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Mode II Shear Behavior of A Glass Fabric/Epoxy and A Multi Scale Glass Fabric/Epoxy Thick Beam Composite Containing Multiwalled Carbon Nanotubes

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Abstract

The mode II shear behavior of a glass fabric/epoxy composite and a multiscale hybrid glass fabric/epoxy composite containing Multi Walled Carbon Nano Tubes (MWCNT) are compared here in the interlaminar mode of loading. The thick composites were fabricated through the vacuum bagging technique and two sets of specimens were prepared with and without carbon nano tubes. One set of each of these specimens were prepared with a Teflon® film crack initiator at the end of the specimens exactly at the centroidal line of the laminate to study the fracture behavior and strain energy release rates. This study addresses the issue of testing thick laminated composites and thick multiscale laminated composites in Mode II Inter Laminar Shear (ILS). The Inter Laminar Shear Strengths (ILSS) of these composites were evaluated. The theories on the strain energy release rate are also revisited and useful conclusions drawn regarding the applicability of the same to multiscale composites loaded in the ILS test set up. Thus, a comparison is made between the mode II behavior in flexure and ILS of these composites.

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Keywords: MWCNT, Glass/epoxy, Multiscale composites, Thick laminates, ILSS, Mode 2 strain energy release rate

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1. **Introduction**

Glass fibre is a material that is composed of numerous fine strands of glass. Its properties such as high surface area to weight ratio, good thermal insulation, etc. have made it highly useful in the manufacture of various composite materials. Unlike carbon fibre, glass fibre can undergo more elongation before it breaks.

Multi-walled carbon nanotubes (MWCNT), allotropes of carbon, have remarkable properties such as high tensile strength and elastic modulus that can be exploited to our advantage by using them in the manufacture of composites. Normally, MWCNTs are used as reinforcements in composites to strengthen the specimens.

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When MWCNTs are used in the manufacture of composites, the specimens can be classified as multiscale composites as the glass fibres are of micrometre dimensions and the MWCNTs are of nanometre dimensions. Thus a multiscale composite is one in which the constituents vary largely in size and the best benefits of a nanoscale reinforcement are achieved with cost in mind as microscale reinforcements are also used. Creating a sandwiched composite consisting of glass fibre and MWCNT held together by epoxy resin and hardener brings together the cumulative advantages of the different constituents: the MWCNT adds good tensile strength and stiffness to the composite and the glass fibre provides reasonable strength, thermal and electrical properties. These sandwich structures find wide applications in electronic packaging, thermal insulation, structural members and products with cladding.

The ability to predict failures in laminated fibre reinforced composites becomes paramount because of their increasing applications in various fields. The most common failure mode for laminated composites is delamination (separation of adjacent layers). Several tests have been developed for evaluating delamination over the years [1,2], but only a few of them have qualified for standardization. Some of the approved tests are the Mode I delamination test with Double Cantilever Beam (DCB), the End Notch Flexure (ENF) test and the ILSS test [3,4,5]. The strain energy release rate approach has been used in most studies of composite laminates involving delamination. The critical strain energy release rate G_c, is a measure of fracture toughness and may be different for three types of loading: Mode 1-opening, mode 2-in plane shear and mode 3-out of plane shear. Though quite a bit of literature is available on the strain energy release rate and fracture toughness of DCB and ENF specimens [6,7,8] not a single research paper is available on the measurement of the same for ILSS specimens though the later can be closer to mode 2 shear (depending upon the number of layers , span and the beam thickness) than the former which is actually mode 2 shear with large deflection, moments and beam curvature and hence only a mode 1 , mode 2 mixed mode test at longer spans.

In this investigation, a comparative study of the Inter Laminar Shear Stress (ILSS) and strain energy release rate between a glass / epoxy laminate and a glass /epoxy laminate containing MWCNT is carried out. The laminate used here is a thick beam, whose average thickness is around 6.5 mm. This study addresses the issue of testing thick laminated composites and thick multiscale laminated composites in Mode II Inter Laminar Shear (ILS). The Inter Laminar Shear Strengths (ILSS) of these composites were also evaluated. The theories on the strain energy release rate and fracture toughness applicable to ENF specimens that are tested at longer spans, are also revisited and useful conclusions have been drawn regarding the applicability of the same to multiscale composites loaded in the ILS test set up. Thus, a comparison is made between the mode II behavior in flexure and ILS of these composites.

2. Experimental Methods

2.1 . Raw Materials and Properties

The materials used in the experiment were 26 layers of glass fabric 260 GSM along with GY 257-A140 epoxy resin and hardener. 3 grams of MWCNT supplied by Quantum Materials Corp, Bangalore, were also used. Each layer of the glass fabric is approximately 0.3 mm thick with an appropriate volume fraction of the resin coated on it. Every glass fabric layer was completely coated with the epoxy resin and hardener and flattened to remove any air bubbles with a roller before the next fabric layer was laid perfectly in place on top of it. 13 such layers were arranged after which the 3 grams of MWCNT were evenly spread across exactly half the area of the top most glass fabric layer.

A Teflon tape of width 18 mm was placed along the length of the entire specimen such that it covered a half of the area that did have MWCNT as well the other half which did not. Once the MWCNT was in place, 13 more layers of glass fabric were set in place with the epoxy resin and hardener between them. A pre cracking technique is employed here to study the strain energy release rate and fracture toughness of the ILS specimens. A 20 micron thin Teflon film is used for pre-cracking which is likely to make crack development more realistic as the tip of the starter crack will be pointed and a thicker film otherwise would have produced undesirable effects like a blunt and thick starter crack tip that would have initiated a delamination at a thicker resin rich layer

Fig.1. Glass/Epoxy layers with and without MWCNT

A starter crack always causes fibre bridging, which can affect testing results [3]. The total weight of the 26 layers of the 260 GSM Glass Fabric weighed 681 grams. The density of the glass fabric was 2.52 g/cc and that of the resin was 1.2 g/cc. The mixture of resin and hardener was done in a 2:1 ratio. The volume fraction of the fabric was decided to be 0.40 and the resin and hardener contributed to the remaining 0.60. Using these volume fractions, the volume of the fabric was found to be 270.24 cc, that of the resin to be 270.24 cc and the hardener to be 135.12 cc. The MWCNT, with an outer diameter of 50 nm and length of 30 micrometres has an average density of 1.5 g/cc, the range being 0.2 to 2.20 g/cc. Therefore, the volume of MWCNT used was 2cc. Adding this, the total volume of the specimen was 677.6cc. Hence, the volume fraction of MWCNT was found to be 0.295%. Since only half of the whole specimen was sprinkled with MWCNT, the actual volume was 0.588 %. As ILSS is a matrix dominated parameter, the volume fraction of the matrix was maintained as 0.60 , more than that of the reinforcements.

2.2. Vacuum Bagging Technique

The entire specimen was then placed in a plastic bag for vacuum bagging . A vacuum pump was attached to the plastic bag and the air evacuated from the bag, thus creating a vacuum inside and allowing an atmospheric pressure of 1 bar to act on the vacuum bag. A bleeder layer was laid which served the purpose of absorbing excess resin. This atmospheric pressure on the laminate helps remove any existing air bubbles within the laminate and removes any excess resin such that an optimum fibre to resin ratio is obtained. Thus a consolidated laminate could be obtained.

Fig. 2. The Vacuum Bagging Technique

2.3. Specimen Details

Once the vacuum bagging process was complete, the laminate was set aside to cure for a period of 72 hours. On completion of the room temperature curing process, the laminate was cut into 48 different Specimens such that 8 different variations of samples were obtained from the same laminate :

- Specimens with MWCNT and with Teflon insert with a 1:5 thickness to width ratio.
- Specimens with MWCNT and no Teflon insert with a 1:5 thickness to width ratio.
- Specimens without MWCNT and with Teflon insert with a 1:5 thickness to width ratio.
- Specimens without MWCNT and no Teflon insert with a 1:5 thickness to width ratio.
- Specimens with MWCNT and with Teflon insert with a 1:2 thickness to width ratio.
- Specimens with MWCNT and no Teflon insert with a 1:2 thickness to width ratio.
- Specimens without MWCNT and with Teflon insert with a 1:2 thickness to width ratio.
- Specimens without MWCNT and no Teflon insert with a 1:2 thickness to width ratio.

The choice of the two thickness to width ratios were based on an earlier investigation on interfacial shear of fibre tows embedded in an epoxy matrix [9]. A higher width was known to produce a valid pull out that could be used in the evaluation of Interfacial Shear Stress (IFSS). Each specimen had a length of 55 mm and an average thickness of 6.5 mm. As the specimen thickness is higher than 6 mm which is the upper limit set by the ASTM D 2344 standard on ILSS testing [5], a span to depth (thickness) ratio of 5:1 was followed in place of the conventional 4:1 ratio . Besides, the high number of layers and the necessity of a 18 mm starter crack at the end demanded the choice of a slightly longer span to study the crack propagation in the ILS mode.

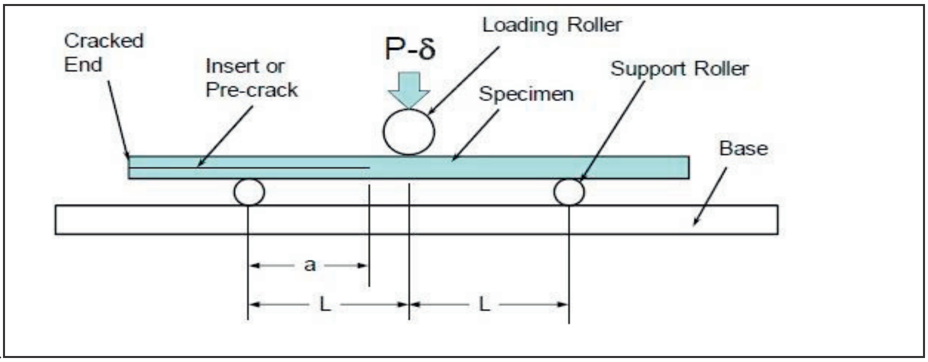

Figure. 3. The ILSS Specimen Geometry , where crack length a= 18 mm, thickness of the specimen= 6.5 mm & span length= 2L = 33 mm.

2.4. ILSS Tests and Parameters of Evaluation

Upon machining the laminate and obtaining the variations in sample types, the Inter Laminar Shear Stress (ILSS) was found using the Three Point Bending Flexure test. The standard followed for the procedure was the ASTM D2344, with a few modifications that were necessary to adapt the test to a thicker beam and evaluate the strain energy release rates as described before. At this juncture it is noteworthy that there is currently no published literature or standard available to evaluate the strain energy release rates of ILSS specimens. This investigation attempts to address this issue. Here, an Instron 8801machine was used to carry out the three point bending flexure test. Each sample was positioned on the two lower points while the third upper point was used to impart the normal force on the samples such that Mode II inter-laminar shear took place between the layers of the laminate. Once the set up was in place, the test was carried out at a cross head velocity of 1 mm/min. and using a digital output, the load and the displacement were recorded and the ILSS for each sample was calculated.

Inter laminar shear strength of all the 48 samples were calculated using the formula [5]:

$$ILSS = 0.75 \left(\frac{P}{bd}\right) \quad \text{-----------} \quad (1)$$

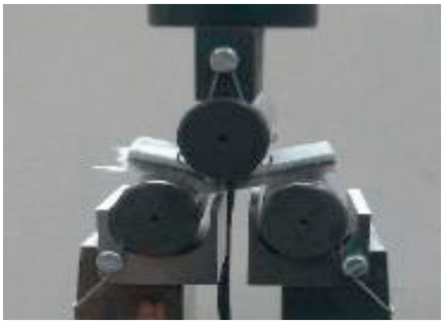

Fig.4. ILSS test using Instron Machine.

Where P is the load in Newton, b is the breadth in mm and d is the thickness in mm.

Upon calculating the shear strength of each of the samples, the Direct Beam Theory and Russell's Theory were used to calculate the Mode II Strain Energy Release Rate of each of the specimens with a Teflon pre-crack, which is a pre-requisite.

The formula used by the Russell's Theory is [10]:

$$G_{II} = \frac{9P^2a^2}{16b^2E_{II}h^3} \tag{2}$$

Where G_{II} is the strain energy release rate in Mode II , P is the considered load, "a" is the crack length, "b" is the breadth, E_{II} is Young's Modulus in the longitudinal direction and 2h is the depth or thickness of the beam.

The formula used by the Direct Beam Theory is [11]:

$$G_{IIc} = \frac{9a^2P\delta}{2B(2L^3 + 3a^3)} \tag{3}$$

Where G_{IIc} is the strain energy release rate in Mode II, P is the considered load, δ is the deflection, L is half-span length, `a' is the crack length and B is the width.

The calculation of E_{11} was done by assuming, E_f= 35GPa, V_f = 0.4, E_m= 1.5GPa, V_m=0.6

Where E_f is the fabric elastic modulus (The fabric here is bidirectional and woven. The E_f of unidirectional fibres is 70 GPa and so for the same volume fraction of fibres, the elastic modulus is 35 GPa for a bidirectional plain weave with equal warp and weft along the 11 direction). V_f and Vm are the volume fraction of the fibre and matrix. E_m is the matrix elastic modulus. By rule of mixtures,

$E_{11}= E_f*V_f+ E_m*V_m$ = 14.9 GPa for the laminate in the present investigation. (4)

The strain energy release rate in Mode II is calculated either according to the maximum load criteria (i.e the maximum load in the load- deflection plot of the pre-cracked ILSS specimen) or the onset of non-linearity criteria (which is the load at the onset of non-linearity in the load-deflection plot of the pre-cracked ILSS specimen) .

3. Results

The average, maximum and minimum ILSS values for all the 8 set of parameters used in this investigation are shown in Figures 5 and 6. Out of the 48 samples that were tested for ILSS and Mode II strain energy release rates, all the samples were evaluated for their ILSS and only 6 samples showed Mode II delaminations that could be evaluated for strain energy release rates using the two aforesaid theories [10,11]. It is worth mentioning here that no previous published attempt has been recorded in the evaluation of mode 2 strain energy release rates from ILSS tests and hence the effort. The MWCNT samples with a higher width exhibited about 10 % higher ILSS values compared to those with a 1:2 thickness to width ratio. As the interlacing points of the fabric along the width are more for wider samples indicating more redistribution of shear stress along the width, it is likely that the wider MWCNT samples exhibit higher ILSS. The nanotubes in the midplane of a thick specimen probably contribute to more crack pinning, detour and blunting events, thereby providing an elementary understanding as to the noticeable increase in its ILSS values compared to the unfilled samples. An earlier investigation with graphite additions strengthens this view [12]. Teflon inserts do not seem to influence any of the ILSS values. The samples that failed in mode 2 delamination are listed below.

- MT-4: Sample with MWCNT and with Teflon insert, 1:5 thickness to width ratio
- M-4: Sample with MWCNT and no Teflon insert, 1:5 thickness to width ratio
- M-3: Sample with MWCNT and no Teflon insert, 1:5 thickness to width ratio
- t-5: Sample without MWCNT and with Teflon insert, 1:2 thickness to width ratio
- T-5: Sample without MWCNT and with Teflon insert, 1:5 thickness to width ratio
- t-1: Sample without MWCNT and with Teflon insert, 1:2 thickness to width ratio.

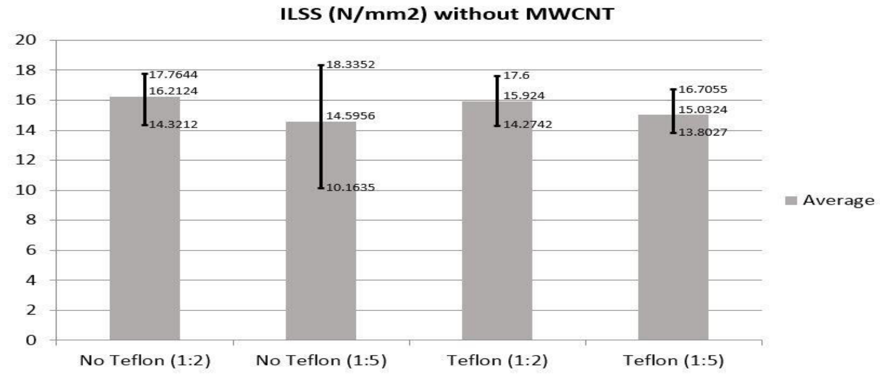

Fig.5. Inter laminar shear strength (in MPa) of samples without multiwalled carbon nanotubes

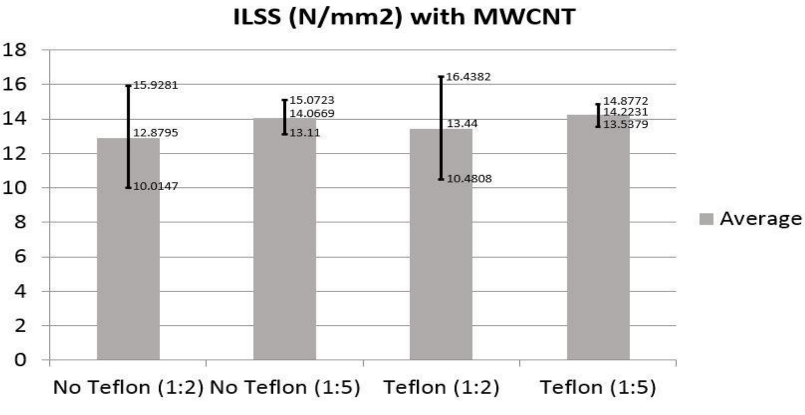

Fig.6. Inter laminar shear strength (in MPa) of samples with multiwalled carbon nanotubes

The values of strain energy release rates were calculated using equations 2 and 3 at the point of maximum load as well as at the point of onset of non-linearity in the load vs. deflection curves using both Russell's Theory [10] and Direct Beam Theory [11]. Figures 7-12 show the strain energy release rates evaluated using the different approaches. It is seen that the values are higher for the direct beam theory than Russell's theory by a factor of ~2. As there is no appreciable difference between the point of onset of non-linearity when compared to the maximum load in general for the glass/epoxy and the multiscale composite, the strain energy release rates for the onset method are always lower than the values from the maximum load method.

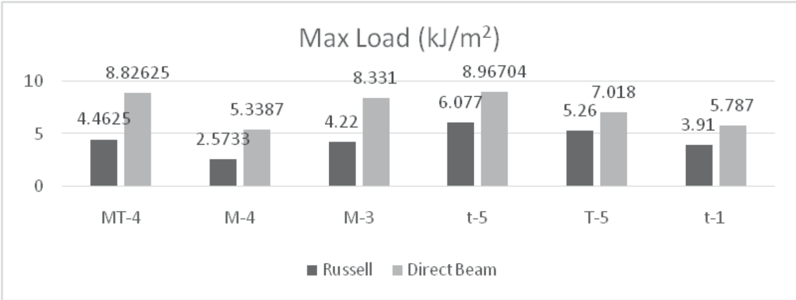

Fig.7. Mode 2 Strain Energy Release Rate values at Maximum Load

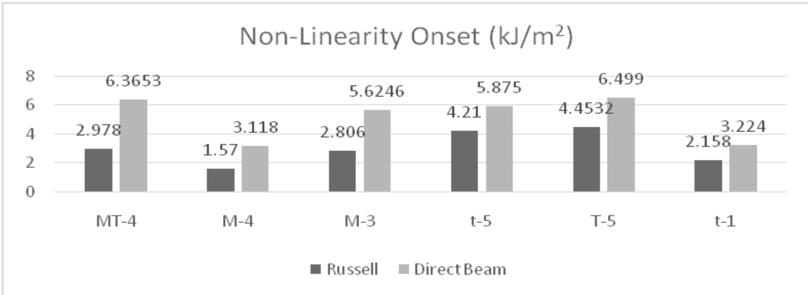

Fig. 8. Mode 2 Strain Energy Release Rate at Non-Linearity Onset

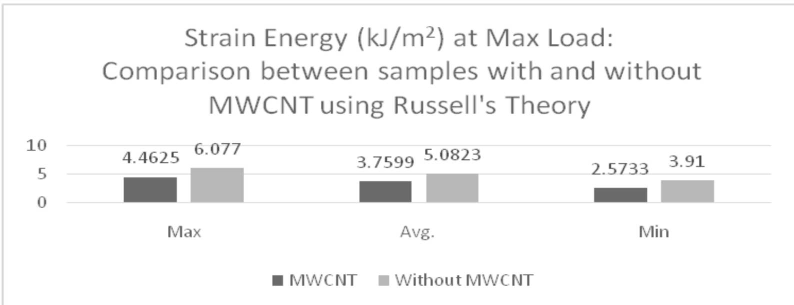

Fig. 9. Mode 2 Strain Energy Release Rate Comparison at Max Load using Russell's Theory

The strain energy values that have been calculated by using the direct beam theory are always more than the values that have been calculated by using Russell's theory as the former involves the terms span and crack length, both being smaller denominators compared to the E_{11} used in Russell's theory that does not take shear deformation into account for an approximated curvature . A careful analysis of the theories in question proves this argument. As the Russell's theory computes the strain energy release rate values keeping the P^2/E_{11} values in mind, small differences in the maximum load contribute to noticeable differences in the strain energy release rates for the samples with and with out MWCNT as both the elastic modulus and the load change with nanotube additions. The values obtained from the direct beam theory are identical for the two composites investigated because of closer loads and the term P and not P^2 appearing in the numerator. Though a pre-requisite for the ILS test, the shear term is not considered in the Russell's theory which is only meant for ENF tests at longer spans. In Table 1, we have used the strain energy release rate values that have been calculated using Russell's theory for ILS specimens and the percentage differences in the strain energy release rate values that were obtained from Ref [13] and Ref [14] for glass/epoxy ENF specimens.

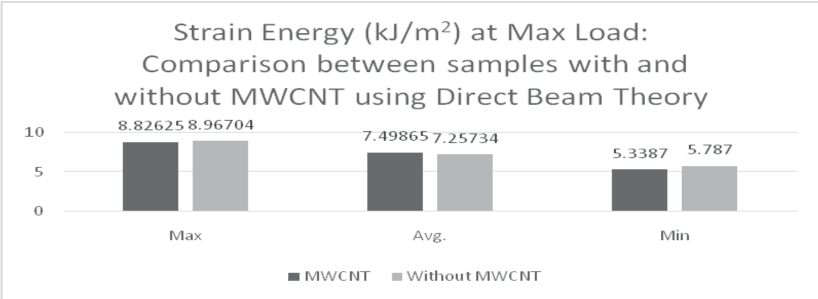

Fig. 10. Mode 2 Strain Energy Release Rate Comparison at Max Load using Direct Beam Theory

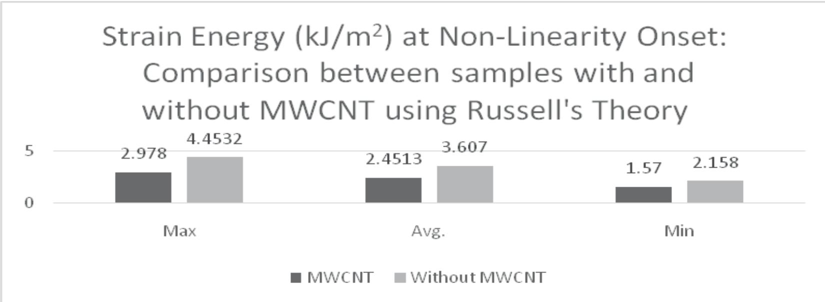

Fig. 11. Strain Energy Release Rate Comparison at Non-linearity Onset using Russell's Theory

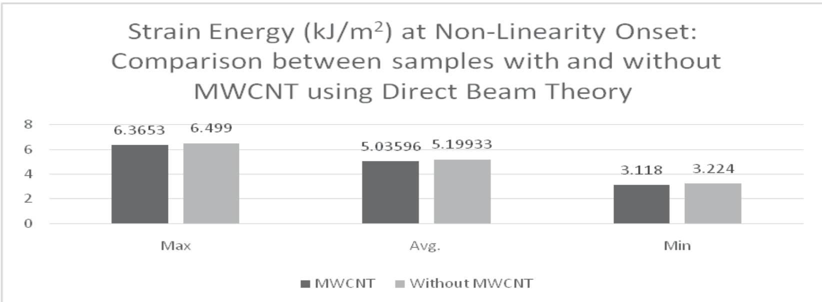

Fig. 12. Strain Energy Release Rate Comparisons at Non-Linearity Onset using Direct Beam Theory

Figures 13 (a) to (e) depict the micrographs of the failed ILSS specimens obtained through observation under an optical microscope. 13 (a) and (d) show the fracture at an interlacing point site due to load redistributions as discussed earlier. Figure 13 (b) shows a transverse crack running through the axial fibre reinforcements, due to specimen failure in ILS. Figure 13 (c) exhibits a much observed Mode 2 delamination between the layers. As ILSS tests were seen to produce multiple delaminations between the layers in thick samples as in the present case, the same is representatively shown in Figure 13 (f) as a macrograph of three different delaminated specimens. Figure 13 (e) reveals the resin fracture features of a layer surface that delaminated in ILS. The crack growth direction is quite visible along the axial reinforcement direction that also shows oblong resin fracture features transverse to the crack direction, indicating a stick slip type of failure at the resin rich zones which substantiates the view that the ILS failures are matrix dominated at a matrix volume fraction of 0.6 . It is proposed to optimize the ILSS test [5] and the specimens to evaluate Mode 2 strain energy release rates at appropriate spans, widths , number of layers and thicknesses in a more consistent manner and formulate a model for a more reliable evaluation of the same for the ILSS test as it demands a smaller specimen size and the required failure modes can also be more easily designed than the ENF test. The challenge lies in overcoming the difficulties faced in the ENF test on achieving a pure mode 2 shear and the required propagation characteristics of a mode 2 crack with out large deflections, curvatures and rotational moments routinely observed in laminated specimens with longer support spans as described in ref. [4,10,11].

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Table 1

A Comparison of Strain Energy Release Rate Values for ILSS , Ref. [13] and Ref. [14]

Sample	Russell	Paper [13]	% Diff.	Paper [14]	% Diff.
MT-4	4.4625	2.90576	34.88	2.5	43.97
M-4	2.5733	2.90576	-12.93	2.5	2.84
M-3	4.22	2.90576	31.14	2.5	40.75
t-5	6.077	2.90576	52.18	2.5	58.86
T-5	5.26	2.90576	44.75	2.5	52.47
t-1	3.91	2.90576	25.68	2.5	36.06

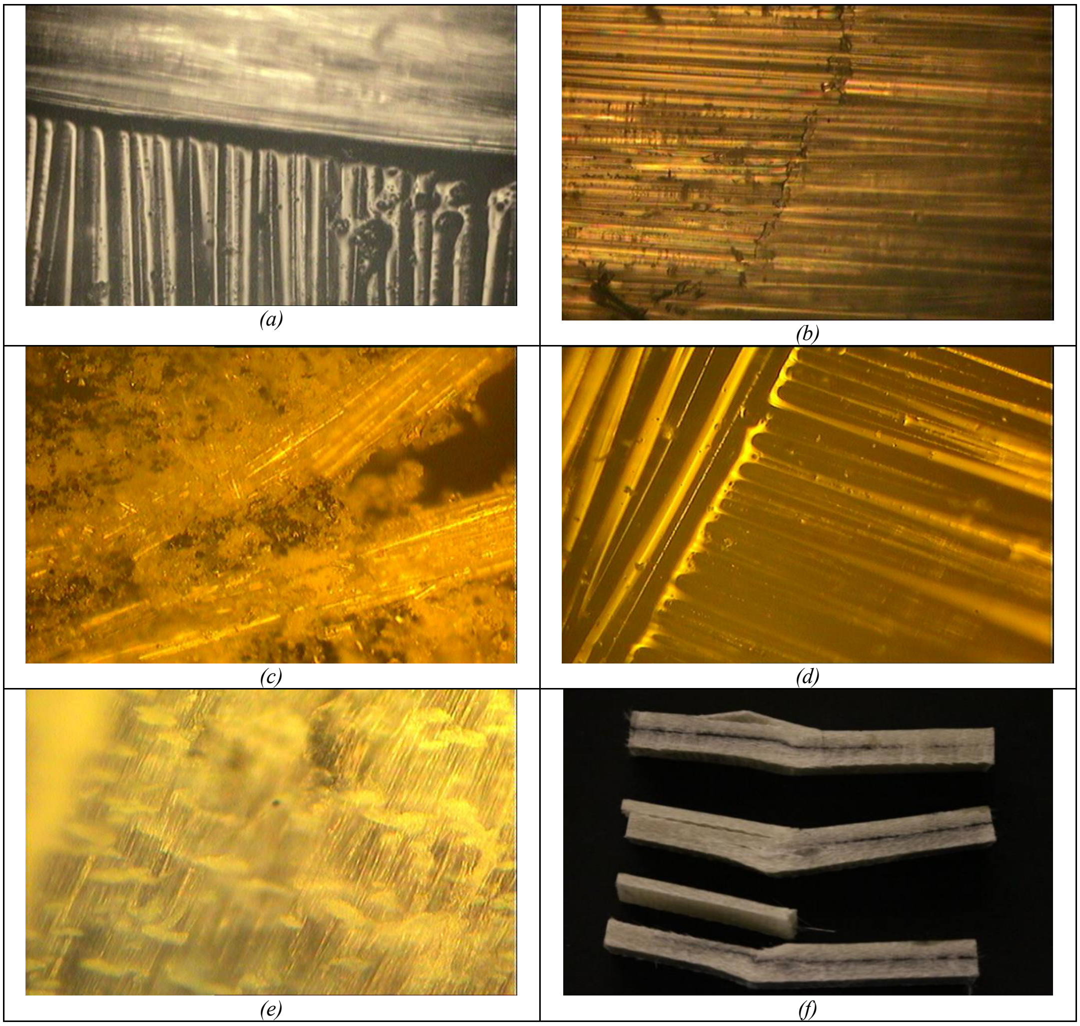

Fig. 13. Micrographs of Faiiled ILSS Specimens, (a) Specimen M4 (b) Specimen M4 (c) Specimen N1 (d) Specimen N4 (e) Specimen t1,

(f) Macrograph of failed IISS specimens

4. Conclusions

- Average inter laminar shear strength value of specimens with no MWCNT and a depth to width ratio of 1:2 is greater by 11.07% than that of the wider specimens. But, the case is reversed when MWCNT is added. The ILSS of the specimens with a width ratio of 1:5 is greater than that of the narrow specimens by ~ 9.22%.
- The strain energy release rate values are higher for the direct beam theory than Russell's theory by a factor of ~2. As there is no appreciable difference between the point of onset of non-linearity when compared to the maximum load in general for the glass/epoxy and the multiscale composite, the strain energy release rates for the onset method are always lower than the values from the maximum load method.
- The strain energy release rate values that have been calculated using Russell's theory for ILS specimens vary noticeably for the glass/epoxy and the MWCNT multiscale composite in comparison to the values obtained from the direct beam theory.
- Mathematical and fractographic evidence could be drawn to support our observations.

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