INTERFACE STRENGTH IN GLASS FIBRE-POLYPROPYLENE MEASURED USING THE FIBRE PULL-OUT AND MICRODEBOND METHODS

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SUMMARY

Interface strength in glass fibre-polypropylene was measured using both fibre pull-out and microdebond methods. Excellent compatibility between two methods was obtained. The data from microdebond test could be divided into two groups according to whether or not there was constant interfacial friction after debonding

Keywords: interfacial shear strength, glass fibre-reinforced polypropylene

1 INTRODUCTION

Use of glass fibre-reinforced thermoplastic polymer composites has been rapidly increasing in a great many applications due to their high performance, mass processability and recyclability[1]. It is well-known that optimisation of the fibre-matrix interface is vital to achieve the desired performance in composite materials. Therefore over the years there have been a great deal of attempts to develop techniques which could accurately measure fibre-matrix adhesion levels in composites[2]. One of the generally accepted manifestations of adhesion is in the mechanically measured value of interfacial shear strength (IFSS)[3]. Although a number of direct methods have been available to determine IFSS such as the pull-out test, the microdebond test, the push-out test and the single fibre fragmentation test. There seems no overall consensus among these techniques and large scatter in the results seems to be a common issue which has been inhibiting the development of effective data reduction[4].

It appears that those experimental techniques have been extensively employed on thermosets based composites rather than thermoplastics and sample preparation for these techniques is not optimised for use with thermoplastic matrices[3]. Nevertheless comparing results obtained by different measurement methods may provide a better understanding of interfacial behaviour also in thermoplastic composites. The present

work is focusing on this interest and trying to get a further understanding of correlation between interfacial properties of glass fibre-reinforced polypropylene (GFPP) and data variation in the results.

2 EXPERIMENTAL

2.1 Sample Preparation

To minimise the uncertainties, only bare glass fibre (water sized glass fibre from Owen Corning; fibre diameter 17.5μm) and homopolymerised isotactic polypropylene (SABIC®PP 579S; MFR=47g/10min at 230°C and 2.16kg) were involved in the present work. The cardboard usually used in a single fibre tensile test was employed as a sample holder in both fibre pull-out and microdebond test as shown in Figure 1. For microdebond sample a single fibre first was glued at the contact points between the fibres and the window cut. Then a small piece of polypropylene fibre was transferred on the surface of the suspended glass fibre. The polypropylene loosely hung on the fibre and could shake off easily. Thus a soldering iron was used to slightly heat the polypropylene so that it could firmly attach to the fibre. Finally the whole assembly was put into an oven with the temperature 220ºC. After a certain amount of time in the oven the samples cooled down at ambient temperature. As for fibre pull-out specimens, the difference was that fibre had been embedded in the matrix on a hot plat before being put on the card. When the polypropylene melted it could penetrate into the card and eventually formed a strong bond with it.

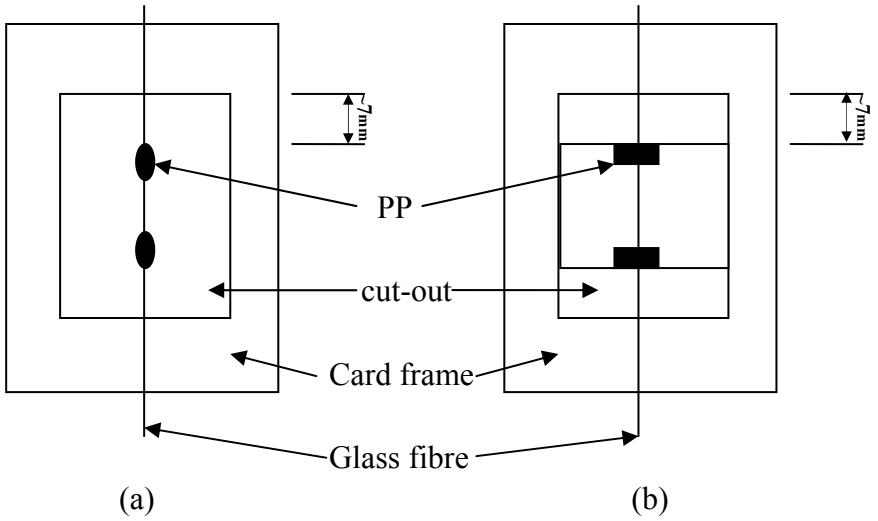


Fig. 1 Schematic representation for (a) microdebond sample and (b) fibre pull-out sample

2.2 Microdebond tests

To do the microdebond test we manufactured a device which has two movable knife edges controlled by a pair of micrometer heads with accuracy ~1um. A stereo-microscope was utilised to ease the positioning of knife edges and monitor the testing process. A single-column tensile tester with 10N load cell was used to carry out the test with the rate of fibre end displacement set to 0.1mm/min. The fibre with bonded resin droplets was mounted in the machine and pulled out of the droplet while the droplet was constrained by the knife edges (see Fig. 2). The tested samples were examined again under a microscope to see if pure debonding process had occurred.

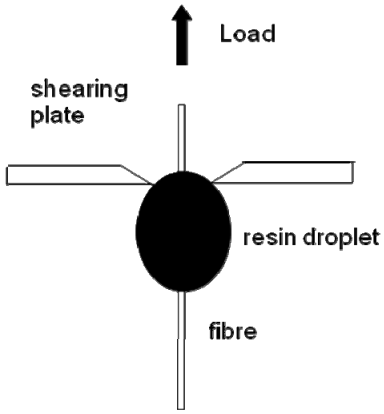


Fig. 2 Schematic representation of microdebond test

2.3 Fibre pull-out tests

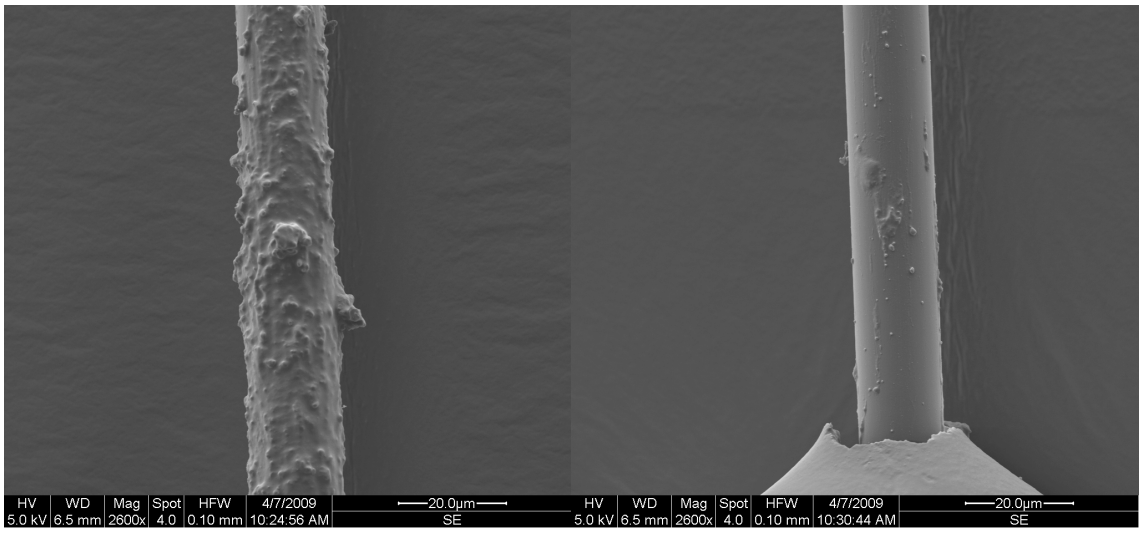
Single fibre pull-out tests were conducted with the same testing rate as in microdebond tests.

3. RESULTS AND DISCUSSION

3.1 Observation on tested specimens

Microscopy observation made on tested specimens in microdebond tests has divided them into two categories (A and B) as shown in Figure 3. About 2-7 micrometres thickness of residual resin was observed around the debonded area of the fibre in the group A with decreasing friction after the peak load was reached. Few tested samples with a much lower amount of residual resin were found in the group B, most of which

leave a clean debonded surface. According to further observation on matrix indentations caused by knife edges and the test results shown in section 3.3, such a difference does not arise from knife edges. No residual resin was observed on tested samples in fibre pull-out test.



(a) (b)

Fig. 3 SEM photograph of different debonded surface: (a) category A and (b) category B

3.2 Comparison of IFSS measured using fibre pull-out and microdebond test

Both microdebond and single fibre pull-out techniques have been utilized to determine the IFSS of GFPP. The results of measurements of the IFSS of polypropylene homopolymer and bare E-glass fibres obtained using these two methods are presented in the same plot as shown in Figure 4. Excellent agreement on the conventional data-reduction technique (e.g. the averaged IFSS) between two methods was obtained. This may imply that apparent IFSS could be an adequate quantitative parameter which could characterise the actual mechanism of interfacial failure in glass fibre-thermoplastic composites. The fact that both fitting lines do not tend to go through the origin will be addressed in the following section.

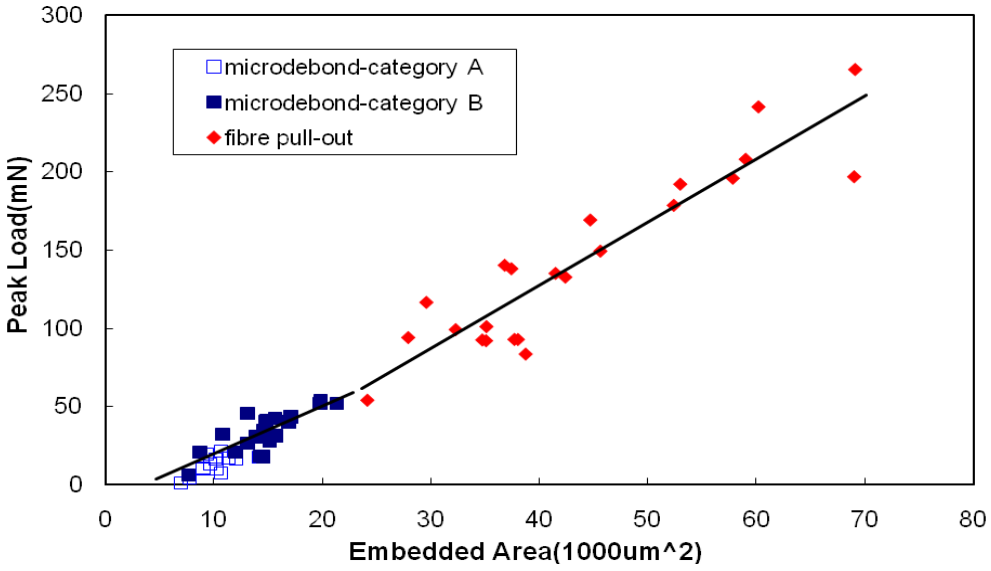


Fig 4 Peak load vs embedded area from both microdebond and pull-out test on single glass fibre-reinforced polypropylene.

3.3 Effect of matrix thermal history on IFSS

Apart from the good compatibility between two methods, the data from microdebond test apparently can be divided into two groups according to two categories of tested samples specified earlier. Indentation observed around the area far from where the fibre enters the resin droplet indicates that the microdebond failure that could occur at a weaker region than the interface is unlikely to be triggered by the knife edges. In addition the data from pull-out test which does not have such a systematic effect on the matrix correspond to tested samples all with a clean debonded area. Therefore more microdebond tests were conducted with variations of thermal history in matrices by changing their duration of stay in the oven with the same temperature from 4 minutes to 6 minutes. The result is shown in Figure 5

It can be clearly seen that the extra 2 minutes heating processs has made a significant impact on interface strength between bare glass fibre and homopolymer polypropylene studied in the present work. The 6 minutes set has an overall lower peak load than the 4 minutes set in the same range of droplet size. No tested sample of category A could be

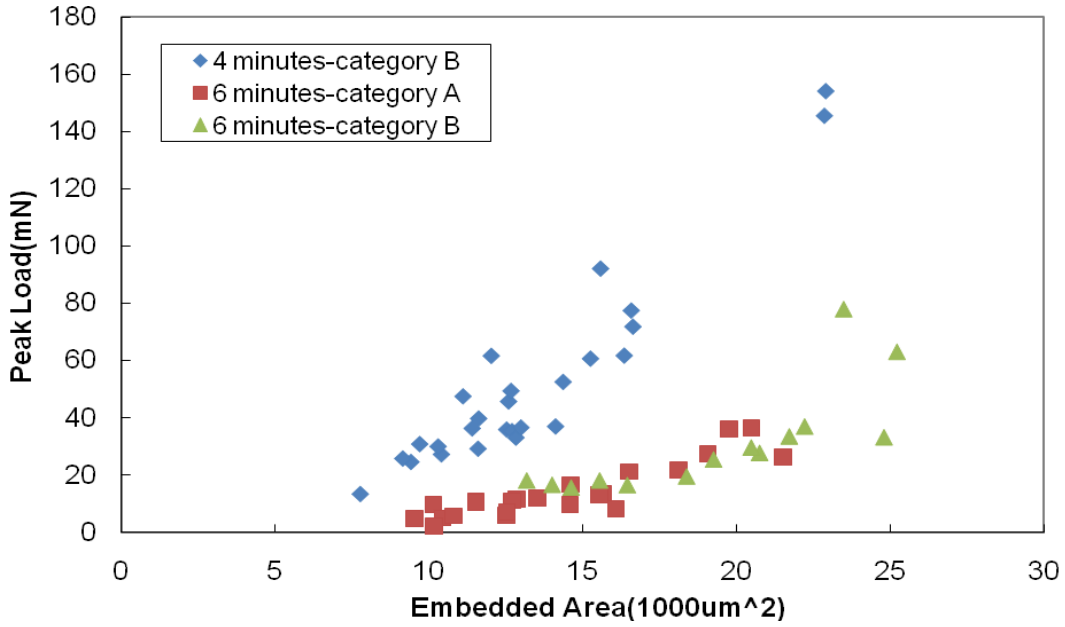


Fig. 5 Comparison of effect of matrix thermal history on interface strength of GF-PP measured using microbond method

found in the 4 minutes set, while about half of tested samples appears as category A in the 6 minutes set and they ahso seem to be affected by droplet size. As droplet size in the 6 minutes group increases it becomes more likely to have tested samples of category B than the other situation. The additonal thermal history has not only converted adhesive interfacial failure into cohesive matrix failure but considerably reduced the interface strength as well. This phenominon could be interpreted by the variation in matrix mechanical properties due to thermal oxidation and degradation. Small polypropylene droplets natually are more vulnerable to thermal penetration and more sensitive to oxygen attack at elevated temperature due to their relative high fraction of surficial molecules. Additionally, small droplets have less probability to possess an adequate amount of stabilisers than big ones. Therefore when the droplets with various size go through the same thermal treatment, the small ones could suffer from more severe thermal oxidation and even thermal degradation, which could dramatically reduce the degree of crystallinity of the polypropylene since high crystallinity requires high tacticity, which implies the presence of long, uninterrupted, stererospecific sequences along the chain[5]. As the tacticity along the polymer chain is reduced by either the addition of oxygen atoms on polymer chains or chain breakage, the crystallinity is expected to decrease. Unlike most glass fibre-thermosetting systems, there may be little or no chemical reaction across the interface between the bare glass fibre and neat polypropylene[6]. Instead the shrinkage stresses built around the interface during fabrication of thermoplastic composites are regarded as the major contribution to the stress transfer capability at the interface[7][8]. The shrinkage stresses at the interface

could be reduced as the degree of crystallinity of polypropylene decreases. As a result the decrease in crystallinity could lead to the degradation of both matrix mechanical properties and shrinkage stresses. In our case when the thermal condition was fierce relative to small droplets (e.g.6 minutes and 220°C), the deterioration of mechanical properties was so severe that polypropylene shear strength could be less than its interface strength with glass fibre. The cohesive matrix failure then would occur. As the droplets became bigger, it would be more possible for them to maintain sufficient crystallinity and in turn mechanical properties, which would provide a matrix shear strength higher than its interface strength with fibre. Adhesive interfacial failure then would occur. When the thermal treatment was relative mild (e.g.4 minutes and 220°C), even the smallest droplets would be able to maintain sufficient mechanical properties and prevent the matrix failure during the test. However, the interface strength could be still reduced to some extent, depending on the droplet size or the size of resin block in fibre pull-out test, which may also account for the fact that linear fitting lines in both methods did not go through the origin. The same thermal treatments were applied to fibre pull-out test and no significant difference was found. This is probably due to resin size was too sufficient to exhibit any dramatic effect as seen in microdebond test. Another heating process, 2 minutes and 220°C, was also applied to both tests. No measurable droplets were formed for micodebond tests and the lower IFSS was obtained in fibre pull-out tests.

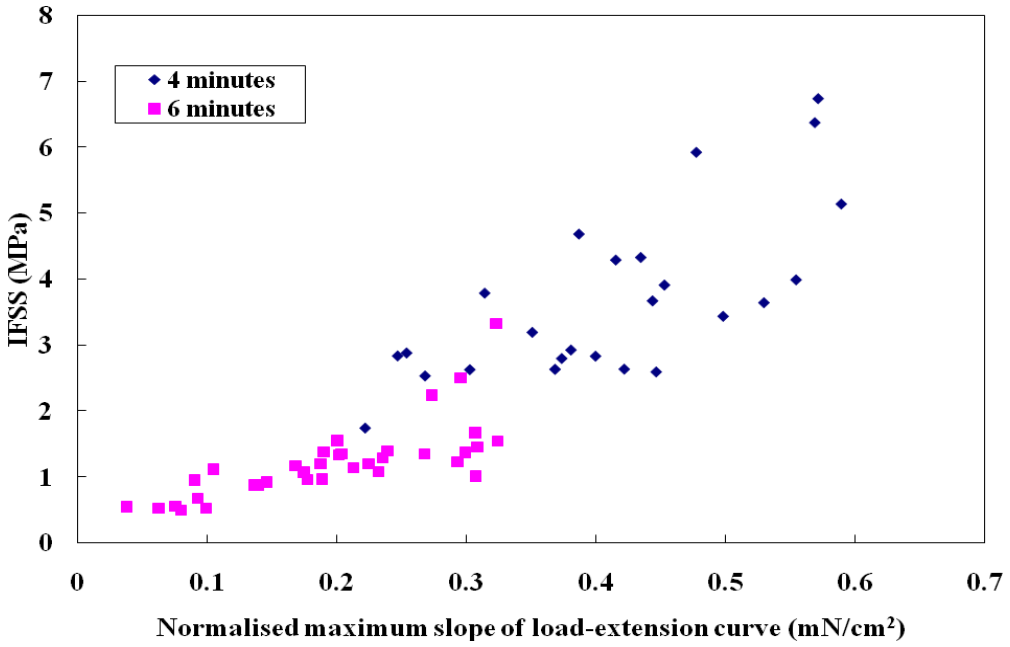


Fig. 6 Comparison of correlation between IFSS and experimental setup compliance in microdebond tests for 4 minutes and 6 minutes sets respectively

To further examine the above statement, the embedded length normalised maximum slopes of load-extension curves recorded in microdebond tests were manually estimated.

IFSS vs maximum slope of load-extension is plotted in Figure 6. The free fibre length was roughly kept the same throughout all the tests as shown in Figure 1. Thus the variation in the compliance of experimental setup may reflect the change in matrix stiffness. Figure 6 shows that the IFSS tends to rise as normalised matrix stiffness increases in both groups and overall, the 4 minutes group with a higher IFSS also has a higher normalised matrix stiffness than the other. This fairly agrees with the interpretation given to Figure 5.

4 CONCLUSIONS

Interface strength in glass fibre-polypropylene was measured using both fibre pull-out and microdebond methods. Excellent compatibility between two methods was obtained which may imply that apparent IFSS is an adequate quantitative parameter which can characterise the actual mechanism of interfacial failure in glass fibre-thermoplastic composites.

The data from microdebond test could be divided into two groups according to whether or not there was constant interfacial friction after debonding. Further investigation has revealed that such a division could be interpreted by the variation in matrix mechanical properties due to the effect of thermal oxidation and degradation on polymer crystallinity. This explaination was indirectly supported by the correlation between IFSS and maximum slop of load-extension curve normalised by embedded length. Further work will be focused on providing direct evidences to prove the hypothsis proposed in this work.

ACKNOWLEDGEMENTS

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