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Effect of Chopped Fibers on the Mode I and Mode II Interlaminar Fracture Toughness of Glass Fiber Reinforced Composites

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Research Article	ABSTRACT
Keywords:	The separation of layers in laminated composites called delamination is the main mode of failure under transverse and shear loadings. Interlaminar fracture toughness is a material property that describes resistance to delamination. Increasing the interlaminar fracture toughness is one of the main motivations in the laminated composite field. In this regard, chopped glass fibers were interleaved between the layers of laminated composites to improve the interlaminar fracture toughness. The chopped glass fibers were processed to take the form of a tissue, which is a thin nonwoven mat material composed of randomly-oriented short glass fibers. This tissue is then placed between the layers. The vacuum infusion method was used for the manufacturing of composite samples. To measure the Mode-I and Mode-II interlaminar fracture toughness of the composite, a double cantilever beam (DCB) test, and an edge notch flexure (ENF) test were conducted, respectively. The addition of chopped glass fibers has enhanced the Mode I and Mode II fracture toughness by 64% and 27%, respectively. The influence of the chopped glass fiber on the crack path or fracture surface was investigated using scanning electron microscopy (SEM).
Polymer-matrix composites Delamination Interlaminar fracture toughness Chopped glass fibers	
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1. Introduction

Fiber-reinforced polymer (FRP) composites have been finding increasing application areas in the aerospace, automotive, civil, and marine sectors. Depending on the application area, FRP parts work under different loading conditions such as tension, compression, bending, twisting, or a combination of these loading conditions. FRP composites have different failure modes by nature. One of the failure modes is delamination more sensitive to bending and twisting loading conditions. The delamination failure mode is related to the interlaminar fracture toughness of the FRP composites. Different methods [1-3] such as fiber surface modification [4-6], Z-pinning [7, 8], stitching [9-14], 3D weaving [15], interleaving [16-42], matrix-toughening with micro/nanofillers [43-46] have been considered to improve the interlaminar fracture toughness of FRP composites. Z-pinning, stitching, and fiber surface modification techniques result in a reduction in the in-plane mechanical properties. Matrix-toughening in out-ofautoclave applications has problems related to high resin viscosity. Interleaving is a technique of introducing a new layer between plies of laminates. This new layer can be thermoset or thermoplastic films [18, 25-27, 35], electrospun nanofiber veils [17, 19, 21, 22, 36-38], a layer using spraying/sifting/spreading method [23, 24, 29, 34, 39], and dry or prepreg tissue of chopped fibers [16, 30].

Zhou et al. [18] used modified epoxy including short carbon fibers (SCFs) and carbon nanotube-grown short carbon fibers (CNT-SCFs) as an interleaf. An ethanol synthesis method was considered to grow carbon nanotubes on short carbon fibers. The length of the fibers was 0.75 mm. Significant improvement of 73% and 125% in Mode I interlaminar fracture toughness was gained by SCF and CNT-SCF modified epoxy as compared to the control laminates, respectively. It was also found that higher areal density of SCF and CNT-SCF decrease the GIC, which may leave defects between the interleaf and adjacent plies. Hojo et al. [27] considered carbon fiber/epoxy laminates with self-same epoxy interleaf to investigate the effect of resin-rich layer on Mode I, Mode II, and fatigue crack growth behavior. The thickness of the epoxy interleaf was 50 micrometers. It is observed that the resin-rich region did not have any effect on the Mode I properties of these epoxyinterleaved CFRP laminates. On the other hand, the Mode II interlaminar fracture toughness was 1.6 (initial value) and 3.4 (propagation value) times higher than that for the control laminates, which may be due to the fact that the damaged zone size for the epoxy-interleaved laminates is much larger than that for the control laminates.

Different material systems for nanofiber mats processed using the electrospinning method were considered in the literature such as PA 6.6 [19, 20], PCL [21, 36], PA 6.9 [17], thermoplastic polyurethane [22], PSF [38]. Daelemans et al. [17] investigated the effect of orientation of PA 6.9 nanofiber mats. Three different orientation configurations were used: nanofibers oriented parallel and transverse to the crack growth direction and a random deposition of nanofibers. Laminates

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interleaved with a random deposition gave the highest Mode II interlaminar fracture toughness due to effective nanofiber bridging. Beylergil et al. [19] manufactured PA 66 interleaved laminates using electrospinning and vacuum infusion technique to explore the effect of interleaving on the mechanical properties. Three-point bending, tensile, compression, interlaminar fracture toughness tests (Mode I and Mode II), and Charpy impact were conducted. According to the results, there was an improvement in the mechanical properties considered except for the tensile strength of the laminates. Nanofibers caused a decrease in the tensile strength of the laminates.

Spraying/sifting/spreading is another interleaving technique. Ning et al. [23] applied epoxy paste including carbon black (CB) as an interleaving material to the laminate. Mode I and Mode II fracture toughness values were considerably improved by applying 15 g/m2 CB/epoxy paste. The reason behind this improvement may be that CBs cause more irregular and rough fracture surfaces due to crack deflection and plastic deformation, which results in higher driving force and energy consumption. In addition, CBs may release the crack-tip stress triaxiality and promote matrix shear plasticity. Li et al. [24] sifted the powder of vapor-grown carbon fiber (VGCF) on the mid-plane of the stacked prepreg material for the reinforcement of the interface. Diffusion processes were triggered through temperature and high pressure in the autoclave. The conducted DCB test and bending test revealed that VGCF improves the interlaminar fracture toughness, bending modulus, and bending strength of the laminate. The observed zigzag patterns on the fractured surface lead the higher fracture toughness. Ning et al. [29] used surface-modified VGCF to improve Mode II interlaminar fracture toughness between Aluminum and glass fiber-reinforced composite material. ENF test results indicated that surface-modified VGCF improves the Mode II interlaminar fracture toughness by 425.16 %. A solvent spraying method was used by Rodríguez-González et al. [39] to apply multiwalled carbon nanotubes (MWCNTs) on the carbon/epoxy prepreg material to improve Mode I fracture toughness of the composite laminates. 52% improvement in the average GIC initiation was achieved for the laminates modified with oxidized MWCNTs compared to the base samples.

In this study, chopped glass fibers were interleaved between the layers of the glass fabrics to improve the Mode I and Mode II interlaminar fracture toughness of the laminates. The chopped glass fibers were processed into a non-woven glass tissue (NWGT) form. The prepared NWGT was laid on the mid-plane of the laminate. Double cantilever beam (DCB) and end-notched flexure (ENF) tests were conducted on the laminates manufactured with and without NWGT to see the effect of NWGT on interlaminar fracture toughness properties.

2. Experimental

2.1. Materials and Manufacturing

A biaxial E- glass fabric of 300 g/m2 (LT300 0/90 Metyx Composites, Turkiye) was used as a reinforcement material and an epoxy resin (MGSTM LR285) was used as a matrix material for the manufacturing of the composite laminates. Chopped glass fibers (SGFs) were prepared by cutting the same biaxial E-glass fabric.

The non-woven glass tissue (mat) was prepared by a combination of wet-lay and hand lay-up methods. The predominant method for turning glass fibers into nonwovens is a wet-lay method. Wet laying entails spreading the proper type and quantity of fiber in water, transferring it over a continuously moving fine mesh screen, and then producing a mat through filtration. The resulting mat or wet-laid sheet still includes 80% water and 20% fiber. [47]. The dispersion of fibers in water is a crucial step in the wet-lay nonwoven manufacturing process. In this technique, the fibers must be dispersed in water to produce a suspension, and they must float and remain evenly distributed in the suspension until the web is laid. It is commonly believed that the higher the dispersion of fibers in water, the higher the quality of the resulting webs. Certainly, fiber dispersion in water has a significant impact on the final quality of wet-laid nonwoven fabrics [48, 49]. A uniform dispersion may require the addition of surfactants and a viscosity modifier to water. For consistent fiber dispersion in the wet-lay process, the fiber length/diameter (L/D), fiber stiffness, type/amount of crimping, fiber wettability, and mechanical agitation of the mixture are critical criteria [47,49]. In this study, a dispersion agent was not used for the elimination of surface modification. Raw glass fibers were obtained from the Biaxial E- glass fabric. The woven glass fibers were manually chopped into short glass fibers around 6 mm in length. The chopped glass fibers of 12 g weight were dispersed in 1000 ml water. The dispersion was stirred by using an overhead stirrer for 6 hours. The dispersed fibers were allowed to settle down on a 150x300 mm filter paper by using the hand lay-up technique. The fibrous web dried in a hot air oven at 120°C for 15 minutes. The density of the prepared web was 2.7 g/cm2 and the average thickness of 0.927 ± 0.15 mm. The preparation procedure can be seen in Fig. 1.

The stacking sequence of the laminates was [0/90]6s. A Teflon film was inserted in the mid-plane of the laminate to form an initial crack. The processes of chopped fibers in a form of a tissue mat were laid on the mid-plane beside the Teflon film, as shown in Fig. 2(a). Vacuum-assisted resin transfer molding method was used to manufacture the laminates. The laminates with chopped fibers and without chopped fibers were manufactured in the same batch, as indicated in Fig 2(b). The laminates manufactured were then cut using a water-cooled diamond disk to obtain samples of ENF and DCB tests. The dimensions of the test samples were determined according to the standards of ASTM D5528 and ASTM D7905 for DCB test and ENF test, respectively.

2.2. DCB and ENF Test Procedure

To investigate the Mode-I and Mode-II interlaminar fracture toughness, DCB and ENF tests were carried out using a tensile testing machine with a 1 kN load cell. Tests were conducted under constant 1 mm/min cross-head speed in tension for the DCB tests and in compression for the ENF tests.

Three samples were tested for each type of laminate for DCB tests but one of the samples from each batch failed from the aluminum tab. The dimensions of the samples and the test set-up for the DCB test are shown in Fig. 3 (a) and (b), respectively. The initial delamination length (a_o) was 50 mm. According to ASTM D5528, an initial loading was applied to



Fig. 1 Preparation of non-woven glass tissue (mat)



Fig. 2 (a) The placement of the chopped fibers, and (b) vacuum assisted resin transfer molding

samples before the actual test to create a virgin crack front. Thus, the initial loading was stopped after an increment of delamination crack growth of 3 mm. After crack growth of 3 mm, specimens were unloaded at a crosshead rate of 25 mm/min. A new pre-crack length (ao + 3 mm) was recorded after unloading. For the actual tests, samples were reloaded at a crosshead speed of 1 mm/min until the crack propagated about 30 mm from the crack tip. During this operation, load, opening displacement, and crack length were recorded. To observe crack propagation and obtain the R curve a camera

was used. The mark lines on the DCB samples were traced every three seconds through images recorded using the camera.

To calculate the values for critical Mode-I interlaminar fracture toughness (G_Ic) at pre-crack and during propagation, the Modified Beam Theory (MBT) data reduction method as stated in ASTM D5528 was considered. The equation to calculate the G_Ic is given in Eq.1.

$$G_{lc} = \frac{3P\delta}{2b(a+|\Delta|)} \tag{1}$$

where *P* is the applied load, δ is the load point displacement, b is the specimen width, a is the delamination length, Δ is a value that is determined experimentally by generating a least squares plot of the cube root of compliance (C^{1/3}) as a function of delamination length. The compliance, C, is the ratio of the load point displacement to the load applied.

Six samples were tested for each type of laminate for ENF. The dimensions of the samples are schematically shown in Fig. 4 (a) and (b). In the ENF tests, the compressive load was applied from the top roller. The loading rollers uniformly contacted the samples across their width.



Fig. 3 (a) Schematic representation of a DCB sample, and (b) test set-up





Fig. 4 Schematic representation of a ENF sample (a), and test set-up of the test (b).

The Mode-II interlaminar fracture toughness was obtained using the compliance calibration method. Prior to the actual ENF tests, the compliances from two different crack lengths (a=20 mm and a=40 mm) were obtained through the appropriate placement of the samples in the fixture using mark lines. The G_IIc is calculated using Eq (2).



$$G_{IIc} = \frac{3mP_{max}^2 a_o^2}{2B} \tag{2}$$

where m is the compliance calibration coefficient, P_{max} is the maximum force from the ENF test a_o is the crack length used in the fracture test (30 mm), and B is the specimen width.

3. Results and Discussion

3.1. Mode-I Interlaminar Fracture Toughness

Fig. 5 (a) shows load versus displacement curves obtained for the laminates with and without chopped fibers. Laminates with chopped interleaving tissue exhibited higher interlaminar failure loads. Mode-I interlaminar fracture toughness was characterized by a resistance behavior called R-curve that includes an initiation value and a region of propagation as the crack extend until a plateau region. R-curves for the control and chopped reinforced laminates are shown in Fig.5 (b). The Mode-I initiation interlaminar fracture toughness increased from 0.75 kJ/m2 for the control samples up to 1.23 kJ/m2 for the control samples, the increase in fracture toughness was 64%, as shown in Fig.6.

To understand the toughening mechanism, images were captured using a scanning electron microscope (SEM). The fracture morphology of the control and the chopped fiber interleaved laminates was shown in Fig.7 (a) and (b), respectively. The control laminates display characteristic brittle fractures of the epoxy material (see red arrows) between glass fibers (see yellow arrows). The cracks propagated preferentially along the interface between glass fibers and the epoxy material, yielding lower fracture toughness values. On the other hand, this smooth spread of crack was disturbed by randomly distributed chopped fibers through fiber bridging, and short fiber cracking, as shown in Fig. 7 (b). Also, crack propagated inside the interleaf interface as shown in Fig. 7 (b). Different orientations of chopped fibers in the interleaf can be seen in the figure. Deviations in the crack orientation were one of the



Fig. 5 (a) Load-displacement curves of the control and modified laminate DCB samples, and (b) R-curves for the control and modified laminate



Fig. 6 Mode-I interlaminar fracture toughness comparison.

factors that responsible for the higher interlaminar fracture toughness.

3.2. Mode-II Interlaminar Fracture Toughness

Crack growth is not stable in the ENF test so the initiation value of the Mode-II interlaminar fracture toughness was obtained from the preimplanted Teflon insert. Fig.8 shows load-displacement curves obtained from ENF tests. The laminates with chopped fiber interleaf gave higher load values as compared to the control samples. Mode-II interlaminar fracture toughness values are given in Fig. 9. Introducing chopped fiber interleaf to the laminates increases Mode-II fracture toughness value by 27%. The average value of Mode-II fracture toughness was 2.85 kJ/m² and 3.63 kJ/m² for the control and the chopped fiber interleaved laminates, respectively.



Fig. 7. SEM images captured from the control sample (a), and the chopped fiber interleaved laminates (b).





Fig 8. ENF test results for the control samples and the chopped fiber interleaved samples.

Fig 8. ENF test results for the control samples and the chopped Fig 9. Mode-II interlaminar fracture toughness comparison.

4. Conclusions

Chopped glass fibers were interleaved between the layers of the laminated composite to improve the interlaminar fracture toughness. The non-woven glass tissue (mat) was prepared by a combination of wet-lay and hand lay-up methods. This tissue was then laid down on the mid-layer. To manufacture the laminate samples, the vacuum infusion method was used. Mode I and Mode II interlaminar fracture toughness of the composite were obtained using a DCB test, and an ENF test, respectively. The addition of chopped glass fibers has enhanced the Mode I and Mode II fracture toughness by 64% and 27%, respectively. The influence of the chopped glass fiber on the crack path or fracture surface was investigated using scanning electron microscopy (SEM). Deviations in the crack orientation and fiber bridging were the factors responsible for the higher interlaminar fracture toughness. The effect of the thickness of the interleaf tissue and the chopped fiber length on the interlaminar fracture toughness might be investigated in a future work.

Declaration

Author Contribution: Conceive-K.C.; Design- K.C., S.S.; Supervision- K.C.; Experimental Performance, Data Collection and/or Processing- K.C., S.S, K.U.; Analysis and/or Interpretation- K.C., K.U.; Literature Review- K.C., S.S.; Writer- K.C., S.S.; Critical Reviews- K.C., S.S.

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