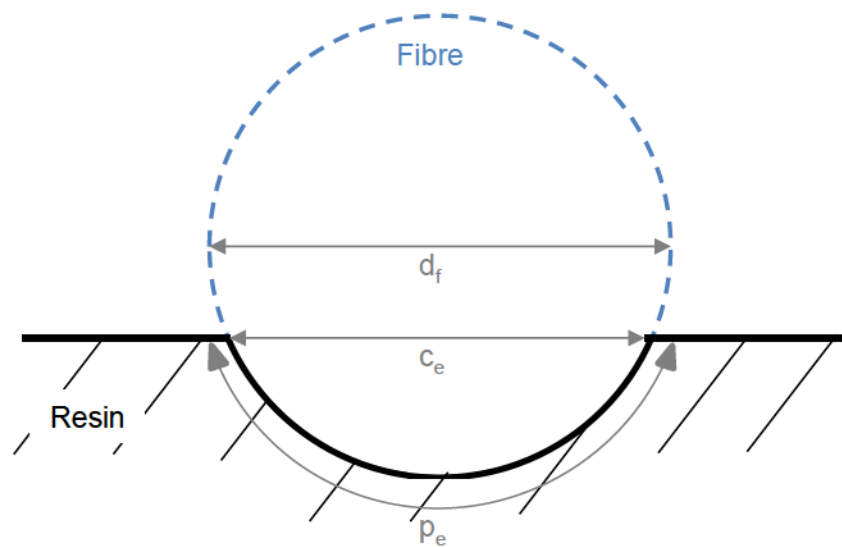


Figure 5: Diagram showing key geometry of a part embedded fibre

A model sample is shown in Figure 5 with the key dimensions that are required in order to calculate the embedded perimeter p_e of the fibre: d_f the fibre diameter and c_e the embedded chord of the fibre. By spin coating a thin, controlled layer of resin it is possible to ensure that (depending on diameters of fibres selected) fibres will be embedded by less than 50 % of their circumference.

4.3. Theoretical background

The energy of fracture for peel test specimens is calculated from the average force of debonding divided by the sample width over which it acts. For large and regular shaped materials this dimension may be well defined and easily measured, but for the single fibre peel test it is more challenging. The embedded perimeter, which is an arc of a circle, must either be measured directly or calculated from other dimensions. This was achieved by obtaining d_f and c_e (Figure 5): fibre diameter was obtained using optical microscopy and c_e by measuring the profile of the depression left behind after a fibre was peeled. This was achieved using a stylus profilometer and importing the profile into Origin to measure. A typical profile is shown in Figure 6.

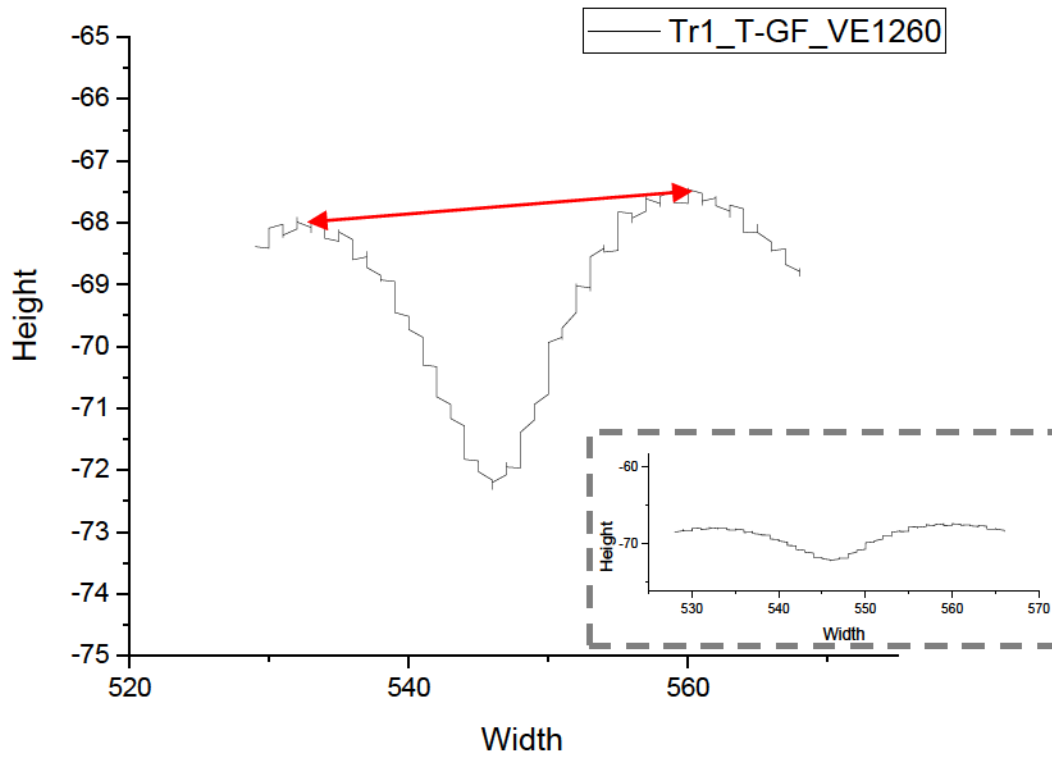


Figure 6: Typical profilometry data of GF_{ITA} (diameter approx. $34 \mu\text{m}$) after fibre debonding. Inset (dotted line) shows 'true' proportions of the profile on equally scaled x and y axes (dimensions μm)

With these dimensions the embedded perimeter of the fibre specimen can be calculated at a number of points along the length for which average peel force (\bar{F}) is known and stable, according to (2)

$$l_e = \frac{\pi d_f}{180} \sin^{-1} \left(\frac{0.5c_e}{r} \right) \quad (2)$$

The fracture energy G_I^c for peeling at 90° is found from (3) or if an oblique peel angle φ° is used then according to (4).

$$G_I^c = \frac{\bar{F}}{l_e} \quad (3)$$

$$G_I^c = \frac{\bar{F}(1 - \cos\varphi)}{l_e} \quad (4)$$

4.4. Testing procedure

Fibre peel tests were performed at extension of 1 mm/min and an oblique angle of 30° , in order to maximise the detectable debond force. This force increases with decreasing angle but is unreliable once $\varphi < 30^\circ$ [22]. Testing was carried out using a single column Testometric M250-2.5CT tensile machine with a 5 N load cell. An aluminium base plate with dovetail was manufactured: this allowed different

test fixtures with a range of angles to be easily substituted. Test fixtures were made from PLA using a 3D printer.

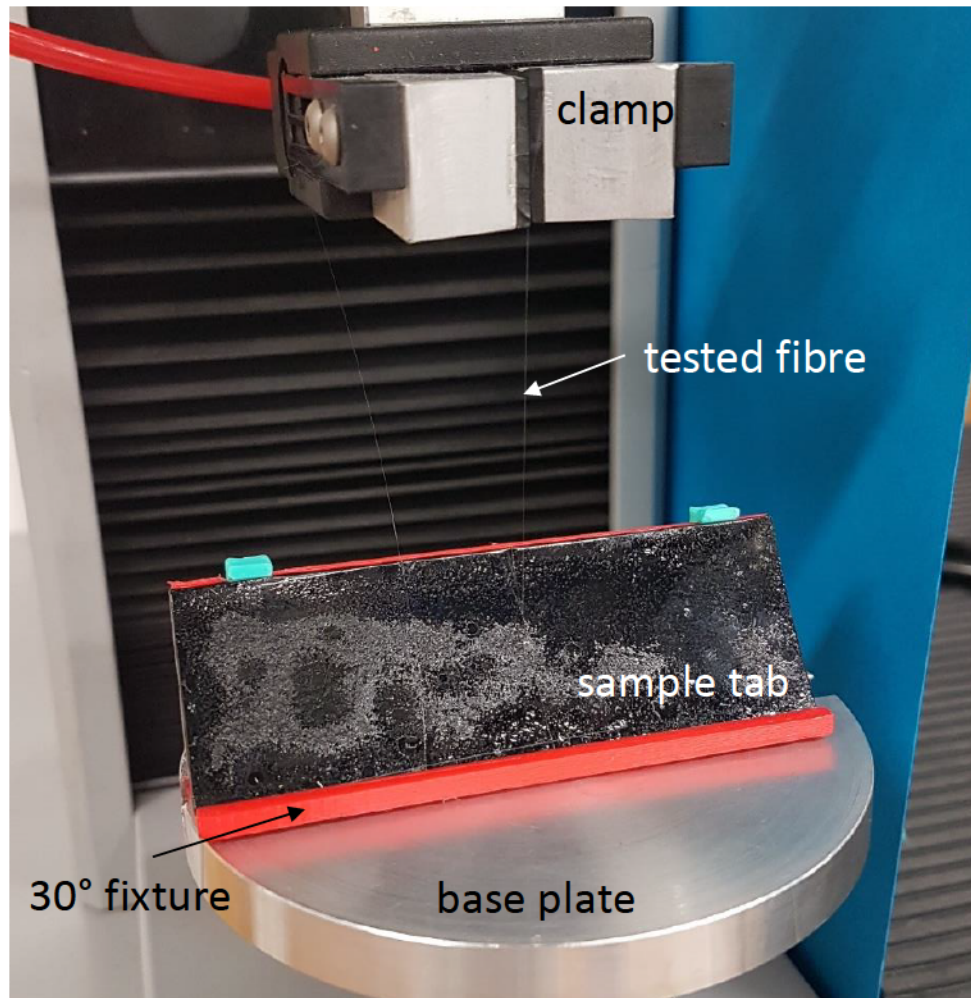


Figure 7: Image showing single fibre peel setup at 30°

The image in Figure 7 shows the 30° fixture with a sample mounted; in this case DER 332 was spun on to a black PMMA slide. In the image the tested fibre can be observed; it is clamped directly in the rubberised pneumatic grips of the instrument. Due to the very small debonding forces involved it does not appear that fibre slippage during testing is a problem. This is a significant advantages as it allows direct fibre gripping rather than the use of a slip foil and adhesive to be applied to each test sample before loading. A diagram showing the progression of the test is shown in Figure 8.

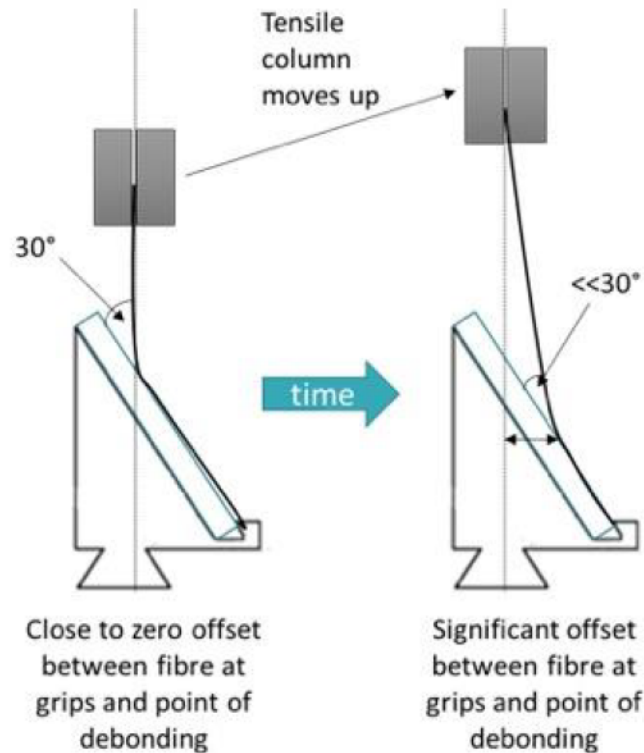


Figure 8: Progression of SFPT experiment showing change in angle between fibre and resin surface over time

The current test setup does not allow for compensatory movement of the sample in sync with the vertical displacement of the fibre/tensile column. Therefore, the debond angle changes gradually throughout a test. In the 30° case, for example, fibre slack that is required during mounting must first be displaced in the early moments of the test. During this time the angle exceeds 30° . The specimen is observed and, when the fibre is parallel with the tensile column, the absolute (machine) displacement is recorded. Average force data from ± 0.5 mm of this point only are considered. The right hand part of Figure 8 shows that, as the test progresses further, the angle between fibre and matrix decreases below that defined by the test fixture.

4.5. Initial results

The load extension plot for an unsized glass fibre (GF_{ITA}) is shown in Figure 9. This plot shows some typical features that can occur during single fibre peel testing. There is an initial period in which slack in the fibre is taken up before it bears any load. The 'middle' portion of the test is of most interest as the useable data are found here. In this case, the fibre was observed to be parallel with the column of the tensile machine at extension of approximately 3 mm. The force data around this point, therefore, were used for analysis of the debond energy.

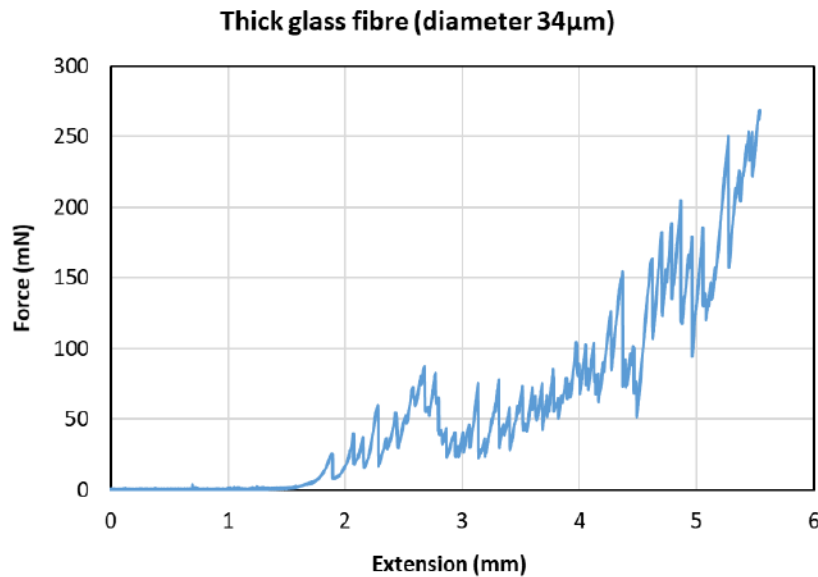


Figure 9: Load-extension plot of GF_{ITA} in DER 332 peeled at 30°

The average force from 3-3.5 mm was 44.2 mN and the fibre was estimated to be 50% embedded from images obtained by optical microscopy. From (3) the fracture energy was estimated as 27.7 J/m². Images of the fibre taken post-test, however, suggest that there is likely significant error in this value. Residual matrix was observed along its length; it is possible, therefore, that both debonding at the fibre-matrix interface and matrix cracking contributed to the force detected.

Future investigations using this test method will focus on approaches to minimise this effect. The resin distribution method has been optimised to ensure a layer is created which is thinner than the fibre radius. Testing at additional angles of 45, 60 and 90° will also be carried out.

5. Conclusions

Micro-mechanical testing of the interface between fibres and polymer matrices remains a useful approach in spite of the known (and at times unanticipated) challenges. It was shown that application of the microbond test to investigation of glass fibre/vinyl ester specimens presents several issues which must be overcome in order to achieve accurate and representative values of apparent interfacial shear strength. Initial cure of samples must be performed under an inert atmosphere and, thereafter, post-curing at elevated temperature is also necessary. An unexpected interaction was discovered between the size on a resin-compatible glass fibre and the cyanoacrylate based adhesive commonly used to restrain fibres for specimen preparation. Deposition of the adhesive from its vapour state on to the fibre appears to be the cause; however, the mechanism by which this significantly reduced the adhesion between fibre and resin is not yet understood.

A new micro-mechanical test method for characterisation of interfacial adhesion in mode I, the single fibre peel test, was introduced. It is intended to address some criticisms and limitations of the pre-existing micro-mechanical interface tests which induce mode II failure.

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References

- [1] Drzal LT, Madhukar M (1993) Fibre-matrix adhesion and its relationship to composite mechanical properties. *J Mater Sci* **28**:569–610. doi: 10.1007/BF01151234
- [2] Jones FR (2010) A review of interphase formation and design in fibre-reinforced composites. *J Adhes Sci Technol* **24**:171–202. doi: 10.1163/016942409X12579497420609
- [3] Dey M, Deitzel JM, Gillespie JW, Schweiger S (2014) Influence of sizing formulations on glass/epoxy interphase properties. *Compos Part A Appl Sci Manuf* **63**:59–67. doi: 10.1016/j.compositesa.2014.04.006
- [4] Zhandarov S, Mäder E (2014) An alternative method of determining the local interfacial shear strength from force-displacement curves in the pull-out and microbond tests. *Int J Adhes Adhes* **55**:37–42. doi: 10.1016/j.ijadhadh.2014.07.006
- [5] Sørensen BF (2017) Micromechanical model of the single fiber fragmentation test. *Mech Mater* **104**:38–48. doi: 10.1016/j.mechmat.2016.10.002
- [6] Piggott MR (1997) Why the fibre/polymer interface can appear to be stronger than the polymer matrix. *Compos Sci Technol* **57**:853–857. doi: 10.1016/S0266-3538(96)00151-0
- [7] Piggott MR (1997) Why interface testing by single-fibre methods can be misleading. *Compos Sci Technol* **51**:965–974.
- [8] Graupner N, Rößler J, Ziegmann G, Müssig J (2014) Fibre/matrix adhesion of cellulose fibres in PLA, PP and MAPP: A critical review of pull-out test, microbond test and single fibre fragmentation test results. *Compos Part A Appl Sci Manuf* **63**:133–148. doi: 10.1016/j.compositesa.2014.04.011
- [9] Kim IC, Yoon TH (2000) Enhanced interfacial adhesion of carbon fibers to vinyl ester resin using poly(arylene ether phosphine oxide) coatings as adhesion promoters. *J Adhes Sci Technol* **14**:545–559. doi: 10.1163/156856100742735
- [10] Yang L, Thomason JL, Zhu W (2011) The influence of thermo-oxidative degradation on the measured interface strength of glass fibre-polypropylene. *Compos Part A Appl Sci Manuf* **42**:1293–1300. doi: 10.1016/j.compositesa.2011.05.011
- [11] Thomason JL, Yang L (2011) Temperature dependence of the interfacial shear strength in glass-fibre polypropylene composites. *Compos Sci Technol* **71**:1600–1605. doi: 10.1016/j.compscitech.2011.07.006
- [12] Thomason JL, Yang L (2014) Temperature dependence of the interfacial shear strength in glass-fibre epoxy composites. *Compos Sci Technol* **96**:7–12. doi: 10.1016/j.compscitech.2014.03.009
- [13] Minty R, Yang L, Thomason JL (2018) The influence of hardener-to-epoxy ratio on the interfacial strength in glass fibre reinforced epoxy composites. *Compos Part A Appl Sci Manuf* **112**:64–70. doi: 10.1016/j.compositesa.2018.05.033
- [14] Petersen H, Minty R, Thomason JL, et al. (2018) The amine:epoxide ratio at the interface of a glass fibre/epoxy matrix system and its influence on the interfacial shear strength. *Compos Interfaces* 1–13. doi: 10.1080/09276440.2018.1511107
- [15] Miller B, Muri P, Rebenfeld L (1987) A Microbond Method for Determination of the Shear Strength of a Fiber/Resin Interface. *Compos Sci Technol* **28**:17–32. doi: 10.1016/02663538(87)90059-5
- [16] Yang L, Thomason JL (2012) Development and application of micromechanical techniques for characterising interfacial shear strength in fibre-thermoplastic composites. *Polym Test* **31**:895–903. doi: 10.1016/j.polymertesting.2012.07.001
- [17] Bénétuilière T, Duchet-Rumeau J, Gérard JF, et al. (2015) Physico-chemistry of vinylester/glass fiber interfaces used in SMC composites. *ICCM Int. Conf. Compos. Mater.* 2015-July:
- [18] Bryce D, Yang L, Thomason JL (2019) Microscale curing performance of epoxy resin in the microbond test [presentation]. *5th Int. Conf. Mech. Compos.*, Lisbon, Portugal
- [19] Scott TF, Cook WD, Forsythe JS (2002) Kinetics and network structure of thermally cured

- vinyl ester resins. *Eur Polym J* **38**:705–716. doi: 10.1016/S0014-3057(01)00244-0
- [20] Wargacki SP, Lewis LA, Dadmun MD (2007) Understanding the chemistry of the development of latent fingerprints by superglue fuming. *J Forensic Sci* **52**:1057–1062. doi: 10.1111/j.1556-4029.2007.00527.x
- [21] Alimuddin MA (1999) Peel Test for the Evaluation of Environmental Effects on Fiber-Polymer Interface. University of Toronto
- [22] Alimuddin MA, Piggott MR (1999) Fracture Toughness of Fiber-Polymer Interfaces Estimated From Single Fiber Peel Tests. *Polym Compos* **20**:655–663.
- [23] D6862-11 (2010) Standard Test Method for 90 Degree Peel Resistance of Adhesives. Annu B ASTM Stand. doi: 10.1520/D3167-10R17.1
- [24] D903-98 (2003) Standard Test Method for Peel or Stripping Strength of Adhesive Bonds. Annu B ASTM Stand 1–3. doi: 10.1520/D0950-03R11.responsibility
- [25] D1876-08 (2015) Standard Test Method for Peel Resistance of Adhesives (T-Peel Test). Annu B ASTM Stand. doi: 10.1520/D1876-08R15E01.Copyright
- [26] D1781-98 (2012) Standard Test Method for Climbing Drum Peel for Adhesives. Annu B ASTM Stand 1–4. doi: 10.1520/D1781-98R12.2
- [27] Réquillé S, Le Duigou A, Bourmaud A, Baley C (2018) Peeling experiments for hemp retting characterization targeting biocomposites. *Ind Crops Prod* **123**:573–580. doi: 10.1016/j.indcrop.2018.07.012