

## Effect of Multiple Impacts on GFRP Laminates Exposed to Hydrolytic Ageing using Acoustic Emission Monitoring

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### ABSTRACT:

*In this study, pristine, single impacted and double impacted GFRP specimens were subjected to ageing by immersion in seawater at ambient temperature for different periods of time. The dominating failure mode of the aged specimens was found using acoustic emission monitoring. The water absorption capacity and flexural strength of specimen subjected to ageing were assessed to study the trend of variation. A reduction in residual strength was observed between single and double impacted specimens. Stress cycle plots from AE monitoring indicate that the onset of failure occurs sooner as the ageing time progresses. From those of impacted specimens, it can be seen that the onset of failure and damage initiation occurs sooner in double-impacted specimens than in single-impacted counterparts. Parametric peak frequency analysis shows that fiber-matrix debonding was the predominant mode of failure.*

### KEYWORDS:

*GFRP; Epoxy resin; Drop weight impact; Seawater degradation; Acoustic emission monitoring*

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## 1. Introduction

Glass Fiber Reinforced Plastics (GFRP) laminated composites are widely used in ships, boats and underwater vehicles. Polymeric matrix composites differ from other materials in which water may easily migrate even at room temperature, generating a variation of the material's structure, morphology, and composition. This phenomenon occurs only in the matrix or at the fiber-matrix interface. In many cases, it could lead to an irreversible degradation of the material in the humid aging that includes both chemical aging and physical aging. The epoxy matrices show moisture sensitivity due to interactions between some polar groups of the macromolecule and the water molecules, which leads to a reduction of both glass transitions temperature and mechanical properties. This sensitivity increases with the increasing degree of cross-linking, and also with the polarity and concentration of the molecular groups.

For a given temperature and a given ageing time, mechanisms other than simple diffusion can take place within the material such as:

- (1) Hydrolysis of the macromolecular chains, which leads to the formation and migration of low molecular weight chains;
- (2) Hydrophilisation, which tends to increase the equilibrium water uptake through the development

of local and/or overall swelling and or softening of the matrix;

- (3) Interfacial de-cohesion, which induces a degradation of the composite.

In the case of epoxy matrix composites, the ageing mechanisms fall into first two groups.

Visco et al. [1] evidenced that the diffusion through micro-cracks, pores, defects in the material is better than capillary flow of water molecules along the fibre/matrix interface. They noticed that the mechanical degradation of polyester resins was not provoked only by hydrolytic ageing but mainly by crack and blister formation. Therefore, the knowledge of water diffusion mechanisms and their effect upon mechanical performance into the advanced composites is significant in order to simplify the material design and to improve short and long term durability of the material. Among epoxy polymers, aromatic amine-cured epoxies show the highest sensitivity to ageing.

In order to study the effect of water absorption in the composite, the following steps are necessary [2]:

- (a) Identify the reversible or irreversible mechanisms through physical and analytical observations;
- (b) Establish a kinetic model based on the previously established mechanics scheme;
- (c) Predict the properties from the structural state using polymer physics or other tools.

The generally accepted mechanism for water in polymers is an activated absorption-diffusion process.

Lee and Peppas [3] gave an excellent review of the mechanism involved in the water transport in epoxies. The molecules first dissolve into the polymer surface and then diffuse through the bulk of the polymer by a series of activated steps. Apicella et al. [4] suggested that there are three absorption modes as follows:

- (1) Bulk dissolution of water in the polymer network;
- (2) Moisture absorption onto the surface of the excess free volume elements;
- (3) Hydrogen bonding between polymer hydrophilic groups and water.

Adamson [5] postulated that the transport of moisture below the glass transition temperature,  $T_g$ , is a three-step process in which the water first occupies the free volume voids, then becomes bound to the network sites causing swelling and finally enters the densely cross-linked regions. Barrie et al. [6] included the possibility of clustering of water molecules at high moisture levels. It has also been proposed that the equilibrium water concentration is mainly governed by the available free volume or that water molecules occupy micro voids and other defects [5, 7 and 8].

Previous studies show that water molecules are linked by strong hydrogen bonds to hydrophilic groups such as hydroxyl and amine groups. Another study shows that water neither appears to be bound to polar groups nor stay as free water but instead forms clusters. Water absorption can be modelled by the superposition of the Flory-Huggins or Henry's law that gives the diluents concentration in terms of the water activity for the polymer diluents solution and Langmuir expressions for hydrogen bond formation and absorption in holes. This absorption process generates softening of the glass transition temperature and a reduction of the mechanical properties [9-10]. The various absorption modes of a glassy polymer greatly influence the kinetics of absorption. Glassy polymers exhibit complex mass transfer behaviour. Both concentration gradient-controlled diffusion and relaxation-controlled swelling contribute to the rate and extent of penetrated absorption.

The physical phenomena that simultaneously occur are dissolution, diffusion, swelling and relaxation, together with deformation and stress build-up in the matrix. At each immersion temperature, two parts can be distinguished on the absorption curves as follows:

- (a) At shorter times, the moisture content increases linearly first, and then reaches a pseudo-equilibrium state. The water uptakes at this pseudo-equilibrium state are approximately the same whatever the immersion temperatures being considered. Such features are characteristic of a thermally activated Fickian absorption behaviour;
- (b) At longer times, a slow positive deviation from the Fickian pseudo-equilibrium state occurred. These non-Fickian water uptakes increase with immersion temperature. Such continuous slow absorption processes have been reported by numerous authors, and can be attributed either to slow relaxation processes of the glassy epoxy network or to the filling of hygro-thermally induced voids.

Only one surface of each composite specimen is exposed to seawater. This exposed surface is subjected to impact testing under different conditions [11]. A series of experiments on dry and wet composite specimens were conducted to characterize the impact damage, and the residual compressive strength. Results from seawater exposure indicate that the compression-after-impact strengths of the wet specimens reduced by around 10% compared to the baseline dry specimens over 29-month seawater exposure. Therefore, durability of current marine composite is better than the previously measured properties. Our simple seawater tank durability experiment is expected to provide accurate justification for the required maintenance period of new composite ship structures [12]. Due to low fibre-matrix adhesion, the prevailing failure modes at low impact energy were fibre/matrix debonding and interfacial cracking. The compression strength suffered significant reductions with water absorption (28%) and impact (max. 42%).

Composites consisting of a flexibilised epoxy resin, reinforced with woven glass, carbon and polyester fibers, respectively, were soaked in seawater for 3 months at 10°C and at 20°C. All composites experienced an increase in mass, a reduction in the effective flexural modulus and a corresponding increase in flexural loss factor. The carbon fibre reinforced material was found to be the least affected [15]. The absorption resistance of the composites also depends upon polyester resin used in composite manufacturing. Isophthalic resin bonded well to glass fibres so that seawater absorption resistance, flexural stiffness and strength, as well as shear strength was higher than that of orthophthalic one [1]. Water uptake behaviour has been compared for the polyester, phenolic and vinyl ester GFRPs and neat resin castings. The phenolic GFRP displayed anomalous uptake behaviour and loss of flexural strength [13]. To create a better understanding of the initiation, growth and interaction of the different types of damage, damage monitoring during mechanical loading is very important. Acoustic emission (AE) is the only non-destructive test technique capable of detecting all of the above mentioned damage types in composites [14].

This paper bring out the results and analysis of reduction in flexural strength of the GFRP composite laminates when immersed in seawater by the characterization of water absorption quantity of the material and the change in the flexural properties of the impacted laminates as compared to pristine laminate.

## 2. Experimental procedure

Laminates were fabricated from E-Glass (300 gsm) and epoxy resins supplied by Mark tech Composites Pvt. Laminates consist of 16 symmetrical bidirectional plies of fabric impregnated with epoxy resin by sealant-vacuum bagging at 1 atmospheric pressure for 2 hours, curing at room temperature for 8 hours and subsequent post-curing at 100°C for 2 hours to get the laminates. The specimens were cut using water jet cutting. A total of 16 layers were used to get a thickness of  $3.4 \pm 0.2$  mm. The dimension of the specimen is 120 mm x 20 mm for flexural testing as per ASTM D790. To investigate the effect of impact damage on moisture absorption, i.e. the

acceleration of moisture absorption due to pre- induced impact damage, it is necessary to avoid coating on the specimen. Moreover impact damage may remove the coating from the site of the impact when compared to other places which may give misleading results.

Specimens were impacted in the FRACTOVIS PLUS 7526 impact machine as shown in Fig. 1. The impact velocity and offset distance was maintained at 1.5 m/s and 300 mm respectively. The impact temperature (29°C) and the energy (2.167 J) was maintained constant throughout the test. The CEAST data acquisition system of the machine acquires the impact parameters such as peak force, impact energy and deformation instantaneously so that the dynamic characteristics of the impact event were obtained. The pristine, single impacted and double impacted specimens were immersed in a container with seawater at room temperature for 100 days as shown in Fig. 2. Regular weight gain measurements were carried out. Water uptake was calculated as weight gains related to the weight of the dry specimen. Four composite specimens were tested and results are averaged.



Fig. 1: Fractovis Plus drop weight impact setup



Fig. 2: GFRP specimens immersed in seawater bath

Three point bending test was conducted at 0.25 mm/min to characterize the mechanical properties of laminates in the Tinius Olsen 100 kN Universal Testing Machine as shown in Fig. 3. AE characteristic was used to estimate the contribution of each failure mode towards the overall failure and also to find the predominant one

amongst them. Modal analysis was carried out as a preliminary step to determine the number of peak frequency clusters present in the AE data after which a MATLAB code was used to form separate clusters for each mode of failure.

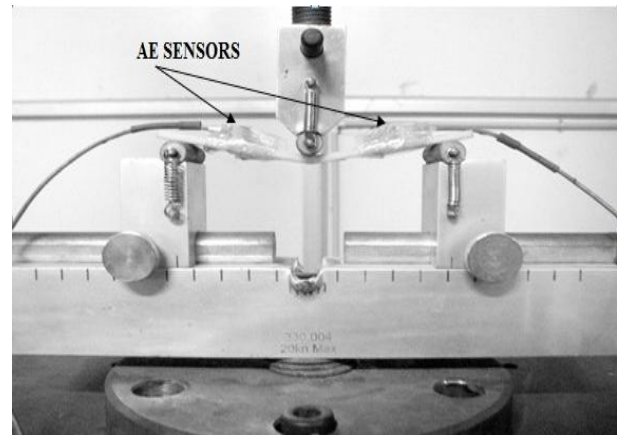


Fig. 3: GFRP specimen under static flexural test

### 3. Result and discussions

The hygroscopic response of the composite specimen was measured in terms of parameters like coefficient of diffusion ( $\text{mm}^2/\text{s}$ ) which is the rate of transport of water molecules into the composite [15], loss factor ( $\eta$ ) [16], weight gain [17] and relative absorbance change of water band ( $\Delta A^{\text{FTIR}}$ ) [18]. Nevertheless, in this study, water uptake of the material was characterized by percentage of weight gain ( $M$ ) of the material as a function of time. The specimens were weighed and checked for increase in mass from their dry mass which was recorded progressively with time. It was assumed that the initial moisture content of the specimens was zero and that the increase in mass was solely due to the intake of water so that the percentage increase in mass of the specimens could comfortably be taken as their water absorption capacity.

#### 3.1. Effect of impact on water absorption

The internal damage generated by a low-velocity impact on a composite plate is shown in Fig. 4. The impact can cause a significant reduction in the strength of the material, without creating any obvious damage on the impacted surface.

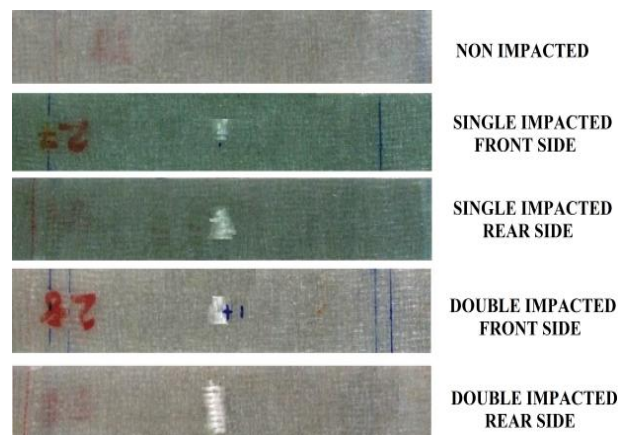


Fig. 4: Severity of single and double impact on GFRP specimens

The material gets more porous after every impact which serves as a passage to the entry of water into the composite. This is the reason why low-velocity impact attracts a great deal of attention in damage formation. Figs. 5 (a) and (b) show the contact force vs. deformation and energy vs. time response of GFRP specimens subjected to single and double impacts. It was evident from the results that the absorbed energy and permanent deformation increases as the number of impact progresses. As expected the double impacted specimens retained more amount of damage when compared to the single impacted specimens. A well-known fact that can be proved from Fig. 6 is that the flexural strength decrease on impact and the residual strength reduces further with an increase in the number of impacts.

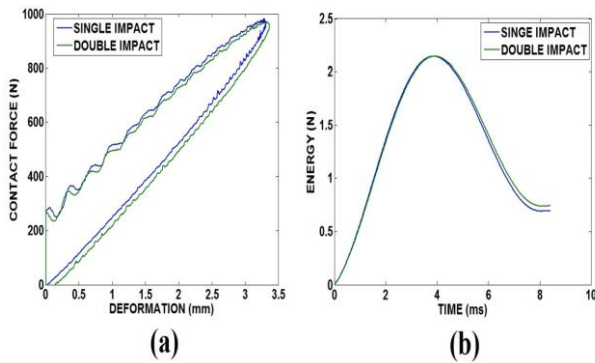


Fig. 5: (a) Contact force vs. Deformation and (b) Energy vs. Time response of GFRP specimens subjected to repeated impacts

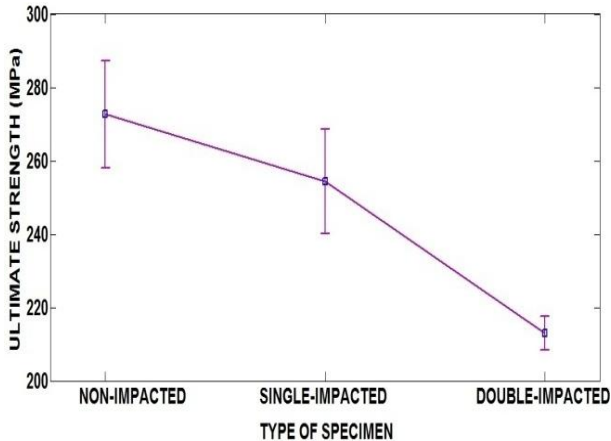


Fig. 6: Residual flexural strength of dry GFRP specimens

Fig. 7 compares the weight gain of impacted and pristine specimens. The weight gain profile traverse in a more or less parallel manner for single-impacted and pristine specimens whereas the double-impacted specimen shows higher escalation. This was due to the greater extent of damage occurred that led to more water seepage, i.e. the water absorption has increased due to impact and with number of impacts. This shows that water uptake by a specimen would occur at a faster rate after being subjected to an impact since it accelerates the capillary action through the deformed specimen.

Fig. 8 shows the plot of ultimate flexural strength values of aged and dry impacted and pristine specimens. Ultimate flexural strength decreases for single-impacted and double-impacted when compared to the pristine

specimen at the end of 100 days. Impact was seen to have an appreciable change in the water absorption behaviour of GFRP specimens and can be visualized as one of the prominent accelerating mechanisms for ageing. As a consequence, components fabricated with composite parts under water have to be monitored frequently for damage due to impact loads so that replacement/repair can be made immediately in order to prevent a catastrophic failure.

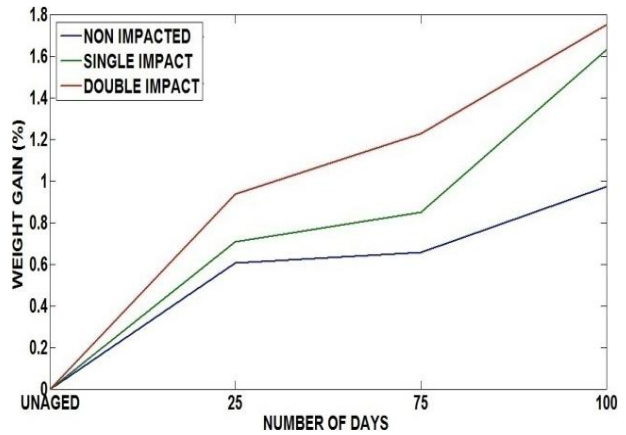


Fig. 7: Weight gain (%) over time for different categories of specimens

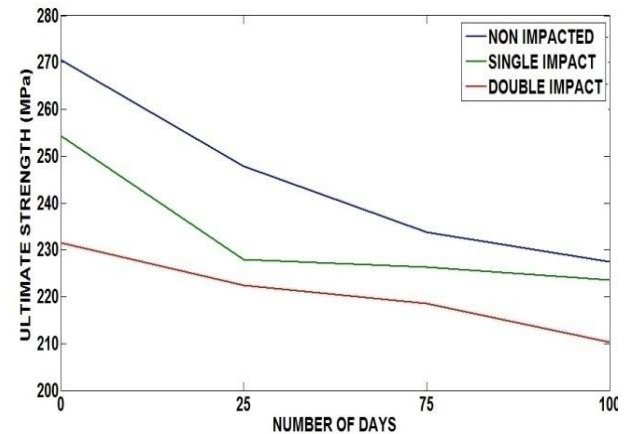


Fig. 8: Flexural strength for different categories of specimens

### 3.2. Effect of ageing on AE parameters

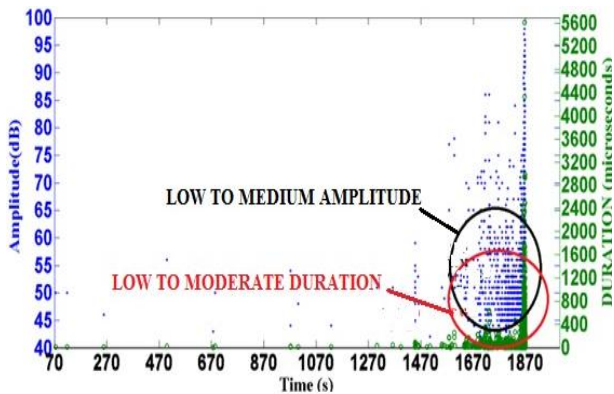
The quality of an AE signal depends mainly on the nature of elastic waves that are generated from the material during failure. Since irreversible changes such as softening of the matrix, weakening of the fiber-matrix interface and accumulations of salt occur due to ageing, it is inevitable that the AE waves undergo mutation in terms of their acoustic parameters. The variation in the percentage of AE hits acquired at different amplitude ranges such as 45-55 dB, 56-65 dB, 66-75 dB, 76-85 dB, 86-95 dB, 96-99 dB for specimens aged under progressive immersion times are given in Table 1. The threshold amplitude was set at 45 dB. It was observed that the acquisition of hits was predominant in the lower amplitude range of 45-55 dB. The percentage of AE hits was found to increase in that range as ageing progresses. There was a decrease in the number of hits in the medium and higher amplitude ranges from 55 dB to 99 dB. This change could be attributed to the lowering in the amplitude of elastic waves (dampening effect)

generated during the breaking of the specimens which in turn was due to the presence of moisture being ingested into the matrix. As it was seen that the range of amplitude in which the AE waves were recorded gets lowered due to ageing, the threshold amplitude might have to be adjusted under certain circumstances for proper acquisition because inappropriate setting of threshold will ultimately result in erroneous interpretations.

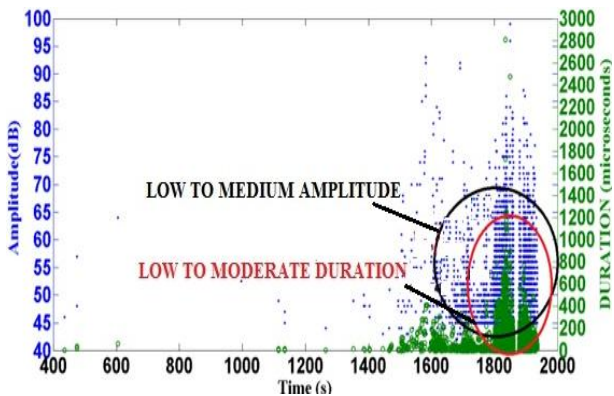
**Table 1: AE hits (%) acquired at different amplitude ranges**

AE Hits (%) Amplitude (dB)	Immersion time (days)			
	0	25	75	100
45-55	73.72	77.40	79.06	80.22
56-65	22.36	12.88	16.85	14.82
66-75	2.91	5.65	2.62	3.63
76-85	0.72	3.17	0.86	0.76
86-95	0.28	0.59	0.35	0.41
96-99	0.08	0.29	0.37	0.22

The AE parameters duration and amplitude were used in parametric studies to analyze the failure modes. To investigate the effect of ageing on them, these two parameters were recorded and plotted over time. It can be noticed in Figs. 10(a) and (b) that the scattering of points has occurred at low to medium amplitude range and low to moderate duration which is the characteristic feature of fiber-matrix debonding [19]. Moreover, the range of duration was found to increase which explains that the ageing process extends the time the AE signal remains over the threshold.



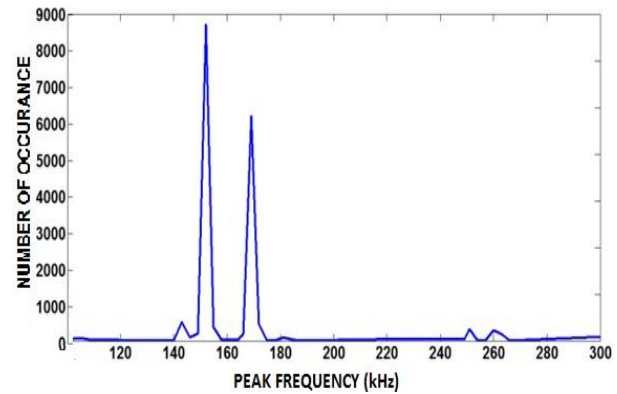
**Fig. 10(a): Amplitude vs. Time for dry (100 days) specimens**



**Fig. 10(b): Amplitude vs. Time for aged (100 days) specimens**

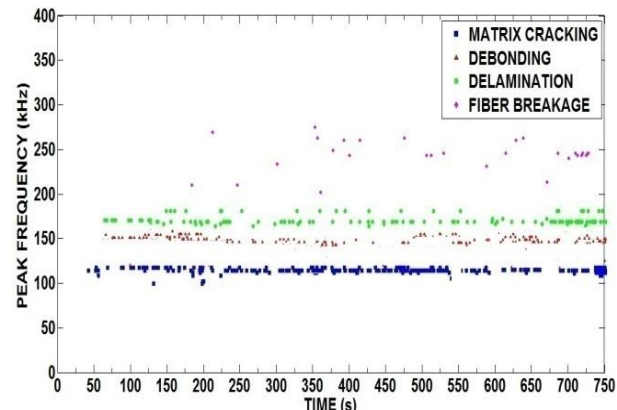
Peak frequency, an AE characteristic was used to estimate the contribution of each failure mode towards

the overall failure and also to find the predominant one amongst them. Modal analysis was done as a preliminary step to determine the number of peak frequency clusters present in the AE data after which a MATLAB code was made use of to form separate clusters each corresponding to a mode of failure. Four major peak frequency clusters exist. Each peak relates to a failure mode and the one with highest peak (maximum number of occurrences within the given time domain) was taken as the major failure mode. Fig. 11 gives a prediction that the fiber-matrix debonding was the dominant failure mode corresponding to the second peak, which in general occurs at a moderate peak frequency range [20] with low to medium amplitude range and low to moderate duration [23] as shown in Fig. 10.



**Fig. 11: Number of occurrence vs. Peak frequency**

Clustering of peak frequency points has occurred at a moderate frequency range as shown in Figs. 12(a) and (b). This clearly explains that debonding was the major failure mode. Other modes such as delamination, matrix cracking and fiber failure together contribute to the rest of the failure propagation. As it is known that delamination and debonding occur at a slightly overlapping peak frequency range, failure discrimination between them is rather tedious. But, debonding could be given an upper hand in this case considering the fact that the fiber-matrix interface gets weakened thereby reducing the interfacial strength due to water absorption, which is one of the mechanisms that leads to ageing. This is in accordance with the fact that the degrading effect of fluid absorption on the matrix of the composites is only secondary, compared to the damage of the fiber-matrix interface [5].



**Fig. 12(a): Peak Frequency vs. Time for dry (100 days) specimens**

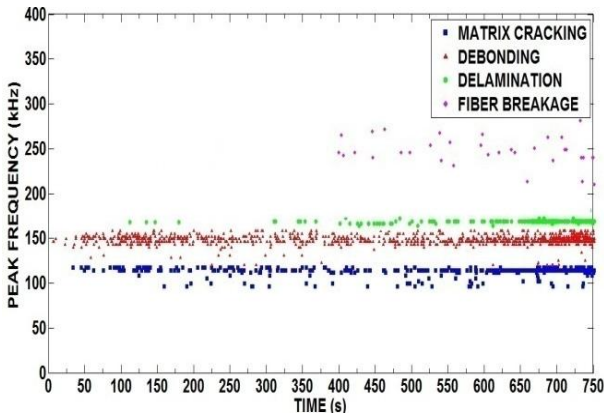


Fig. 12(b): Peak Frequency vs. Time for aged (100 days) specimens

Fig. 13 shows the load-cycle plots for dry and aged specimens from a MATLAB code which correlates the time and stress data from UTM and the AE time which depicts various regions starting from the damage initiation point through propagation and ultimately the failure of the material. The point of failure was indicated at the point where the stress curve starts declining and the curve of cumulative counts rises steeply both of which occurring simultaneously at the same recorded time. Counts are nothing but the number of times the AE signal crosses the detection threshold. Counts at every instant, when added cumulatively give rise to cumulative counts, which governs the start and ultimate point of failure during loading of the material.

The stress curve slope starts decreasing at a value of 295 MPa and 240 MPa for dry and aged specimens respectively. Cumulative counts curves have started rising at a time of 2050 s and 1870 s for dry and aged specimens, which mark the failure point. The point of failure occurs in advance in the aged pristine specimens as compared to their dry counterparts. Similar phenomenon was observed between dry and aged impacted specimens. In pristine dry specimens, the damage initiation and occurrence of failure was much delayed than in impacted specimens. Finally, the point of failure occurs earlier for aged specimens due to the deterioration in the ultimate strength irrespective of the specimen being impacted or not.

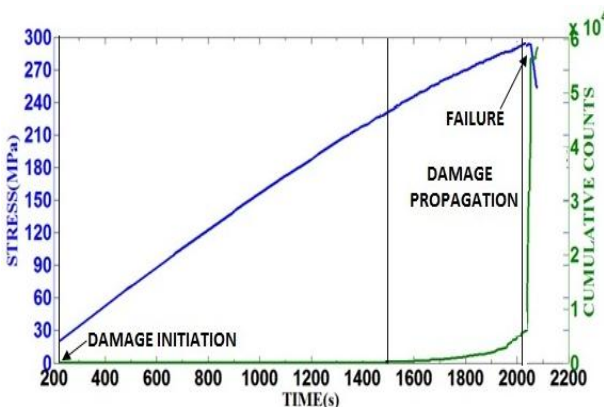


Fig. 13(a): Stress cycle plot for dry (100 days) specimens

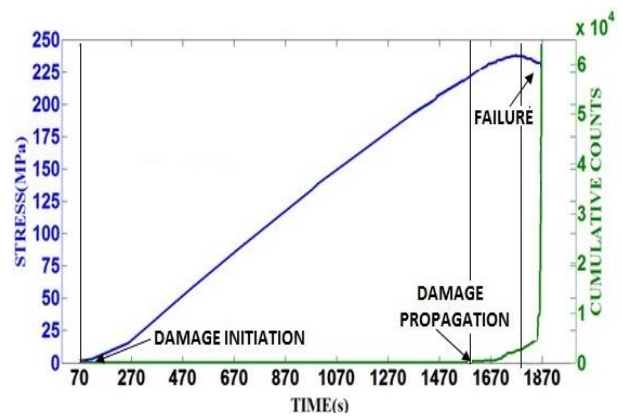


Fig. 13(b): Stress cycle plot for aged (100 days) specimens

#### 4. Conclusions

The hydrolytic ageing of composite materials weakens their mechanical properties. In addition to that, weight gain and strength reduction were a monitored for impacted GFRP specimens over a certain time period both of which were worst with an increase in the number of impacts. Thus, impact was seen to have an appreciable change in the water absorption behaviour of GFRP specimens and can be visualized as one of the prominent accelerating mechanism in ageing. AE testing of aged specimens demonstrated that the range of AE parameters such as amplitude, duration and cumulative counts in a given time domain depicts the change in the nature of the emitted AE signal because of the water uptake by the specimen. The signatures of the AE signals associated to delamination and interfacial debonding are significantly modified by the hydrolytic ageing. The results clearly put forward that the amplitude of the debonding from the modes of failure contributes more to the overall failure of the laminate.

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