Numerical and experimental analysis of fracture toughness improvement using multi-walled carbon nanotube modified epoxy in fiber metal laminate joints

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***Abstract*— Fiber metal laminates (FML) are a new class of laminated composite materials having superior mechanical properties such as excellent strength to weight ratio and fatigue life. These characteristics have significantly increased their applications in aerospace industry and high performance sports equipment. Laminates in FML are bonded together by adhesive joints using high grade epoxy resins. These adhesive joints are weak regions and may result in inter-ply failure i.e., delamination due to debonding of adhesive in joint under transverse loading conditions. This work aims at strengthening of adhesive joints using modified epoxy resins containing different weight percentage (wt %) of multiwalled carbon nano-tubes (MWCNTs). Finite element analysis (FEA) was used for simulating mode-I fracture in adhesive joint. ABAQUS v13.0 is commercially available software used for this analysis. Simulation in this paper was done using extended finite element method (XFEM) approach as this numerical technique is very popular and successfully used for precisely simulating delamination failure due to debonding. The simulations were carried out on double cantilever beam (DCB) test as per ASTM D5528 standard. Multiple simulations were carried out and in these simulations Double Cantilever Beam (DCB) was adhesive bonded by epoxy containing 0 wt% (neat epoxy), 1 wt% and 2 wt% MWCNTs. The results were obtained as force-displacement curves for each simulation carried out by XFEM and these curves were used for development of toughness-delamination curves. Later these results were compared with the results of specimen bonded by neat epoxy.**

Keywords—Fiber metal laminates (FML); Multiwalled carbon nanotubes (MWCNTs); Delamination; Finite element analysis (FEA); XFEM; Double cantilever beam (DCB)

I. INTRODUCTION

Laminated composites materials are being widely used in multiple engineering applications like in aerospace industry as well as in sports industry for making lighter and stronger equipment[1-2]. Laminated composites have excellent mechanical properties such as high specific strength, stiffness and strength to weight ratio and excellent fatigue life [3-5]. Fiber metal laminates (FML) is a class of laminated composites which consist of thin sheets of metals adhesively bonded to fiber reinforced layer. Due to their superior mechanical properties FML have replaced conventional alloys in many applications[6-7]e.g. GLARE has replaced aluminum alloy for making fuselage of commercial airliner[8]. However, there are some limitations regarding mechanical properties of FML due to which they are not fully utilized. Delamination is one of the major problem occurring in FML deteriorating their strength, stiffness, compressive load bearing capabilities and fatigue life[9-11]. Delamination occurs when laminated composite is subjected to transverse tensile loading scenarios. Laminates have excellent in-plane mechanical properties while in transverse loading, matrix (adhesive) is the main load bearing component. As matrix is weaker as compared to both metallic as well as composite laminates high transverse load results in failure. Delamination can be result of fracture within the adhesive, fracture within reinforcement or debonding due to softening of adhesive layers that bond reinforcement layers together[12-16]. Several mathematical models have been developed to model fracture behavior and strain energy release rates to predict delamination failure. Extended finite element method (XFEM) is one of these models which works by enhancing conventional shape functions with extra functions derived from partition of unity concept[17-18]. XFEM model can be applied to both cohesive zone modeling (CZM) and virtual crack closure technique (VCCT) and it can also be used to predict load carrying capability of cracked part[19-20]. In this paper it is applied for evaluation of load carrying capability of cracked part. The fundamental equation governing displacement (u) is written as:

$$u = \sum_{i=1}^{N} N_i\,(x)\ [u_i + H(x)a_i + \sum_{\alpha=i}^{4} F_\alpha\,(x) b_i^\alpha\,] \quad (1)$$

where H(x) is discontinuous jump function and F_α (x) is neartip function. XFEM models are applied to double cantilever beam test to predict the inter-ply fracture damage (delamination). This test can be employed to evaluate load-displacement curves related to mode-I, mode-II, mode-III and mixed mode fractures. Commercially available ABAQUS v6.13.0 is a powerful tool for finite element analysis and above mentioned XFEM function is applied to double cantilever beam test as per ASTM standard D5528.

In this research, aim was to modify matrix (epoxy adhesive) by addition of functionalized MWCNTs to increase the stiffness and fracture toughness of adhesive bond in transverse loading direction. As functionalized carbon nanotubes form strong bond at interface with epoxy, this strong interface between dispersions-matrix results in improvement in load bearing capabilities of the matrix which further improves the fracture toughness of adhesive bond. This hinders delamination as debonding due to matrix softening is minimized. N.Yu et al. reported fracture toughness improvement by 1.29-1.69 times to that of pure epoxy by addition of 1wt%-3wt% carbon nano tubes [22]. Borowski et al. reported 25%, 20% and 17% improvement in interlaminar fracture toughness by addition of 0.5, 1.0 and 1.5 wt% MWCNTs to epoxy resin respectively [21]. Multiple samples of modified epoxy were prepared by mixing MWCNTs. Neat epoxy bonded specimens were also prepared and used as standard specimens to compare toughness-delamination values to that of modified epoxy bonded specimens. First, tensile specimen were made of neat epoxy as well as of modified epoxy. These specimen were used for tensile test to determine modulus of elasticity. Modulus of elasticity was used for simulating mode-I fracture on double cantilever beam using XFEM shape functions in ABAQUS v6.13.0.

II. MATERIALS AND METHODS

A. *Experimentation*

The epoxy resin used for adhesive bonding for making double cantilever beam (DCB) was LY-5052. Its elastic modulus is 1.85GPa. MWCNTs used in this study were manufactured by 'Cheap Tubes'. These MWCNTs have diameter of 35nm-45nm. MWCNTs were examined under SEM for morphology and diameter measurements. Fig. 1 shows the SEM image of MWCNTs.

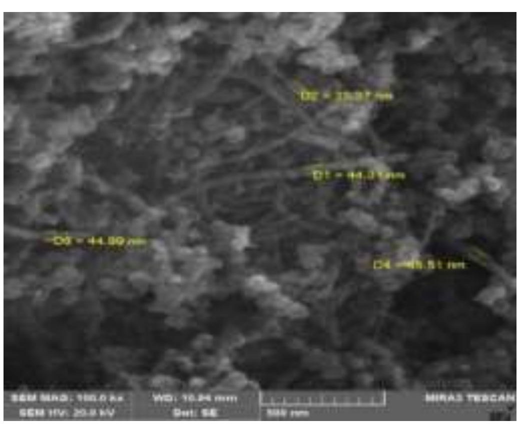

Fig. 1. SEM micrograph of MWCNTs

The purity level of these MWCNTs was more than 95%. MWCNTs were functionalized using mixture of concentrated H_2SO_4 and HNO_3. Functionalization promotes formation of -OH hydroxyl group on MWCNTs. These -OH form hydrogen bonding with epoxy groups. Hydrogen bonding between MWCNTs and epoxy results in formation of strong interface which improves mechanical properties. MCWNTs were added to epoxy resin at weight percentage basis. Mixture was homogenously mixed by employing mechanical stirring for 30 mins.

Tensile test specimen were made. The tensile tests gave elastic modulus values of neat epoxy and modified epoxy containing 1% and 2% MWCNTs respectively. These results are given in Table 1.

Sample ID	**Table 1 : Mechanical properties of epoxy**		
	Epoxy resin LY-5052	***Elastic modulus***	***Tensile strength***
	% of MWCNTs	/ GPa	/ MPa
1	0	1.85	26.2
2	1	2.14	30.5
3	2	2.29	32.5

For double cantilever beam experimentation, FML composite structure was developed, for upper and lower face-sheets 1 mm thick sheets of AA 2024 were used. Composite layers were sandwiched between these metallic layers. The thickness of composite part was 2 mm as 8 of woven carbon fiber-epoxy lamina each having 0.25 mm thickness were stacked over each other to form 2 mm thick composite part. Carbon fibers layers were impregnated with epoxy resin and these layers were laminated with metallic layers followed by vacuum bagging to press the laminated together for curing of epoxy. The elastic modulus values for composite laminates and aluminum 2024 sheet are 70GPa and 73Gpa, respectively. The pre-crack is introduced between composite part and one of the metallic layer by using tape between these laminates before curing. The length of pre crack is 40 mm. The length and width of double cantilever beams is 130mm and 25 mm respectively. For clamping DCB into universal testing machine (UTM) piano hinges are attached to pre-cracked end on upper and lower metallic face sheets. DCB are marked for measuring delamination length. Fig. 2 illustrates DCB sample for experimentations.

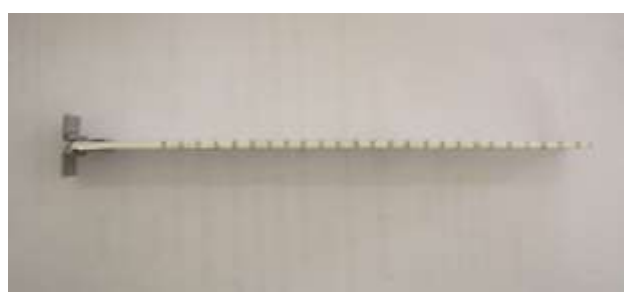

Fig. 2. Double cantilever beam configuration

Three of these beams are developed, first with neat epoxy and second and third with 1wt% and 2wt% MWCNTs modified epoxy. These beams were clamped to UTM fixture and pre-crack tape was removed prior to testing as shown in Fig. 3.

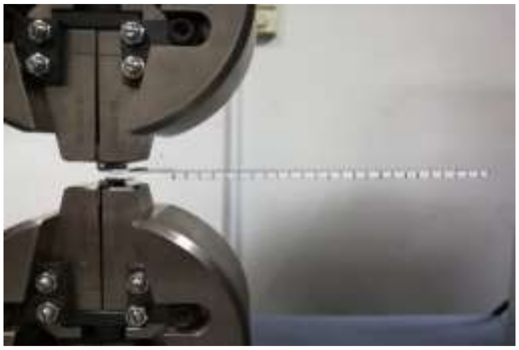

Fig. 3. DCB clamped in UTM fixture

Opening displacement rate of beams was set to 2mm/min and force-displacement values and corresponding delamination length was recorded.

For FEM numerical simulations, XFEM approach is used to model and simulate double cantilever test. Two beams are adhesively bonded together by modified epoxy as shown in Figure 4.

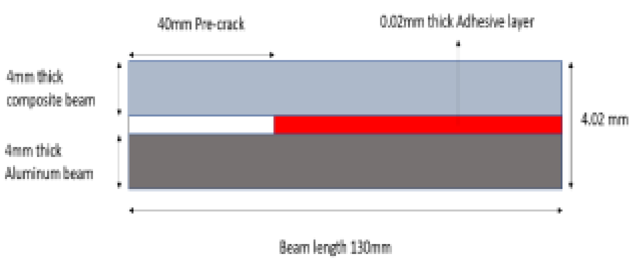

Fig. 4. DCB schematics for ABAQUS

Double cantilever beam was developed in ABAQUS v6.13.0, upper beam was assigned as composite layer while lower was selected as aluminum alloy layer and their respective stiffness values were assigned. For dimensions, the length of beams was assigned as 130mm, the thickness of both upper and lower layer was 2mm and the thickness of adhesive layer was 0.2mm.The depth of plane was selected as 25mm as the simulations were performed in 2D model. Pre-crack of 40mm was used.

2D models of double cantilever beam were developed in ABQAUS, the damage in model was evaluated by traction separation law across fracture region. For damage initiation maximum principal stress criteria (MAXPS) was used. For damage evaluation linear model of traction-separation law was used. Pre-crack length of 40 mm was used and crack path is defined parallel to adhesive element. The fracture initiation occurs at the center of the elements. It is assumed the fracture will occur only between the elements of adhesive joint layer. Fiber bridging was also taken under consideration as this induces resistance to opening of beams, as a result additional force has to be applied. The crack tip was selected as per XFEM. Model developed in ABAQUS is shown in Fig. 5.

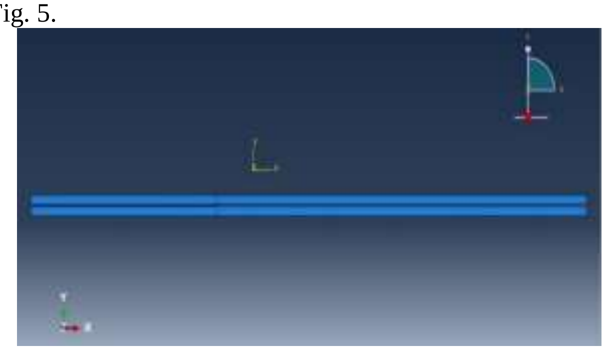

Fig. 5. DCB XFEM 2D model in ABAQUS

Meshing was done for whole assembly, for upper and lower beams continuum shell elements of size 0.5 mm were used and for adhesive joint 0.1 mm sized elements were used for meshing of assembly.

Finally three models were developed first model was adhesive bonded with neat epoxy, second and third models were adhesive bonded with modified epoxy mixed with 1 wt% and 2 wt% MWCNTs respectively. Double cantilever beam test was run and results were obtained as load-displacement plots.

III. RESULTS AND DISCUSSION

Mode-I fracture (opening mode) was observed in DCB as upper and lower beams move apart on application of tensile force in y-directions. The crack started propagation as load and displacement from UTM cross head increased. This resulted in increase in delamination length. Load-Displacement and corresponding values of delamination were recorded and registered for the three experiments.

For numerical simulation results for DCB tests, Fig. 6 illustrates the distribution of Von Mises stress on three DCB specimens. The Load displacement plots for DCB specimen developed by neat epoxy, 1 wt% and 2 wt% modified epoxy are shown in Fig. 7. The load-displacement values were used for construction of Critical strain energy release rate (G_{IC}) vs delamination length curves for mode I fracture. The value of G_{IC} at a point can be calculated by formula given below.

$$G_{IC} = \frac{3P\delta}{2b(a+|\Delta|)} \qquad (2)$$

where : P = load;

δ = opening displacement;

a = delamination length;

b = width of beam;

$|\Delta|$ = correction factor and can be calculated by least square plot of cube-root of compliance $C^{1/3}$ ($C = \delta/P$).

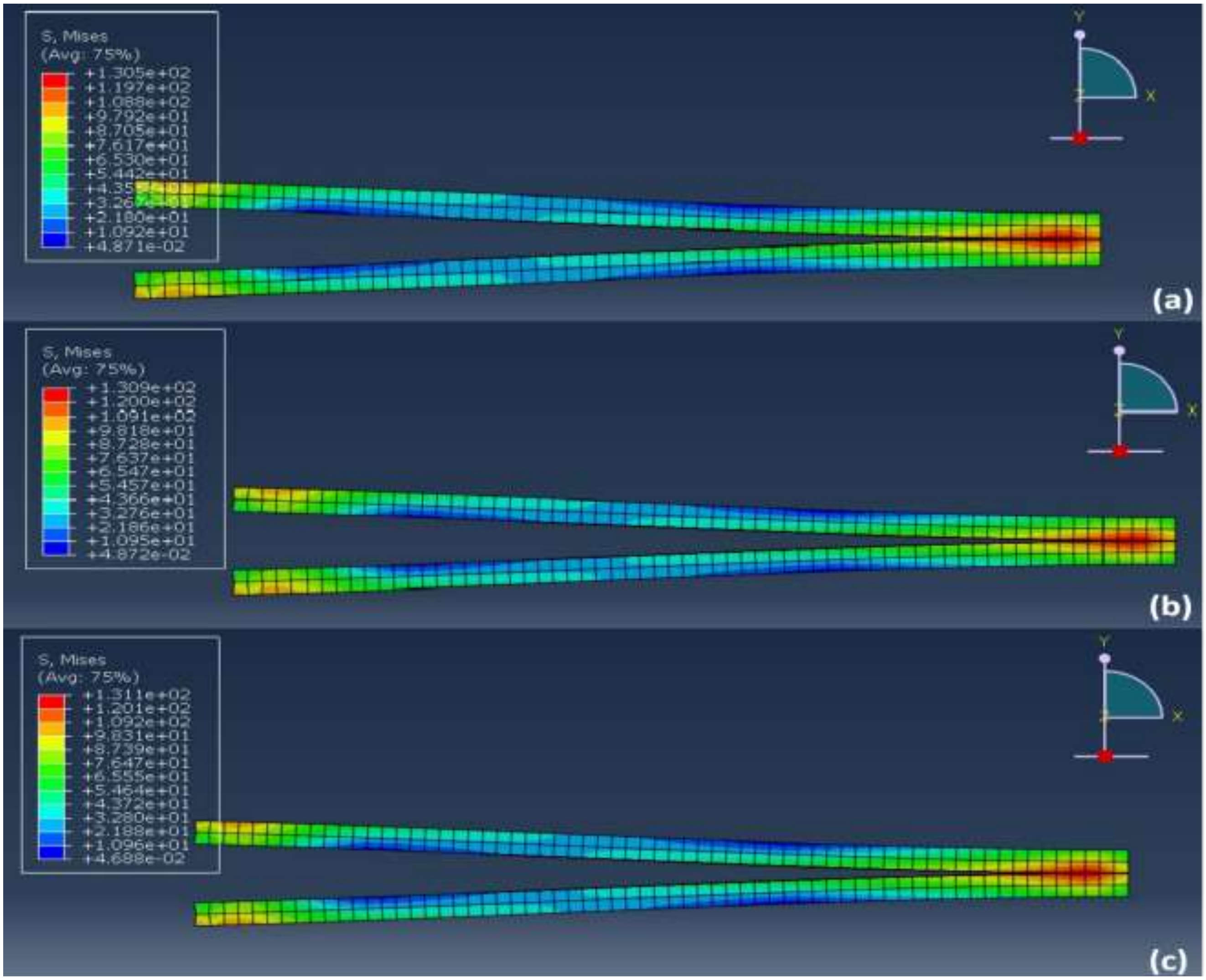

Fig. 6. Von mises stress distribution for DCM bonded by (a) neat epoxy (b) 1% MWCNTS modified epoxy (c) 2% MWCNTs modified epoxy

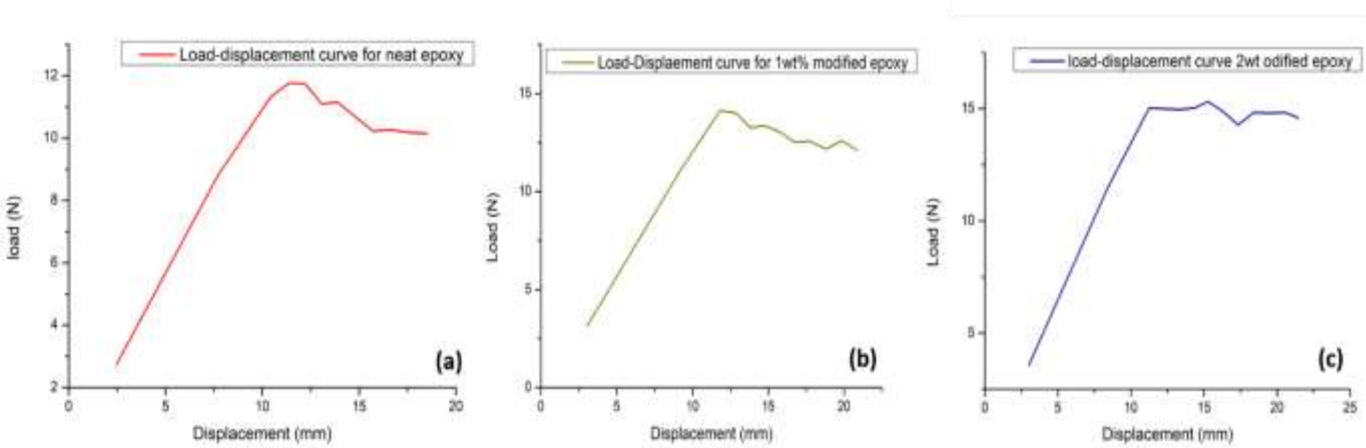

Fig. 7. Load-Displacement curves for (a) DCB bonded with neat epoxy (b) DCB bonded with 1 wt% modified epoxy and (c) DCB bonded with 2 wt% modified epoxy

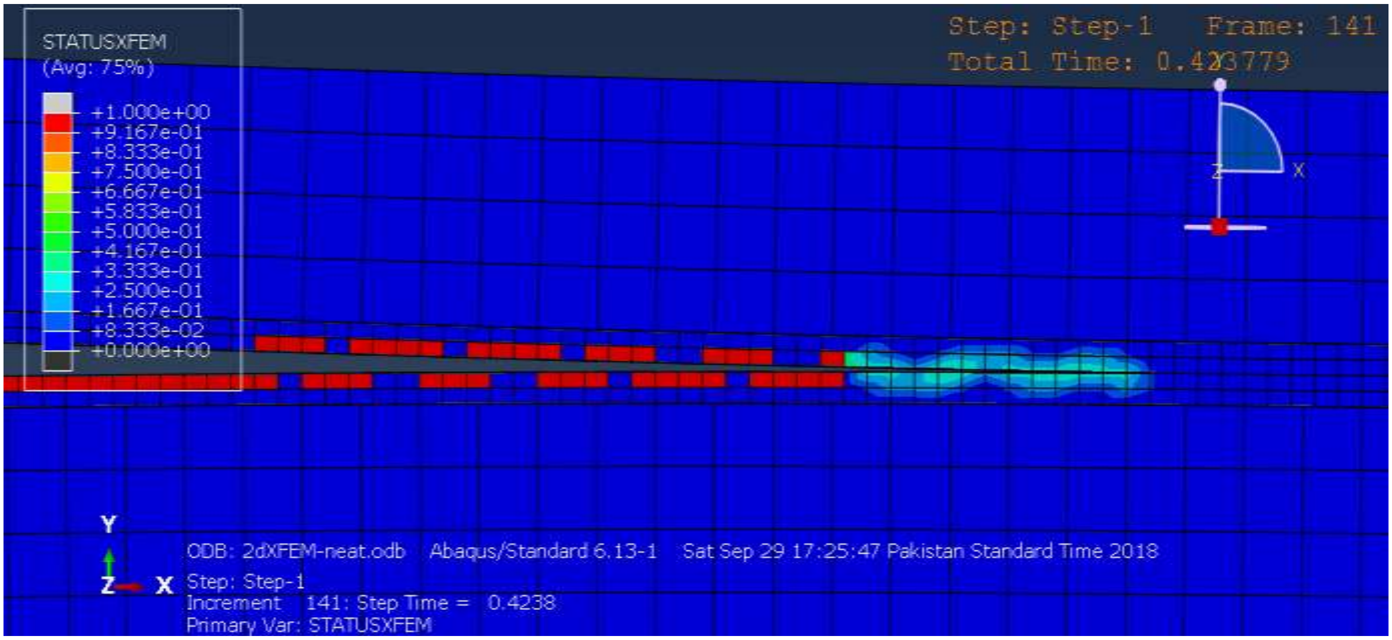

Fig. 8. Simulated crack profile and its stress distributions in ABAQUS simulation

The crack profile and stress distribution around it can be seen in Fig. 8

Load-Displacement curves were obtained from above three simulations. The displacement value was the opening displacement of beam and was calculated by selecting unique nodal displacement. The three load-displacement curves from simulated DCB tests were merged on a single plot and shown in Fig. 9

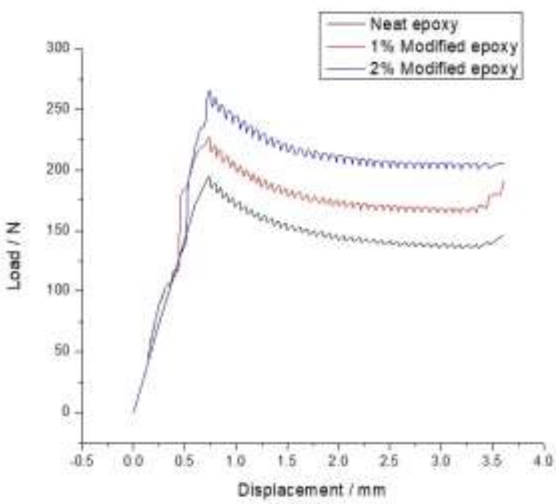

Fig. 9. Load displacement curves for three DCB simulations

Finally G_{IC}-delamination length curves were derived from values obtained from load-displacement curves. These values were compared with respective simulated results. Fig. 10 illustrates G_{IC} vs delamination length plots for experimental results as well as for numerically simulated results (Dashed lines represent experimental results).

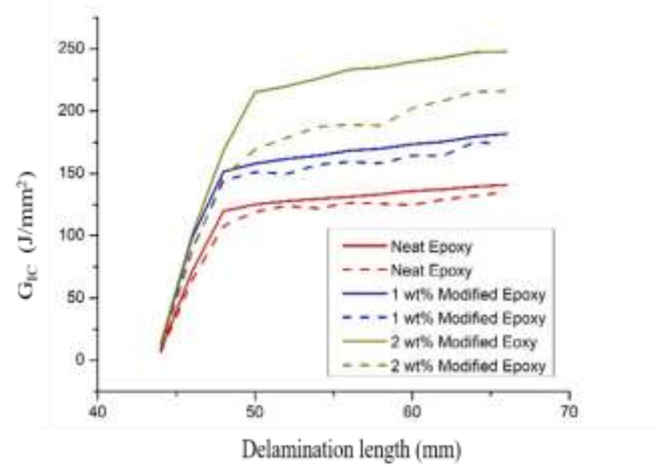

Fig. 10. Fracture toughness-Delamination curves (Dashed lines represent experimental results)

IV. CONCLUSION

Delamination study of FMLs was conducted using both experimental and numerical approach. The fracture toughness for mode 1 delamination was improved by addition of MWCNTs. The improvement in fracture toughness is due to addition of functionalized MWCNTs nano fillers. These nano fillers form hydrogen bonds with epoxy resin. Mechanical locking and hydrogen bonding results in stronger interface, as a result load transfer to MWCNTs during crack bridging improved significantly which results in improvement in fracture toughness. According to numerical results, addition of 1 wt% and 2 wt% of MWCNTs in epoxy resulted in increase of fracture toughness (GIC) by 30% and 70% respectively. Experimental results showed good agreement with simulated results for neat epoxy and 1 wt% modified epoxy bonded sample, whereas only 45% improvement in fracture toughness was observed experimentally for 2 wt% modified epoxy bonded sample. This difference in experimental and numerical values may be due to many factors the most prominent is homogenous mixing of MWCNTs in matrix which is not possible experimentally for higher wt% of these nano fillers, as agglomeration occurs which restricts homogenous distribution of these fillers. The results were comparable with the results obtained by similar studies conducted in past years mentioned above, using functionalized MWCNTs for improving delamination fracture toughness. This indicates validity and accuracy of XFEM model. The difference in improvement of fracture toughness values may be due to many factors such as percentage functionalization and functionalization precursors used for MWCNTs, type and characteristics of MWCNTs, the mixing process being employed and characteristics of epoxy being used.

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