COMPARISON STUDY OF FIXED AND VARIABLE BLADE INTERFACIAL TESTING SYSTEMS FOR THE CHARACTERISATION OF THERMOSET COMPOSITE

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Abstract
The microbond test is widely used in the characterisation of fibre-matrix interfacial shear strength (IFSS), however a lack of standardisation means that methodology, sample preparation and equipment vary across research groups. Most commonly, a set of adjustable parallel blades are used to apply loading to the microdroplet, introducing variation in blade placement between users. Prior research indicates that factors such as blade placement, droplet embedded length, and fibre surface variation all contribute to the high scatter in results. The present work focuses on establishing a direct comparison between Dia-Stron's LEX820 IFSS equipment with a fixed blade setup, and a variable blade rig system in which the blade gap is controlled using a digital micrometer. Results of the comparison study showed that the use of a fixed blade system resulted in significant differences in the shear strength determined, with the variable blade system and fixed blade system having an average IFSS of 47.7 MPa and 41.8 MPa respectively. Furthermore, investigation into the effect of the fibre gauge length is conducted, with initial findings indicating that the gauge length may have an effect on the measured interface strength.

1. Introduction

Composite materials have become a staple in aerospace and automotive industries due to their manufacturing flexibility and the ability to tailor their properties to the requirements of the load the component will experience. As such, the development of our understanding of the fundamental properties that contribute to the load-bearing capability of composites is crucial. The optimisation of composite properties is intrinsically linked to the optimisation of the stress transfer capability of the fibre-matrix interface. One accepted mechanically measurable value which can be used to characterise the strength of the interface is the interfacial shear strength (IFSS). With interface strength being affected by the chemistry of the fibre surface and matrix system applied, the accurate quantification of the IFSS is vital to the tailoring of these properties [1-3].

The microbond test provides an estimation of the IFSS through the shear debonding of a droplet on a single fibre by means of a set of parallel blades [4]. Due to a lack of standardisation of the test, sample preparation, equipment and methodology differ between research groups, as evidenced in the round-robin investigation conducted by Pitkethly et al. [5]. Most commonly, microvise rigs consisting of a set of parallel blades controlled by a micrometer are used [6-8], however, alternatives such as the TMA microbond technique [3], circular loading configurations [9] and automated adjustable blade testing systems [10] have also been developed.

Whilst the microbond test provides valuable insight into the load-bearing capabilities of a given composite, several sources of error that contribute to the high level of scatter in the results must be considered and mitigated throughout. Possible sources of error noted throughout the literature include accurate dimensioning of embedded length and fibre diameter, scaling issues during sample preparation, and position of the shearing blades [10, 11].

Variations in blade position result in changes to the stress state applied through the droplet, with several studies documenting the location of the peak shear stress changing with changing blade placement [12-15]. Chou et al. observed that with increasing blade separation, the IFSS was found to be overestimated [12]. Furthermore, a finite element study considering the effect of various geometrical parameters found that issues in blade position could be further compounded by any axial misalignment in the blades, as fibre bending can occur as a result [16].

Whilst the effect of the placement of the variable blades has been widely reported, there remains a gap in the literature with respect to the efficacy of a fixed blade setup and the effect that such a system may have on the results. In the present work, microbond tests are conducted using the Dia-Stron LEX820 fitted with an IFSS module with a fixed blade setup, and the Instron 3342 universal tensile tester with a variable blade rig system fitted.

2. Experimental

2.1. Materials

The experiments were carried out using silane-sized Advantex glass fibres, taken from the inside of a larger roving, with nominal tex of 2400 g/km and the average diameter of 17 µm. The epoxy resin used was DER 332 bisphenol-A diglycidyl ether (DGEBA) cured with Triethylenetetramine (TETA) hardener.

2.2. Sample Preparation

Single fibres were extracted from the bundle of fibres taken from the roving, and were initially mounted on a card frame, containing a 20 mm length window, using double sided tape. The fibre was then set in place with a droplet of Loctite super glue gel at each end of the card, and left to cure for 24 hours. Another set of samples were prepared in a similar manner, with a card frame containing a 40 mm length window being used, to allow for testing of the effect of fibre gauge length on the test.

The matrix mixture was then prepared, using the stoichiometric ratio at 2000 mg epoxy resin and 286 mg hardener. The mixture was degassed for 10 minutes in a vacuum chamber, removing any air-bubbles and improving the homogeneity of the mixture. A scalpel, modified with a small length of steel wire at 125 µm, was used to apply small droplets of resin to the fibre by gently touching the tip of the wire to the fibre. This method of application deposits several droplets onto the fibre with each touch of the wire, with there being a large variation in droplet sizes due to the lack of control over the volume of resin applied.

On completion of the droplet application the samples were then cured in an oven, with the same cure cycle used for each set of samples. The temperature was first ramped up to 60°C over 20 minutes, at which point this temperature was maintained for an hour. Upon completion of the first cure cycle stage, the temperature was then ramped up to 120°C over 30 minutes, and maintained for 2 hours. The samples were then left in the oven to cool overnight before being removed.

Each sample was studied using an optical microscope at 250x magnification, with images then being taken of each fibre and the droplets on each fibre. ImageJ, an open-source image analysis software, was used to take measurements of the fibre diameter (D_f), droplet diameter (D_d), and droplet embedded length (L_e) of each sample. These measurements are subsequently used in the post-processing of the results of the maximum debond force (F_{max}) from the microbond tests to calculate the IFSS, as shown in Equation 1.

$$IFSS = \frac{F_{max}}{\pi . D_f . L_e} \tag{1}$$

2.3. Microbond Test

Microbond tests were conducted across two machines; the variable blade tests used an Instron 3342, fit with a specially designed variable blade rig [6]. Tests for the fixed blade system were carried out on the Dia-Stron LEX820 IFSS equipment with a fixed blade setup.

The variable blade setup used in the current work made use of two blades set in a microvise rig, placed below the hook of the load cell. The sample was placed onto the hook, with the fibre being situated between the blades. A stereo microscope was set up in front of the rig, to allow the droplet to be tested to be identified and positioned just below the blades. Micrometers on either side of the microvise are then adjusted to bring the blades into contact with the fibre. The test was then set to run at a constant crosshead speed of 0.1 mm/min, and the machine would gradually pull the fibre upward, at which point the droplet was subjected to a shearing force. The droplet deformed until the shear force exceeded the interface strength, at which point, the droplet would debond.

Dia-Stron's LEX820 fixed blade setup consists of a slot plate, with a gap of 20 µm, fixed on top of the load cell, with the piston attached to the tab pocket situated above. The distance from the end of the tab down to the droplet to be tested is measured and the value input into the machine program, resulting in the piston moving into the required position. The sample is then set in the tab pocket with the fibre situated in the blade gap. The piston then pulls the fibre upward at a constant rate until the droplet has debonded. At the end of each machine's testing process, a graph is produced with the maximum force prior to debonding determined, as shown below in Figure 1.

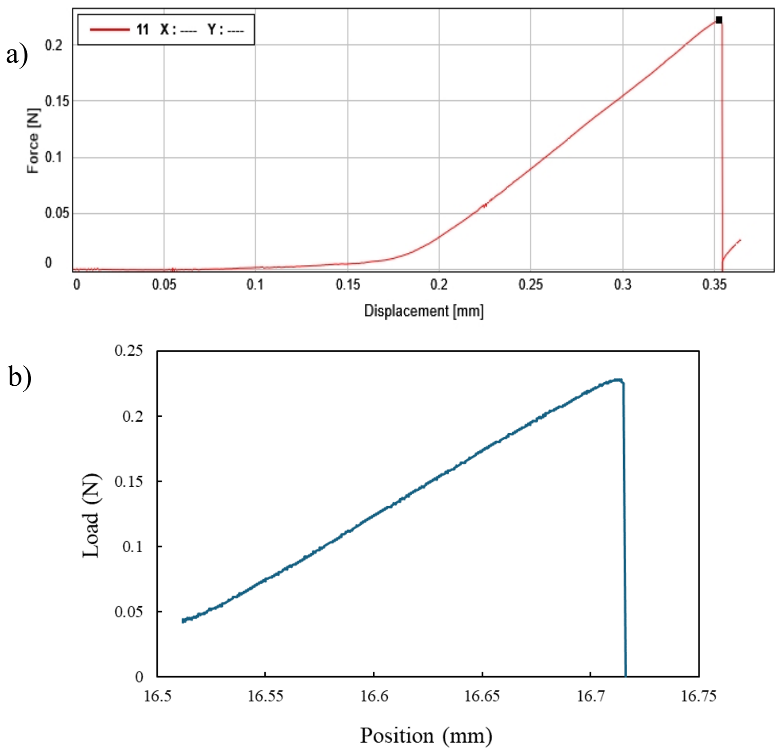

Figure 1. Graph for successful debond as output on a) Instron and b) Dia-Stron

Two sample sets were tested on each machine, with the 20 mm gauge samples being used for the basic comparison between the adjustable and fixed blade setups, whilst the 40 mm gauge samples were used to test the effect of increasing the fibre-free length. The fibre-free length tests required the testing of several droplets along the length of each fibre, with the position of each droplet to be noted.

3. Results

The maximum load recorded for each droplet tested was collated and plotted on a maximum load against embedded area plot, as shown in Figure 2. It was found that the Instron's variable blade setup resulted in an increased IFSS value compared to Dia-Stron's fixed blade system, with the resulting average IFSS values being 47.7 MPa and 41.8 MPa respectively. Furthermore, it was observed that the fixed blade system exhibited a reduced level of scatter, as indicated by the increased R^2 value displayed in the Dia-Stron results.

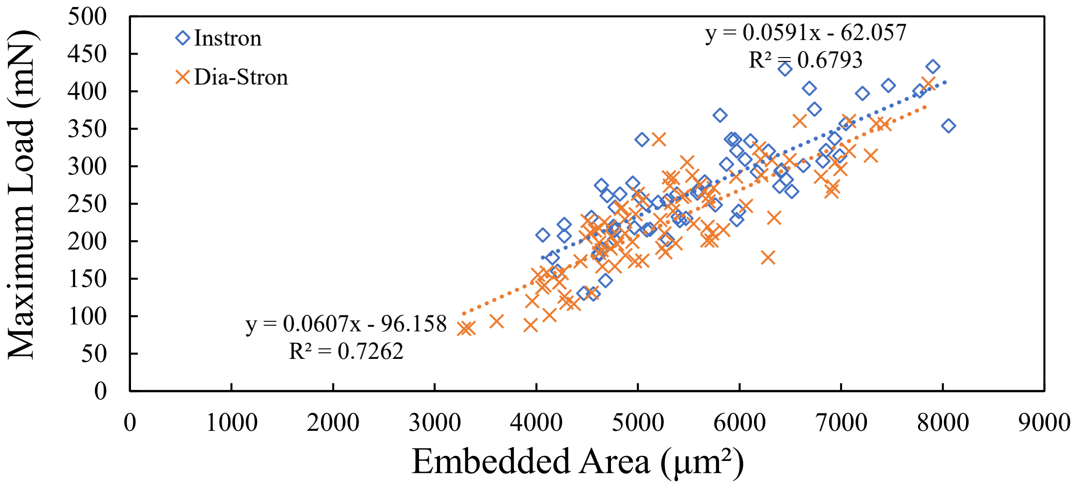

Figure 2. Maximum load vs embedded area for each microbond test system (20 mm gauge fibres)

Following the comparison study of the different blades, tests to determine the effect of the fibre-free length were conducted. Initial results show different trends occurring for each machine. Tests carried out on the Instron show a slight decrease in the average IFSS along the length of the fibre. In contrast, Dia-Stron results display an increase in average IFSS along the length of the fibre.

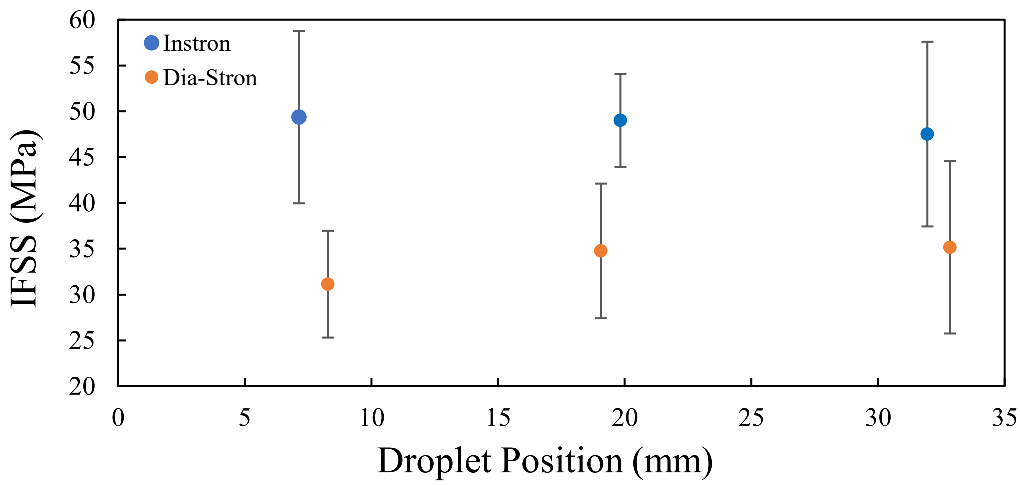

Figure 3. Comparison of increasing fibre-free length for each microbond test system (40 mm gauge fibres)

4. Discussion

Past studies looking at the effect of various experimental parameters suggested that the separation of the blades has an effect on the IFSS, with Chou et al. stating that a blade gap width close to that of the fibre diameter was likely to produce a more accurate result [12]. As such, a decrease in IFSS when the test is conducted with a fixed blade system is to be expected, as a consistent blade gap ensures a reduced level of variability throughout a series of tests. Furthermore, the reduced level of scatter displayed in the Dia-Stron results is likely due to the consistent placement of the blades on the droplets, with the main factors affecting the blade placement in a fixed blade system being the fibre and droplet diameters. Measured fibre diameters ranged from 15 µm up to 18.4 µm, meaning that the free distance between the fibre and blade ranged from 2.5 µm to 0.8 µm on either side of the fibre. With the presence of the meniscus on each droplet, it is likely that the blades come in contact with the droplet near the top, resulting in a non-uniform stress distribution, in which the top of the droplet experiences a higher stress concentration and subsequently promotes debonding at a lower force.

Further tests focussing on the effect of the fibre-free length were conducted with initial findings indicating that the free length of the fibre may have an effect on the estimated IFSS, however differences between the averaged IFSS at each position along the fibre do not show statistical significance. Furthermore, the fibre-free length had a different effect when tested on each machine. Tests carried out on the Instron variable blade system displayed a slight decrease in the average IFSS with increasing fibre-free length. A possible explanation for this trend is that as the length of the fibre is increased, the presence of potential surface flaws is increased along with fibre strain, as postulated by Burn [17]. However, whilst this effect could explain the decrease in results from the Instron system, it does not account for the results from the Dia-Stron system.

The increase in IFSS, particularly going from the top to middle sections of the fibres tested in the Dia-Stron system is thought to be a result of possible axial misalignment of the fibre when droplets positioned below 5 mm are tested. As stated by Zhao et al., axial misalignment can cause the fibre to bend, resulting in failure of the interface at a lower force [16]. As the fibre-free length is increased, the fibre can be better positioned between the blades to ensure alignment. The negligible difference in results of the middle and bottom droplets suggests that the fixed blade system may be more compatible with increased fibre lengths.

5. Conclusions

It is evident that the microbond test has inherent limitations, with a major source of error being the effect of the blade positioning with respect to the droplet. The results presented in this work indicate that the use of a fixed blade microbond test system can aid in the mitigation of this error, improving the reliability of the results obtained. A comparison study of the Instron system with an adjustable blade rig and the Dia-Stron LEX820 fixed blade system highlighted the significant difference in resulting average IFSS values obtained, with the IFSS estimated using the Instron system being higher by 5.9 MPa. Furthermore, it was observed that the use of the fixed blades resulted in a reduced level of scatter.

Initial studies into the effect of the fibre-free length on the microbond test suggested that a relationship between the free length and the interface strength may exist. Results from tests conducted with the adjustable blade system were as expected, with the IFSS decreasing slightly over the length of the fibre. In contrast, the fixed blade system displayed an increase in IFSS from the droplets at the top of the fibre down to the middle of the fibre. One potential explanation for this is that droplets positioned at the top of the fibre are more likely to be tested at an angle in the fixed blade setup, resulting in a lower debonding force. Whilst a considerable level of research has been done into factors that affect the microbond test, little work has been done on the effect of the fibre-free length. As such, further research into the effect of the fibre-free length on the microbond test would be beneficial towards the potential standardisation of the test.

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