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Interlaminar Fracture Properties of Multi-Layer Woven Silk Fibre/Polyester Composites

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Abstract: The effect of the number of layers of woven silk fabric on the interlaminar fracture toughness of silk/polyester composites which is a measure of the resistance of the material to delamination crack propagation has been studied. The multi-layer woven fabric silk/polyester composites were produced by compression moulding technique with increasing layers of silk of between 8 and 14 layers in thermoset polyester. The composite panels have been tested for mode I interlaminar fracture using double cantilever beam specimens (DCB) testing method. It was found that the interlaminar fracture toughness, G_{IC} of the composite increases as the number of silk layers increases. Stable crack propagation is observed during the tests and the crack propagation areas showed all the fibres were bare with no matrix covering them as were seen at 100x and 500x magnification using Scanning Electron Microscopy. Failure occurred at the fibre-matrix interface with no fibre bridging observed between the two fracture surfaces. The smooth clean surface of the silk fibres is the result of weak interfacial debonding and the increases in interlaminar fracture toughness is due to improve strength of the composites panel. The results give the indication of the effect of the layers because the thicknesses of all the specimens are the same. In order to increase the interlaminar fracture toughness of woven silk fibre/polyester composites, multiple layers of woven silk fibre up to 14 plies has proven to be effective.

Key words: Double cantilever beam specimens, scanning electron microscopy, crack propagation, interfacial debonding, compression moulding, fracture surfaces

INTRODUCTION

Composite materials are well known as an excellent structural materials. Composite materials consist of two or more materials (i.e., fiber and matrix) combined to give superior performance compared to the properties of the individual components (Baral *et al.*, 2008; Mathews and Swanson, 2007; Mouritz *et al.*, 1999). Woven fabric silk fibre reinforced polyester fabricated in this study has been used as structural materials for solar car body due to their cost and weight advantages compared to carbon fibre/epoxy. Manufacturing of structural materials made from woven silk fibre/polyester composites calls for an improved technique to improve the interlaminar fracture toughness of the composites. The interphase between fibers and polymers has a strong impact on composite properties (Wetherhold *et al.*, 2007). For woven silk fiber/polyester composites, the interphase properties are determined by a surface coating, the so-called sizing, which is applied to the silk fibres during manufacture. Another method to increase the interlaminar fracture toughness is by increasing the number of layer of silk fibre so that increased stiffness will also increases interlaminar fracture toughness of the composites.

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Delamination is the most predominant and life limiting failure mechanism in composite materials, hence limiting the use of composites for structural applications. Laminated composite structures subjected to static, impact and/or cyclic loads may suffer significant inter-laminar damage as a result of insufficient interlaminar toughness. There are three basic modes of deformation by which a load can operate on a crack. Each will effect a different crack surface displacement. In mode 1, or the opening mode, displacements of crack surfaces are perpendicular to the crack plane and open the crack. In mode 2, or the shearing mode, the surfaces slide over each other in the direction parallel to the crack. Lastly in mode 3, or the tearing mode, the surfaces slide over each other in the out-of-plane direction. Delamination propagation is caused not only by interlaminar normal (mode-I) and in-plane shear (mode-II) stress fields but also the inter-laminar out-of-plane shear (mode-III) stress field (Murakami *et al.*, 2008; Stevanovic *et al.*, 2003). Delamination can be considered using fracture mechanics approach with the critical strain energy release rate as a criterion.

All three individual and/or mixed interlaminar toughness must be measured in order to characterize fully the interlaminar fracture properties of composite laminates. The mode-I fracture toughness of laminated composites is successfully measured by double cantilever beam (DCB) test method (Carlsson and Pipes, 1987; Wang *et al.*, 1984; Hug *et al.*, 2006; Yoshihara and Kawamura, 2006). The mode-II fracture toughness is also obtained by the comparatively simple end notched flexure (ENF) test method (Carlsson and Pipes, 1987; Suemasu *et al.*, 1987; Wood, 2007). Mixed in-plane shear and normal (mode-I and II) fracture tests can be also conducted by cracked lap shear (CLS) tests (Carlsson and Pipes, 1987; Becht and Gillespie, 1988). Experimental techniques for measuring mode-III toughness have been proposed by Becht and Gillespie (1988) and Tvergaard and Hutchinson (2008). However, they are not as practical as those proposed for measuring the mode-I and II toughness. An interesting method for measuring Mode III interlaminar toughness, called the edge crack torsion (ECT) test, has been proposed by Lee (1993). The distribution of the energy release rate along the crack front and necessary condition to obtain uniform energy release rate etc. are not well discussed. To utilize the method, more a rigorous equation for estimating the G_{III} value should be given and necessary conditions must be well discussed. However, the ECT test method is still attractive for measuring the mode-III fracture toughness, because the experiments are simple and easy to conduct, like the DCB and ENF test methods.

The aims of the research is to study the relationship between the number of layers of woven silk fibres with the mode I interlaminar fracture toughness (G_{IC}) of the woven silk fibre/polyester composites. Knowledge of the composite's resistance to interlaminar fracture is useful not only for product development and material data, but measurement of the interlaminar fracture toughness of the composite is useful for establishing design allowable for damage tolerance analyses of composites structures. In order to achieve cost effective design, this will allow the optimum number of layers required for certain applications to be determined such as the minimum number of layers required for solar car body or other structural applications where the delaminations is the main problems.

MATERIALS AND METHODS

The material used was a silk fibre reinforced polyester composite with four different number of silk fibre layers. The panels were prepared by a compression moulding technique. In order to initiate delamination, a treated release film of thickness 13 μm was placed at laminate mid-thickness during moulding. The thickness of the film was sufficiently thin so as to minimize disturbance of the composite during manufacture. The length of the starter film from the loading line was chosen to be 50 mm so that the influence of the hinges can be neglected.

All specimens were nominally of 20 mm width and 4 mm thickness. The specimens had piano hinges blocks bonded to the starter defect end to permit load introduction. The hinge and the specimen

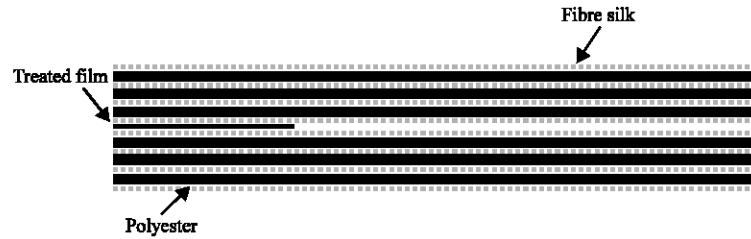


Fig. 1: Cross-section of the laminated composites

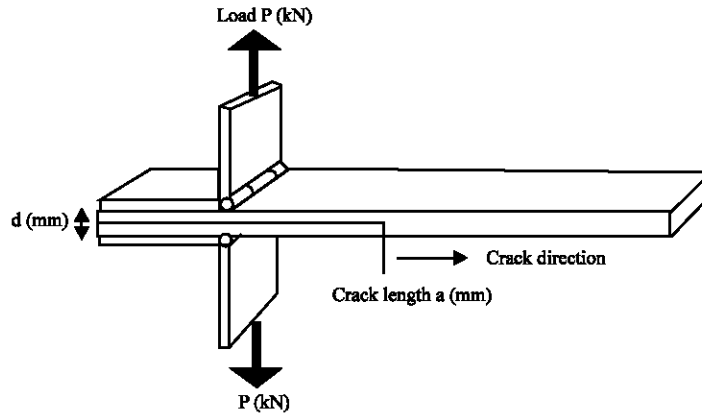


Fig. 2: Geometry of the double cantilever beam specimen

were lightly abraded using a sand paper since the load required to delaminate the specimens in these tests are quite low. Bonding of the loading tabs was done immediately after surface preparation. The bonding adhesive used was a room temperature cure epoxy adhesive. In order to minimize errors in the applied moment arm, the distance from the hinge pin to the center line of the specimen arm did not exceed 10 mm. The number of silk layers used for the composites are 8, 10, 12 and 14 layers. Figure 1 shows the cross-section of the laminated composites.

The tests were conducted at the Structural laboratory, Universiti Kebangsaan Malaysia in February 2008 according to the European Structural Integrity Society (ESIS) Protocol for mode I interlaminar fracture testing of composites (Davies, 1993). The detailed geometry of the Double Cantilever Beam specimen is shown in Fig. 2. All tests were conducted on an Instron Universal Testing machine. A 5 kN load cell was used since the maximum load never exceeded 5 kN. The specimen was loaded at a crosshead rate of 5 mm min^{-1} . At this loading rate, crack propagation can be followed visually and recorded reasonably well. Load and displacement data were recorded continuously by a personal computer at a rate of 10 points per sec.

To monitor the position of the crack tip, both sides of the specimen were coated with the white correction fluid to enhance the visibility of the propagating crack. At intervals of 5 mm, the delamination length measured visually on a scale marked using the correction fluid, was noted together with the corresponding load and displacements. The point on the load-displacement plot at which the crack first propagated from the insert was also recorded. When crack has propagated at least 60 mm from the starter film, the test was stopped and the specimen was then completely unloaded.

The fracture surfaces of the specimen was then analysed on both side of the ply using the scanning electron microscope. The micrographs of the fracture surface of the specimens are very

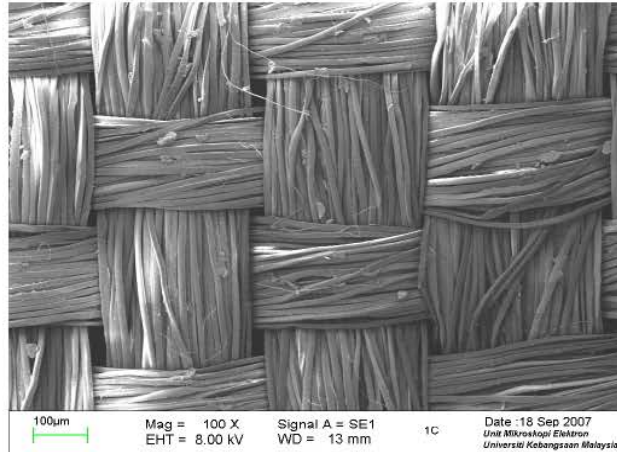


Fig. 3: Scanning electron microscopy image of the silk fibre at 100x

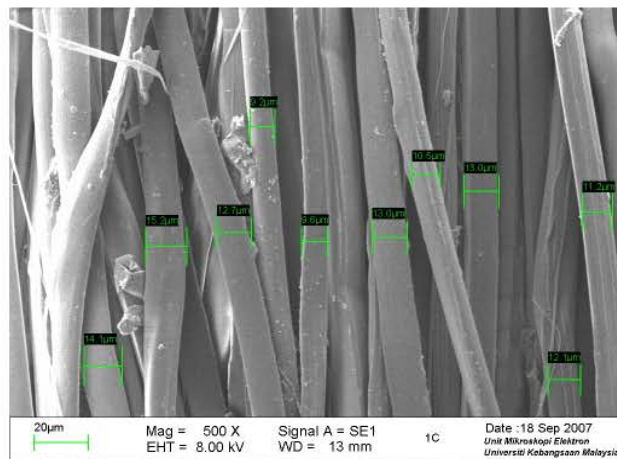


Fig. 4: Scanning electron microscopy image of the silk fibre at 500x

important in giving information on the fibre-matrix failure mechanism. Figure 3 and 4 show the Scanning Electron Microscopy images of the silk fibre at 100x and 500x. The average value of the silk fibre was determined to be around 12 µm.

Data Analysis

The propagation values of the interlaminar fracture toughness were determined using the corrected beam theory. The simple beam theory expression for the compliance of a perfectly built-in DCB specimen is as follows:

$$G_{ic} = \frac{3Pd}{2Ba}$$

where, P is the load, d is the displacement, B is the specimen width and a is the crack length.

The formula will underestimate the compliance because the beam is not perfectly built-in, hence the beam must be treated as containing a slightly longer crack, $a+D$ and D may be found experimentally by plotting the cube root of compliance, $C^{1/3}$, as a function of crack length as shown in Fig. 6 for 10 layers silk fibre. G_{IC} is then given by:

$$G_{IC} = \frac{3Pd}{2B(a+D)}$$

where, P is the load, d is the displacement, B is the specimen width and a is the crack length and D is the value of intersection between the plot line and the x-axis.

RESULTS AND DISCUSSION

The average values of G_{IC} determined for a different layers of silk is shown in Table 1. Stable crack growth was observed over all the specimens and is shown on the graph of load (P) against displacement (d) in Fig. 5 for 10 layers woven silk fabric. Increasing the number of silk layers were found to increase the stiffness and interlaminar fracture toughness in woven silk fibre/polyester composites. The results show that the propagation values away from the point of crack initiation were very much higher than that of initiation values. The toughness values at initiation from the insert are required because in most woven double cantilever beam specimens, fibre bridging occurs as the delamination progress along the length of the beam and increases the energy required to propagate the delamination further. The initiation values of G_{IC} are also of great interest since they are the lowest values and therefore offer the most conservative values for the design considerations (De Morais and De Moura, 2006).

Fibre bridging resulted in a significant increase in G_{IC} values (Huang and Hull, 1989; Martin, 1995). Stable delamination are observed in the load-displacement curve for all composites specimens. Instead of the load decreasing rapidly from the onset of delamination growth, the delamination grew slowly and the load did not decrease. The cause for the stable delamination growth is that the increase in interlaminar fracture toughness cause by fibre layers occurs at a greater rate than the decrease of the strain energy release rate. However, values of G_{IC} obtained from the delamination growth from the insert is unaffected by fibre layer. The characteristics of fracture development after fiber-matrix separation (debonding along the fiber/matrix interface) gives further indication of the interface strength.

Results obtained shows an increases of G_{IC} (initiation) from 0.436 to 1.687 kJ m^{-2} and an increases from 0.847 to 1.872 kJ m^{-2} for G_{IC} (propagation). Davies (1993) conducted a series of mode 1 double cantilever beam tests on IM6/PEEK composites. Their results indicated that the mode 1 propagation values of G_{IC} are dependent on specimen thickness. This thickness effect is related to the contribution of multiple cracking and fibre bridging which results in higher values being obtained on thicker specimens.

Figure 5 and 6 show the graph of load against displacement and the graph of $C^{1/3}$ versus crack length (a) for corrected beam theory for 10 layer woven silk fibre/polyester composites. All the sample specimens show the same pattern of graph of load against displacement as shown in Fig. 5. From the values of maximum load and the load at the point of non-linearity, the interlaminar fracture toughness,

Table 1: Initiation and propagation interlaminar fracture toughness, G_{IC} for all composition of silk/polyester

Material	G_{IC} (Initiation) kJ m^{-2}	G_{IC} (Propagation) kJ m^{-2}
8 plies silk/polyester	0.436	0.847
10 plies silk/polyester	1.030	1.530
12 plies silk/polyester	1.244	1.638
14 plies silk/polyester	1.687	1.872



Fig. 5: Graph of load (P) against displacement (d) for 10 layers silk fibre

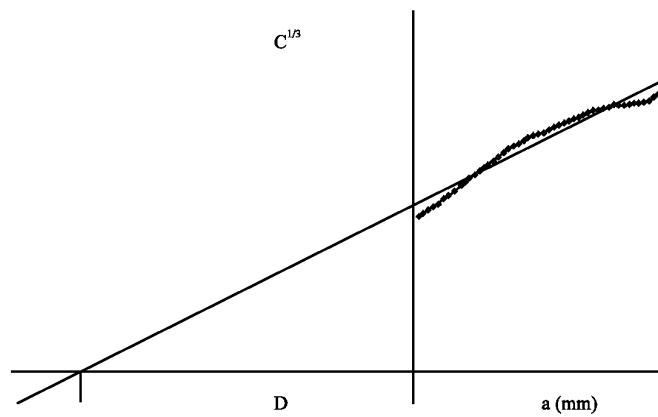


Fig. 6: Graph of $C^{1/3}$ versus crack length (a) for corrected beam theory

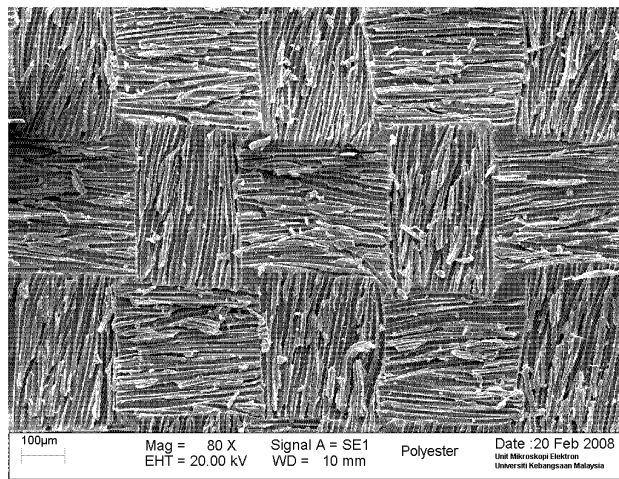


Fig. 7: The matrix side of fracture surfaces showing the fibre pull out at 80x magnification

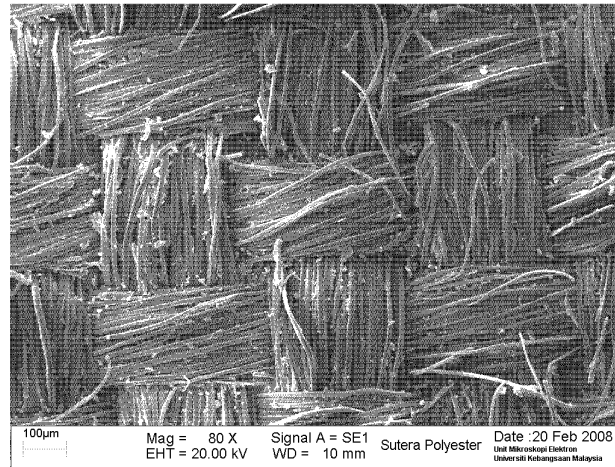


Fig. 8: The fibre side of fracture surfaces showing the fibre with very small matrix fragments at 80x magnification

G_{IC} at recorded length was calculated. The interlaminar fracture tests on the DCB specimens resulted in a linear loading curve up to initiation from the insert. The values of G_{IC} obtained in the current experiments show very little scatter suggests that the test set-up is reliable.

Figure 7 and 8 shows both sides of the silk fibre/polyester fracture surfaces for 8 plies silk/polyester composites. The propagation areas show that all the fibres are bare with very little matrix covering them as seen at higher magnification. The smooth clean surface of the fibres are the result of weak interfacial bonding. Similar results are apparent for all the other silk fibre/polyester composite layer arrangement. The SEM micrographs explained why the stable crack growth was observed over all the specimens for the different fibre layers arrangement and no crack jump phenomenon occurred during the tests.

CONCLUSION

This experiment shows that increase in the number of layers of fibre is influencing the interlaminar fracture toughness of woven silk fibre reinforced polyester composites for the specimens with the same thickness of 4 mm. The measured value is attributed to the toughness of the matrix over a reduced section caused by the presence of the non-adhering fibres. The results give the indication of the effect of the layers because the thicknesses of all the specimens are the same. In order to increase the interlaminar fracture toughness of woven silk fibre/polyester composites, extra layer of woven silk fibre can be used. However, for the manufacture of woven silk fibre/polyester for solar car applications, due consideration must be given to the high costs of silk fibre if more layers of silk are required for improved interlaminar fracture toughness properties of these composites.

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