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Microbond testing of the interface in glass fibre vinylester composites

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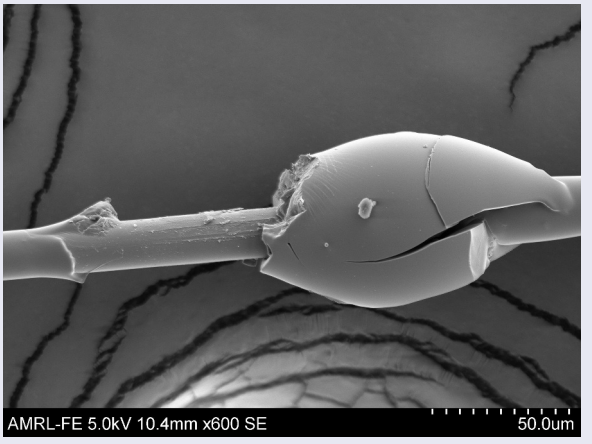
ABSTRACT

The microbond test was used to investigate the interface strength (IFSS) in various glass fibre-vinylester (VE) composite systems and a number of issues related to the sample preparation were identified. Cure schedules that produce well-reacted VE polymers on the macroscale did not result in cured microdroplets. Hence, the microbond test could not be carried out on samples with the same cure history as macroscale composites. Testable microdroplet samples could only be obtained when resin cure was carried out under an inert atmosphere. Higher IFSS values were obtained by raising the final temperature of the cure schedule. Glass fibres with a full sizing gave significantly higher apparent IFSS values comparedf to bare fibres or fibre coated with only silane coupling agent. It was discovered that the measured IFSS of VE compatible glass fibres was approximately doubled when fibres were mounted using epoxy glue instead of cyanoacrylate glue. This phenomenon appears to be related to the deposition of cyanoacrylate vapours onto the surface of the fibres during sample preparation. It is concluded that great care must be taken in ensuring that effects observed using the microbond test are evidence of real material characteristics and not artefacts of sample preparation.



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1. Introduction

The stress transfer capability of the interface region between matrix and reinforcement fibre is one of the most important factors in the successful application of any composite material and there has been significant research effort made to elucidate its role [1–4]. The vast majority of fibres require surface modification to improve their processability and interfacial compatibility with polymer matrix materials. 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. 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 composite performance and may eventually lead to a situation where the composite can no longer fulfil its intended function.

One accepted mechanically measurable value for quantifying fibre-matrix adhesion is the apparent interfacial shear strength (IFSS). Over the past several decades, a number of micro-mechanical tests have been created and subsequently improved to measure IFSS. 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. The work reported here forms part of the larger EU Horizon 2020 funded DACOMAT (Damage Controlled Composite Materials) project whose objective is the development of more damage tolerant and damage predictable low-cost composite materials for use in large load carrying constructions such as bridges, buildings, wind-turbine blades, and offshore structures [5]. The project has focussed on the investigation of glass fibre reinforced vinyl ester resins for these applications.

Vinyl ester (VE) resins are widely used in structural components in a broad range of applications, usually in the marine, offshore, and civil infrastructure sectors. Although typical vinyl ester systems may provide somewhat lower mechanical performance levels compared to many epoxy systems, they generally exhibit relatively fast fabrication cycles, ease of processability, lower material cost, and may still perform adequately in many composite infrastructure applications [6,7]. During their service-life vinyl ester composites may well be exposed to a variety of hygrothermal conditions and therefore it is essential to have a strong understanding of the response of the composite interface region to such environmental exposure. However, the number of studies on the interface and environmental exposure of vinyl ester composites is limited and knowledge about their durability and life expectancy is limited [7–9].

We have been employing microbond testing to research the state of the interface, both in the short term and after longer term environmental ageing, in glass fibre reinforced vinyl ester composites. A challenging aspect of such micromechanical testing is the preparation of appropriate small-scale samples that still reflect the properties and performance of the same materials in a macroscopic composite. The microbond test, for example, has been applied relatively widely to different matrix materials [4,10,11] but it is not unusual to encounter effects that must be mitigated against to obtain reliable test data. For example, the results obtained for interfacial shear strength may vary, due to degradation in thermoplastics [12], with relatively small changes in testing temperature

[13,14] and due to small variations in the stoichiometry in thermoset systems [15]. The scaling issue challenge in the preparation of microbond droplets that properly reflect the properties of macroscale composites is one that should not be underestimated. There is no single uniform solution to these challenges and each different fibre–matrix combination may be subject to unexpected and previously unpublished artefacts of sample preparation that can affect the obtained IFSS values much more significantly than any material-related effects that may be under investigation. Such has been the case in this investigation of glass fibre-vinyl ester IFSS. In this paper, some specific and previously unknown challenges encountered in the preparation of samples for microbond testing of several glass fibre-vinylester systems are presented and discussed.

2. Materials and Methods

2.1. Materials

The experiments were conducted using a number of boron-free E-glass fibres. Bare (water-sized) fibres were taken from rovings supplied by Owens Corning. Fully sized commercial fibres were taken from various rovings supplied by 3B-fibreglass. SE3030 is a commercially available roving sized with a vinyl-ester compatible sizing. Two other rovings with experimental sizings that were modifications of the 3030 sizing were also investigated. Sizing S001 was modified in order to improve reactivity with VE resin and sizing S002 was modified in order to increase the toughness of the fibre-matrix. A number of other vinyl-ester glass fibre compatible rovings were acquired at a later stage in the study for comparative studies. The full range of fibres investigated in this study are listed in Table 1. Microbond experiments were conducted using a low viscosity bisphenol epoxy-based vinyl ester resin (VE-1260) with a styrene content of 48–52%, supplied by Polynt Composites Ltd. All resins were formulated with 2.5% PBC-21 UN 3103 Organic peroxide type C initiator from United Initiators GmbH as a curing agent. The epoxy resin droplets were made using DER 332, a diglycidyl ether of bisphenol-A, cured with a stoichiometric (14.3 phr) amount of triethylenetetramine (TETA) tetra-functional amine [15] both sourced from Sigma Aldrich. The resin was mixed and degassed under vacuum for approximately 10 mins before application. The cure cycle utilised consisted of two isothermal stages, 60°C for one hour followed by two hours at 120°C, with intermediate heating rates of 2°C/min.

Table 1. Details of glass fibres used in this investigation.

Fibre ID	Manufacturer	Sizing	Measured Avg. Fibre Diameter	Nominal Tex
OCb	Owens Corning*	None	16.3	1200
OCs	Owens Corning*	MoPTMS silane	16.6	1200
SE2020	3B-Fibreglass	Epoxy compatible	18.1	2400
SE3030	3B-Fibreglass	Multi-compatible	17.7	1200
S001	3B-Fibreglass	VE development sizing	17.7	1200
S002	3B-Fibreglass	VE development sizing	17.8	1200
111AX11	3B-Fibreglass	Multi-compatible	20.0	2400
R25HX22	3B-Fibreglass	Multi-compatible	18.1	1200
2002	Nippon Electric Glass	Multi-compatible	17.4	2400
2026	Nippon Electric Glass	Multi-compatible	17.5	2400

*manufactured on a pilot plant bushing

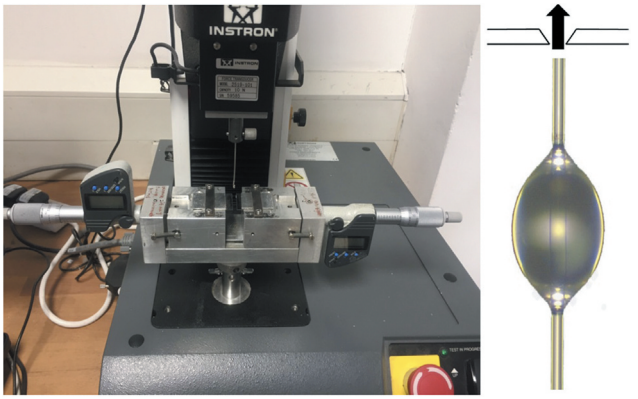

Figure 1. Photo of microbond test setup and typical sample.

2.2. Microbond testing

The IFSS between reinforcement fibres and VE or epoxy resin droplets was measured using an in-house designed microbond rig. The experimental setup has been previously described in detail [16]. A schematic of the system used is shown in Figure 1. The basic procedure for the microbond test involves a single fibre being pulled from a restrained droplet of cured matrix while measuring the force required to detach the fibre. The microbond testing jig was designed around an Instron 3342 universal tensile testing machine equipped with a 10 N load cell and a microvice with adjustable shearing blades. Shearing blade horizontal movement was controlled at a resolution of 1 μm by a pair of adjustable parallel micrometers mounted on either side of the microvice [16]. Micrographs of cured droplets were collected using a Leitz Ergolux optical microscope at 200x magnification. Fibre diameter (D_f) and embedded length (L_e) were measured with ImageJ software. Microbond tests were performed at a constant crosshead speed of 0.1 mm/min and Instron Bluehill software was used to record crosshead displacement and applied load. Successful debonding or instances of droplet plastic deformation were confirmed by in-situ observation of droplet loading using 45x magnification stereo microscopy and a live camera feed. The apparent IFSS (τ_{app}) was calculated using Equation 1.

$$\tau_{app} = \frac{F_{max}}{\pi D_f L_e} \tag{1}$$

where F_{max} is the peak in the load-deformation plot during debonding. At least 20 individual tests were carried out for each condition to obtain an average IFSS value. Error bars shown in the results are the 95% confidence limits on these averages.

2.3. Microbond sample preparation

The sample preparation process comprised several steps that are of particular importance due to the influence they can have on the value of IFSS obtained. First, individual fibres were isolated and fixed to card frames with an adhesive so that resin droplets can be

applied in a controlled manner. The standard adhesive used for this purpose was a cyanoacrylate gel superglue that was allowed 24 h to fully react. Next, the resin under investigation 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. Samples were initially cured at room temperature under three different atmospheres, air, air saturated with styrene, and nitrogen. Nitrogen cured samples were also postcured in an air convection oven at 60°C for 24 h, 80°C for 3 h, and 100°C for 1 h.

3. Results and discussion

3.1. Effect of sample curing conditions

Vinyl ester resins are capable of achieving similar mechanical performance as some epoxies, but often with more straightforward processing requirements. Although a significant volume of data regarding the microbond testing of many fibre-polymer systems can be found in the literature, research on polyesters and vinyl esters are less prevalent [11,17]. Typical values for the apparent IFSS of glass fibres with epoxy resins are in the range 40–50 MPa when proper cure is achieved [15]. Values obtained for the IFSS of the unsized fibres and SE3030 in VE resin are given in Figure 2. It can be seen that very low values of apparent IFSS were obtained when the initial curing was carried out under an air atmosphere. Observation of the droplets during testing revealed that it was not possible to accurately measure an IFSS value for these samples due to the plastic deformation of the droplet. In many cases, the droplets simply deformed and eventually slipped through the gap between the blades. The droplets clearly had not cured and solidified sufficiently to transmit the applied load to the interface, and so there was no possibility to measure a credible IFSS by this method. It is worth mentioning at this point that the suppliers data for the mechanical properties of macroscopic sample of this resin

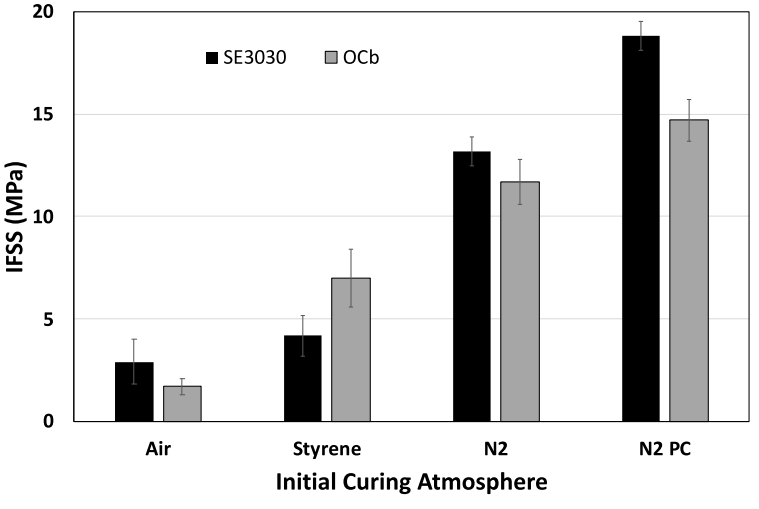

Figure 2. Influence of atmosphere on IFSS of GF-VE1260 system.

system cured under the same conditions are Young's modulus of 3.2 GPa and a tensile strength of 80 MPa and hence should be perfectly capable of producing a testable microbond sample.

Notwithstanding the name 'microbond' test, it should be noted that the mass of these polymer 'microdroplets' extends down well into the nanogram range, especially for the smaller droplets. Microscale curing issues of thermosetting resin systems have been reported in the literature prior to the development of the microbond test [18] and have been identified as areas for improvement in some of the earliest critical reviews of microbond [19] and micromechanical [20] testing methods. However, there has been little significant effort to address this phenomenon directly, despite the growing usage of the microbond test. Bryce *et al.* have recently published evidence for significantly lower degrees of cure in microdroplets made using a range of epoxy systems [21]. Work by Ash *et al* attributed incomplete polyester microbond sample curing and subsequent data scatter to evaporation of 50–60% of the initial styrene content out of the droplets [22]. Dirand [23] and Laurikainen [24] reported a similar hypothesis relating to evaporation of styrene out of vinyl ester microbond samples. Significant problems were noted during their microdroplet sample preparation using the picolitre scale volumes, which resulted in poor curing of the resins. Based on the observations made on the behaviour of their VE resin, and on previous work presented on microdroplet testing of a similar VE resin, the cause of the problem was identified as vaporization of the styrene from the resin droplets [23]. Laurikainen claimed that this effect could be counteracted by placing the curing droplets in a high styrene content atmosphere such as a closed container [24]. More recently Bénéthuilière *et al* also stated that they used this method successfully when preparing microdroplets with VE resin [17].

The results shown in Figure 2 for samples cured in a saturated styrene atmosphere for 24 h at room temperature do show a small, but significant, increase in the measured IFSS for the two fibres. Nevertheless, observation of the debonding process also revealed significant plasticity in these droplets and the low IFSS values obtained are a strong indication that curing the droplets under a styrene atmosphere did not solve the under-curing issue with this VE resin system. It has also been shown that the presence of oxygen can significantly inhibit the polymerisation reaction of vinyl ester resins [25,26] and the strength of vinyl ester resin cured in an open mould exposed to the air has been shown to be significantly reduced by up to 65% [26]. It was reported that this effect could become increasingly pronounced as specimen thickness is reduced and could even become 'catastrophic' for very thin samples [26]. Consequently we also prepared microdroplet samples with the initial 24 h cure under inert nitrogen. As can be seen in Figure 2 this procedure appeared to allow sufficient curing of droplets such that they could bear load during the microbond test. The IFSS values for both OCb and SE3030 fibre increased significantly yielding values in the range 12–14 MPa.

DSC analysis was used to investigate the glass transition temperature (Tg) and excess enthalpy of samples of VE resin that experienced different post-cure temperatures in order to quantify any residual under-cure in the polymer specimens. The average mass of a microbond droplet is in the range of approximately 0.2–2.0 μg and hence of insufficient size to produce detectable Tg values in DSC. Therefore, DSC samples were not taken directly from microbond specimens but were prepared from the same batch of resin and distributed on release film to produce droplets with mass of 10–15 mg suitable for DSC

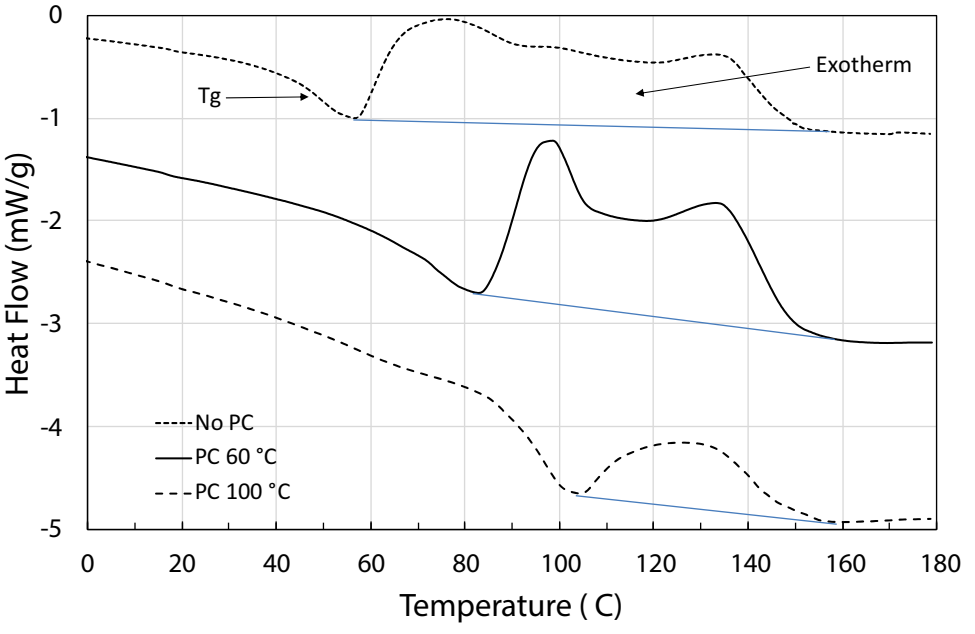

Figure 3. DSC analysis of VE1260 resin system after different levels of postcure.

Table 2. DSC analysis of vinylester samples with different cure history.

Post-cure after 24 h at room temperature under N_2	Tg (°C)	Exotherm initiated at (°C)	Exotherm enthalpy (J/g)
		58/98.6	276.0
No Post-cure	49.3	58	70.2
24 h at 60°C	75.1	86	31.2
24 h at 60°C, 3 h at 80°C, 1 h at 100°C	94.2	106	7.9

analysis. Examples of typical DSC heating runs are shown in Figure 3 and the data analysis results are presented in Table 2. The room temperature cured samples all exhibited a significant exotherm when heated above their Tg and the Tg of the polymer increased with increasing post-curing. This indicates that the curing reaction for this VE resin does not reach completion with only a room temperature cure. It can be noted that low-temperature curing is apparently associated with multiple peaks in the supra-Tg exotherm and that the number of these peaks appears to reduce as the postcure temperature is increased. This phenomenon has been reported by others and Li *et al.* has associated this behaviour with the presence of a two-phase structure in the cured VE resin [27,28]. Elevated temperature post-curing significantly increased the polymer samples Tg and also significantly reduced the level of supra-Tg exotherm indicating a significant increase in the degree of cure for these samples.

The effect of addition of a post-cure regime to the microbond sample preparation on IFSS can be seen in the final set of data in Figure 2. A post-cure procedure of 24 h at 60°C, 3 h at 80°C, 1 h at 100°C at elevated temperature significantly increased the IFSS for both fibre types to around 15 MPa for the unsized fibre and 19 MPa for the commercial SE3030 fibre. Hence, the most critical step in obtaining testable VE specimens for microbond testing, with this VE resin system, was the initial curing under an inert

atmosphere. This significantly reduced the oxygen inhibition effect that hinders the VE polymerisation reaction. It was also found that post-cure at elevated temperature is necessary to increase the degree of cure and mechanical properties of room temperature cured droplets.

3.2. Effect of the glass fibre sizing

The effect of the glass fibre surface coating on the apparent IFSS is examined in Figure 4 which compares the obtained average apparent IFSS values for five different glass fibres in the VE1260 resin with identical full cure schedule (24 h at room temperature under N_2 followed by 24 h at 60°C, 3 h at 80°C, 1 h at 100°C). The IFSS values obtained were all in the range of approximately 15–21 MPa. It is noted that this is a relatively low level of IFSS compared to typical GF-epoxy systems [1,4,14,15] and also low in comparison with other published studies with different GF-VE systems [17]. The main difference shown in the results shown in Figure 4 is that all of the fibre coatings, whether silane only or full sizing, significantly (unpaired two sample t-test at 95% confidence level) increased the apparent IFSS of the GF-VE system by approximately 33%. Interestingly there was no significant difference between the fully sized fibres and the fibres coated only with methacryloxypropylytrimethoxysilane (MoPTES). Within the group of three fibre from 3B-fibreglass the experimental sizing S02 did produced a small but significant increase in IFSS compared to the standard SE3030 product.

3.3. Effect of fibre mounting adhesive

A further goal of the research had been to investigate the IFSS of the same specimens after water exposure. In order to accomplish the necessary water treatment, it was necessary to mount the microbond sample on steel washers [29,30] as opposed to the card windows that are commonly used by those engaged in single fibre micromechanical

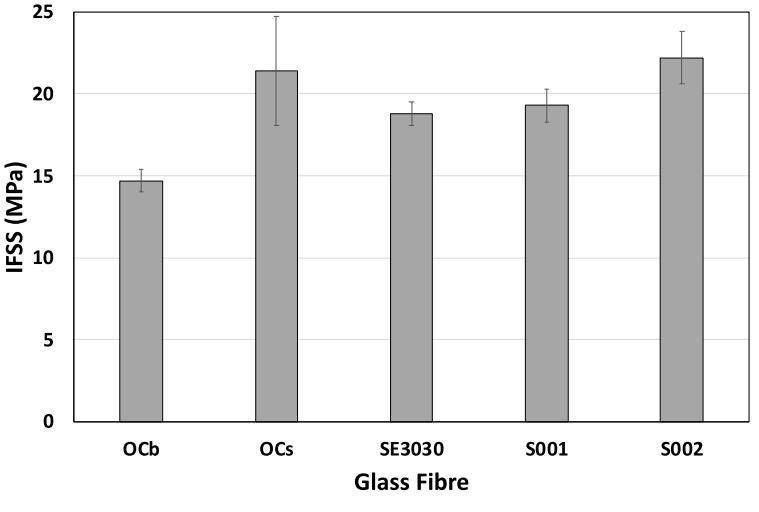

Figure 4. Influence of glass fibre size on IFSS of GF-VE1260 system.

experimentation. Somewhat serendipitously this also required switching from cyanoacrylate (CA) glue for securing the fibre to the steel washer to an epoxy (EP) based glue. Most surprisingly, it was found that this method of mounting the single fibres for microbond test specimen preparation resulted in microbond IFSS values for the SE3030 VE compatible glass fibre almost double that of the previous samples that were mounted on card windows with CA glue. By repeating the experiment with EP glue fixing to a card mounting it was quickly established that this was not related to the steel substrate but appeared to be related to the type of glue used to mount the single fibre samples. Figure 5 shows the peak force versus embedded area results for these two sets of data comparing the influence of the sample mounting adhesive. Despite the normal level of scatter observed in typical microbond data, the large difference in performance is clearly evidenced in this figure. The average values for the IFSS of the EP glued samples are compared with the previous CA glued samples in Figure 6. In our 15 years of experience using the microbond test in many different fibre-matrix systems such a large effect of the sample mounting adhesive has never been observed before. Extensive searching of the existing literature did not reveal previous evidence of any such phenomenon.

Figure 6 presents further results on the investigation of the fixing adhesive on the obtained IFSS values. It can be seen that for the unsized fibres in VE there is no effect of the mounting adhesive on the level of IFSS obtained (t-test p-value = 0.30). This could be an indication that the issue is related to the fibre sizing. Interestingly the fibres coated with MoPTMS silane, which is commonly used in VE compatible sizings [31], also shows no significant effect of the mounting adhesive on the IFSS (t-test p-value = 0.48). Hence, if the adhesive is interacting with the sizing then it would appear that it is an interaction with the film former (or other non-silane components). As we have used the CA adhesive extensively in our investigations of IFSS if epoxy systems we also checked for any evidence of a mounting adhesive effect. Two sets of data based on epoxy microdroplets are also presented in Figure 6. When using a fibre with an epoxy compatible sizing

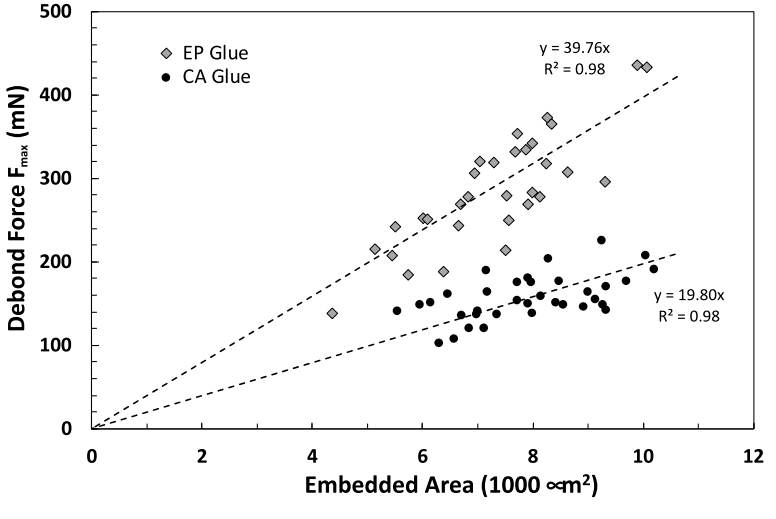

Figure 5. Influence of fibre mounting adhesive on the IFSS of the SE3030-VE1260 system.

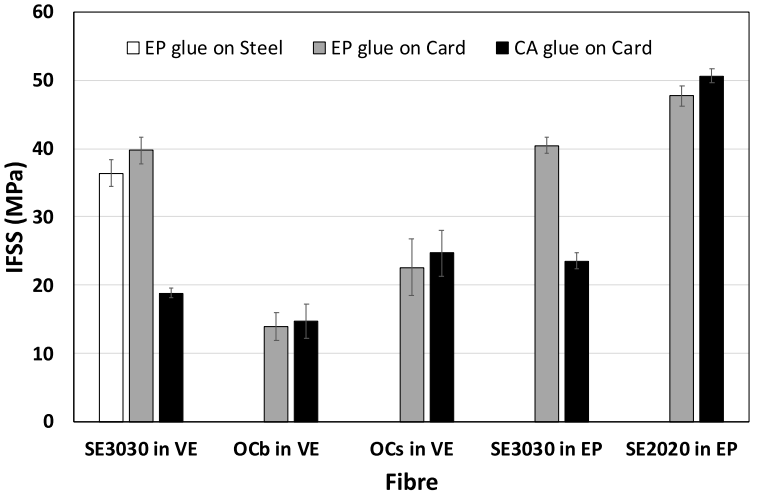

Figure 6. Influence of fibre mounting adhesive on the IFSS of various GF-VE1260 systems.

(SE2020) there was no evidence of a mounting adhesive effect on the IFSS. However, when using the VE compatible sized fibre (SE3030) in epoxy droplets the large effect of the mounting adhesive is also obtained. Hence, it can be concluded that the negative interaction of the CA adhesive is not with the components of the epoxy resin systems or the epoxy compatible sizing but appears to be only with some component of the VE compatible sizing or possibly the VE1260 resin.

The investigation was further extended to check whether this phenomenon was particular to only one VE compatible sizing formulation. Figure 7 presents results for the IFSS of a range of commercially available VE compatible glass fibres from different manufacturers (presumably with different sizing formulations) where fibre samples have been mounted to cardholders with the two different adhesives. The first notable

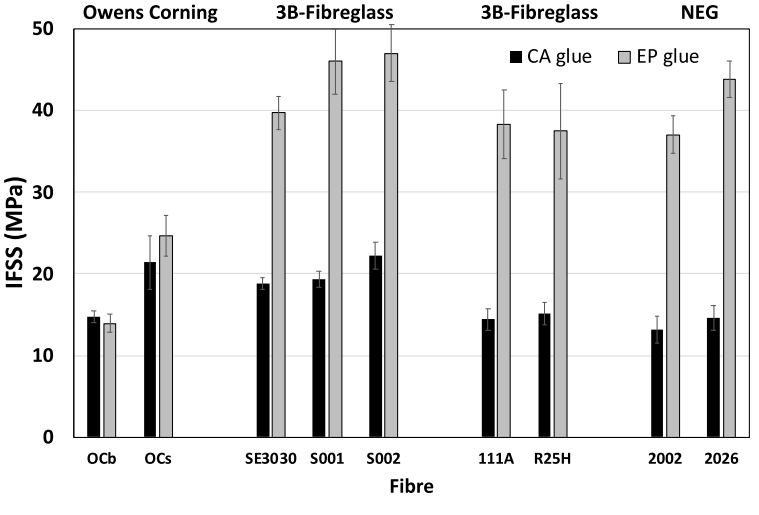

Figure 7. Influence of fibre mounting adhesive on IFSS fibres from various manufacturers.

observation about the results in Figure 7 is that all of the commercially sized VE compatible glass fibres exhibit the same sensitivity to the sample preparation fixing adhesive in their microbond IFSS performance. Secondly, it can be seen that the result of the comparative exercise to evaluate the effect of fibre sizing on IFSS in VE resin would have two very different conclusions depending on which type of adhesive was used to secure the test fibre samples. Using CA adhesive would result in a conclusion that VE compatible sizing formulations add very little to the measured IFSS over a bare fibre surface. However, using EP adhesive to secure the fibres it can be concluded that sizing does significantly increase the IFSS in the GF-VE system. It is worth mentioning again that the sample fixing effect in this system appears to be unknown in the literature and was only discovered in this project by serendipity. This certainly raises questions about the microbond test as a screening tool in the study of fibre-matrix adhesion and also highlights the major issues that occur when dealing with the effects on unknown sizing formulations on micromechanical performance.

3.4. Effect of the VE resin formulation

The influence of the fibre-mounting adhesive type of the measured IFSS has also been checked in two other VE resin systems. As part of the DACOMAT project the research team is also evaluating a number of new VE resin formulations. Of these VE1263 that is a rubber modified resin and VE1256, which is VE1260 with a low shrink additive added to the formulation have been evaluated with three 3B-fibreglass glass fibres in the microbond test. The results are presented in Figure 8 where it can clearly be seen that the large difference in IFSS performance related to the fibre mounting adhesive observed with the VE1260 resin is also obtained with these two developmental VE resins systems. It can also be seen that the maximum value of apparent IFSS is obtained with the S002 development fibre in the VE1263 resin system when the fibres are mounted using the EP-based adhesive. The problem with any further interpretation of this result is the fact that

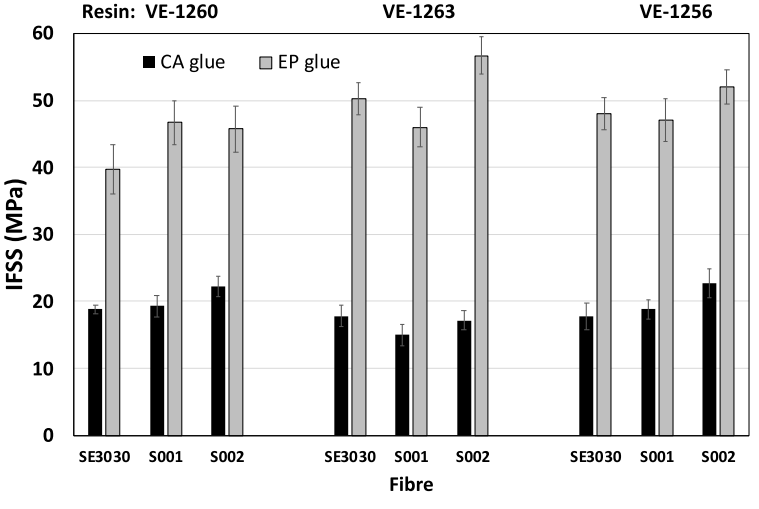

Figure 8. Influence of fibre mounting on IFSS of different VE resins.

the largest effect observed on the IFSS in the GF-VE systems is due to the fibre mounting adhesive. Hence, one is left to question if any of the IFSS results obtained actually reflect the nature of the fibre–matrix interface in a macroscopic composite or whether as yet other, still unidentified, issues with microbond sample preparation are the main cause for any observed differences in the IFSS results.

3.5. Possible mechanisms of the fibre fixing adhesive – IFSS interaction

Given the widespread use of CA glue in the mounting of single fibres for microbond testing (and fibre tensile testing) it is apposite to consider the possible nature of the observed negative interaction. It seems likely that, in order to affect the IFSS measurement, glue molecules must find their way to the point on the test fibre surface where the microdroplet is applied. Given the care taken in sample preparation and the fact that all fibres in a batch of microbond sample are affected it seems reasonable to eliminate any accidental contamination of individual test fibres. Two possible hypotheses are either wicking of the glue from the card contact point along the test fibre or deposition of glue vapours onto the fibre surface.

Two experiments were carried out to test these hypotheses. A batch of SE3030 fibres was mounted on steel washers using EP glue at the normal points. Once the EP glue had set, drops of superglue were applied to the steel frame used to hold the washers in place. One drop of glue was applied close to, but not in contact with, each mounted fibre. Microdroplet sample preparation with VE1260 and testing then was then carried out as normal. The average IFSS obtained was 12.4 MPa, similar to the low values obtained when the fibre were secured with the CA glue. Hence, the close proximity of the CA glue lowered the IFSS results despite there being no direct contact with the test fibre to enable wicking to take place. In a further experiment a series of card window mounted samples was prepared where the SE3030 fibres were secured to the card alternating CA and EP glue on a single A4 sized piece of card that contained 6 × 5 fibre mounting windows. Microdroplet sample preparation with VE1260 and testing then was then carried out as normal. The average IFSS obtained for the CA glue mounted fibres was 14.6 MPa and for the EP mounted fibres it was 14.7 MPa. Hence, in both of these experiments simply having liquid CA glue in the vicinity of EP glued fibres was enough to reduce the IFSS performance by 50%. These results would seem to indicate that the wicking hypothesis is unlikely to be the explanation of how the glue reached the test length of the fibre.

It is known that CA adhesive produces vapour that can react with a variety of chemical compounds on any nearby surface. This phenomenon is the basis of the CA fuming method used by criminal investigators for developing latent fingerprints [32]. A further range of scouting experiments was carried out which confirm that CA vapours appear to deposit on the fibre test surface during mounting and prior to application of the polymer droplet. This deposit appears to negatively interact with either some component common to VE compatible fibre sizings from different manufacturers or may possibly interfere locally with the curing reactions of the applied VE resin droplet. Thomason has analysed over 500 sizing patents from various glass fibre manufacturers and indicated that VE compatible sizings often contain VE and/or epoxy emulsions as film formers and usually contain MoPTMS as at least one of the silane coupling agents [31]. Unfortunately, the exact chemical formulation of glass fibre sizings in this study is kept confidential by glass

manufacturers and so further investigation of specific chemical interactions with CA is not possible. However, what is clear from our investigation is that all fibre samples affixed with CA glue have the potential to have CA vapours deposited on the surface of the fibre sizing. How, or whether, this will affect the results of further microbond testing appears to be a function of the chemical formulation of the fibre sizing and the polymer matrix.

4. Conclusions

This investigation of the use of the microbond test to measure the apparent IFSS in the glass fibre – vinylester composite system has revealed a number of issues related to the sample preparation that can significantly affect the outcome of the test. Firstly, it is clear that there exists a scaling issue with the curing of vinylester resin since cure schedules that produce well-reacted VE polymers on the macroscale do not result in cured microdroplets. Consequently, it was not possible to carry out the microbond test on samples with the same cure schedule as macroscale composites. This problem was not solved by curing in a styrene saturated environment in order to reduce styrene evaporation from the micro-droplets. Initial room temperature curing of droplets under an inert atmosphere may partially alleviate the issue and results in testable microbond samples. However, these droplets still exhibit relatively low IFSS values. Higher IFSS values may be obtained by applying elevated temperature stages to the sample cure schedule. Using this cure schedule it was found that glass fibres with a full sizing gave significantly higher apparent IFSS values compared to bare fibres or fibre coated only with silane coupling agent.

The IFSS values obtained with glass fibre – vinylester composites can be significantly changed by using a different glue when mounting single fibre microbond samples. The measured IFSS of VE compatible glass fibres was approximately doubled when the fibre samples were mounted using an epoxy glue in place of a cyanoacrylate glue. This phenomenon appears to be related to the deposition of cyanoacrylate vapours onto the surface of test fibres. This contamination then inhibits the level of IFSS obtained after the resin droplet is applied to the glue contaminated fibre surface. We conclude that there may often be many unexpected pitfalls that lie in wait of the unwary experimentalist preparing samples for the microbond test. Great care must be taken in ensuring that effects observed using the microbond test are evidence of real material characteristics and not artefacts of sample preparation.

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