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PET interleaving veils for improved fracture toughness of glass fibre/low-styrene-emission unsaturated polyester resin composites

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ABSTRACT: The use of interleaved polyethylene terephthalate (PET) veils to increase the interlaminar fracture toughness of glass fiber-reinforced, low-styrene emission, unsaturated polyester resin composites, was investigated. PET, being chemically similar to the unsaturated polyester resin, was expected to exhibit good wetting and strong interaction with the matrix. Composite laminates were manufactured by hand lay-up, with the veil content varying up to 7%. The effects of PET veils on the interlaminar shear strength, flexural strength, flexural modulus, glass transition temperature, damping parameters, and Mode-I interlaminar fracture toughness of the composite were studied. The veils were found to enhance most of these properties, with only minor negative effects on flexural stiffness and T_g. The PET/resin bonding did indeed prove to be strong, but the enhancement of fracture toughness was not as much as expected, because of the weaker glass/resin interface providing an alternative crack propagation path. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 42877.

KEYWORDS: composites; mechanical properties; polyesters; thermosets

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INTRODUCTION

The energy absorption capability of composite materials is critical for many applications in the automotive, consumer goods, marine, wind energy, and construction industries. Thermoset composites, although extensively used, have relatively brittle interlaminar interfaces, which makes them prone to delamination. This limits their energy-absorption capability, under impact loading. Unsaturated polyester and vinylester thermoset resins are widely used matrices for composites, but "styrene emission" during their processing presents a problem, as it is highly hazardous. "Low-styrene emission" resin grades are commercially available and the use of such resins is desirable from a health and safety viewpoint. However, several researchers have reported that the resin formulations used to limit styrene emission are significantly more brittle than traditionally employed resins. For example, the mechanical properties of composites based on low styrene emission unsaturated polyester resins were reported by Baley *et al.*[1] for marine applications. They observed that low styrene emission grades were significantly more brittle than the traditional resins used in marine industries. Perrot *et al.*[2] concluded that low styrene emission unsaturated polyester resins when used as matrix in glass fiber reinforced composites exhibit lower damage resistance. An easier damage initiation and propagation is observed in such composites with extensive damage zones compared to their traditional counterparts. The impact damage resistance of such composites need to be improved so that they can be more widely used. Skrifvars *et al.*[3] studied the morphology of low styrene emission glass fiber/unsaturated polyester laminates by polarization microscopy and established that this technique can be useful to understand how the wax additives affect the styrene emission and delamination resistance in the laminates. Suitable measures should be taken with such low emission grades of resins to minimize the associated problems like enhanced brittleness, delamination, etc.

Delamination can be inhibited by using advanced textile technologies such as knitting, braiding, through-thickness stitching, and weaving, using suitable fiber preforms.[4] Delamination resistance can also be improved by tailored alignment of the fibers. The mode I fracture toughness of multi-directional laminates with 0/90, 0/45, 45/-45 fiber alignments, measured in the form of critical strain energy release rate (G_{IC}), by double cantilever beam (DCB) tests, were reported to be 30% higher than unidirectional laminates.[5] The use of particulate fillers in the interlayer can also increase the mode-I delamination resistance of polymer composites. Different fillers like nanoclays, rubber particles, carbon nanotubes (CNTs), and carbon nanofibers have all shown promise for improving G_{IC}.[6–11] A disadvantage of the above techniques though is that they tend to complicate

Table I. Percentage by Weight of Composite Components for Panels

	Short beam shear and 3PB panels			
Abbreviation	Constituents	% PET veil	% Glass fiber	% Resin
G	All glass layers	0	79.4	20.6
GPETG	One PET veil between each glass layer with no veil on the outer surfaces	4	73.2	22.8
PETGPET	One PET veil between each glass layer with one veil placed on each of the two outer surfaces	5.4	69.7	24.9
GPETPETG	Two PET veils between each glass layer with no veils placed on the outer surfaces	6.2	59.9	33.9
DCB panels				
G	All glass layers	0	73.5	26.5
GPETG	One PET veil between each glass layer with no veil placed on the outer surfaces	4	66.5	29.5
GPETPETG	Two PET veils between each glass layer with no veil on the outer surfaces	7.1	63.8	29.1

the manufacturing process or place limits on the usable lay-ups. One common way of improving the mode-I fracture toughness of composites, which does not adversely affect processing, is by introducing interlayer veils. Several studies have been conducted on interleaved carbon fiber reinforced composites,[12–17] but very few on glass fiber composites],[18–21] which is the subject of this article.

The role of embedded interleaving materials during the interlaminar crack propagation in E-glass/epoxy composites was studied by Yasaee *et al.*.[19] The materials investigated as interleaves were thermoplastic films, thermoplastic particles, chopped fibers, E-glass/epoxy prepreg strips cut to size, thermoset adhesive films, and thermoset adhesive particles. The interleaving strips were placed on the way of crack propagation in the midplane of DCB specimens. The Mode II interfacial toughening through these discontinuous interleaves was also studied by the same authors.[20] The results indicated that the Mode-I fracture toughness of the composites was not much influenced by the incorporation of such interleaving materials, but the Mode-II fracture toughness was significantly enhanced by the introduction of more tortuous crack paths. They suggested that bridging of inserted veil fibers between the two delaminating plies during Mode-I crack propagation might be an efficient way of increasing the Mode-I fracture toughness in laminates.

Suzuki *et al.*[21] studied the influence of silane coupling agents on the interlaminar fracture in glass fabric reinforced unsaturated polyester laminates. They used methacryl silane as a representative coupling agent which reacts chemically with the resin during curing, and epoxy silane as another coupling agent, which does not. Their main objective was to understand the role of glass fiber/matrix interfacial adhesion on the Mode-I crack propagation behavior in the composites. The fracture toughness and crack propagation behavior were dependent on the types and concentrations of the silane coupling agents.

In our previous work[22] we reported on the use of polyamide veils as interleaving materials in glass fiber composites, with a low styrene emission grade of unsaturated polyester resin. A laminate containing two layers of polyamide veils between each glass layer exhibited an increase of up to 170% in G_{IC} over the values obtained for the all-glass fiber composite. Thus, the interleaving technique can be an effective way of improving interlaminar fracture toughness in glass fiber reinforced composites. However, the chemical compatibility and bonding of the interleaving material with the matrix resin is likely to play a major role in the delamination behavior. In the present study, a low styrene emission grade of unsaturated polyester resin has been used as the matrix component, and chemically similar PET veils have been used as the interleaving materials. The performance of the resulting composites has been investigated with short beam shear (SBS), three point bend (3PB), DCB tests, and dynamic mechanical analysis (DMA). The DCB fracture surfaces were examined under the scanning electron microscope (SEM) to understand the role of PET veil in the mid plane during the mode-I crack propagation.

EXPERIMENTAL

Materials

Crystic 2-446PA, a room temperature curing, low styrene emission, unsaturated polyester resin from Scott Bader was used to manufacture composite laminates. The catalyst used was Butanox LPT-IN (Akzo Nobel). A multi-axial glass fiber, Ahlstrom 42052L, was used as the reinforcement material. A 45 g/m^2 apertured polyethylene terephthalate (PET) veil (Syntex SLA45 Polyester) was used as the interleaving material. The thickness of the veil was 0.55 mm +/− 0.1 mm.

Fabrication of Composite Laminates

Interleaved and noninterleaved composite specimens were manufactured by hand layup technique using metal moulds. Laminates with six glass fabric layers were made for the SBS and 3PB tests, while laminates with eight layers of glass fabric were manufactured for the DCB tests. The laminates were laid up in

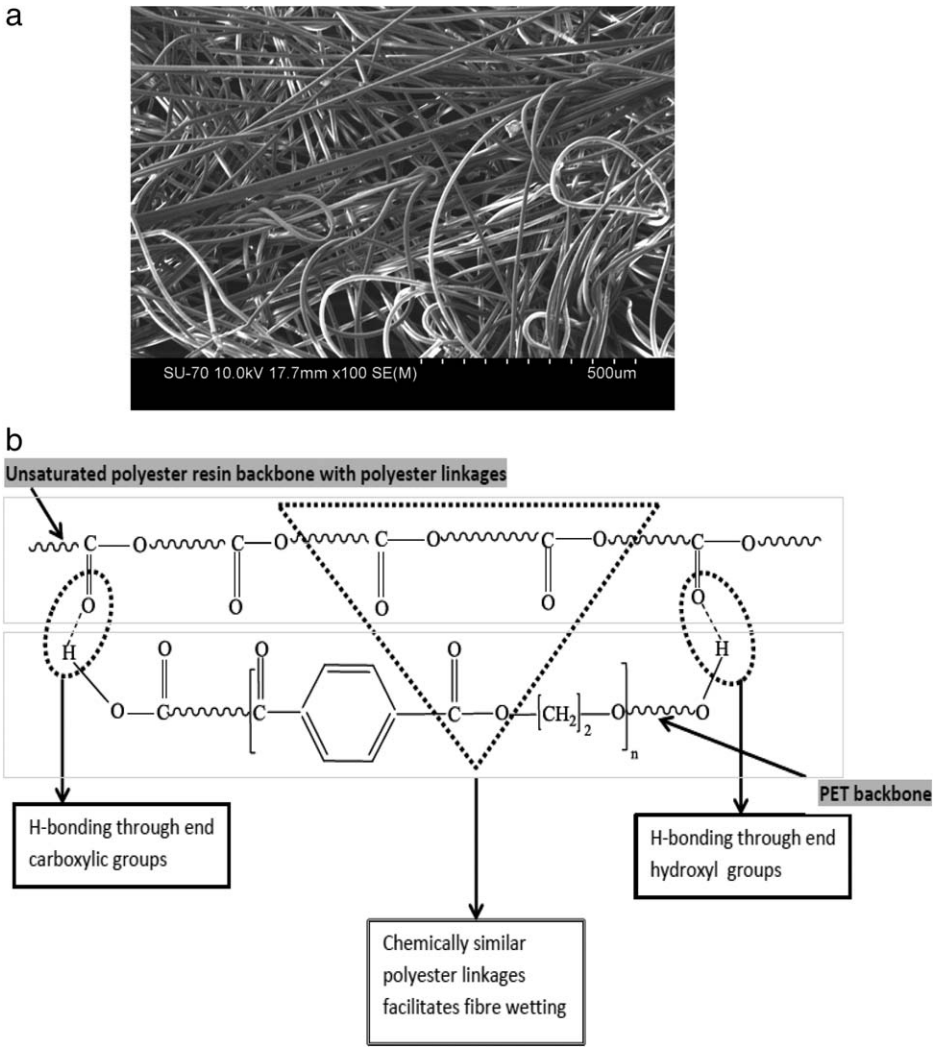

Figure 1. (a) SEM micrograph of the PET veil. (b) Possible chemical interaction between the PET veil and unsaturated polyester resin.

a unidirectional $[0]_n$ layup and formed by compression moulding in a hydraulic press. The laminates were cured at room temperature for 24 hours, followed by postcuring at 80°C for 3 hours. The amount and position of the interleaving PET veils were varied in four types of laminates. Table I shows the abbreviations of the different types of laminates manufactured and their respective constituents. The veil content varied from 0% (G) up to 6% (GPETPETG) by weight in the SBS/3PB laminates, and from 0% up to 7% in the DCB laminates.

Characterization

Short beam shear (SBS) testing was conducted with a span-to-thickness ratio of 4:1 on a multi-purpose Tinius Olsen machine in accordance with ASTM D2344. Five specimens were tested from each set. 3PB testing was carried out in accordance with ASTM D7264 on the same machine with a span-to-thickness ratio of 32 : 1. Five specimens were tested from each set. Dynamic mechanical analysis (DMA) was done in 3PB mode over a temperature range of 30°C to 125°C with 1 Hz frequency and 10 microns amplitude in accordance with ASTM D5023.

The DCB testing was conducted in accordance with ASTM D5528. A unidirectional layup of glass fiber reinforcements was used. Both sides of the test specimens were coated with a thin layer of Tipp-Ex correction fluid prior to testing, from the end of the PTFE insert, to help in detection of the visual onset of delamination. After the precrack length, thin vertical lines were drawn on each specimen every 1 mm for the first 5 mm, and then every 5 mm for the remaining length of the sample. Loading blocks were attached to the samples to apply the load during testing. The specimens were tested at a constant rate of 5 mm/min. The crack growth was tracked visually and also with the use of a LaVision two-dimensional (2D) Digital Image Correlation (DIC) camera, with results analyzed via LaVis 7.4 processing software.[22] Five specimens of were tested from each set. Fracture surfaces obtained from the DCB tests were examined under a Hitachi SU-70 Field Emission Scanning Electron Microscope (FESEM). The samples were coated with a thin layer of gold prior to analysis, using an EMITech sputter coater for 40 s. An acceleration voltage of 10 kV was used.

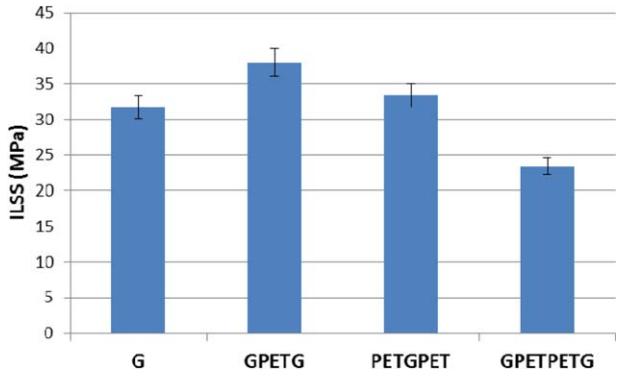

Figure 2. Interlaminar shear strength (ILSS) values of the composite specimens. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RESULTS AND DISCUSSION

The PET fibers in the veil were approximately 20 μm in diameter and randomly distributed and oriented [Figure 1(a)]. PET (thermoplastic polyester) fibers and the unsaturated polyester resin (thermoset polyester), both being of same chemical nature (polyester), are expected to have strong interaction with one another, which is likely to enhance the overall composite properties. Figure 1(b) illustrates potential interactions, which include improved fiber wetting because of chemically similar polyester linkages, and hydrogen bonding between carboxylic groups and also between hydroxyl groups.

Short beam shear (SBS) tests provided the interlaminar shear strength (ILSS) of the various specimens, as shown in Figure 2. The ILSS of the G, GPETG, PETGPET and GPETPETG laminates were found to be 31, 38, 33 and 23 MPa respectively. GPETG and PETGPET exhibited an increasing trend in the ILSS values. The PET veil between the two glass fiber plies being flexible in nature and being strongly bonded with the adjacent plies, takes part in energy absorption under shear and improves the ILSS. However, a 25% drop in ILSS was seen in GPETPETG with two PET veils in the interlaminar region, which is similar to our findings on polyamide veils in the same glass/resin system.[22] It is apparent from the results that the interlaminar region between the two adjacent PET veils, both being flexible in nature, might act as a weak point under shear stress and the ILSS is reduced.

The flexural strength and modulus are presented in Figure 3(a, b) respectively. The reference laminate G exhibited a flexural strength of 448 MPa. This value compares well with the flexural strength of a similar GF/unsaturated polyester resin composite reported by O'Donovan *et al.* (466 MPa)[22] and Velmuruganet *et al.* (378 MPa).[23] An increase in flexural strength was observed for all the interleaved specimens. The flexural strength increased by 13%, 21%, and 15% in GPETG, PETGPET, and GPETPETG laminates. Such an increase in flexural strength can be attributed to the similar chemical nature of the veil and the matrix resin which helped them to strongly interact with one another. Note that a drop in flexural strength, up to 23%, was evident in our previous work with a similar GF/unsaturated polyester resin system with interleaving polyamide veils.[22] The enhancement in

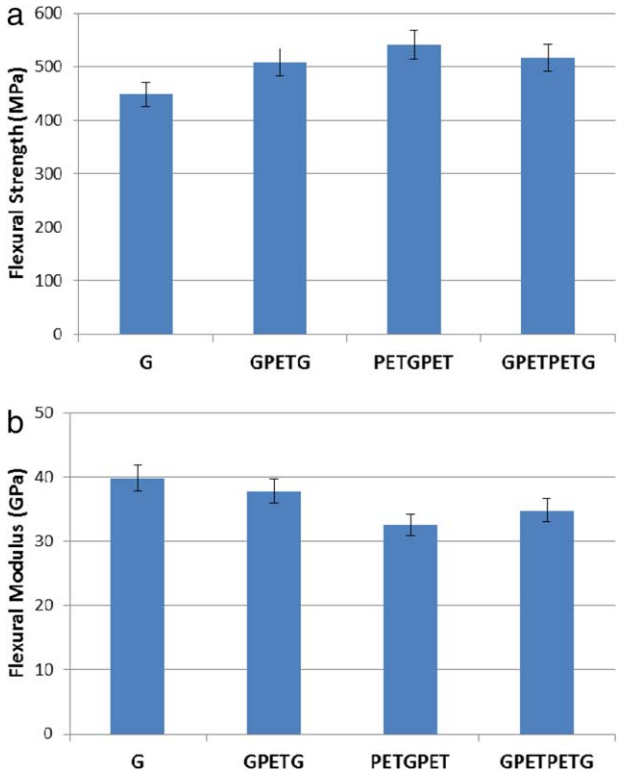

Figure 3. (a) Flexural strength and (b) flexural modulus of the composite specimens. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

flexural strength observed here reflects the additional advantage of using chemically similar interleaving veils with the matrix resin. The flexural modulus of G, GPETG, PETGPET, and GPETPETG samples were found to be 39.8, 37.8, 32.6, and 34.8 GPa respectively. This represents a 5–18% drop in flexural modulus with the incorporation of PET veils, with highest drop seen for PETGPET laminates where the veils were present on the outer surfaces also. This drop is not surprising since the incorporation of veils results in a more flexible connection between plies, allowing them to deform more independently in an axial direction, and so more easily accommodate the

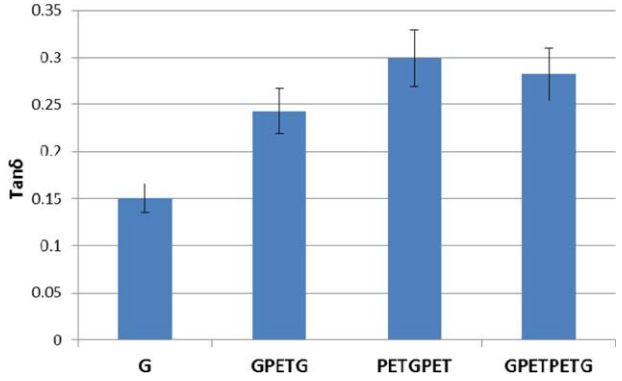

Figure 4. Damping parameter (tanδ) of the composite specimens. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

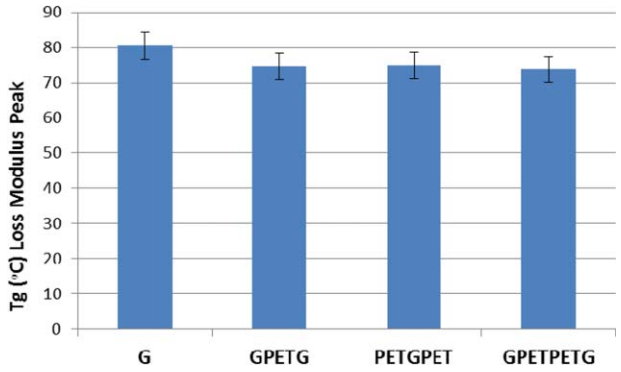

Figure 5. Glass transition temperature (Tg) of the composite specimens measured from the peak of loss modulus curves. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

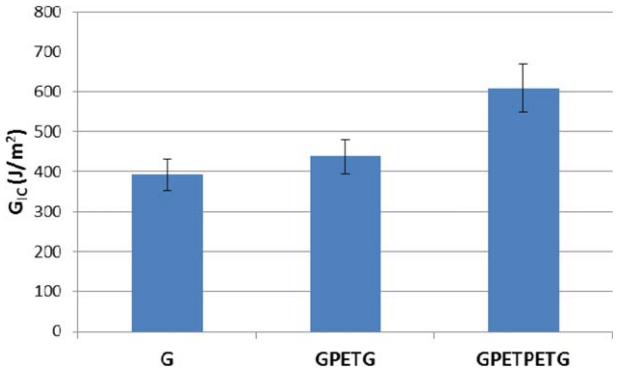

Figure 7. G_{IC} propagation values of the composite samples under DCB testing. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

variations in axial deformation through the laminate thickness that occur during bending.

The peak damping parameter (tan δ) values obtained from the DMA tests showed a clear upward trend with incorporation of veils, as seen in Figure 4. Relative to the reference G panel, the increase in tan δ was 98%, 87%, and 61% for the PETGPET, GPETPETG, and GPETG laminates respectively, indicating higher energy absorption capacity under dynamic loading conditions when veils are introduced. The 38% larger value of tan δ for PETGPET laminates relative to the GPETG laminates demonstrates that the outer surface veils in laminates play a significant role in energy absorption.

The glass transition temperature (T_g) of the laminates is shown in Figure 5. A 6–8% reduction in T_g was observed for the interleaved composites. The glass transition temperature of the PET veil itself was 71°C as measured by differential scanning calorimetry (DSC). The flexibilization of the PET veil fibers above T_g and strong interaction of the resin molecules with the PET fibers through H-bonding may be responsible for the small reduction in T_g of the composites.

The Mode-I type fracture toughness of the G, GPETG, and GPETPETG panels was measured by DCB testing. PETGPET samples were not subjected to DCB testing as they contained one PET veil at the crack propagating plane, similarly to that of the GPETG samples, and hence should give similar results. Figure 6 shows representative load versus displacement curves from the DCB tests. All the GPETPETG samples initially endured much higher loads than the G and GPETG specimens, although later in the test the load dropped to the level seen by the G and GPETG specimens. Consequently the critical strain energy release rate, $G_{ICpropagation}$, shown in Figure 7, was 56% higher for the GPETPETG specimens than for the reference G specimens. In comparison the increase in $G_{ICpropagation}$, for the GPETG specimens over G was only 12%. The $G_{ICpropagation}$ values for G, GPETG, and GPETPETG were 391, 438, and 608 J/m^2 respectively. These values compare well with the previously reported values of glass reinforced composite systems. In our previous work,[22] the $G_{ICpropagation}$ values were found to be 430 J/m^2 for the reference laminate, 780 and 1171 J/m^2 for the laminates with one and two interleaving polyamide veils respectively. The improvement in Mode-I fracture toughness was thus more

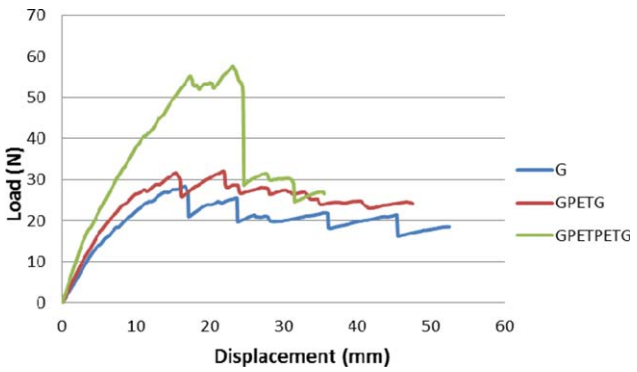

Figure 6. Load vs displacement curves of the composite specimens under DCB test. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

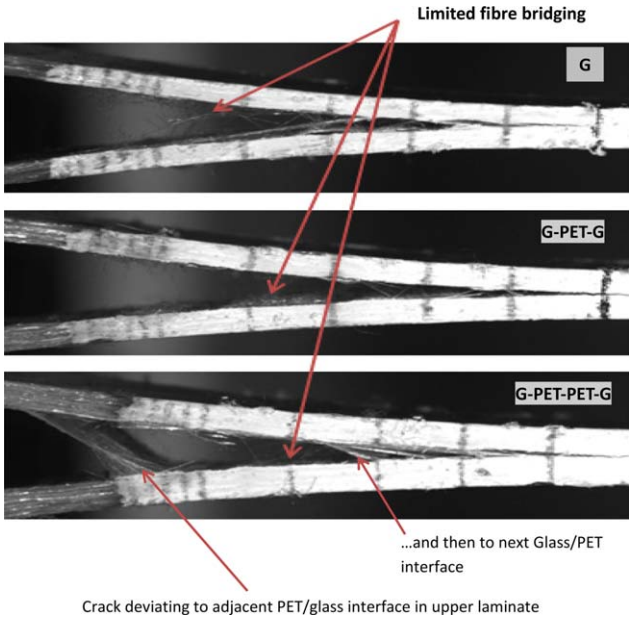

Figure 8. Images from DIC camera indicating limited fiber bridging in the interleaved samples. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

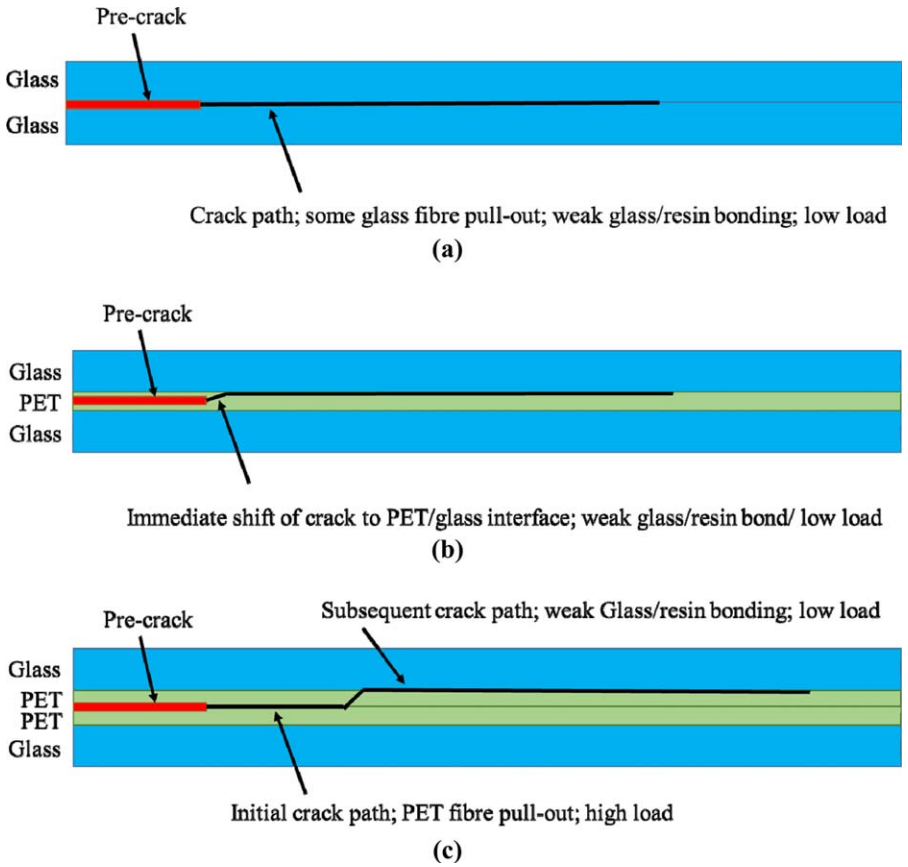

Figure 9. Schematic diagram of the crack propagation in (a) G, (b) GPETG, and (c) GPETPETG DCB specimens. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

pronounced with the polyamide veils than that observed here with the PET veils. The chemical nature of the veil, areal weight, bonding with the matrix and architecture play a major role in arresting the crack growth during Mode I delamination. It is important to note here that the architecture and the areal weight of the PET and the polyamide veils were different in the two cases as they were sourced from market.

Yasee *et al.*[19] reported the $G_{ICpropagation}$ of their reference glass/epoxy laminate to be around 700 J/m^2. With thermoset epoxy film and polyimide thermoplastic film as interleaving materials, the $G_{ICpropagation}$ value increased to approximately 1000 and 1200 J/m^2 respectively. In a recent work, Williams *et al.*[24] studied the effects of different quantities of CNTs incorporated as interlayers between glass/epoxy prepreg plies. They found the $G_{ICpropagation}$ values to be 284 J/m^2 for control, increasing up to 414 J/m^2 with 1.2 gm/m^2 coating of CNT. Compared to various techniques, veil interleaving in composites seems to be a feasible approach with the possibility of translation to larger scale manufacturing.

To explain the load–deflection curves and G_{IC} values in Figures 6 and 7, we turn to images from the DCB tests, shown in Figure 8. One of the key mechanisms through which thermoplastic fibers in interleaving veils typically increase mode-I type fracture toughness of composites is through veil fibers pulling out of the matrix and bridging the crack as it propagates.[12,22] However, as seen from Figure 8, in our tests, bridging of veil fibers played a much less prominent role than is typical for interleaved laminates. To aid the discussion, schematics of the crack propagation in the three cases are shown in Figure 9. In the G specimens, Figures 8(a) and 9(a), the crack propagated an extended distance along the laminate mid-plane, before jumping to adjacent interfaces in the bottom half of the laminate, causing minor load drops each time this happened (see Figure 6). There is evidence of some *glass* fiber bridging in Figure 8(a), indicating the glass fiber/matrix interface was not very strong. In the GPETG specimens, Figures 8(b) and 9(b), the precrack was positioned in the middle of the central PET veil, but the crack immediately moved to the adjacent PET/glass interface and then propagated much as in the G case, with much the same load (Figure 6). Little or no PET fiber bridging can be seen, indicating that pull-out of PET fibers from the matrix was difficult because of strong PET/matrix bonding, as we had hoped would be the case, given the chemical similarity between the PET and the matrix. In the GPETPETG specimens, Figures 8(c) and 9(c), a more complex failure process is evident. The crack initially progressed along the laminate mid-plane, which was a PET–PET interface (left side of picture), and was bridged by PET fibers, which had pulled out of the resin. From Figure 6 we can see that the load was very high during this period, because of the strong resistance to PET fiber pull-out. Had the crack continued along the PET–PET interface, the resulting G_{IC} increase

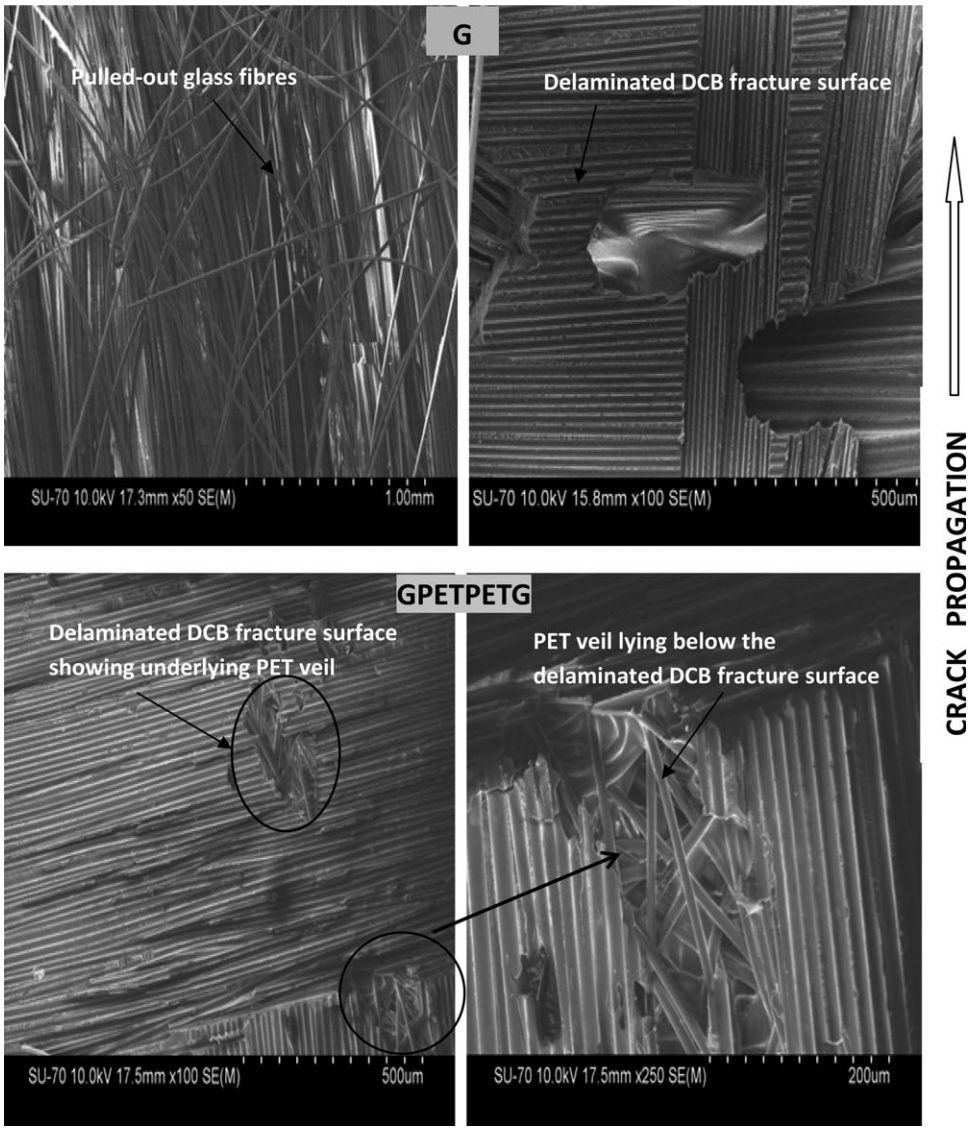

Figure 10. SEM micrographs of the DCB facture surfaces.

over the G laminates would have been much more dramatic than it was. However, a path of less resistance was found whereby the adjacent PET/glass interface in the upper half of the specimen debonded, and the crack then proceeded along that interface. This interface is similar to that along which the crack propagated in the GPETG case so, as would be expected, little or no PET fiber bridging occurred from this point on, and the load dropped dramatically to the level seen in the G and GPETG tests (Figure 6). Later in the test, the crack made further jumps to adjacent Glass/PET interfaces in the upper half of the laminate, and because these were of a similar nature, the resulting load drops were minor. Further corroborating evidence for this behavior is seen in SEM images of the DCB fracture surfaces, shown in Figure 10. The veils remained strongly bonded with the matrix, with very little pull-out of veil fibers, in contrast to the usual behavior of such interleaved composites.[22] Instead, debonding and pull out of the *glass* fibers from the matrix were evident in the form of loose fibers and fiber impressions on the fracture surfaces. The fact that, after the load dropped in the GPETPETG tests, the load–displacement curves were very similar in the G, GPETG and in GPETPETG laminates, supports the hypothesis that the crack propagation took place in all the laminates through the glass-resin interface rather than the veil-resin interface. If the bond between the PET and resin had been somewhat weaker, the crack in the GPETPETG laminate might have stayed on the PET–PET interface maintaining the load for longer, leading to a higher $G_{ICpropagation}$.

The role played by the interleaving PET veil at the interlaminar region was thus found to be significantly different than that observed in our previous work with polyamide veils.[22] Polyamide veils gave a larger increase in fracture toughness (up to 170%) than the PET veils used here, and displayed extensive fiber bridging. However polyamide veils had a significantly deleterious effect on flexural strength, whereas the PET veils used here led to an increase in flexural strength. The PET veils also

resulted in significantly better ILSS results than the polyamide veils in Ref. 22, while tan δ results were similar for both types of veils. While the PET veils used here gave a less dramatic fracture toughness increase than the polyamide veils in Ref. 22, their use led to better overall properties, taking all the various test results into account.

These results thus present the possibility of a choice of interleaving veils depending on the desired composite properties. Such interleaved glass fiber reinforced composites can find application as structural and semi-structural vehicle parts, in construction industries, in sports and consumer goods, where they can offer improved fracture toughness with enhanced crash performance.

CONCLUSIONS

The introduction of a fine, low areal weight thermoplastic veil into a glass fiber laminate can result in a significant increase in Mode-I type interlaminar fracture toughness. The wetting and bonding of the veil fibers with the resin is a key factor which controls the absorption of energy during the interlaminar crack propagation. The mechanism of toughening under Mode-I loading conditions is usually linked to fiber bridging effects that are enhanced by the introduction of the tough thermoplastic veils in the interlaminar regions. However, in this study, fiber bridging was not the dominant effect, and instead strong bonding between the PET fibers and the unsaturated polyester resin resisted crack propagation through the veils and forced the crack to deviate through weaker glass-resin interfaces in the adjacent layers. This effect was particularly significant when two PET veils were incorporated in the interlaminar regions. The incorporation of PET veils led to positive effects on the flexural strength, interlaminar shear strength, mode I type interlaminar fracture toughness (G_{IC}), and the damping parameter of the composites, while the flexural modulus was only slightly reduced. Overall, PET veils show promise as interleaving materials in FRP products to improve their fracture toughness without much sacrifice in their mechanical strength. For matrices like low styrene emission unsaturated polyester resin, which show relatively more brittleness, such interleaving technique can offer significant benefits.

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