Crack suppression by natural fiber integration for improved interlaminar fracture toughness in fiber hybrid composites

Anant Joshi
Department of Mechanical Engineering, KLS VDIT, Halital, Research Center, Department of Mechanical Engineering SDMCET, Dharwad, Visvesvaraya Technological University, Karnataka, India.
anantjoshi85@gmail.com

P S Shivakumar Gouda*
Research Center, Department of Mechanical Engineering, SDM College of Engineering & Technology, Dharwad, Visvesvaraya Technological University, Karnataka, India.
ursshivu@gmail.com

I. Sridhar, Umarfarooq M A, Vinayak Uppin
Research Center, Department of Mechanical Engineering, SDM College of Engineering & Technology, Dharwad, Visvesvaraya Technological University, Karnataka, India.
sridhan74@gmail.com, umarfarooq.ma@gmail.com, vinayakuppin@gmail.com

Jyoti Vastrad
Department of Textile and Apparel Designing, Community College, University of Agricultural Science, Dharwad, Karnataka, India
jyotivastrad@gmail.com

Nabaneeta Gogoi
AICRP on WIA, Assam Agricultural University, Jorhat, India.
nabaneta@gogoi@gmail.com

Abhilash Edacherian
Department of Mechanical Engineering, College of Engineering, King Khalid University, Saudi Arabia
edalheriad@kku.edu.sa

ABSTRACT. In this paper, the effect of integration of natural fibers in UD carbon fiber is studied. The integration of natural fibers in carbon fiber is made via intra fiber hybridization. Natural fiber hybrid composite samples were prepared for Mode I and Mode II fracture tests. XRD analysis was done for the chosen natural fibers to know the crystallinity index and then compared with Carbon and Glass fibers. The fracture test experimental results, revealed that the effect of Jute fiber integration in UD Carbon epoxy...
A. Joshi et alii, Frattura ed Integrità Strutturale, 60 (2022) 158-173; DOI: 10.3221/IGF-ESIS.60.12

A composite was found significant in getting relatively good Mode I and II fracture toughness at the crack initiation without losing its stiffness. In addition to this Kenaf Carbon epoxy composite indicated better crack suppression with 30% higher propagation toughness values as compared to other hybrid combinations and pristine composites. It is observed that integration of jute fibers in UD carbon epoxy composites was significant in achieving good mode I and mode II fracture toughness at the crack initiation without losing its stiffness and also kenaf carbon epoxy composites indicated better crack suppression with 30% higher propagation toughness as compared to other hybrid combinations used.

**KEYWORDS.** Intra-fiber hybridization; Fracture toughness; Crack suppression, Natural fibers.

INTRODUCTION

The environmental and sustainability consciousness has enhanced the incorporation of green technology in the field of the composite science to look for new sustainable hybrid FRP materials. The ever-increasing environmental concerns and protocols for cleaner processes have motivated the scientific research towards new class of eco-friendly hybrid materials. Composite materials are the most inventive engineering materials which are used in variety of industrial applications. In contrast, with conventional isotropic materials like monolithic metals and alloys, the load bearing ability of fiber reinforced composites can be augmented for required application by embedding or aligning the fibers in a proper manner with suitable matrices/bonding materials. By using two or more types of fibers within the same matrix can give tailored properties and this technique is commonly known as Fiber hybridization. The hybridization can be achieved by using combination of synthetic and natural fibers namely, Glass, Carbon, Aramid, Jute, Flax, and Kenaf etc. The need for such hybrid materials is increasing as modern-day composite materials are required to attain multiple and desirable properties like stiffness, strength, and toughness etc. Furthermore, applications like automotive and aerospace the requirement of strength, stiffness and impact resistance are significant. Along with these desirable properties, light weight, low cost, and sustainability are extremely important to compete in the market. This will also promote circular economy in composite manufacturing sectors. Specifically in laminated composite materials, delamination is one of the significant failure modes in service, due to which the performance of composite material drops to a greater extent. Therefore, altering the interlaminar fracture toughness of high-performance composites, particularly for composites with brittle matrices, becomes an important task in order to use these composites in primary and secondary load bearing structures. A novel pre-preg coating method was used to improve the interlaminar fracture toughness in carbon fiber epoxy composite laminates, using reactive liquid rubber. Epoxy Terminated Butadiene Nitrile (ETBN) liquid rubber were incorporated between Glass and Carbon fiber pre-preg interfaces using automatic draw bar coating technique to achieve improvement in interlaminar critical energy release rates ($G_{IC}$ and $G_{IIC}$) by 140% in mode-I and 32% in mode-II loadings respectively [1, 2]. Additionally, various methods like toughened resins, interleaving [3], fiber surface treatment, and through-thickness reinforcement (e.g., 3D weaving, stitching, z-anchoring and tufting) have been identified to improve delamination resistance of composite materials [4]. Natural and synthetic fibers are gradually being used as reinforcements in many applications. Synthetic fibers are known for their mechanical properties, whereas natural fibers are environment friendly, low cost and possess good vibro-acoustic properties [5]. Many industries are investing in green and sustainable technologies, hence natural fibers have been gaining lot of consideration and are being used in applications like car interior, sporting equipment, etc. To date, their applications have been restricted to primary structural components of automotive and aerospace due to low tensile modulus of natural fibers. Furthermore, it was reported [6] that interlaminar fracture toughness has improved by 67% in mode I and more than 40% in mode II through hybridization of Carbon fibers with ultra-high molecular weight Dyneema fibers. Additionally, interlaminar shear strength and interlaminar fracture toughness of twisted Flax fibers with Glass fiber reinforced hybrid composites were higher than those of Glass Fiber composites [7]. In addition to this optimum Cellulose coating on Glass fiber with surface density of 320 g/m² has contributed a good compromise to get improved fracture toughness for mode I and for mode II loadings [8]. Improvement in curved beam strength (CBS) and interlaminar radial stress of a UD Glass
epoxy L-bend composite laminate by incorporating Kenaf in the range of 5–10 wt% and GO in the range of 1–2 wt% were loaded at each ply at the curvature of a L-bend [9]. The mode-I interlaminar fracture toughness of Flax, Glass hybrid fiber woven composites was studied by using a DCB test. Thus, the hybridization gives better interlaminar fracture toughness, $G_{IC}$, during crack propagation despite an initial value of $G_{IC}$ close to that of Glass Fiber Reinforced Epoxy (GFRE) but lower than the Flax Fiber Reinforced Epoxy (FFRE) composites [10]. The incorporation of Cotton fibers in the range of 10 to 25% with Glass fibers exhibited 43% improvement in Mode I fracture toughness as compared with pristine Glass fiber composite. Similarly, Mode II fracture toughness enhanced up to 59.8% with the addition of Cotton fibers in the range of 10 to 25% [11]. The interlaminar fracture toughness under mode I loading increased by 32% in hybrid composite with Cork and UD Carbon–epoxy in comparison with Carbon-epoxy composite. The mode II toughness is of the same order of the Carbon-Epoxy laminates, although it was observed that the insertion of a Cork layer leads to a stable crack growth, even for quite unstable configurations of the ENF test [12, 13].

By knowing the fiber hybridization effects on interlaminar fracture toughness through various techniques adopted by different investigators as discussed in the above section an attempt was made in the present study on the influence of Intra-fiber hybridized architecture for suppressing the crack growth by evaluating interlaminar fracture toughness in hybrid fiber composites. Further, a crystallinity of the natural fibers was evaluated by X-Ray Diffraction (XRD) test. SEM examination was also done to know the failure mechanisms at the fiber and matrix interface in composites.

**EXPERIMENTATIONS**

**Materials**

Standard grade unidirectional (UD) Carbon fiber and Glass fiber was supplied by Mark Tech Composites Pvt. Ltd, Bangalore, India. The epoxy (Lapox L-12) and polyamine hardener (K-6) was supplied by Atul Industries Ltd, Gujrat, India. Kenaf, Jute and Hibiscus. Natural fibers were taken from the University of Agricultural Sciences, Dharwad, India and processed with 3% NaOH treatment to achieve good surface functionalities and to attain proper interfacial bonding between the fibers and matrix [20].

![Figure 1: The schematic of hybrid plies consisting of (a) Carbon-Glass (b) Carbon-Kenaf (c) Carbon-Hibiscus and (d) Carbon-Jute Fibers.](image-url)
Intra-Fiber Hybridization of Natural fibers

Prior to intra-fiber hybridization, UD Carbon strands of known quantity (25wt%) were taken out from woven UD Carbon ply. Then, same quantity of UD Glass/Kenaf/Jute/and Hibiscus fiber strands were inserted manually between UD Carbon strands to make hybrid plies. The schematic of intra-fiber hybridized configurations is shown Fig 1(a) to (d).

Construction of Hybrid Composite Panels

The two hybrid plies comprised of Carbon-Glass (CG), Carbon-Kenaf (CK), Carbon-Hibiscus (CH) and Carbon-Jute (CJ) were coated with measured quantity of epoxy and then placed at the mid-section. Above and below the hybrid plies an equal number of UD Carbon plies of same size were placed to form a required thickness of hybrid composite panels. A separation film of 14-micron thickness is introduced at the mid plane between two chosen hybrid plies to form pre-crack for Mode-I and Mode-II fracture tests. The schematic of stacking sequence with hybrid plies and separation film is shown in Fig.2. Finally, the stacked plies of UD Carbon epoxy and hybridization of Carbon-Glass-Epoxy (CGE), Carbon-Kenaf-Epoxy (CKE), Carbon-Hibiscus-Epoxy (CHE) and Carbon-Jute-Epoxy (CJE) were pressed by keeping dead weight and allowed to cure under room temperature for an about 24hours. The cured hybrid panels were cut in accordance with ASTM D 5528 standard for Mode I loading and ASTM D 7905 standard for Mode II loading. Five samples were prepared and tested for each configuration. The details of the laminate codes and their compositions are shown in Tab.1.

Figure 2: Schematic stacking sequence and hybrid plies with separation film

| Laminate Codes           | UD Carbon (wt%) | UD Glass (wt%) | Kenaf (wt%) | Jute (wt%) | Hibiscus (wt%) | Epoxy (wt%) |
|-------------------------|-----------------|----------------|-------------|------------|----------------|-------------|
| Carbon-Epoxy (CE)       | 50              | --             | --          | --         | --             | 50          |
| Glass-Epoxy (GE)        | --              | 50             | --          | --         | --             | 50          |
| Carbon-Glass-Epoxy (CGE)| 25              | 25             | --          | --         | --             | 50          |
| Carbon-Jute-Epoxy (CJE) | 25              | --             | 25          | --         | 25             | --          |
| Carbon-Hibiscus-Epoxy (CHE) | 25           | --             | --          | 25         | 25             | --          |
| Carbon-Kenaf-Epoxy (CJE)| 25              | 25             | --          | --         | --             | 50          |

Table 1: Hybrid laminate codes and their fiber compositions

TEST METHODS

X-Ray Diffraction (XRD)

X-ray powder diffraction (XRD) is a quick analytical technique mainly used for identification of phase of a crystalline material. This also gives an information on unit cell dimensions. The material which is to be tested is finely milled, homogenized. This test will give average bulk composition of the sample. XRD technique employs the X-ray
scattering phenomenon to clarify the crystal structure of crystalline/semicrystalline materials with scattering of X-rays by intermittent array of atoms give rise to definite diffraction patterns that gives a qualitative image of atomic arrangements within the crystal lattice. Powder X-Ray Diffraction (P-XRD) is one such characterization tool that offers the advantage of simultaneous characterization of both the precursor and end products with a comprehensive qualitative presentation of their micro-structural behaviours.

**Mode-I Test**
Double Cantilever Beam (DCB) specimens for Mode I test were prepared in accordance with ASTM D5528 [14]. The method of using separation film as pre-crack and it is found to be very effective as it presents the sound conditions of real crack [15]. The dimensions of DCB sample are shown in Fig. 3. The calibrated uniaxial testing machine of 10kN capacity with cross-head displacement speed of 2mm/min was set during Mode I test. Replicates of five DCB samples were tested for each combination of hybrid composites. While carrying out DCB tests crack growth was recorded using a high-definition SONY digital video camera with resolution of ±0.5mm. The modified beam theory (MBT) is used to determine the critical strain energy release rate \( (G_{IC}) \) as per ASTM standard using Eqn. (1).

\[
G_{IC} = \frac{3P\delta}{2B(a+|\Delta|)} 
\]

where ‘P’ is load, ‘\(\delta\)’ is load point displacement, ‘B’ is specimen width, ‘a’ is delamination length, ‘\(\Delta\)’ is correction factor and is determined experimentally with the help of generating a least square plot of the cube root of compliance \( (C^{1/3}) \) as a function of delamination length.

**Figure 3: DCB specimen geometry**

**Mode-II Test**
Mode II interlaminar fracture toughness \( (G_{IIC}) \) were determined in accordance with ASTM D 7905 [16] using three-point end notched flexure (ENF) specimen. Tests were accomplished using Tinius Olsen UTM of 10 kN capacity with cross head speed of 1 mm/min. The configuration of the specimen used for test is shown in Fig.4. The span length of all the specimens while testing was maintained 100 mm as per ASTM D 7905. \( G_{IIC} \) values were determined for non-precracked specimens based on compliance calibration method as per the procedure given in ASTM D 7905. The relationship between the beam compliance and the crack length is given by Eqn. (2) as

\[
C = A + ma^3 
\]

where A and m are the Crack Compliance coefficients, C – Compliance of beam, a – Crack length

\( G_{IIC} \) can be determined using Eqn. (3) as follows:

\[
G_{IIC} = \frac{3mP^2a_0^2}{2h} 
\]
where \( a_0 \) – Delamination length for ENF test, \( P_{\text{max}} \) – Maximum load value in a Load- displacement curve, \( b \) - Width of the specimen

Prior to ENF test, compliance of the specimen is determined experimentally for three different crack lengths by shifting the specimen horizontally either to left or right. These experimentally determined Compliance from different crack lengths were utilized to determine the compliance coefficients ‘A’ and ‘m’ by befitting a least square linear equation between \( C \) and \( a^3 \). ENF tests for compliance were carried out at crosshead speed of 1 mm/min and the compliances for three crack lengths are determined from Load-displacement curves.

As per the procedure given in [17], three vertical lines are marked at distance of 10 mm, 20 mm and 30 mm from one end of the pre-crack and named as B, C and A respectively as shown in Fig. 5 (a). These three lines B, C and A are used as central loading points, which corresponds to crack length of 40 mm, 30 mm and 20 mm respectively. Initially the specimen is positioned in 3 point bending fixture with ‘A’ as loading point with crack length of 20 mm as shown in Fig. 5(b). Then specimen is gradually loaded and corresponding load-displacement curve is recorded until load reaches to predetermined values of 90 N [16]. Similar procedure is followed at points ‘B’ and ‘C’. Specimen loaded at location ‘B’ and ‘C’ with corresponding crack lengths are shown in Fig. 5(c) and Fig. 5(d) respectively.

Finally, the specimen is repositioned with crack length equal to 25 mm which is shown in Fig. 5(e), it is steadily loaded and corresponding load-displacement plots were recorded. The testing is continued until the pre-crack grows and a sudden drop in load is observed. The maximum load obtained from this test is utilized to determine the Mode II fracture toughness using Eqn. (3).

![Figure 4: Major dimensions and loading of three-point ENF Specimen](image)

![Figure 5: Compliance calculation using three different crack lengths (a) ENF specimen marked with vertical lines (A, B and C) (b) ENF specimen with loading at Point A (c) ENF specimen with loading at Point B (d) ENF specimen with loading at Point C (e) ENF specimen loading configuration for determination of Mode II fracture toughness.](image)
Fracture Morphology

The delaminated fracture surface of composites at the fiber matrix interface were analyzed using Zeiss GeminiSEM-300 operated at 30 keV. The composites were coated with gold using sputter coating machine and samples were cut to a size of 10mm x 10mm in accordance with vacuum chamber of the scanning electron microscope (SEM) and fixed on a stand with special arrangements using two-sided adhesive tape.

RESULTS AND DISCUSSIONS

Summarized results of XRD, Mode-I, Mode-II and SEM investigations of Intra-fiber hybridized natural fiber composites are discussed with proper justifications under the following sections.

X-Ray Diffraction (XRD)

NaOH treated Kenaf, Jute and Hibiscus Natural fibers were studied for their physical structure using advanced diffractometer with Cu-Kβ radiation. Fibers were powdered to make them usable in XRD stage and measurement was recorded in the range of 10-100° at 40kV and 30 mA with scan speed of 10°/min. Natural fibers mainly consists of cellulose, lignin and hemi-cellulose. Cellulose exhibit crystalline structure whereas lignin and hemi-cellulose exhibits amorphous structure. From the XRD data which is shown in Fig.6 a major crystalline peak were observed at 22.5° and amorphous peaks was also seen for all the natural fibers in the range of 15.8° to 16.5°. The synthetic fiber composite materials viz. pristine glass and carbon fiber composites have inherent amorphous nature as the glass and carbon fibers does not exhibit any sharp peaks.

![Figure 6: XRD curves for (a) Kenaf (b) Hibiscus and (c) Jute fibers](image)

The XRD curves indicated that the cellulose content of Natural fiber reinforced hybrid composites exhibited crystalline nature but lignin and hemi-cellulose content exhibited amorphous nature. The percentage crystallinity and crystallinity index can be determined by using the relations (4) and (5).
Percentage Crystallinity = \( \frac{I_{2\theta}}{I_{2\theta} + I_{\text{am}}} \) (4) 

Crystallinity Index = \( \frac{I_{2\theta} - I_{\text{am}}}{I_{2\theta}} \) (5)

where \( I_{2\theta} \) represents major diffraction intensity at \( 2\theta \) and \( I_{\text{am}} \) represents amorphous diffraction intensity at \( 2\theta \). The calculated Crystalline Index and Crystalline Percentage for chosen Natural fibers were listed in Tab. 2. The percentage Crystallinity of all the Natural fibers represented in Tab. 2 are higher with respect to Carbon and Glass fibers [18,19]. Thus, the natural fibers have greater strength, higher elongation and water intake with specific area which is available within the fiber surface for chemical reactions.

|             | Maximum Intensity at I002 | Angle (2θ) at I002 | Maximum Intensity at Iam | Angle (2θ) at Iam | Crystallinity Index (CI) | Percentage Crystallinity (%) |
|-------------|---------------------------|-------------------|--------------------------|-----------------|--------------------------|-----------------------------|
| Kenaf       | 4680                      | 22.5              | 2200                     | 15.8            | 0.52                     | 68.02                       |
| Hibiscus    | 4490                      | 22.4              | 2400                     | 15.5            | 0.46                     | 65.16                       |
| Jute        | 4600                      | 21.5              | 2410                     | 16.8            | 0.47                     | 65.62                       |
| Glass [18]  | 5900                      | 20.0              | 3800                     | 15.0            | 0.35                     | 60.82                       |
| Carbon [19] | 4400                      | 28.2              | 3200                     | 26.1            | 0.27                     | 57.89                       |

Table 2: Crystallinity index and Percentage Crystallinity of Kenaf, Hibiscus, Jute, Glass and Carbon fibers

**Mode I Interlaminar Fracture Toughness**

The load versus displacement curves for Double Cantilever Beam (DCB) specimens with hybrid combinations of Carbon-Glass, Carbon-Kenaf, Carbon-Hibiscus and Carbon-Jute and Pristine Glass and Carbon Epoxy composites are plotted in Fig. 7.

![Figure 7: Load versus Displacement Curves for Mode I test](image)

The plain Carbon-Epoxy (CE) composite has highest load bearing capacity with respect to Glass-Epoxy (GE) composite and other hybrid combinations. However, CGE hybrid composite performed significantly lower than GE, but gave slightly higher stiffness than GE. The Natural Fiber Hybrid Composite (NFHCs) comprising of Carbon-Jute (CJE) and Carbon-
Kenaf (CKE) have exhibited relatively lower load bearing capacity with respect to CE composites. Nevertheless, CJE has maintained same stiffness as that of CE, but CHE has significantly reduced its stiffness as compared to CE, this is witnessed by looking at the slope of load extension curves in Fig.7. Further, CHE and GE composites showed similar trend with respect to their load capacity and stiffness behaviour.

Fig.8 demonstrates the delamination resistance curves (R-Curves) for all the combination of hybrid and pristine composites. R-Curves were constructed using mode-I experimental test data wherein the recorded crack extension at initiation (for every 1mm up to 5mm) and propagation (for every 5mm up to 60mm) with respect to recorded time in video camera was taken by matching the test machine time for corresponding load and displacement values. Then \( G_{IC} \) values were calculated and plotted R-Curves (Fig 8). As per the Fig 8, the CKE hybrid composite exhibit a large increase in fracture toughness as the crack propagates which can be attributed to relatively higher fiber bridging seen in Fig.9d. However, CE has got higher toughness at the crack initiation than GE and other NFHCs (CGE, CHE, CKE & CJE). But, NFHCs have performed relatively better in their toughness values during crack propagation.
Figure 10: Mode I Interlaminar Fracture Toughness (a) $G_{IC}$, (b) $G_{IP}$ values and (c) Schematic of Fiber bridging mechanism at the crack initiation region.

The amount of fiber bridging is also one of the observations during crack propagation in achieving improved fracture toughness in Hybrid composites (Fig. 9). It is observed that CGE and CHE composites exhibited low fiber bridging (in Fig 9 (a) & (b)) whereas CJE has indicating moderate fiber bridging (Fig 9 (c)). However, it was realized that the amount of fiber bridging was higher in case of CKE composite. Hence, enhanced Mode-I propagation fracture toughness is observed in actual test results.

The summarized initiation and propagation fracture toughness values ($G_{IC}$ & $G_{IP}$) are plotted in Fig 10 (a) and (b). These toughness values are in good agreement with the amount of fiber bridging which is seen in Fig 9(a) to (d) during crack propagation. Similarly, the bridging of fibers at the crack initiation is shown in schematic Fig 10(c). In Fig 10, it is observed that pristine GE and CE composites have exhibited higher toughness values during crack initiation and CKE has exhibited significantly higher values during crack propagation, this enhancement in toughness values are in line with the earlier investigations presented in [21-23] which is based on intra-fiber hybridization of UD Flax, Glass and Carbon fibers for making composites. In addition to this, the toughness values (Fig. 10a) are similar to previous finding [24] on the influence of different fiber architectures for improving mode-I interlaminar fracture toughness in flax epoxy composites.

Mode II Interlaminar Fracture Toughness
The load versus displacement curves of Mode II ENF tests for all the hybrid composites along with Pristine Glass epoxy and Carbon epoxy composites are plotted in Fig.11.

The pristine CE and GE composites exhibited higher load bearing capacities among all the composites presented in Fig 11. However, it is surprise to see from slope of the curves that the stiffness remains same in CE, GE, CJE and CKE composites under Mode II loading. Further, CJE and CKE Hybrid composites have found reasonably good toughness when compared to CE and GE composites. In case of CGE and CHE it was seen that there was a significantly reduced stiffness with respect
to other composites chosen for this study. This reduced stiffness might be one of the reasons for integrating higher elongation fibers (Glass and Hibiscus) in UD Carbon epoxy composites. The hybrid epoxy composites comprised of Carbon-Jute (CJE) and Carbon-Kenaf (CKE) performed higher load bearing capacity which is almost equivalent to CE and GE epoxy composites. The Fig.12. shows Mode II interlaminar fracture toughness values with respect to chosen composites for this study. It is evident that CE and GE composites have almost similar mode II fracture toughness values, whereas CKE hybrid composite has exhibited 25% higher toughness than CE and GE composites. It is also seen that CJE and CGE exhibited relatively higher values of interlaminar fracture toughness as compared to CE and GE composites. Finally, it was concluded that by reinforcing Kenaf fibers with UD carbon fibers through Intra-fiber hybridization technique will give better Mode-II interlaminar fracture toughness. Hence, the Natural fibers can pose tough competition in partial replacement of synthetic fibers in developing high performance Hybrid composites for damage tolerant design applications.

Figure 11: Load versus Displacement Curves for Mode II Loading

Figure 12: Mode II Interlaminar Fracture Toughness (G_{IC}) values
Fractography

Fractographic analysis of fractured surfaces of NFHCs were done to know the two-phase morphology promising with regular toughening mechanisms such as fiber pull-out, debonding and plastic deformation of matrix around the fibers using SEM examination. The SEM images shown in Fig.13 characterizes the failure behaviour of CGE hybrid composite under Mode-I and Mode-II loadings. The propagation fracture toughness values evaluated for CGE Hybrid composites were moderately higher than that of the CE and GE composites. The presence of UD Glass fibers in UD Carbon fibers at the crack interfacial region showed better fiber matrix bonding with low fiber breakage (Fig. 13b) requiring additional energy to debond the fibers from matrix has resulted an improved toughness of the CGE hybrid composite leading to relatively higher propagation values. But, the initiation toughness values of CGE were significantly low with respect to CE and GE composite due to large number of delaminated fibers from the matrix which is seen in Fig. 13a.

Figure 13: SEM images showing fractured surface of Carbon - Glass Fiber Hybrid Composites (CGE) in Mode I Loading (a) Crack Initiation, (b) Crack Propagation and (c) Fractured surface under Mode-II loading.

SEM image in Fig.13c represents the fractured surface of CGE hybrid composites under Mode II loading. The estimated fracture toughness values were significantly higher in CGE hybrid composite than that of CE and GE composite. The presence of micro-buckling/fiber kinking due to interfacial surface traction under mode II loading might be one of the reasons for enhanced Mode II toughness values in CGE hybrid composite as compared to CE and GE.

The Fig.14 (a) to (c) represents SEM images of fractured surface of CJE hybrid composites under Mode-I and Mode-II loading. The absence of fiber imprints was seen during crack initiation which helps for ease of fiber delamination (Fig 14a) from the matrix. Hence, lower $G_{IC}$ values were observed than that of other hybrid composites chosen in this study during actual Mode-I fracture test. In addition to this, huge number of fibers gets separated from the matrix with some fiber breakage which is seen (Fig.14b) during crack propagation, hence lower $G_{IP}$ values. From Fig 14(c), it is clear that, the presence of micro-buckling or fiber kinking during Mode-II test leads to relatively better $G_{IIC}$ values when compared to CE and GE composites. But, $G_{IIC}$ values in CJE composite is almost very close to other hybrid combination chosen in the present investigations.
Figure 14: SEM Images showing fractured surface of Carbon-Jute Hybrid Composites (CJE) under Mode-I loading a) At Crack Initiation b) At Crack Propagation and (c) Mode-II fractured surface.

Figure 15: SEM Images of Carbon-Kenaf (CKE) Hybrid Composites during (a) Crack Initiation (b) Crack Propagation under Mode-I load and (c) Mode-II fractured surface.
Fig. 15 (a) to (c) displayed SEM images of CKE hybrid composite fractured under Mode-I and Mode-II loadings. It is evident from Fig. 15 (a) that there was a huge fiber separation with no fiber imprints during fiber delamination from the matrix leads to lower $G_{IC}$ as compared to CE and GE composites. But, during crack propagation it is observed in Fig.15b that there exists huge plastic deformation of the matrix between Kenaf and Carbon fibers with low fiber breakage leads to significant enhancement of $G_{IP}$ values as compared to all other hybrid composites including CE and GE composites. Furthermore, the presence of fiber kinking and with low fiber debonding and shear hackles is seen in Fig.15c, which leads to significant enhancement of $G_{IC}$ values in CKE hybrid composite than that of other hybrid and pristine CE and GE composites.

Fig. 16 (a) and (b) shows fractured surface SEM image of CHE hybrid composite under Mode I loading. Specifically, it is seen in Fig.16 (a) that there is a clear fiber delamination with low fiber bridging during crack initiation leads to reduced $G_{IC}$ values with respect to other composites. But, during propagation the CHE hybrid composite has shown slightly better $G_{IP}$ values than that of $G_{IC}$ at crack initiation zone. This is due to presence of better fiber imprints and good interfacial bonding between fibers which is observed in Fig.16 (b). Nevertheless, the CHE Hybrid composite fractured under Mode-II loading has exhibited low quantity of fiber breakage (Fig.16c), indicating almost similar $G_{IC}$ values when compared to CE and GE composites, but the performance of CHE with respect to other hybrid composites was found relatively low and this was true from our experimental results.

CONCLUSIONS

The interfacial fracture toughness of selected NFHCs was improved by the incorporation of various natural fibers in UD carbon fibers. Hybrid plies were prepared successfully through intra-fiber hybridization technique. Then, the hybrid composites CGE, CJE, CKE, CHE along with pristine CE and GE composites were manufactured under room temperature curing catalyst. XRD, Mode-I and Mode-II fracture toughness evaluation reveals that:
The percentage of crystallinity was found good and almost same in all the NaOH treated natural fibers. The integration of natural fibers in synthetic fibers realized that the suppression of crack growth was observed during Mode-I propagation and also in Mode-II loading. The Mode-I fracture propagation toughness ($G_{IP}$) was controlled by the presence of Jute and Kenaf fibers in UD Carbon epoxy composites. The enhancement of $G_{IP}$ is found almost 30% higher than that of CE and GE composites. The Mode-II fracture toughness ($G_{IIc}$) of CKE hybrid composite was found 25% higher with respect to CE and GE pristine composites.

Finally, it was concluded that the natural fibers are potential enough to replace partially with synthetic fibers for the development high performance hybrid fiber composites. This will not only reduce the cost but will also make the composite lighter in weight without compromising on their strength aspects. Furthermore, the natural fibers are bio degradable and available in abundant quantity. Thus, there is an ample scope to build a new class of composite materials which can be used in practical applications like structural, industrial, aerospace and automotive etc.

ACKNOWLEDGEMENTS

Authors gratefully acknowledge the funding and support provided by ARDB-DRDO Government of India, New Delhi, with Grant-In-Aid scheme of ARDB/01/1051938/M/1 under Structural panel to complete the research work. The authors of this paper appraise their gratitude to the Department of Mechanical Engineering, KLS VDIT, Haliyal and SDM College of Engineering and Technology, Dharwad for their inspiration and support throughout the investigations.

REFERENCES

[1] Shivakumar Gouda, P.S., Chatterjee, V., Barhai, P.K., Jawali, D., Rahatekar, S., and Wisnom, M.R. (2014). Improved fracture toughness in carbon fibre epoxy composite through novel pre-preg coating method using epoxy terminated butadiene nitrile rubber. Materials & Design 62, pp. 320-326. DOI: 10.1016/j.matdes.2014.05.018.

[2] Shivakumar Gouda, P.S., Williams, D.J., Yasaee, M., Chatterjee, V., Jawali, D., Rahatekar, S., and Wisnom, M.R (2016). Drawdown prepreg coating method using epoxy terminated butadiene nitrile rubber to improve fracture toughness of glass epoxy composites. Journal of Composite Materials 50(7), pp. 873-884. DOI: 10.1177/0021998315583317.

[3] Yasaee, M., Bond, I.P., Trask, R. S., and Greenhalgh, E. S. (2012). Mode I interfacial toughening through discontinuous interleaves for damage suppression and control. Composites Part A: Applied Science and Manufacturing 43(1), pp. 198-207. DOI: 10.1016/j.compositesa.2011.10.009.

[4] Pinto, M.A., Chalivendra, V.B., Kim, Y.K., and Lewis, A.F. (2013). Effect of surface treatment and Z-axis reinforcement on the interlaminar fracture of jute/epoxy laminated composites. Engineering Fracture Mechanics 114, pp. 104-114. DOI: 10.1016/j.engfracmech.2013.10.015.

[5] Kede, H., Tran, L., Kureemin, U., Teo, W.S., and Lee, H.P. (2019). Vibroacoustic behavior and noise control of flax fiber-reinforced polypropylene composites. Journal of Natural Fibers 16(5), pp. 729-743. DOI: 10.1080/15440478.2018.1433096.

[6] Zhao, Y., Cao, M., Lum, W.P., Tan, V. B. C., and Tay, T. E. (2018). Interlaminar fracture toughness of hybrid woven carbon-Dyneema composites. Composites Part A: Applied Science and Manufacturing 114, pp. 377-387. DOI: 10.1016/j.compositesa.2018.08.035.

[7] Yongli, Z., Li, Y., Ma, H., and Yu, T. (2013). Tensile and interfacial properties of unidirectional flax/glass fiber reinforced hybrid composites. Composites Science and Technology 88, pp. 172-177. DOI: 10.1016/j.compscitech.2013.08.037.

[8] Uppin, V.S., I, Sridhar., and Shivakumar Gouda, P.S. (2016). Interlaminar fracture toughness in glass-cellulose reinforced epoxy hybrid composites. In IOP Conference Series: Materials Science and Engineering, 149(1), p. 012113. IOP Publishing.

[9] Gumgol, U., Umarfarooq, M.A., Huddar, D., Vastrad, J.V., Wilkinson, A., and Shivakumar Gouda, P.S. (2019). Influence of Kenaf and GO on interlaminar radial stresses in glass/epoxy L-bend laminates. SN Applied Sciences 1(1), pp. 1-8. DOI: 10.1007/s42452-018-0108-6.
[10] Saidane, E.H., Scida, D., José, M.P., and Ayad, R. (2019). Mode-I interlaminar fracture toughness of flax, glass and hybrid flax-glass fibre woven composites: Failure mechanism evaluation using acoustic emission analysis. Polymer Testing 75, pp. 246-253. DOI: 10.1016/j.polymertesting.2019.02.022.

[11] Kanakannavar, S., Savanur, S., Sridhar, Shivakumar Gouda, P.S., and Veereshkumar, G.B. (2018). Improved Delamination Behaviour in Glass-Cotton Reinforced Hybrid Composites. Materials Today: Proceedings 5(11), pp. 24984-24996. DOI: 10.1016/j.matpr.2018.10.299.

[12] Fernandes, R.L., de Moura, M., Silva, F.G., and Dourado, N. (2014). Mode I fracture characterization of a hybrid cork and carbon–epoxy laminate. Composite Structures 112, pp. 248-253. DOI: 10.1016/j.compstruct.2014.02.019.

[13] de Moura, M., Fernandes, R.L., Silva, F.G., and Dourado, N. (2015). Mode II fracture characterization of a hybrid cork/carbon-epoxy laminate. Composites Part B: Engineering 76, pp. 44-51. DOI: 10.1016/j.compositesb.2015.02.010.

[14] AC09036782, Anonymus, ed. Standard test method for mode I interlaminar fracture toughness of unidirectional fiber-reinforced polymer matrix composites. ASTM Internat., (2007).

[15] Dias, G. F., de Moura, M., Chousal, J. A. G., and Xavier. J. (2013). Cohesive laws of composite bonded joints under mode I loading. Composite Structures 106, pp. 646-652. DOI: 10.1016/j.compstruct.2013.07.027.

[16] ASTM D7905/D7905M-14:2014. Standard test method for determination of the Mode II interlaminar fracture toughness of unidirectional fiber-reinforced polymer matrix composites. West Conshohocken, PA: ASTM.

[17] Shivakumar, K. N., Panduranga, R., Skujins, J., and Miller, S. (2015). Assessment of mode-II fracture tests for unidirectional fiber reinforced composite laminates. Journal of Reinforced Plastics and Composites 34(23), pp. 1905-1925. DOI: 10.1177/0731684415602335.

[18] Adekomaya, O., Adediran, A.A., and Adama, K. (2018). Characterization and morphological properties of glass fiber reinforced epoxy composites fabricated under varying degrees of hand lay-up techniques. Journal of Applied Sciences and Environmental Management 22(1), pp. 110-114. DOI: 10.4314/jasem.v22i1.20.

[19] Chen, J., Liu, R., and Xiong, X. (2019). Friction and wear behaviors of SiCNF modified carbon/carbon sealing materials. Journal of Materials Research and Technology 8(6), pp. 5133-5139. DOI: 10.1016/j.jmrt.2019.08.036.

[20] Shahzad, A., and Nasir, S, U. (2017). Mechanical properties of natural fiber/synthetic fiber reinforced polymer hybrid composites. In Green biocomposites, Springer, Cham, pp. 355-396. DOI:10.1007/978-3-319-46610-1_15.

[21] Göktas, D., Kennon, W.R., and Potluri, P. (2017). Improvement of Mode I interlaminar fracture toughness of stitched glass/epoxy composites. Applied Composite Materials 24(2), pp. 351-375. DOI 10.1007/s10443-016-9560-x.

[22] Ravandi, M., Teo, W.S., Yong, M.S., and Tay, T.E. (2018). Prediction of Mode I interlaminar fracture toughness of stitched flax fibre composites. Journal of materials science 53(6), pp. 4173-4188. DOI: 10.1007/s10853-017-1859-y.

[23] Ravandi, M., Teo, W.S., Tran, L. Q. N., Yong, M. S., and Tay, T. E. (2016). The effects of through-the-thickness stitching on the Mode I interlaminar fracture toughness of flax/epoxy composite laminates. Materials & Design 109, pp.659-669. DOI: 10.1016/j.matdes.2016.07.093.

[24] Bensadoun, F., Verpoest, I., and Vuure, A.V. (2017). Interlaminar fracture toughness of flax-epoxy composites. Journal of Reinforced Plastics and Composites 36(2), pp. 121-136. DOI:10.1177/0731684416672925.