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Failure Mode Discrimination in Mode II Fracture of Glass/Epoxy Laminates Using Acoustic Emission Technique

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Abstract

The intention of this work is to discriminate the failure modes in Mode II fracture of glass/epoxy laminates on the basis of the frequency of the elastic waves emitted when the material is strained. Failure investigation was performed on end notched flexure (ENF) samples prepared with a Teflon insert in the mid-section to act as delamination initiator, by performing acoustic emission (AE) monitoring on four-layer glass/epoxy laminates with different fibre stacking sequences, namely [0/0/0/0], [0/90/90/0], [+45/-45/+45/-45], [0/+45/-45/90] and [90/+45/-45/0]. Unidirectional [0/0/0/0] and [0/0] laminates were also prepared as lap jointed samples and tested in shear. Moreover, pure epoxy resin specimens were tested in tensile and flexural loading to compare with composite laminates. AE monitoring by the analysis of mode II fracture was able to discriminate all the failure modes, including matrix cracks (longitudinal and transverse), interface failures, fibre micro-buckling and fibre fracture by the measurement of the different primary frequency ranges of AE signals. Considerations on the influence of fibre stacking sequence on the severity of particular failures and on the sequence of events over time leading to failure were also reported.

Introduction

Composite materials find their way from highly expensive aerospace industries to automobiles, boats, pipes and now even in our daily life due to technological improvements. Different types of matrix and reinforcing materials are in use, of which thermosets, such as epoxy, reinforced with glass or carbon fibres still particularly attract the industrial sector because of their high strength and stiffness to weight ratios. However, alongside the aforementioned advantages, composites are prone to delamination failure. Hence the investigation of the delamination damage behaviour and the knowledge of critical strain energy release rate values are of great importance [1]. Arumugam et

al. have detailed the failure modes in Mode I fracture of DCB in GFRP laminates based on the frequency content of various failure modes [2]. Ramirez-Jimenez et al. have identified the failure modes in glass polypropylene composites by clustering AE events using frequency method [3]. AE waveform parameters are used to investigate physical damage of the composite [4-8]. Oskouei et al. used wavelet based signal processing technique to detect the damage types during Mode I fracture of glass/polyester composites [9-10]. Aggelis et al. suggested that qualitative characteristics of the signals like the shape of the waveform would provide information on the type of cracks. For example, shear cracks which normally occur during tensile fracture, emit signals with longer rise time as well as lower average frequency [11]. Chen et al. showed that in flexure of unidirectional glass fibre-reinforced composites bending strength is dominated by composite shear strength and fibre/matrix adhesion [12]. Ireman et al. conducted delamination fracture experiments on double cantilever beam (DCB), end notch flexure (ENF), mixed mode bending (MMB), crack lap shear (CLS), single edge notch (SEN) with embedded artificial delaminations or impact damages and analysed energy release rate, stress-based failure criteria and damage models [13]. Kempf et al. analysed the influence of fibre orientation, the effect of fibre-matrix adhesion and resin fracture toughness on damage initiation and propagation [14]. Unnthorsson et al. presented an AE based failure criterion equivalent to the stiffness based criterion and endurance limit in fatigue [15]. Sung-Choong Woo et al. suggested that the direction of the main crack propagation in SEN laminated composites is affected by the fiber orientation and is irrespective of the initial notch direction [16]. Liu et al. studied the influence of different lay-up and hole sizes on the acoustic emission and the related failure properties with the acoustic emission signal features such as the energy, counting and amplitude [17]. Qing-Qing Ni et al. proved that the frequencies of AE signals remain unchanged irrespective of the propagation distance and suggested that the combined use

of Fast Fourier Transform (FFT) and wavelet transform (WT) could be a powerful tool for discriminating micro-failures and for elucidating the fracture mechanisms [18]. Cluster analysis is often used to discriminate various damage mechanisms [19]. In particular, Kordatos et al. analysed the relation between the fatigue limit of a ceramic matrix composite and acoustic emission data [20]. Investigation was done on the AE wave propagation and velocity in glass/epoxy composite materials to aid in the damage spot identification on the specimens. The results were compared with visually observed cracks and error was reduced as much as possible on the basis of the amplitude correction [21].

In the present work, delaminations are artificially induced by using Teflon inserts between the layers, so that mode II delamination test is carried out on laminates with different fibre stacking sequences using acoustic emission monitoring. Different types of failure modes are discriminated based on the frequency of the signals that are emitted during subsequent stages of loading and the dependence of certain failure modes on the orientation of fibre in the stacking sequence. The resistance offered at the interface of different fibre orientation is related to the absolute energy of acoustic emission signals and the number of events related to the interface failures and thus dependence of interlaminar fracture strength on fibre orientation is elucidated.

Materials and Methods

Materials

Unidirectional E-glass fibre cloth with mass density 270 g/m² is used as the reinforcing phase in the composite laminates, where epoxy Araldite resin (LY556) and hardener (HY951) formed the matrix phase. Fibre and epoxy were taken in equal mass proportion and amount of hardener taken is 10% by mass of epoxy resin, so that in the end glass fibre content was 47.5±0.5 wt.%. Hardener is added to epoxy and stirred well. Four-layered 300×300 mm GFRP composite laminates with fibre stacking sequences [0/0/0/0], [0/90/90/0], [+45/-45/+45/-45], [0/+45/-45/90] and [90/+45/-45/0] were fabricated by compression moulding technique. A non-adhesive insert (Teflon) of thickness of 13±1 microns was inserted in the mid-plane to serve as delamination initiator. The laminates were cured at room temperature for 24 hours. End notch flexure (ENF) specimens with dimensions 130×25×5 were cut from each laminate using water jet cutting process. On the edge of each specimen, a Teflon insert of length 30 mm and width 25 mm was disposed, as shown in Figure 1a.

Three-point flexural testing and tensile testing of pure resin samples

Pure epoxy resin specimens were prepared with dimensions 140×12.5×5 mm and were subjected to flexural loading through three-point fixture with a support span of 100 mm using a Tinius Olsen H100KU universal testing machine, with a 100 kN load cell. Tensile testing was carried out on pure epoxy resin specimens of dimensions 260×25×5 through tensile fixture with a gage length of 160 mm and 50 mm was given for gripping on both sides. Five samples were tested for each loading mode (flexural or tensile). Displacement control mode was applied throughout all testing. The crosshead speed was maintained at a rate of 1 mm/min for three-point bending and 0.5 mm/min for tensile testing.

Testing on GFRP laminates

Mode II delamination testing: In Mode II delamination test the specimens are subjected to bending load in transverse direction using Tinius Olsen H100KU Universal Testing Machine, with a 100 kN load cell as in Figure 1b. Five specimens per each stacking sequence were tested. Displacement control mode of testing is preferred as per ASTM D5528-01. The crosshead speed was maintained at a rate of 1mm/min. Crack initiation was identified by tracing marks at the side of the specimen. The loading is similar to simply supported beam with concentrated load at the centre. Teflon insert is at the neutral layer and the layers above the insert are subjected to compression and the layers below are subjected to tension. Shear force is maximal at the neutral layer. Due to this shear force sliding of the layers above and below the neutral layer occurs, which results in delamination.

Lap shear testing: Shear test was carried out on two types of laminates, in a number of five for each type. The first type consisted of single lap jointed unidirectional GFRP, as shown in Figure 2a, with a thick layer of epoxy resin entrapped at the joint. The fact that the laminate has a resin-rich area at the interface would suggest that its dominant failure mode is cohesive shear fracture rather than fibre/matrix debonding. The second type consisted of four-layered unidirectional GFRP laminates, as shown in Figure 2b were fabricated using compression moulding. These are single piece laminates with two notches and no external joint. In this case, a thin layer of epoxy is present at the interface, so to imply that fibre/matrix debonding would rather be dominant over cohesive shear fracture. These tests give the combined debonding and cohesive shear fracture strength of the laminate. In both cases, the load is carried by the small

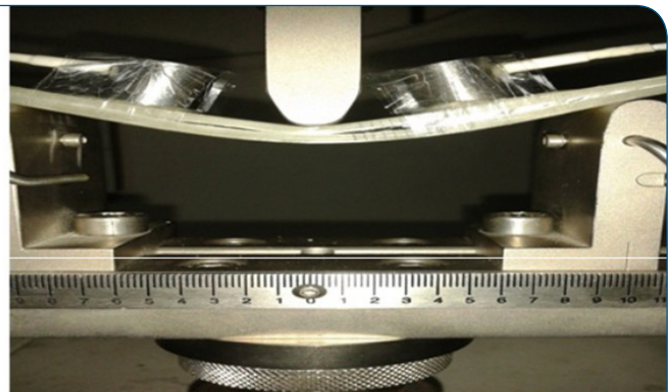
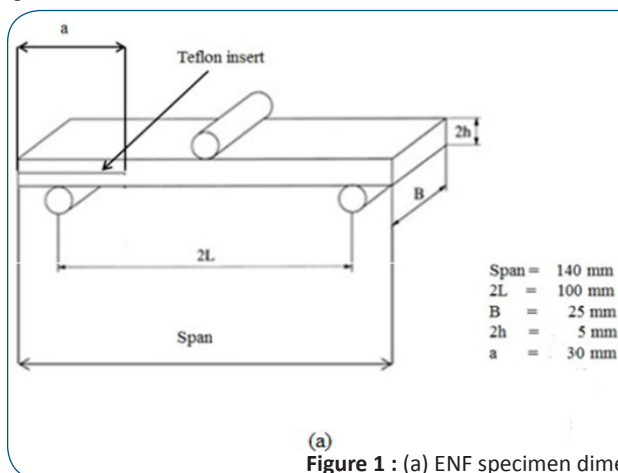


Figure 1 : (a) ENF specimen dimensions; (b) Mode II Delamination testing

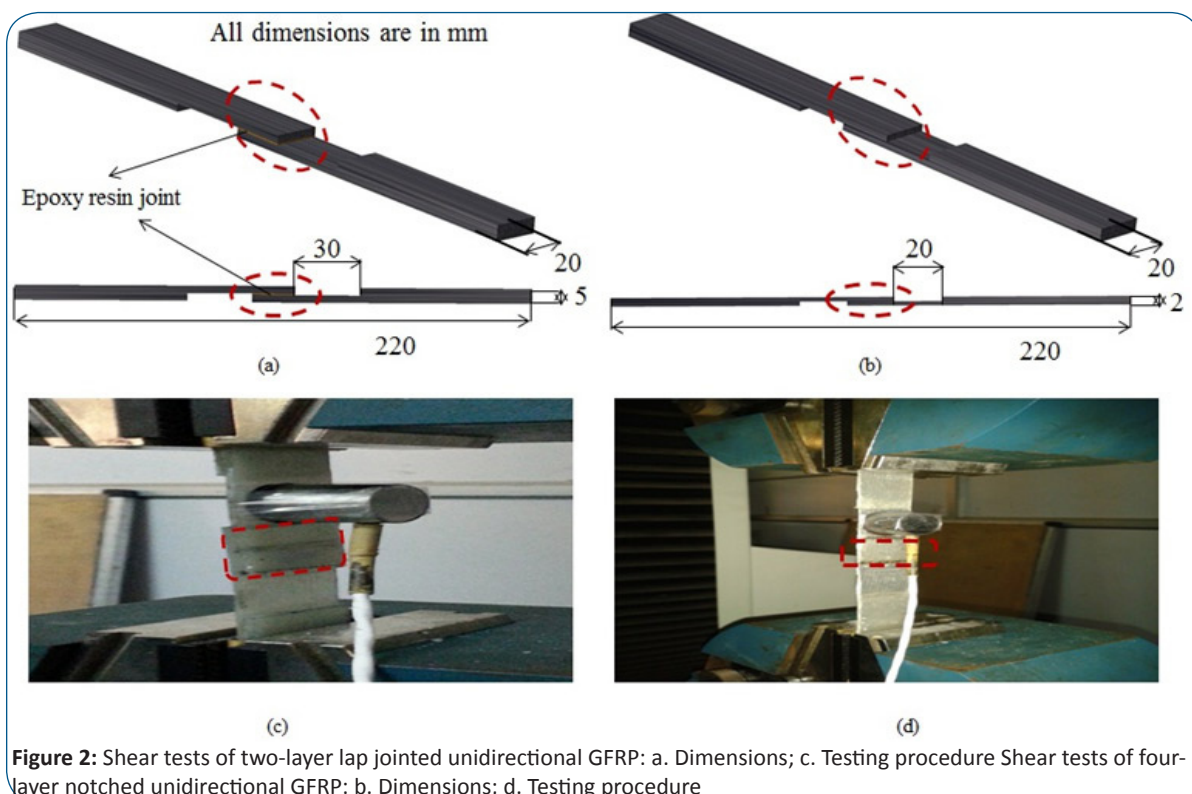


Figure 2: Shear tests of two-layer lap jointed unidirectional GFRP: a. Dimensions; c. Testing procedure Shear tests of four-layer notched unidirectional GFRP: b. Dimensions; d. Testing procedure

area rounded in Figure 2c and Figure 2d. Two acoustic emission sensors were applied on the specimens at a mutual distance of 80 mm. Tensile load was applied using Tinius Olsen H100KU universal testing machine, with a 100 kN load cell. The gage length was maintained as 140 mm and 40 mm was given for gripping. Load was applied in displacement control mode, with a crosshead speed of 0.5mm/min.

Acoustic emission (AE) monitoring

An eight channel AE system, supplied by Physical Acoustics Corporation (PAC), was used for the continuous monitoring of AE data, carried out specifically with a SAMOS E3.10 data acquisition system, again from PAC, with a sampling rate of 3 mega-samples per second (MSPS) and a 40 dB pre-amplification. The amplitude distribution covers the range of 0–100 dB (0 dB corresponds to 1 mV at the transducer output). Ambient noise was filtered using a threshold equal to 45 dB. On all flexural samples wideband sensors, with a quasi-flat response over the 100-900 kHz range, were applied at a mutual distance of 80 mm equidistant from the centre of the sample, while on tensile samples their mutual distance was 120 mm. In particular, attention was concentrated on the time and space localisation of the signals, on their primary frequency content and on cumulative variables accounted for during the tests, such as hits, counts and energy.

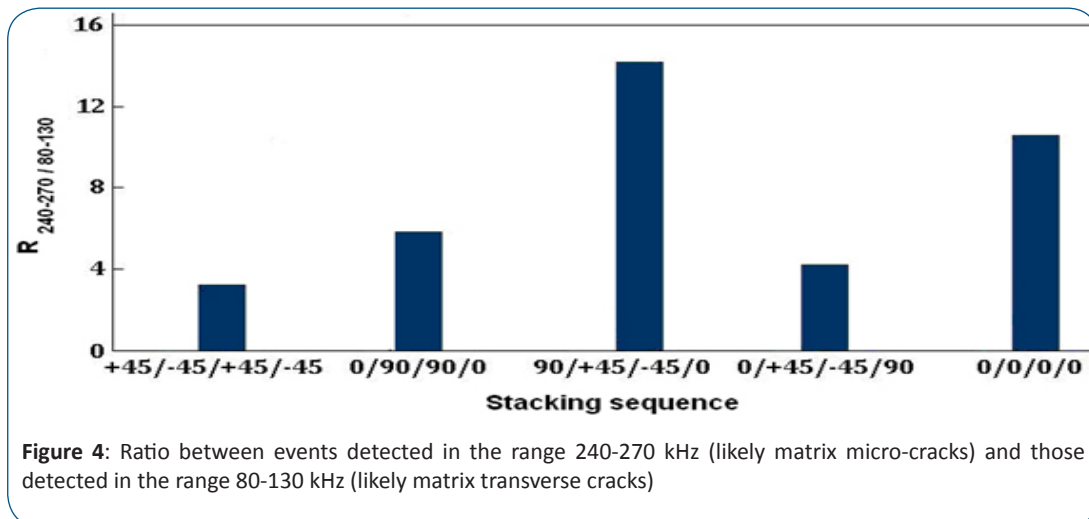
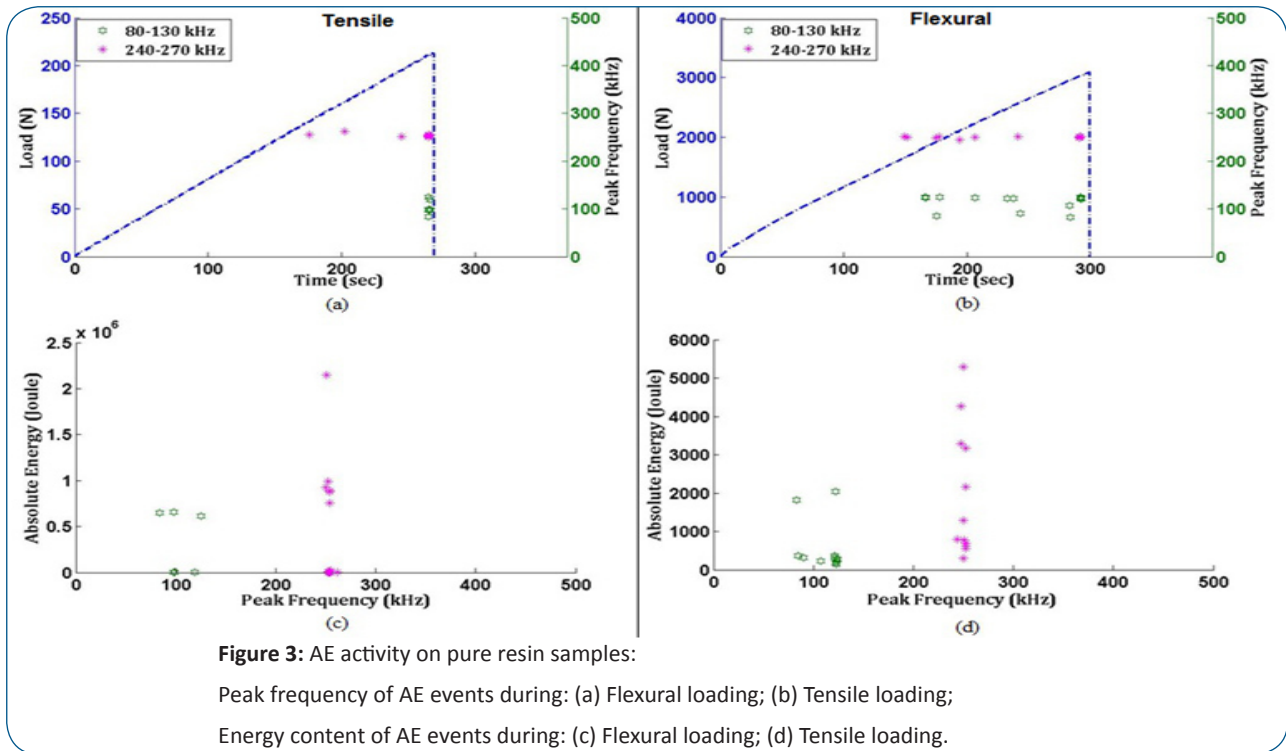
Results and Discussion

AE monitoring on pure resin samples during tensile and flexural tests

By investigating the frequencies of the signals from pure epoxy resin specimens it is clearly seen that events are exclusively concentrated in two frequency ranges: 80-130 kHz and 240-270 kHz, as shown in Figure 3a and Figure 3b, which are referred to flexural and tensile tests, respectively. As for the former frequency range, it has been widely recognised as primary frequencies of

events related to matrix cracking in the transverse direction, for example as the effect of fibres being pulled out, are concentrated in a range centred approximately on 100 kHz [22-23]. A closer observation of the results on pure matrix samples, considering also the energy values of the respective events, as in Figure 3c and 3d, for flexural and tensile loading, respectively, does clarify that in both cases higher energy values are revealed in the case of some 240-270 kHz events, with higher evidence in the case of tensile loading. It can be suggested that the absolute energy of matrix microcrack in the length direction is higher than the matrix transverse crack, which is also true for cracks originating in the tensile region of the flexural sample. So the failure modes occurring on pure matrix samples are identified as matrix micro crack in specimen length direction due to tension with frequency 240-270 kHz and matrix transverse crack with frequency 80-130 kHz in thickness direction.

The comparative analysis of the two matrix fracture modes has also been carried out on the different GFRP laminates produced, where obviously also other failure modes were present, as detailed further later. Figure 4 shows the ratio between matrix micro-cracks, for which signals in the 240-270 kHz range are detected, and matrix transverse cracks, which give rise instead to signals in the 80-130 kHz range, in glass fibre reinforced epoxy laminates with different fibre stacking sequence during ENF testing. It can be clearly seen that in all the laminates the occurrence of 80-130 kHz signals is lower compared to those in the 240-270 kHz range. It can be suggested that the presence of glass fibre in the laminate prevents the transverse cracking in the through thickness direction. This is further confirmed by the fact that laminates with 90 and 45 degree fibre orientation in the bottom layer show comparatively more matrix transverse crack because they allow crack to be propagated through the layer, which is not the case for 0 degree ply.



Failure modes in elastic and plastic region of end-notch flexure (ENF) laminates undergoing fracture

In general terms, it can be observed from Table 1 as end notch flexure tests provide results that are not very different for composites with the various stacking sequences under study, although the presence of +45 and -45 layers is clearly quite detrimental for the final performance, most likely as an effect of premature transverse cracking [24]. During the initial

Stacking sequence	Max. load (N)
0/90/90/0	670±25
90/+45/-45/0	515±30
0/+45/-45/90	550±20
+45/-45/+45/-45	405±25
0/0/0/0	640±55

Table 1: Max. flexural load (N) of different composite materials

stage of ENF samples loading, a clear proportionality between load and displacement can be observed. The departure from proportionality is typically indicated by an abrupt increase in AE cumulative energy, as can be observed in Figure 5. In particular, remaining AE events are divided in three further frequency ranges, in particular 140-190 kHz, 275-295 kHz and 300-320 kHz. When the shear stress in the interface crosses the critical limit rapid failure occurs along with large number of failure events and abrupt hike in absolute energy.

Frequency distribution of ENF laminates under Mode II loading

It is interesting to note that ENF laminates undergoing mode II loading present characteristic and repeatable AE activity as regards the frequency distribution of the events, which can be divided into five ranges, namely 80-130 kHz, 140-190 kHz, 240-270 kHz, 275-295 kHz and 300-320 kHz, the first and third of these, as from Section 3.1, do refer to longitudinal and transverse

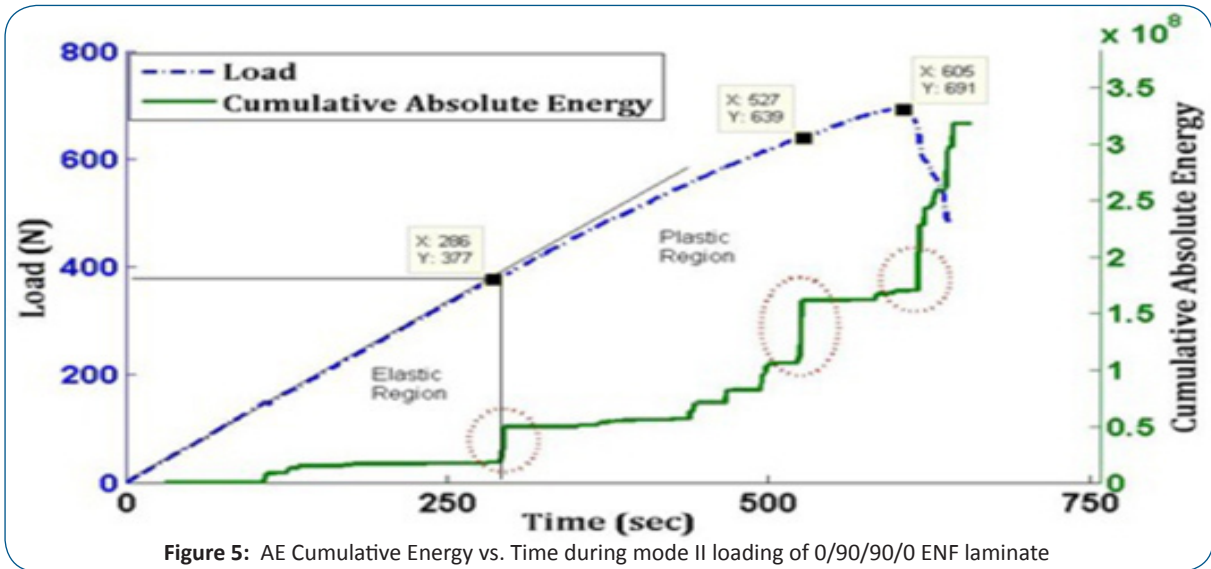


Figure 5: AE Cumulative Energy vs. Time during mode II loading of 0/90/90/0 ENF laminate

matrix cracking, respectively. As from data reported in Figure 6, within the proportional limit the material undergoes elastic strain with a lower number of matrix crack events occurring near the proportional limit. As far as the plastic region is concerned initial stages are dominated by matrix cracking failures and then in course of time accompanied by other failures. The other three frequency ranges will be identified via the considerations developed in the following sections.

Identification of fibre micro-buckling mode

The occurrence of fibre micro-buckling has been recognised as the effect of shear instability in fiberglass, which is very detrimental for its performance under out-of-plane loading [25]. This is likely to occur on the compressed side of the laminate, hence is influenced by fibre orientation of top layers. In mode II loading, the likeliness of fibres in the top layers to buckle would strongly depend on stacking sequence: in practice, with 0 and 45

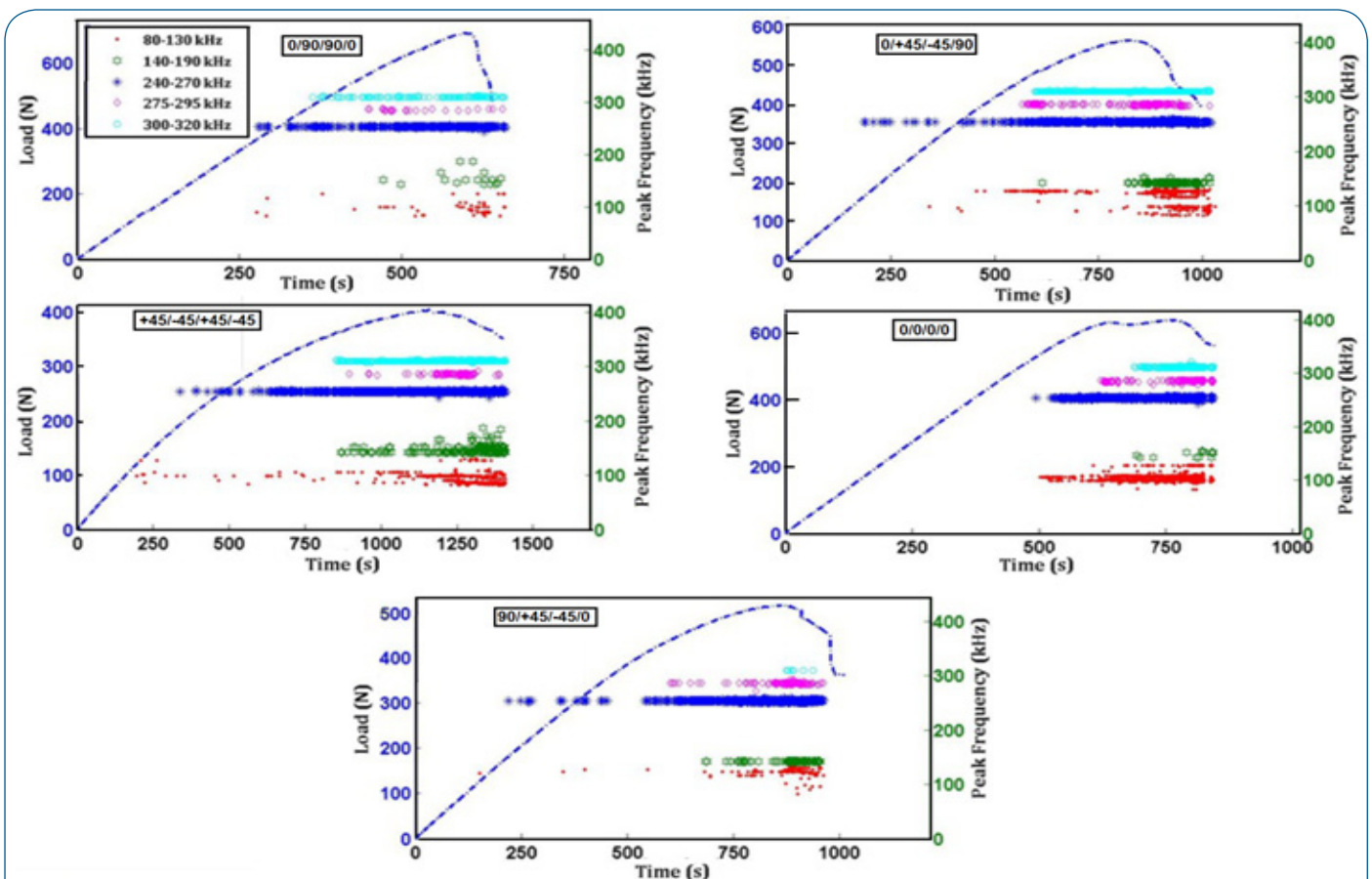


Figure 6: Load vs. Time with AE Peak frequency of laminates with different fibre stacking sequences under Mode II Loading

degree ply on top buckling will be higher and with 90 degree on top buckling is very much unlikely to occur. It has been noticed that AE events with frequency range between 300 and 320 kHz, although found in all laminates, have an occurrence that varies considerably with different fibre stacking sequences. The number of AE events with primary frequency in the range between 300 and 320 kHz in various laminates with different fibre stacking sequences is shown in Figure 7. There is a very significant difference in the number of fevents between 0/+45/-45/90 and 90/+45/-45/0 laminates, in which the top layers are 0 and 90 degree ply, respectively. Therefore this range, often associated with fibre failure, also in studies on carbon fibre composites [26], might be correlated to a considerable extent to breakage occurring as an effect of fibre micro-buckling.

A further confirmation of this comes from AE frequency distribution in lap shear testing of GFRP laminates, which is shown in Figure 8. The 300-320 kHz frequency range, which is available in Mode II fracture of ENF is missing in this case, whereas here frequency range between 350 and 380 kHz is present in shear testing. Under tension load there are no possibilities for fibre to buckle and that is why 300-320 kHz did not occur in shear test, while in contrast the fibre could break under tension load. Moreover, it was described already in [2] that frequency range 350-380 kHz might be related to fibre breakage, hence 300-320 kHz is to be ascribed to micro-buckling.

Discrimination of interface failures

Interface failures may obviously occur only if there is more

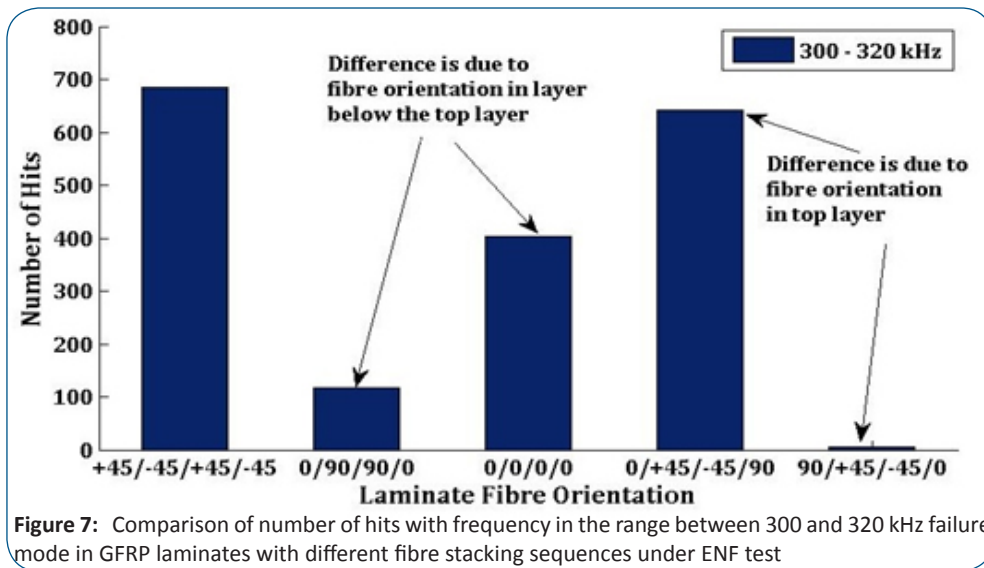


Figure 7: Comparison of number of hits with frequency in the range between 300 and 320 kHz failure mode in GFRP laminates with different fibre stacking sequences under ENF test

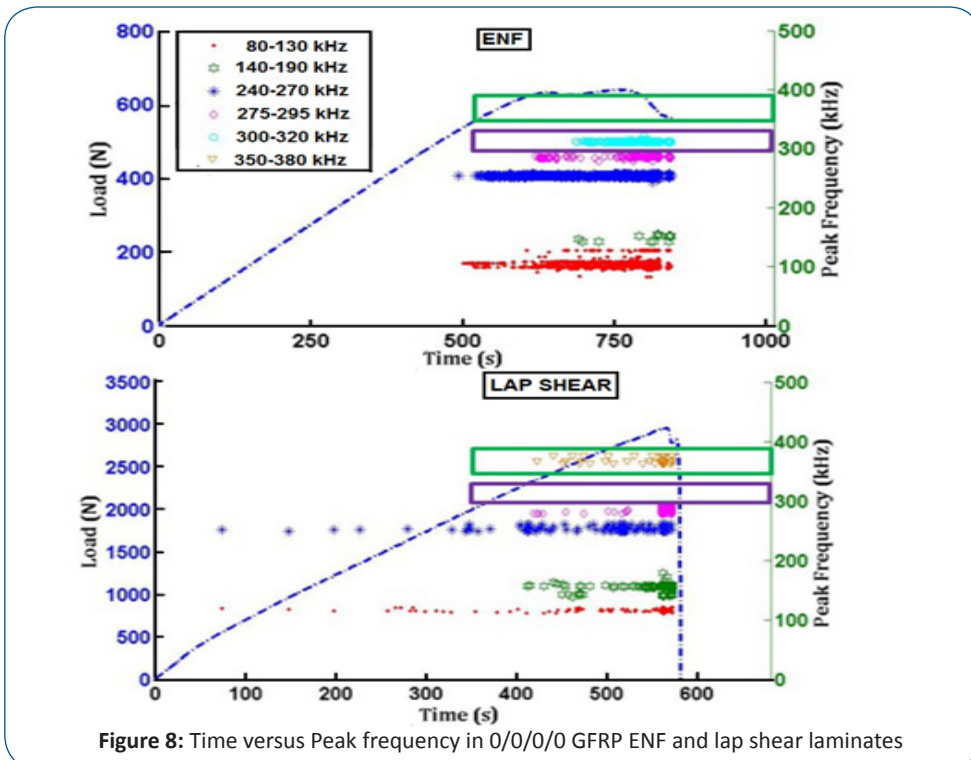


Figure 8: Time versus Peak frequency in 0/0/0/0 GFRP ENF and lap shear laminates

than one layer. AE events detected in both shear and mode II delamination testing of ENF GFRP laminates, have frequencies in two further frequency ranges, 140-190 kHz and 275-295 kHz: however, events in these frequency ranges were not detected during flexural loading of pure resin laminates. So these frequency ranges might be likely related to interface failures, in particular fibre/matrix debonding and cohesive shear fracture. The distribution of frequency content of events at different location of ENF specimen under Mode II loading is shown in Figure 9.

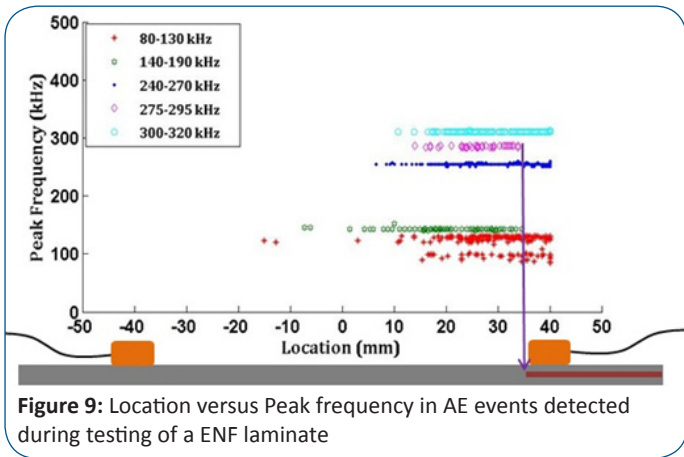


Figure 9: Location versus Peak frequency in AE events detected during testing of a ENF laminate

It can be seen that events in the frequency ranges 140-190 kHz and 275-295 kHz are not present in the region where Teflon is inserted as delamination initiator in the interface, even though it is within the field of acoustic emission sensor. This suggests that those frequencies might be related to interface failures fibre/matrix debonding and cohesive shear fracture. Since in this region the interface is already separated, there is no possibility

of interface failures. In [2], it was detailed that failure modes occurring in Mode I delamination i.e., fibre/matrix debonding and fibre pullout failure are most likely to occur in 150-240 kHz frequency range.

The view of failed interface of resin rich lap jointed GFRP specimen and resin lean GFRP shear specimen are shown in Figure 10. It can be seen from Figure 10b that in resin lean shear specimen a lot of fibre is exposed than in resin rich lap jointed specimen in Figure 10a. This is due to the fact that in resin lean shear specimen the fracture crack propagates through the fibre-matrix interface leaving the fibre exposed. But in resin rich lap jointed specimen the fracture crack propagates mostly through the matrix that trapped between the interface layers which is why no fibre is exposed to the surface. So if comparison is done on the number of failures between 140-190 kHz and 275-295 kHz it might give clear information.

The number of AE events with primary frequency in the ranges 140-190 kHz and 275-295 kHz in resin lean shear specimen of GFRP laminates and resin rich lap jointed GFRP laminates is shown in Figure 11. It can be seen that in resin lean shear laminate the number of events in the frequency range 140-190 kHz is higher than those in the range 275-295 kHz, whereas the opposite is true in resin rich lap joints. This might happen since in lap joints cohesive shear fracture is more likely to occur than fibre/matrix debonding due to the thick layer of resin that allows the fracture crack to be propagated with-in the matrix. So events with frequency in the range between 140 and 190 kHz might be related to fibre/matrix debonding and those in the range between 275 and 295 kHz might be related to cohesive shear fracture. Thus, all failures that were involved in Mode II fracture are discriminated.

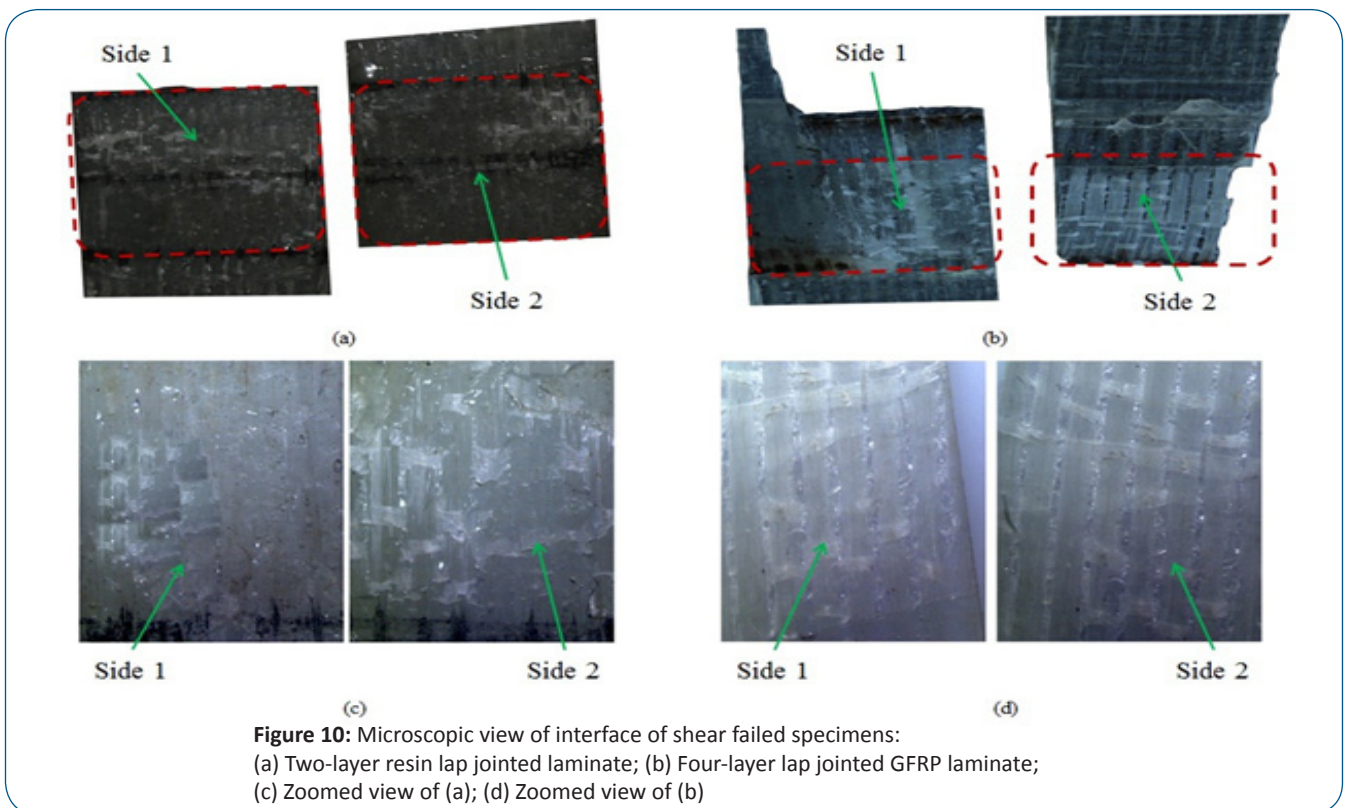


Figure 10: Microscopic view of interface of shear failed specimens: (a) Two-layer resin lap jointed laminate; (b) Four-layer lap jointed GFRP laminate; (c) Zoomed view of (a); (d) Zoomed view of (b)

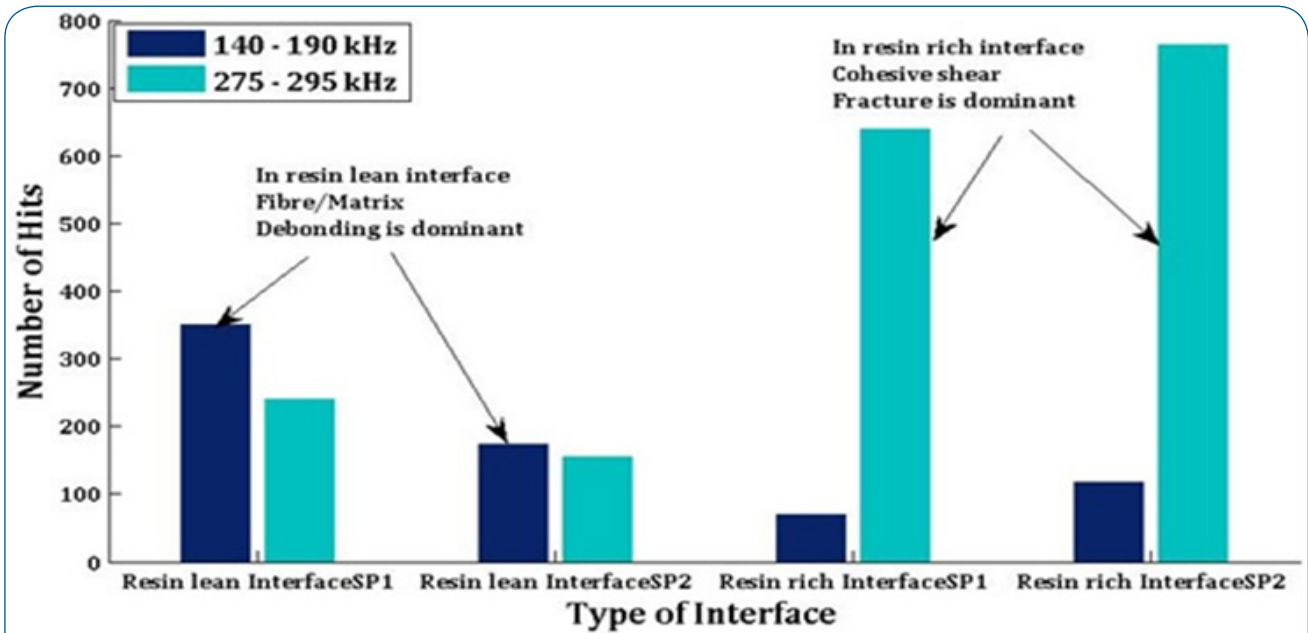


Figure 11: Comparison of number of AE events with frequency in the ranges 140-190 kHz and 275-295 kHz in two resin-lean interface and two resin-rich interface lap jointed laminates, tested in shear

Now, the delamination resistance offered by the interfaces with different fibre orientation has to be investigated to find the best possibility of fibre orientation for better delamination resistance. The shear stress resistance in laminates with different stacking sequences are shown in Figure 12. 0/0/0/0 shows good

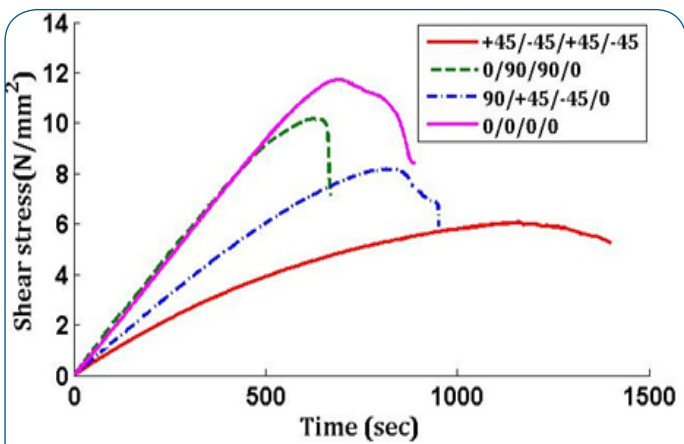


Figure 12: Shear stress curves vs. time for GFRP laminates with different fibre stacking sequence

shear resistance followed by 0/90/90/0, 90/+45/-45/0 and +45/-45/+45/-45 respectively. However, looking at the time taken for ultimate failure, 0/0/0/0 and 0/90/90/0 failed quite abruptly compared to other laminates. This can be attributed to the fact that displacement control mode of testing was applied and that the amount of fibres in the loading direction is substantial. In this loading mode, displacement varies linearly with time but load carried by the laminate depends on the stiffness of the laminate. 0/0/0/0 is stiffer because of the four 0 degree plies and then also 0/90/90/0 is rather stiff, because of the two 0 degree plies and hence they carry more load in short time and will fail quickly. Shear stress is calculated as below:

$$\text{Shear stress} = \frac{F \times A \times \dot{\gamma}}{l \times b}$$

The absolute energy of interface failures which occurred during Mode II fracture is reported in Figure 13a, showing from respective AE events frequencies that fibre/matrix debonding contributes much more than cohesive fracture to the delamination resistance in all laminates. In addition, 0/0/0/0 laminates released more energy before failure thus offered

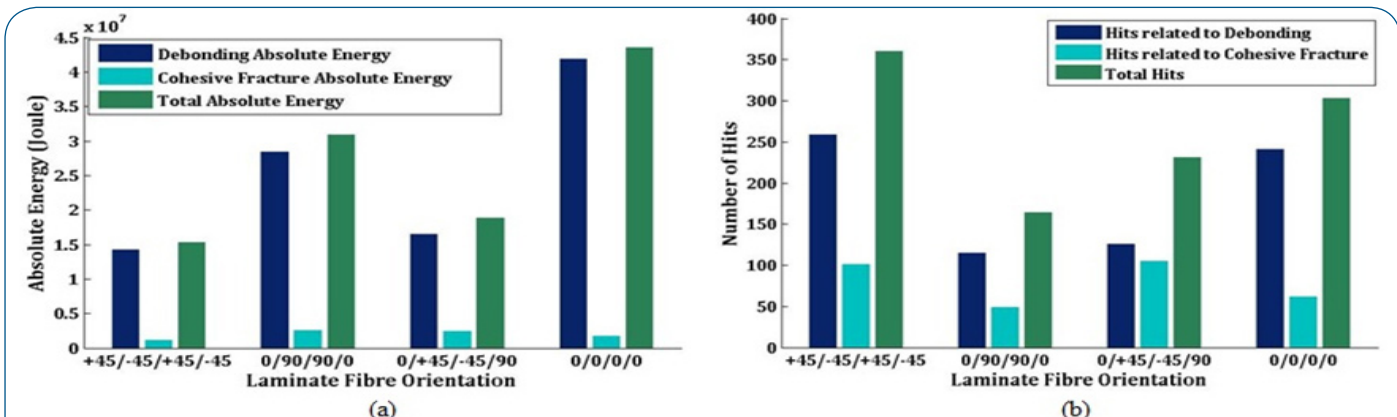


Figure 13: Comparison of: (a) AE energy and, (b) Number of events likely to be attributed to fibre/matrix debonding (140-190 kHz) and cohesive fracture (275-295 kHz) of GFRP laminates

good resistance followed by 0/90/90/0. In contrast, laminates 90/+45/-45/0 and +45/-45/+45/-45 offered more or less the same resistance, suggesting that their interfacial strength is around the same level. In general terms, this indicated that interlaminar fracture resistance depends on the interface fibre orientation. In particular, even though 90/+45/-45/0 and +45/-45/+45/-45 have same fibre orientation in the middle layers, 90/+45/-45/0 shows higher shear stress than +45/-45/+45/-45. This is explained by the fact that 0 ply in 90/+45/-45/0 imparts a higher rigidity to the laminate, though in general terms it leads to precocious failure with respect to +45/-45/+45/-45. Figure 13b shows the number of failure events corresponding to fibre/matrix debonding and cohesive fracture in different laminates. In this case, the ultimate cumulative energy released by 90/+45/-45/0 and +45/-45/+45/-45 are nearly equal, but the number of failure events are much higher in +45/-45/+45/-45 than in 90/+45/-45/0. It is suggested therefore that 90/+45/-45/0 carries more load, while collapsing more abruptly. During final fracture, a lower number of AE events with high energy are detected. Quite to the contrary, +45/-45/+45/-45 laminate carries a lower amount of load for a longer period of time. As a result, a large number of AE events with low energy are in contrast detected.

Conclusions

Failure events occurring during Mode II fracture of E-glass/epoxy laminates are discriminated on the basis of the frequency content of acoustic emission events emitted during fracture. In particular, acoustic emission is seen as able to discriminate between matrix transverse and longitudinal cracks, therefore being useful to investigate the effect of stacking sequence on the relative significance of the different failure modes. In particular, for example, fibre micro-buckling failure was clearly minimised by having 90 degree plies on top layers of laminates. Also interlaminar fracture resistance was proved depending on the fibre orientation of interface layers with 0 degrees plies offering the highest interlaminar fracture resistance. More in general, interlaminar fracture resistance is offered by the combined contribution of bonding between fibre-matrix interface and cohesive strength of the matrix.

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