iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 8 **Issue:** VI **Month of publication:** June 2020

DOI: http://doi.org/10.22214/ijraset.2020.6083

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.429
Volume 8 Issue VI June 2020- Available at www.ijraset.com

Effect of Hygrothermal Aging & Artificially Introduced Delamination on Mode-I Interlaminar Fracture Toughness of E-Glass Epoxy Matrix Composites

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Abstract***: Various types of polymer matrix composites are being considered for use in high temperature applications such as for supersonic aircraft and naval applications. An attempt to find Fracture toughness of hygrothermally aged delaminated E-Glass epoxy matrix composites is done as fracture toughness is important material property to consider in any design applications. Delamination is one of the major failure modes seen in the laminated polymeric matrix composite (PMC). Hydrophilic nature of epoxy polymers can lead to both reversible and irreversible/permanent changes in epoxy upon moisture absorption. The present work deals with the experimental investigation of hygrothermal aging of delaminated E-glass-fibre-reinforced polymer composite and its effect on the fracture toughness due to the exposure to elevated temperatures and wet conditions which resulted in decrease of maximum load required for fracture with increase of temperature thus fracture toughness (G_I) decreased with increase of temperature.***

Keywords: Delamination. Fracture Toughness. Hygrothermal aging. E- Glass. Epoxy.

I. INTRODUCTION

As composite have stunning physical and mechanical property of high strength to weight ratio they are widely used in aircraft ,construction industries and in naval base, where they are likely to be harmed by the environment. It is generally stated that mechanical properties of composite materials are affected by material ageing, which is often accompanied by moisture absorption [8-12]. However, composite laminates are known to be susceptible to delamination failure due to their poor interlaminar strength. The delaminations usually occur through manufacturing defects or low velocity impact damages during service, and they propagate predominantly under mode I, mode II and mixed mode I/II loadings. The growth of the delamination progressively reduces the in-plane strength, stiffness; durability of composite structures and may finally lead to catastrophic failure. Therefore, delamination is one of the major limiting factors in composite structure design, and various techniques have been developed to toughen the inter laminar strength. In most applications it is unavoidable to react with water at different temperature which will influence the mechanical properties of the material. As there will be different service temperatures the properties of the material changes accordingly. Therefore it is important to understand the effect of environment and estimate the durability of composites when subjected to such hard environments. Many studies reported that hygrothermal ageing will definitely influence the fracture toughness of composite laminates. The duration and temperature of the exposure affect the fracture toughness .The hygrothermal effects are generally more noticeable For the specimens immersed in fluid than exposed in humid air. The present study investigated the effects of water immersion at different temperatures on the fracture load and fracture toughness of E-Glass Epoxy matrix composite. The failure criterion of mode-I delamination under the influence of hygrothermal environment was studied.

II. LITERATURE REVIEW

Yian Zhao *et. al* [2] have conducted different delamination tests which include mode-I, mode-II and mixed mode I & II for woven E-glass/Bismaleimide (BMI) composite to find the resistance curve & fracture toughness before ageing and after ageing in seawater at varying temperatures and reported that except in mode-II, fracture toughness decreased with increase of immersion temperature and resistance increased with growth of delamination which is because of more ductility of composite after immersion and plasticization. S. larbi *et.al*[3]studied the effect of time period of ageing in sea water at room temperature of GFRP composites and reported that at the starting of the process the absorption kinetics is fickian and in case of ageing it is non fickian. Reduce in weight of the specimens after ageing is due to effect of high pH value of sea water which causes matrix degradation. Three-point bending test conducted for specimens after oven drying resulted that tensile strength of the specimens reduced due to ageing**.** B.C ray *et.al* [4] reported that at higher temperatures the hygrothermal ageing is faster which resulted in more moisture absorption for both woven and unidirectional carbon fiber composites. Large effect on fiber matrix interface, increase in thermal stress due to ageing at higher temperatures and more duration. Shivakumar *et.al* [5] worked on GFRP composites, by ageing for 200 days in water at different temperatures .The moisture absorption rate is largely dependent on temperature, which results in debonding of fiber and matrix due to swelling and voids created will act as pool for moisture. The hygrothermal ageing for 200 days at 70°C resulted in decrease of flexural strength of the GFRP composite specimen by 30% when compared to specimen which did not undergo any ageing. Masaki Hojo *et.al* [6] studied the delamination fatigue crack growth and fracture toughness of carbon fiber/epoxy laminate with epoxy interleaf. Mode-I and mode-II tests were done for finding the interlaminar fracture toughness and delamination fatigue and reported that in mode-I the results are same for specimens with and without interleaf because toughness of interleaf played a major role . Interlaminar fracture toughness and delamination fatigue crack growth are more for epoxy interleafed composites compared to without interleaf under mode-II tests because interleaf thickness played a major role. Alessi *et.al* [12] reported a fixed fracture toughness tendency for carbon/epoxy composites, when subjected to ageing at 30˚C for 4 week & 8 week immersion of composite specimens in distilled water, but at the same time a decrease in fracture toughness is found when the temperature is increased to 70˚ C.

III. EXPERIMENTAL PROCEDURE:

A. Material & Specimens

The versatility of glass as a fibre makes it unique industrial textile material. Due to the dimensional stability, moisture Resistance, high Strength, fire Resistance, chemical resistance, electrical Properties & thermal conductivity E-Glass fiber is selected. The resin LAPOX L-12 is mixed with hardner K-6 in ratio of 70:10 gm. The E-Glass fiber which is passed through the resin bath mixed with corresponding hardner on the drum is covered with a thin polythene sheet is opened and laid on the table for 24hrs to dry up.

Fig 1. Composite laminate with delamination

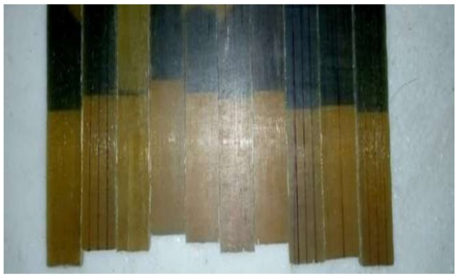


Fig 2. Specimens with delamination

B. Procedure for Manufacturing of Composite Laminates

To prepare a laminate the following procedure is used

1) Smoothing of surface of mould.
2) Cleaning of mould (MS Plate).
3) Applying waxpol to plate.
4) Marking of lamina.
5) Inserting artificial de-lamination.
6) Layup on the MS plate.
7) Curing for 24hours.
8) Extraction of laminate.

Smoothening of surface area is done to remove the extra material or foreign particles over the outer surface of the pipes. We use zero grade emery papers to smoothen the surface of mould. After smoothing of surface of mould, the surface has to be cleaned with acetone to remove the dirt particles. If the surface is not clean then the release agent will not function properly. Releasing agent in this project is waxpol which is applied on the surface of mould. One has to apply in maximum amount on pipes, if not the composite material will stick to the pipes and becomes harder to extract.

The laminas are cut according to the dimensions of the mould. Mould dimensions are 400x400mm so as the laminate dimensions and no of layers of pre-preg is 10. Insertion of de-lamination strip that is a thin polymer layer strip is placed in the middle layer of the laminate that is after the fifth layer. The laminate is left for 24hrs in the room temp for curing and then laminate is extracted carefully. The specimen dimensions are marked on the laminate and the specimens are cut using the cutting machine setup where width of specimen is 25mm &length of the specimens is 114mm.The end of the specimen with delamination (interlayer) is attached with hinges to support the specimen on UTM for conducting double cantilever beam test.

C. Hygrothermal Aging and Observations

Using a heating element, the water was initially heated to a temperature of 100˚C to accelerate the ageing process and the specimens were subsequently aged for periods of 6 hours. Further different specimens are heated to temperatures80˚C, 60˚C , 40˚C respectively for 6 hours duration.

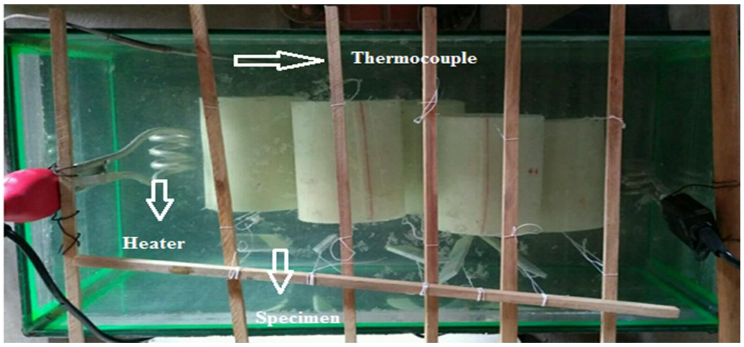


Fig 3. Hygrothermal ageing of composite Specimens

Hydrothermal ageing is expected to affect the fibre-matrix interface , lead to chemical reaction and leaching, thus the compressive response of composite[7].This was investigated by comparing the virgin specimens and the specimens conditioned with accelerated ageing. Water absorption causes the fibre-matrix interface to weaken, which results in fibre-matrix debonding. The primary role of the matrix in the composites is for it to transfer stresses between the fibers to provide a barrier against an adverse environment.

Table 1: Data of specimens subjected to aging.

Temperature (subjected to aging)	Specimen numbers
25^{o}(Virgin)	1,2,3,4,5
40^{o}	6,7,8,9,10
60^{o}	11,12,13,14,15
80^{o}	16,17,18,19,20
100^{o}	21,22,23,24,25

IV. TESTING

A. Mode 1 double cantilever beam (DCB) test for Fracture Toughness

Fig 4. Testing of Specimen.

The mode I delamination toughness is usually measured using the double cantilever beam (DCB) test, which was standardized for fiber reinforced composites by ASTM D-5228 (1994). A schematic of the DCB test is shown in Figure 4, where the specimen thickness is given by t, the crack length is given by a, and the applied load is given by P. The single point loading condition of the DCB provides the ability to use a compliance calibration based data reduction procedure, which is preferred because the only assumptions that must be made are of linear elastic behaviour and of self-similar crack advance. Stable crack advance occurs under displacement-controlled loading (Broek, 1986), which makes continuous measurement of fracture toughness with crack growth feasible.

In this mode 1 fracture test the hinges attached to the specimen are securely inserted into the Universal Testing Machine as shown in the figure above, and subjected to the tensile load. Due to the load the specimen fractures exactly at the delamination layer. The load and displacement are noted at regular intervals to find out the mode 1 fracture behaviour. The same procedure is repeated for all the specimens and results are tabulated.

V. RESULTS& DISCUSSIONS

A. Interlaminar Fracture Toughness

It became a common practise to characterize the resistance to delamination using fracture mechanics. There is competing terminology in literature, such as fracture toughness, average fracture energy, J integral, WOF and critical strain energy release rate. The critical fracture toughness or critical strain energy release rate is the value at the onset of crack propagation. Crack propagation under pure mode-I (opening mode), pure mode-II (shearing or sliding mode) loading has been extensively studied in the literature. Mode-I fracture toughness is given by

$$G_I = \frac{3P\delta}{2Wa}$$

Where

G_I - fracture toughness,

P - Ultimate load

Width of specimen i.e. W = 25mm

Thickness of specimen i.e. T=5mm

' a ' is crack propagation length = 57mm

'δ' is crack displacement

Table 2: Calculation of fracture toughness (G_I)

S.no	Specimen no.	Temperature (T ^{o}C)	Fracture load (P KN)	Displacement (δ)	Fracture toughness (G_i)
1	1	virgin	0.99	7.3	0.00761
2	3	virgin	0.89	9.2	0.00862
3	7	40	0.77	6.7	0.00543
4	8	40	0.71	6.3	0.00471
5	11	60	0.59	4.8	0.00298
6	12	60	0.65	3.7	0.00253
7	16	80	0.59	4.4	0.00298
8	17	80	0.44	5.4	0.00239
9	21	100	0.49	3.5	0.00181
10	22	100	0.47	2.8	0.00139

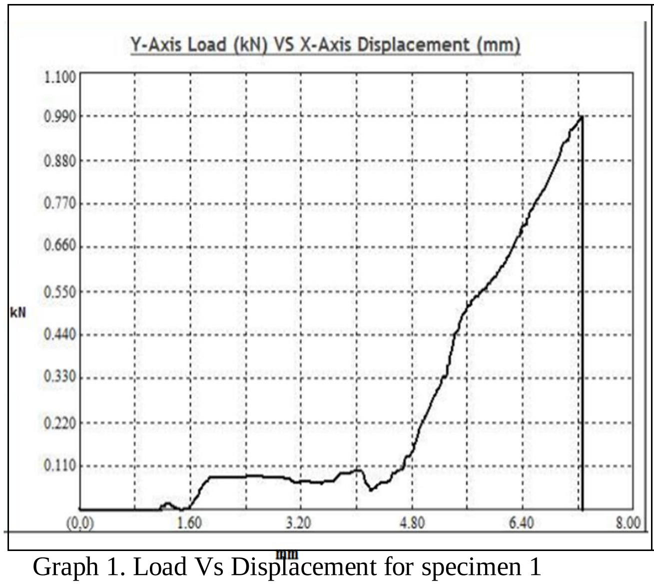
Before and after subjecting to Hygrothermal aging, the specimens are weighed and the values are noted.

It is observed that after Hygrothermal aging the weight of the specimens increased, it is due to the fact that the salt particles get clogged between the space of matrix and fibre. During ageing, diffusion is caused by hydrolysis and plasticisation of the immediate neighbouring molecules in the resin matrix, and swelling was caused by moisture absorption. The rate of diffusion depends on the nature of the resin matrix, fibre orientation, fibre volume fraction, porosity and voids.

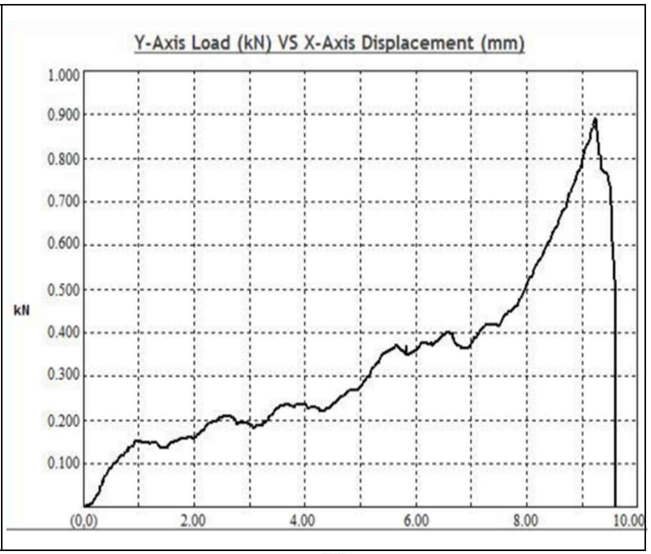
The presence of fibres slowed the movement of the molecules, and hence, lowered the diffusivity factors after the initial stage. Moisture intake caused swelling of the specimens, creating internal stress on the matrix bonding, in turn, leading to the formation of matrix cracks. Moisture absorption caused osmotic cracking inside the matrix and the mechanical properties of the composites deteriorated. Glass fibres showed higher resistance to water absorption, slowing the deterioration.

The following curves represent the load Vs displacement for the specimens

B. Results of virgin specimens 1& 3 at 25^oC

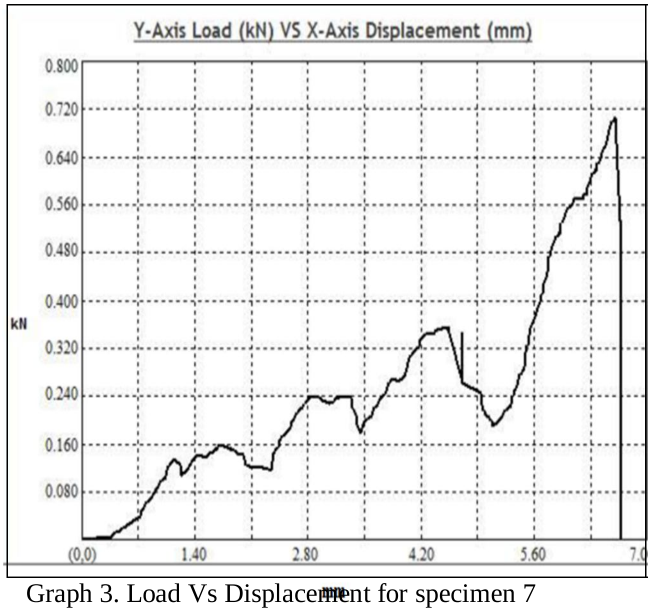


Graph 1. Load Vs Displacement for specimen 1

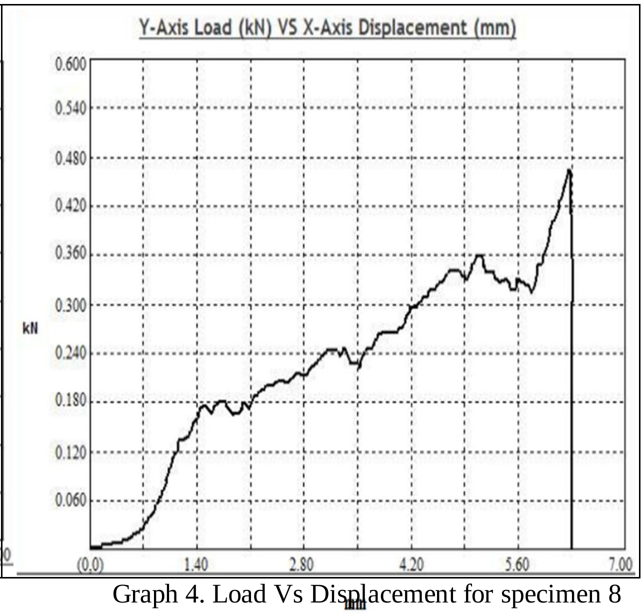


Graph 2. Load Vs Displacement for specimen 3

C. Results of specimen 7& 8 at 40^oC

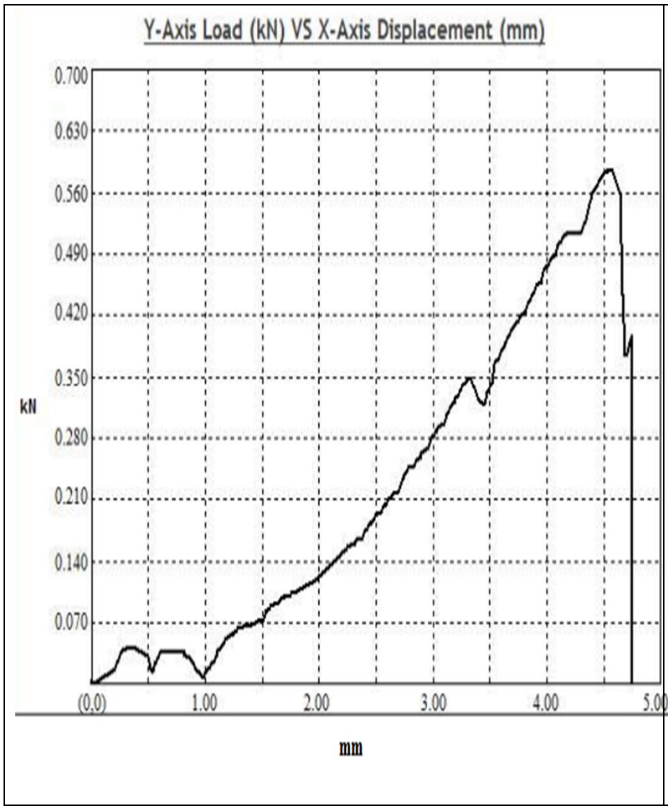


Graph 3. Load Vs Displacement for specimen 7

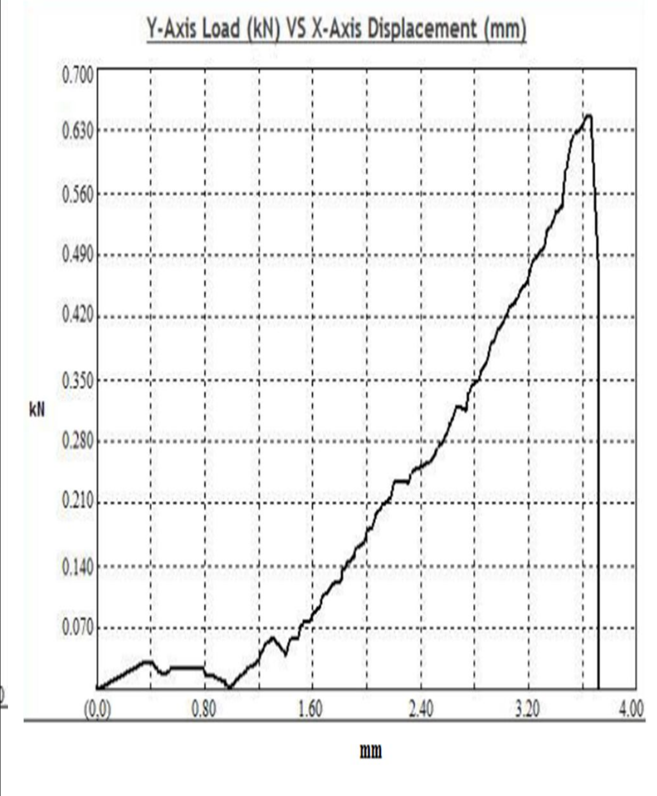


Graph 4. Load Vs Displacement for specimen 8

D. Results of specimens 11& 12 at 60°C

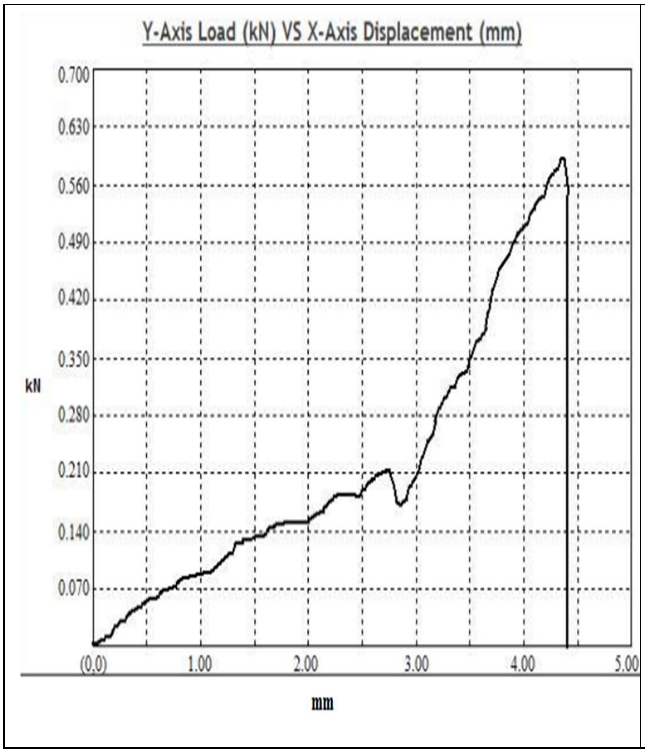


Graph 5. Load Vs Displacement for specimen 11

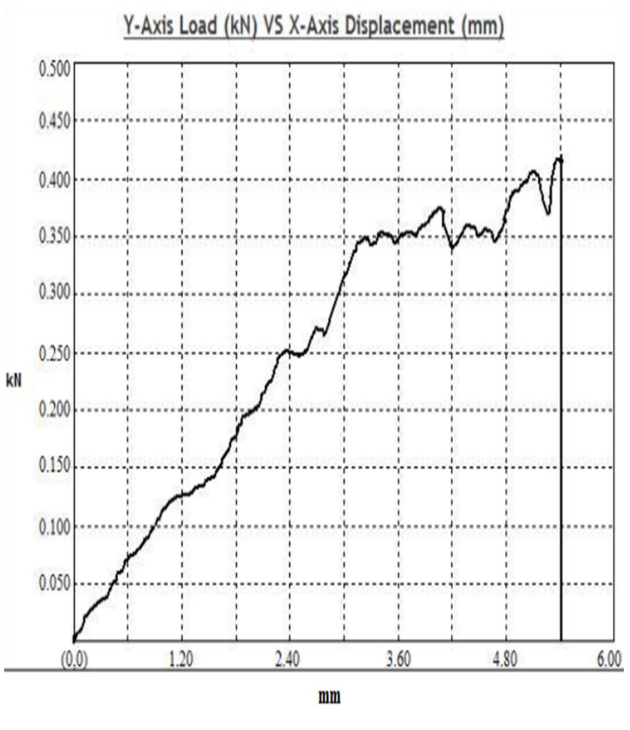


Graph 6. Load Vs Displacement for specimen 12

E. Results of specimens 16& 17 at 80°C

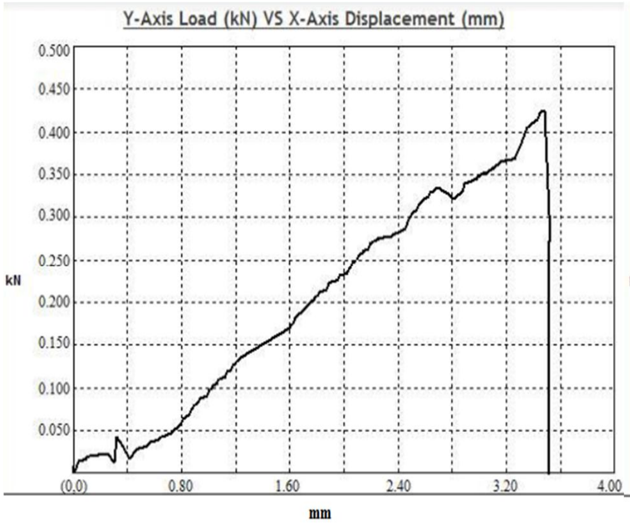


Graph 7. Load Vs Displacement for specimen 16

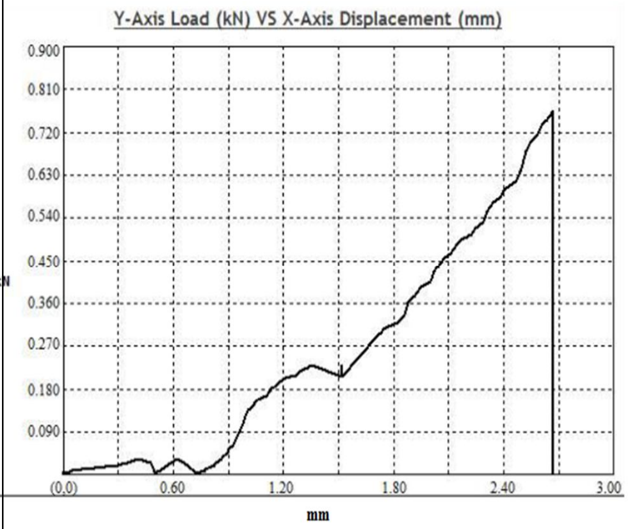


Graph 8. Load Vs Displacement for specimen 17

F. Results of specimens 21& 22 at 100°C



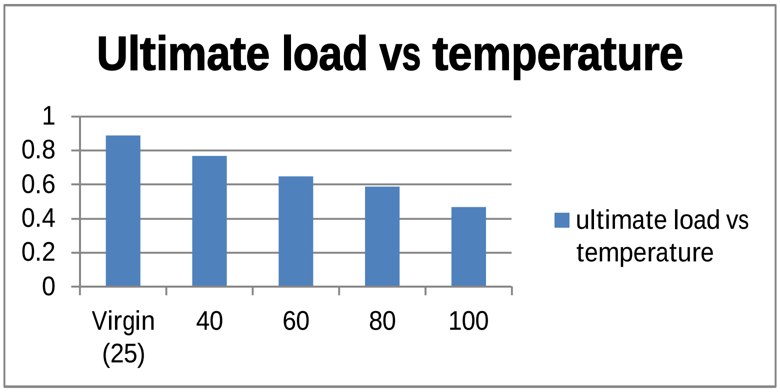
Graph 9. Load Vs Displacement for specimen 21



Graph 10. Load Vs Displacement for specimen 22

Table.3. Variation Of ultimate load Due To Aging at different temperatures

Temperature (°C)	Ultimate load	% decrease in Ultimate load
Virgin (25°C)	0.89	-
40°C	0.77	13.48
60°C	0.65	26.97
80°C	0.59	33.71
100°C	0.47	47.19

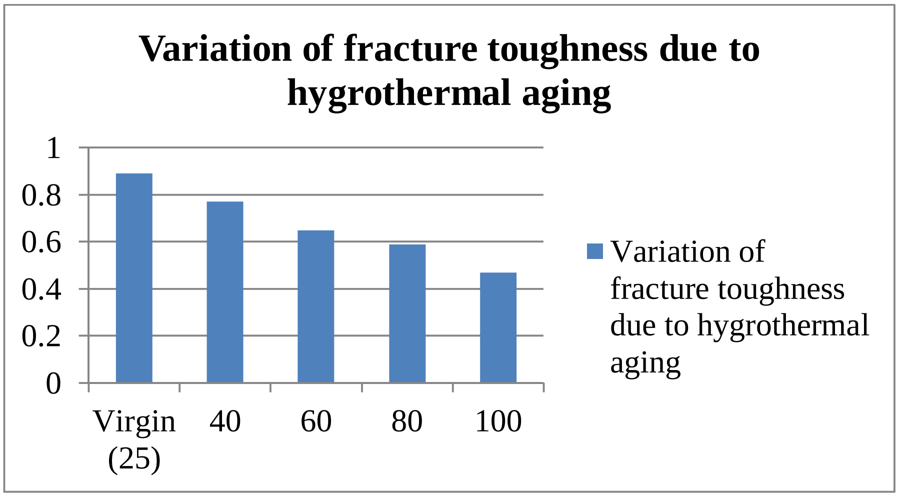


Graph 11. Temperature Vs Ultimate load

Due to hygrothermal aging the ultimate load of the specimens decreased, this is due to degradation of matrix strength because of salt particles.

Table.4. Variation Of Fracture Toughness Due To Aging at different temperatures

Temperature (°C)	Fracture toughness	% decrease in toughness
Virgin (25)	0.00862	-
40	0.00543	37.00
60	0.00298	65.43
80	0.00239	72.27
100	0.00139	83.88



Graph 12. Temperature Vs Fracture Toughness

Due to hygrothermal aging the fracture toughness of the specimens decreased, this is due to degradation of matrix strength because of salt particles.

VI. CONCLUSION

The following conclusions and observations are made from the test results:
As the temperature increases the maximum load required for fracture is decreased.

A. The average ultimate load of virgin specimen is 0.94kN.
B. The average ultimate load of specimens at 40°C is 0.74kN
C. The average ultimate load of specimens at 60°C is 0.62kN
D. The average ultimate load of specimens at 80°C is 0.51kN
E. The average ultimate load of specimens at 100°C is 0.48kN

As the temperature increases the fracture toughness (G_I) is decreased.
1) The average toughness for virgin specimen is 0.0081.
2) The average toughness for specimen at 40°C is 0.0050.
3) The average toughness for specimen at 60°C is 0.0028.
4) The average toughness for specimen at 80°C is 0.0025.
5) The average toughness for specimen at 100°C is 0.0016.
The results revealed that hygrothermal aging conditions at higher temperature had negative impact on mechanical properties of GFRP composites.

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