Mode-I interlaminar fracture toughness enhancement of the glass/epoxy composite by incorporating nano-fillers

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Abstract

Mode-I interlaminar fracture toughness in FRP composites is the resistance of the plies or group of plies against delamination under tensile loading condition. This can be characterized by double cantilever beam approach. The composites have striking mechanical properties in the in-plane direction, but they are weaker in the transverse direction. Also, the commonly used matrix, i.e., the epoxy resin, which intervenes the reinforcing plies, is brittle. Due to these limitations, the application of composites is restricted is still unexplored in some of the areas. By mixing different nano-fillers as MWCNT, MLG, and their hybrid combination in the matrix, it gets toughened and the interlaminar fracture response of the composites is improved by 25.66 %, 15.25 %, and 19.157 % of the control sample. Also, fractography of the delaminated samples was done using environmental scanning electron microscopy to feature different toughening mechanisms.

Keywords: mode-I interlaminar fracture toughness, double cantilever beam, delamination, nano-fillers, and fractography.

1. Introduction

Composite materials are finding increased applications in many engineering fields. In the aerospace industry, the use of composite materials in commercial and military aircraft has increased greatly over the last 20 years. For example, the usage of composites has evolved from less than 5 percent of the structural weight in the Boeing 737 and 747 to about 50 percent in the Boeing 787 Dreamliner. By contrast, aluminum will comprise only 12 percent of the Boeing 787 aircraft. According to Chambers (2003), while the use of composites is less than 10% of the structural weight in the F14 fighter, it has increased to about 40% of the structural weight in the F22 fighter. In the shipbuilding industry, thick-section glass and carbon fiber composites and sandwich composites are more widely incorporated into ship structures than before to fulfill special demands, such as light-weight, good insulation, low maintenance cost, and resistance to corrosion (Daniel and Ishai 2006). In civil structures, such as bridges, the use of carbon fiber reinforced plastics (CFRP) has extended from only internal reinforcement in structures to both internal and external reinforcement. In addition to structures, wide applications of composite materials can be found in automobile parts and frames, trucks, sports equipment, etc. Among these composite materials, the laminated fiber-reinforced composite material is becoming commonplace in primary load-bearing members of structures and machines as a high-performance material. Compared to metallic materials, laminated fiber-reinforced materials can provide not only the primary advantage of high strength to weight ratio, but also offer extra benefits of low coefficient of thermal expansion (CTE), good resistance to corrosion, low maintenance cost, and low pollution. These advantages together make the laminated fiber-reinforced material an attractive candidate for modern structure and machine design. Despite all their advantages, laminated fiber-reinforced composite materials have certain disadvantages as well. Fiber-reinforced polymer (FRP) has different modes of damage, such as fiber breakage, intralaminar matrix cracking, fiber/matrix debonding, fiber pull-out, and delamination (Daniel and Ishai 2006). Among these damage modes, delamination is one of the most important and least understood.

Delamination is defined as the separation of the plies upon loading in the out-of-plane direction. Due to delamination, or interlaminar fracture, there is the wreckage in strength and stiffness of the composite, which alters the reliability and safety of the composite structure [1][2][3]. This delamination initiates and breeds through the matrix layers, which intervene the reinforcing plies in the bonding since it gives the fracture path with low energy. Delamination resistance of a composite structure is measured by a parameter known as the critical energy release rate (G_{IC}), which is termed as interlaminar fracture toughness (ILFT) [4].

There are three different loading conditions in all the three failure, viz. tensile loading in mode I, shearing load in mode II, and tearing load in mode III, as shown in figure 1, and also its mixed combination. Many methods have been devised for the quantification of the ILFT. Among these, double cantilever beam (DCB), as shown in figure 2 and end notched flexure (ENF) are the two most popular methods for the quantification of mode I and mode II delamination behavior [5,6].

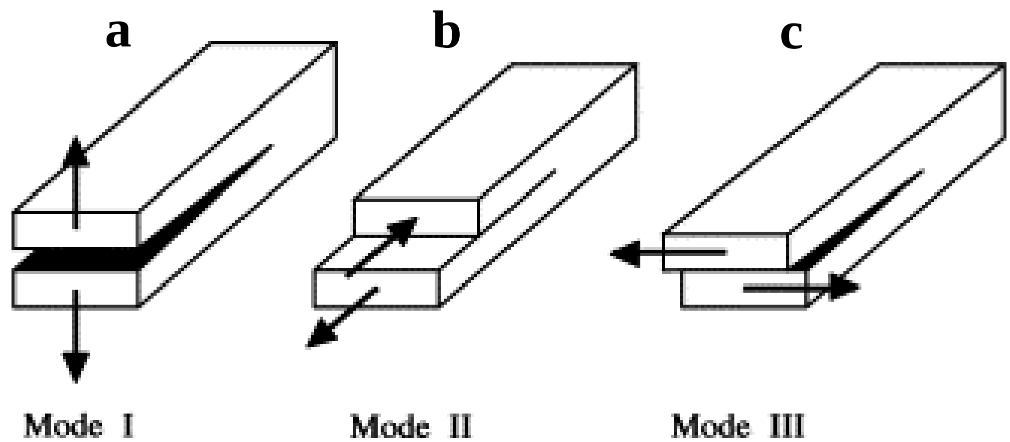

Figure 1. (a) Tensile load, (b) Shear load, and (c) torsion load

Many international organizations and groups are actively involved in carrying out research on *interlaminar fracture toughness* (IFT) testing. Some of them are *i*) ASTM Subcommittee D30.06; *ii*) the Polymers & Composites Task Group of the European Structural Integrity Group (ESIG, formerly the European Group on Fracture); *iii*) the Japan High Polymer Center (JHPC).

Several national and an international standard (ASD-STAN prEN 6033 1995; ASTM D5528-01 2002, ISO 15024 2002; JIS K 7086 1993) already exist for the quasi-static Mode I IFT testing.

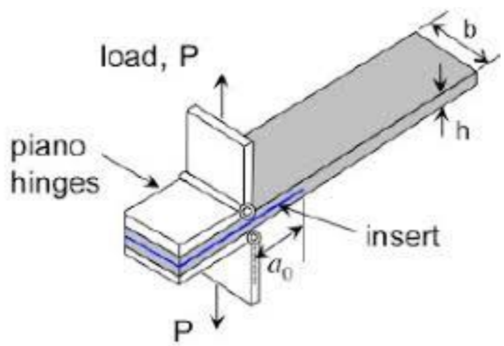

Figure 2. DCB test set-up for Mode-I ILFT

2. Experimental route

2. 1. Laminate fabrication

The reinforcement used in the laminate preparation was unidirectional carbon fiber of 3K plain weave type fabrics manufactured by soller composites and owens corning, respectively. The filament diameter of carbon fiber is 7 µm. The matrix used employed for the fabrication of composites was an epoxy-based resin (diglycidyl ether of Bisphenol A (DGEBA)) manufactured by Atul Industries India, under the trade name of Lapox B-11. The hardener used was triethylenetetramine (TETA) under the trade name of K-6. The properties of reinforcing fiber and matrix are given in table 1.

CFRP sample preparation was done by hand lay-up technique. During hand-lay, releasing sheet was inserted till 75 mm from either end. Followed by curing under a hot press maintained at a pressure of 10 Kgf/cm^2 and temperature of 60℃. Then the samples were cut (to remove unnecessary materials and to get precise dimension) as per ASTM D5528-01 2002 standard [7], as shown in Table 2, using diamond cutter. Finally, post-curing was done by placing in an oven maintained at a temperature of 140 ℃ for 6 hours time duration [8].

2.2. Fracture toughness testing

Mode-I ILFT testing was done by following the following steps:

1. After the fabrication of CFRP laminate, the surface of the laminate was roughened by doing Knurling on it for the first 25 mm from any one side on both surfaces, i.e. top as well as bottom. Also the surface of piano hinges was roughened by Knurling. This roughening was done for the proper gripping between fixture surface and the laminate so that maximum load transfer can take place.
2. Then over the knurled surfaces of laminate and the piano hinges, adhesive “Flex Kwik” manufactured by Pidilite Industries Ltd. was applied. Piano hinges were pasted over both the top and bottom surface. Then it was allowed to be solidified for 24 hours.
3. After solidifying, a meter scale made of paper was pasted over the bottom surface of the laminate. Then marking was made on the front side at a distance of 75 mm from the front end of the laminate. This marking is the endpoint of the pre-crack. Then subsequent markings at the gap of 10 mm were made ahead to it.
4. Finally, the sample was loaded in the tensile fixture of Instron 5967 Universal testing machine.
5. A camera was used for monitoring crack displacement vs. time.
6. The testing was performed at the loading rate of 1 mm/min.

Different theories were proposed for measuring G_{IC}. They are:

1. Beam theory (BT) method

   The G_{IC} is calculated based on the beam theory expression for the strain energy release rate of a perfectly built-in double cantilever beam (i.e., clamped at the debond front).

$$G_{Ic} = \frac{3P\delta}{2ba} \text{ N/mm or kJ/m}^2$$

Where P = load (N)

δ = load point displacement (mm)

b = specimen width (mm)

a = debond length (mm)

The G_{IC} values are computed from the load versus displacement plots based on the non-linear point on the load-displacement plots and using BT equation.

2. Modified beam theory (MBT) method

$$G_{Ic} = \frac{3P\delta}{2b(a+|\Delta|)} \text{ N/mm or kJ/m}^2$$

Where Λ= Correction factor

The beam theory method overestimates the G_{Ic} values because the beam is not perfectly built-in (i.e., rotation may occur at the debond front). In the modified beam theory, the correction factor Λ is incorporated to account for the rotation at the debond front such that considering slightly longer debond length, i.e., a + Λ. The correction factor Λ is determined from the experiment by generating a least-square plot of the cube root of compliance $C^{1/3}$ as a function of debonding length, as shown in figure 3. The compliance, C, is the ratio of the load point displacement to the applied load, δ/P. The G_{Ic} values computed from the load versus displacement plots based on the non-linear point on the load-displacement plots and using MBT equation.

3. Compliance calibration (CC) method

$$G_{Ic} = \frac{nP\delta}{2ba} \text{ N/mm or kJ/m}^2$$

Where n is the slope obtained from the least-square fit of the compliance of the specimen

The GIc value is computed by generating a least-square plot of log δ_i/p_i versus log a using the visually observed debond onset.

4. Modified compliance calibration (MCC) method

$$G_{Ic} = \frac{3P^2C^{2/3}}{2A_1bh} \text{ N/mm or kJ/m}^2$$

Where A_1 is the slope obtained from the least-square fit of a/h versus $C^{1/3}$ of the specimen In the modified compliance calibration method G_{Ic} value is computed by generating a least-square plot of the debond length normalized by specimen thickness, a/h as a function of the cube root of the compliance, $C^{1/3}$, using the visually observed debond onset values.

Also, ASTM 5528-01 suggested the usage of the MBT method, which yields the conservative value of G_{IC} [9].

Table 1. Properties of reinforcing fibers and matrix

Materials	Density (g/cc)	Tensile modulus (GPa)	Tensile strength (GPa)	Stain to failure (%)	Coefficient of Thermal Expansion (10^{-6}/ °C)
Carbon fiber	1.76	230	3.53	1.50	-0.41 (longitudinal)
Epoxy (DGEBA)	1.16	4.50	0.055	4.60	66

Table 2. DCB specimen dimension required in ASTM D5528

Length	Width	Thickness	Initial crack length
L, mm	b, mm	h, mm	a_o, mm
150	25	3	50

3. Results and Discussion

For sample 1

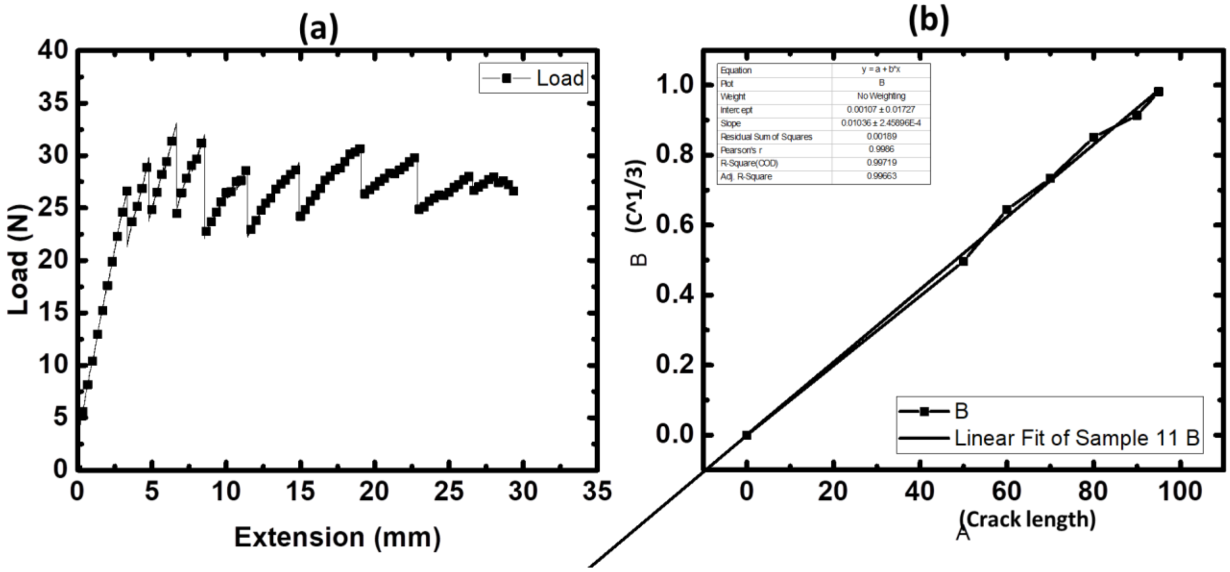

Figure 4. (a) Load vs. displacement curve and (b) Compliance vs. crack length curve

For sample 2

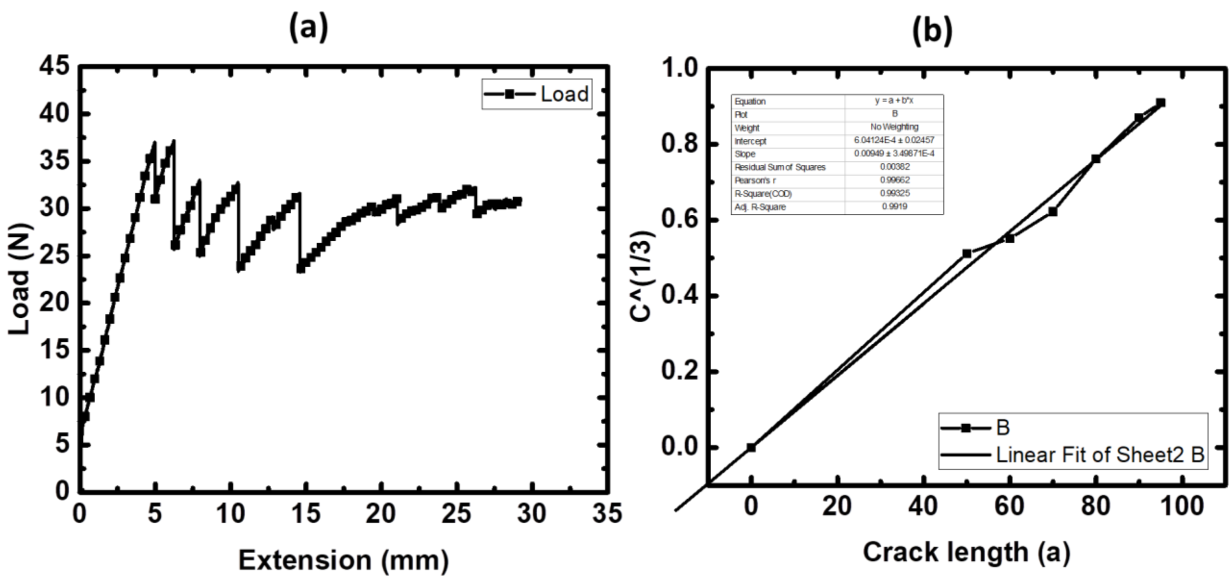

Figure 5. (a) Load vs. displacement curve and (b) Compliance vs. crack length curve

For sample 3

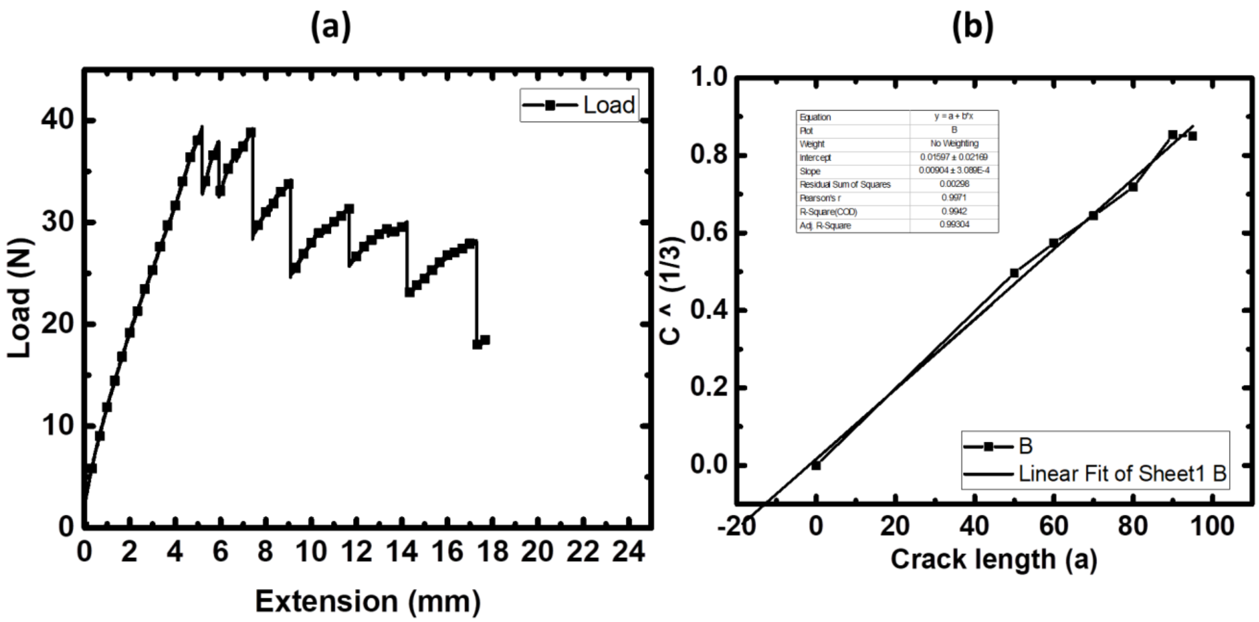

Figure 6. (a) Load vs. displacement curve and (b) Compliance vs. crack length curve

For sample 4

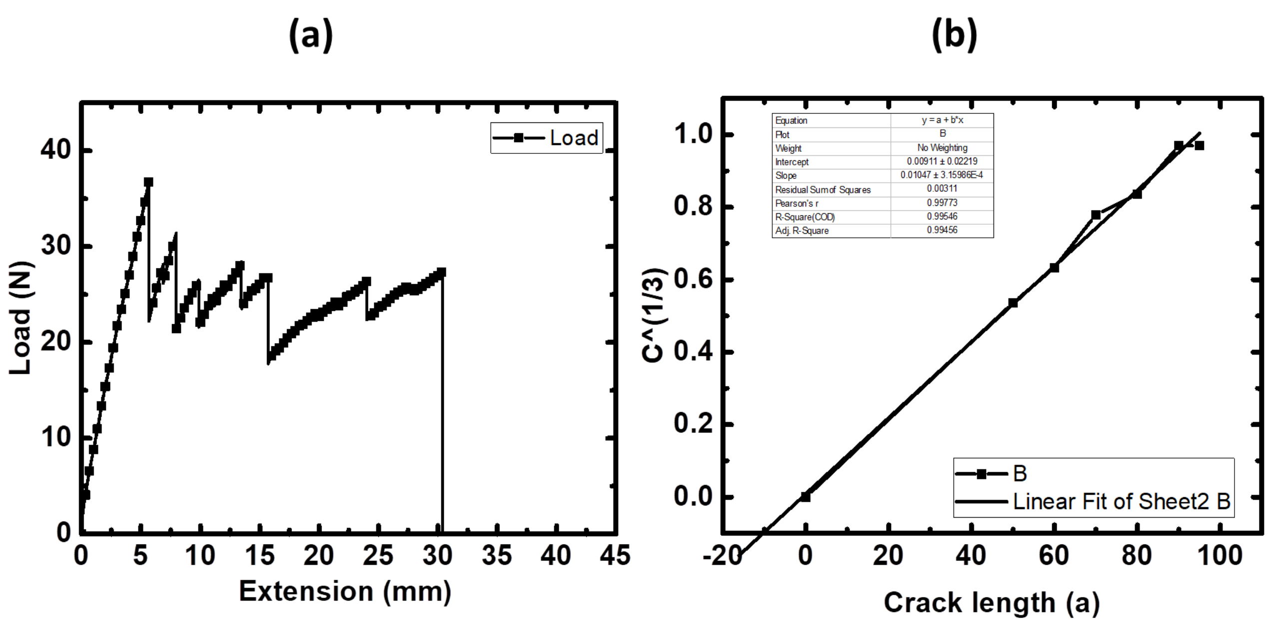

Figure 7. (a) Load vs. displacement curve and (b) Compliance vs. crack length curve

For sample 5

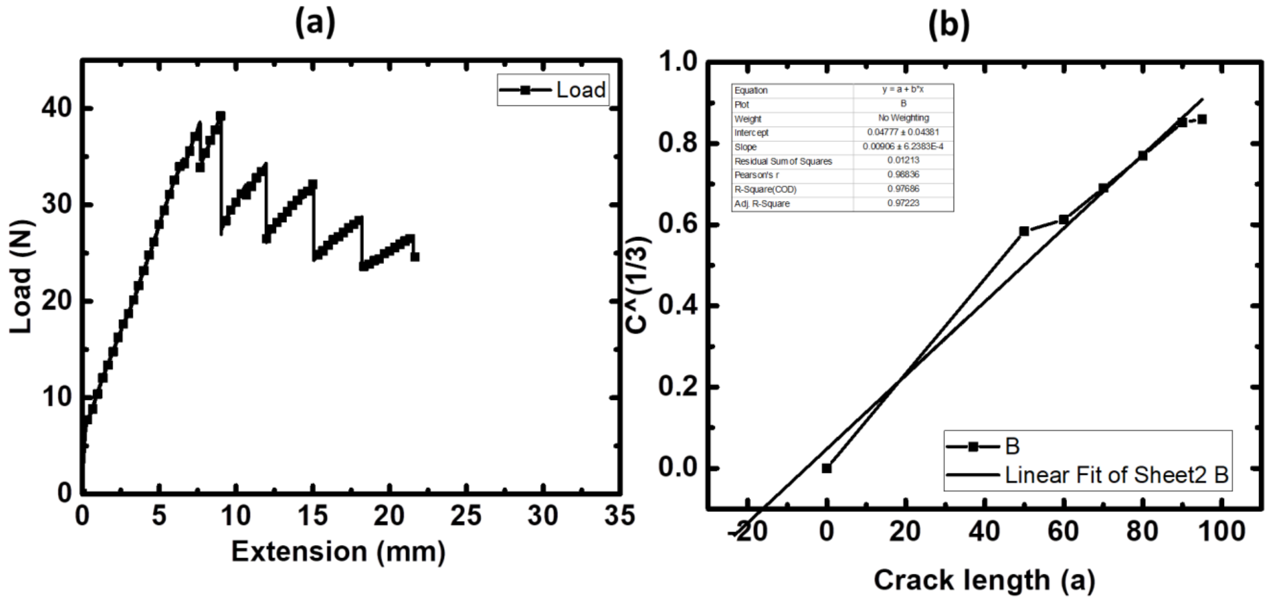

Figure 8. (a) Load vs. displacement curve and (b) Compliance vs. crack length curve

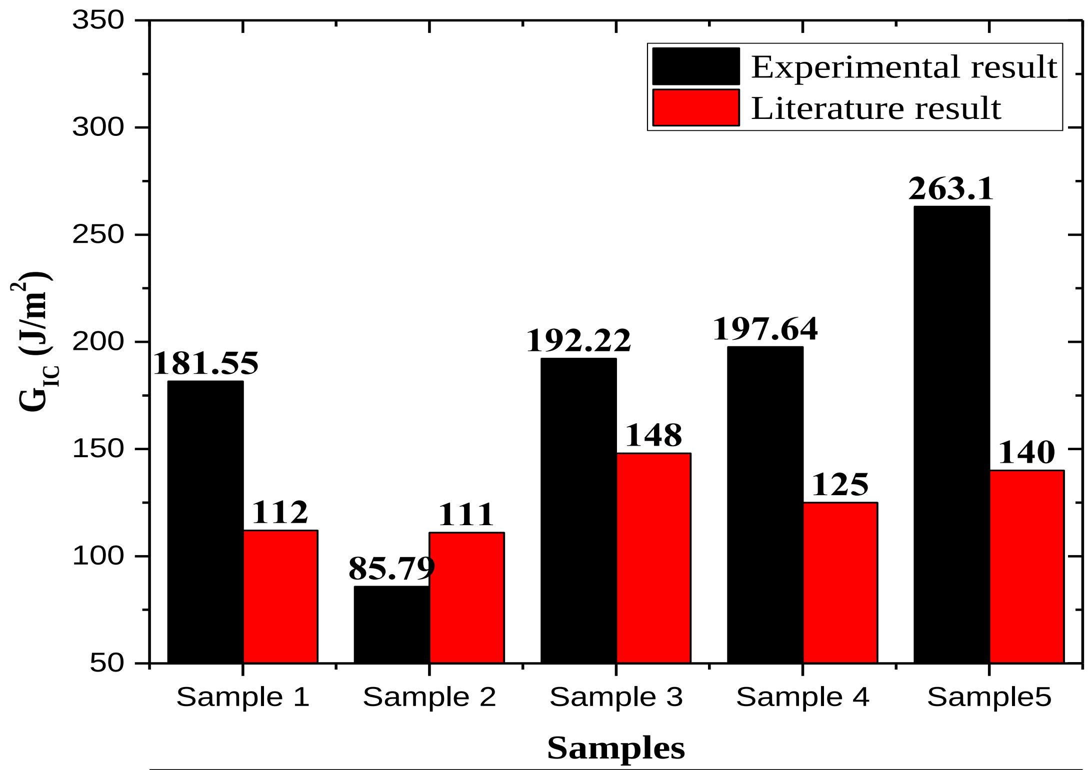

Figure 9. Experimental as well as Literature G_{IC} values

A significant source of non-linearity in the delamination is due to cracking of laminated brittle epoxy matrix and also due to interaction between crack and the fibers [10]. One of the principle reasons for the resistance to delamination is due to fiber bridging [11]. The Non-linearity (NL) G_{IC} value, which is typically lowest of the three G_{IC} initiation values, is recommended for generating delamination failure criteria in durability and damage tolerance analysis [10].

Scope of the work

1. The mode-I interlaminar fracture toughness testing can be done for uni-direction glass fibers, Bi-direction carbon, and glass fibers. Also hybrid combinations of these fibers.
2. Samples testing can be done by varying loading rates as well as temperature.
3. Further, there can be improvement in the fracture toughness value by matrix modification, i.e. incorporating nano-fillers in the matrix phase, also by incorporating Z-pins within the laminate.
4. Further, there is scope of improvement in the fracture toughness value by surface treatment of the fibers, i.e. by incorporating nano-fillers over the fiber surface by electrophoretic deposition (EPD).

Conclusion

Set-up for mode-I interlaminar fracture toughness testing was successfully implemented, and the results were validated.

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