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ENVIRONMENTAL DEGRADATION OF GLASS FIBRE REINFORCED POLYMER COMPOSITE

AUTHOR:	Rahul Chhibber
AFFILIATION:	Thapar University, Patiala
Address:	Patiala, Punjab, India
Tel.:	+91 175 239 3096
EMAIL:	rchhibber@thapar.edu
FURTHER AUTHORS:	Amit Sharma, Abhijit Mukherjee, L. Mishnaevsky Jr.
Reviewer:	Project members
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Abstract: Fibre composites nowadays are being used in various applications from aerospace, military, civil to renewable energy generation. Higher energy demands across the developed and developing world and growing concern regarding environmental pollution has renewed interest in generation of power from eco-friendly sources such as wind. Wind turbine rotor blades are being increasingly made of fibre composites. The increasing use of wind as renewable energy source in tropical part of the world necessitates further research in wind turbine rotor blade design, durability and manufacturing. Hence Effort is required to fully comprehend the GFRP composite response under natural and accelerated moisture and temperature conditions specific to tropical region. Glass Fibre Reinforced Polymer's have been found to be attractive in the Asian region due to their high cost competitiveness. Wide-spread use of fibre-reinforced polymers (FRP) though is hampered in this part of world due to lack of long-term durability and performance data, especially in a tropical environment. The main environmental factors for the deterioration of GFRP are temperature, light, water/moisture, alkalinity and load. Most of the early durability tests were carried out with reference to application of FRP (Fibre Reinforced Polymer) in aerospace. Thus considerable data is available with only one or a combination of some of these parameters. For achieving this objective an experimental setup was prepared. The aim of the experiment was to study the combined effect of chosen parameters moisture, alkali and temperature and also to study the rate and magnitude of damage of GFRP composites. The macroscopic and microscopic behaviour of the GFRP composite specimen exposed to varying hygrothermal loads has been analysed.

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	STATUS, CONFIDENTIALITY AND ACCESSIBILITY								
	Status				Confidentiality		Accessibility		
S0	Approved/Released			R0	General public			Private web site	
S1	Reviewed			R1	Restricted to project members	R1		Public web site	
S2	Pending for review			R2	Restricted to European. Commission			Paper copy	
S3	Draft for commends			R3	Restricted to WP members + PL				
S4	Under preparation			R4	Restricted to Task members +WPL+PL				

PL: Project leader WPL: Work package leader

er **TL:** Task leader

1. Environmental Effects On Fibre Composites



Fig. 1.1 Diffusion path of moisture into composite thickness direction [24]

Fibre composites, are increasingly being used in renewable energy generation applications (wind turbine rotor blades) owing to several desirable properties including high flexural strength, high specific stiffness, lightweight, controlled anisotropy. Despite these advantages over conventional structural materials such as metals, composites are susceptible to heat and moisture when operating in harsh and changing environmental conditions. When exposed to humid environments, Glass fibre–epoxy composites absorb moisture (Fig. 1.1) and undergo volumetric expansion.

Moisture and the stresses associated with moisture induced expansion can result in low damage tolerance, with an adverse effect on long-term structural durability. The amount of moisture absorbed by the epoxy matrix is significantly greater than that by the fibres. Differential volumetric expansion of the matrix and the fibres is observed due to moisture absorption, and this further leads to the generation of localized stress and strain fields in the composite.

Changes in the thermo physical and mechanical properties as well as chemical composition of the epoxy matrix are also observed due to plasticization and hydrolysis resulting from moisture absorption. These changes in the polymer structure lower both the elastic modulus and the glass transition temperature. At the same time, moisture wicking along the fibre matrix interface degrades the fibre matrix bond, resulting in loss of micro structural integrity. The net effect of moisture absorption is the deterioration of matrix-dominated properties such as compressive strength, interlaminar shear strength, fatigue resistance and impact tolerance. These factors lead to reduced damage tolerance and lack of long-term durability.

Maximum moisture content and the diffusivity govern the kinetics of moisture diffusion in homogeneous materials. The maximum moisture content is defined by the net amount of moisture that a fully saturated material contains under steady state equilibrium when exposed to a given environmental condition. It is usually expressed as the ratio of the increase in weight per unit dry weight at the point of saturation. The relative weight gain approaches the maximum moisture content of composite at infinite time.

This process often takes several months, which makes the procedure cumbersome and time consuming. Also the rate of moisture diffusion is governed by the diffusivity. In general, the diffusivity is a strong function of the ambient temperature and a weak function of the relative humidity. In the case of composites, the diffusion process is more complex. The figure 1.2 shows the various effects of moisture diffusion on a composite specimen.



Fig. 1.2 Different effects of moisture diffusion on composites [12]

When a fibre-reinforced composite material is exposed to a hygrothermal environment and mechanical loads, changes in material properties are expected. These changes in material properties are connected to an irreversible material degradation.

The work in this report was an attempt to study the effect of moisture, alkali and heat on Glass Fibre Reinforced Polymer (GFRP) woven fabric (E Glass). The changes occurring in the physical composition and mechanical properties of the composite material under natural and accelerated conditions had been studied.

For achieving this objective an experimental setup was prepared. The aim of the experimentation was to study the combined effect of parameters moisture, alkalinity and temperature and study the rate and magnitude of damage of the chosen composite material which further helped in studying the response of the composite to the given hygrothermal load.

The natural degradation of material takes long time to show appreciable results so we had chosen accelerated degradation condition to have results in short duration of time. The material was pre-stretched to service load conditions in order to form some cracks so as to allow water to seep at a faster rate. The specimen were exposed to hygrothermal loads in water baths and aqueous NaOH baths for pre-decided time periods and then tested for their tensile strength in a universal testing machine. The presented work was mainly carried out on the first two sets of specimen i.e. holding time of 1 and 2 months. Microscopic examination of these specimen was carried out using Scanning Electron Microscopy and Electron Dispersive Spectroscopy to analyse the compositional changes. The image analysis of SEM images of these specimen has also been carried out using Image J to analyse the compositional changes and changes in fibre and epoxy area fractions.

2. Experimental Setup And Experimentation

A set of accelerated aging tests have been carried out to evaluate performance of pre-stretched glass fibres reinforced polymer (GFRP) sheets embedded in epoxy matrix. The field environment very similar to that of tropical climate had been simulated in the laboratory (shown in Fig 2.1).

The specimen were immersed in various baths for different time durations. The novelty of the experiment was that the accelerated environmental exposure had been given to specimen while they were pre-stretched to different service loads. The specimen were removed from the bath after decided time period. The Ultimate tensile strength was measured and a SEM (Scanning Electron Microscope) image was obtained to check the condition of composite material.



Fig. 2.1 Experimental setup

2.1 Test Specimen

Commercially available GFRP sheet had been used for making specimen. The GFRP sheet was placed along the length side (0o orientation) for cutting the specimen.

The following were the specifications of the specimen (refer Fig. 2.2):

Length of specimen : 500 mm Breadth of specimen : 10 mm Thickness of specimen : 0.8 mm (approx.)

The specimen were having "tabs" at both sides only upto a length of 150 mm from respective ends. This left an "effective length" of about 200 mm in centre. We had taken the ASTM Standard D-3039 as a reference to decide the shape and specification of the specimen.



Fig. 2.2 Dimensions of the Specimen

All dimensions are in mm



Fig. 2.3 Actual image of the specimen

The image of the specimen is shown in Fig.2.3. The specimen hung were fully immersed into the bath from a hanger stick with help of thread as shown in Fig.2.4.



Fig. 2.4 Heating Element and RTD sensor in a bath

S.No.	Specimen	Bath type	Bath temperature	Exposure	Percentage of	Specimen
	Designation		(⁰ C)	time	ultimate tensile	count
				(Month)	load at which the	
					specimen is pre-	
					stretched	
1	W-45-1-20-1	Water	45	1	20	1
2	W-45-1-20-2	Water	45	1	20	2
3	W-45-1-40-1	Water	45	1	40	1
4	W-45-1-40-2	Water	45	1	40	2
5	W-45-1-60-1	Water	45	1	60	1
6	W-45-1-60-2	Water	45	1	60	2
7	W-45-1-80-1	Water	45	1	80	1
8	W-45-1-80-2	Water	45	1	80	2
9	W-45-2-20-1	Water	45	2	20	1
10	W-45-2-20-2	Water	45	2	20	2
11	W-45-2-40-1	Water	45	2	40	1
12	W-45-2-40-2	Water	45	2	40	2
13	W-45-2-60-1	Water	45	2	60	1
14	W-45-2-60-2	Water	45	2	60	2
15	W-45-2-80-1	Water	45	2	80	1
16	W-45-2-80-2	Water	45	2	80	2
17	W-55-1-20-1	Water	55	1	20	1
18	W-55-1-20-2	Water	55	1	20	2
19	W-55-1-40-1	Water	55	1	40	1
20	W-55-1-40-2	Water	55	1	40	2
21	W-55-1-60-1	Water	55	1	60	1
22	W-55-1-60-2	Water	55	1	60	2
23	W-55-1-80-1	Water	55	1	80	1
24	W-55-1-80-2	Water	55	1	80	2
25	W-55-2-20-1	Water	55	2	20	1
26	W-55-2-20-2	Water	55	2	20	2
27	W-55-2-40-1	Water	55	2	40	1
28	W-55-2-40-2	Water	55	2	40	2
29	W-55-2-60-1	Water	55	2	60	1
30	W-55-2-60-2	Water	55	2	60	2
31	W-55-2-80-1	Water	55	2	80	1
32	W-55-2-80-2	Water	55	2	80	2
33	N-45-1-20-1	NaOH	45	1	20	1
34	N-45-1-20-2	NaOH	45	1	20	2
35	N-45-1-40-1	NaOH	45	1	40	1
36	N-45-1-40-2	NaOH	45	1	40	2
37	N-45-1-60-1	NaOH	45	1	60	1
38	N-45-1-60-2	NaOH	45	1	60	2
39	N-45-1-80-1	NaOH	45	1	80	1
40	N-45-1-80-2	NaOH	45	1	80	2
41	N-45-2-20-1	NaOH	45	2	20	1
42	N-45-2-20-2	NaOH	45	2	20	2
43	N-45-2-40-1	NaOH	45	2	40	1
44	N-45-2-40-2	NaOH	45	2	40	2
45	N-45-2-60-1	NaOH	45	2	60	1
46	N-45-2-60-2	NaOH	45	2	60	2
47	N-45-2-80-1	NaOH	45	2	80	1

Table. 1 Test matrix Details

48	N-45-2-80-2	NaOH	45	2	80	2
49	N-55-1-20-1	NaOH	55	1	20	1
50	N-55-1-20-2	NaOH	55	1	20	2
51	N-55-1-40-1	NaOH	55	1	40	1
52	N-55-1-40-2	NaOH	55	1	40	2
53	N-55-1-60-1	NaOH	55	1	60	1
54	N-55-1-60-2	NaOH	55	1	60	2
55	N-55-1-80-1	NaOH	55	1	80	1
56	N-55-1-80-2	NaOH	55	1	80	2
57	N-55-2-20-1	NaOH	55	2	20	1
58	N-55-2-20-2	NaOH	55	2	20	2
59	N-55-2-40-1	NaOH	55	2	40	1
60	N-55-2-40-2	NaOH	55	2	40	2
61	N-55-2-60-1	NaOH	55	2	60	1
62	N-55-2-60-2	NaOH	55	2	60	2
63	N-55-2-80-1	NaOH	55	2	80	1
64	N-55-2-80-2	NaOH	55	2	80	2

In above experiment according to the planned test matrix (refer Table 1) overall number of specimen on which the work was done i.e. holding time of 1 and 2 months, amounted to a total of 64 specimen.

The tensile test of unexposed specimen has been first conducted on a Universal Testing Machine to determine the actual failure load (peak load) the specimen can bear. On basis of that failure load the loading weight for specimen has been calculated. The first set of specimen have been stretched to 20%, 40%, 60% and 80% of failure load and exposed to temperature variation of 450and 550C and then have been taken out of bath after a time of 1 and 2 months and were checked for its ultimate tensile strength. Two specimen out of each varied load and temperature combination have been tested for their tensile strength. One failed specimen under each load was also tested for micrograph and compositional details using SEM+EDS.

2.2 Loading values of Specimen (Percentage of Ultimate Tensile Load):

The following Table 2 shows the values at which specimen were stretched to different percentage of ultimate tensile load as mentioned earlier.

Tuble, 2 Bouding values (percentage of Ortimate Tenshe Boud) for speening	2 Loading values (percentage of Ultimate Tensile Load) f	for specimen
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Loading Value (%) of Ultimate Tensile Load	Stress (MPa)
20%	47.0
40%	96.0
60%	155.0
80%	203.0

Ultimate Tensile load assumed as 2 KN.

2.3 Fibre volume fraction and its calculations:

Fibre volume fraction means the amount of fibre present in the composite material. There are several methods available to find but we had chosen the method of the "Ignition Test".

This method determines the mass of the resin by determining the ignition loss of a cured polymer composite. To achieve this, a specimen of known mass has been heated until the resin matrix was oxidised and converted to volatile materials. The ash was removed and the residual (assumed to be the fully fibre) was then weighed. The fibre volume fraction has been determined by knowing accurate values for the density of the fibre and the composites. Any part of epoxy that was not oxidised will affect the calculation of fibre volume fraction. Similarly any volatiles such as water or residual solvent would cause errors unless they were small enough to be ignored. The details of specimen tested and test results are given in Table 3.

The fibre volume fraction was calculated as per formula shown below: [33]

$$V_f = \frac{\rho_m W_f}{\rho_f W_m + \rho_m W_f}$$

Where V_f = volume fraction of fibres W_f = weight of fibres W_m = weight of matrix ρ_f = density of fibres ρ_m = density of matrix

Specime n No.	Length (cm)	Weight of whole specimen in grams	Weight of specimen after burning (i.e. fibre weight) in grams	Weight fraction
1	8.4	1.4021	0.7527	0.54
2	8.4	1.4077	0.7770	0.55
3	10.7	1.7175	0.9913	0.58
4	10.7	1.7130	0.9989	0.58

	Table 3 Details	of spec	imen teste	ed for vo	olume	fraction
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Density of fibre* = 2.6 grams/cm3

Density of Epoxy resin* = 1.3 grams/cm3

*As per the manufacturer's specifications [11]

Using the above data and putting in the equation mentioned above gives:

Fibre volume fraction = 40%

3. Results and Discussion

3.1 Macroscopic Behaviour

3.1.1 Mode of Failure (Macroscopic Visual Observations)

As per the visual observation the initial specimen (Fig.3.1) had fine shiny epoxy coating and the failure of such specimen under uniaxial tensile load showed a broom type failure with long broom fibres which is typical of sudden and violent release of stored elastic energy at failure.

After 1 month (Fig. 3.2 and Fig. 3.3) of exposure in water baths there was heavy visible scale formation on all the specimen. Surface observation showed that epoxy had lost its shine and outer edges on either side showed little fanning out. But still after bending (by hands) the specimen showed considerable strength and flexibility. Broom type failure was visible though with shorter broom fibre.

On the other hand the specimen immersed in aqueous NaOH baths had deteriorated to a large extent, the top surface of some specimen showed the chipping out of epoxy coating which appeared to have gone brittle. Further the specimen had lost their flexibility and were easily broken by bending with hands.

After 2 months (Fig.3.4 and Fig.3.5) of environmental exposure in water baths observations of degradation and heavy scaling were noted with more scale formation on water immersed specimen as compared to first month observations for water immersed specimen. Broom type failure with shorter broom fibre as compared to one month exposed fibres was visible.

For 2 months (Fig.3.4 and Fig.3.5) of environmental exposure in NaOH baths observations of heavy degradation of fibre and scaling were noted with more fibre degradation of NaOH bath immersed specimen as compared to first month observations for NaOH bath immersed specimen. Clean Abrupt failure like cleavage edge was visibly observed. The specimen had lost their flexibility completely and were easily broken by slight bending with hands as compared to one month exposed specimen.





Fig. 3.1 Fresh specimen (a) before tension test (b) after tension test



Fig. 3.2 Specimen taken out after one month of exposure (a) NaOH bath (b) Water bath





Fig. 3.3 Failure in tensile held after one month exposure (a) NaOH bath (b) Water bath



Fig.3.4 Specimen taken out after two months of exposure (a) NaOH bath (b) Water Bath



(a)

Fig. 3.5 Failure in tensile held after two month exposure (a) NaOH bath (b) Water Bath

3.1.2 Ultimate Tensile Strength

The Fig. 3.6 shows the ultimate tensile strength of all the specimen. It is clearly seen that the specimen immersed in water have fairly higher level of strength as compared to the ones immersed in aquous NaOH bath. From the graphs (Fig. 3.7 - Fig. 3.8) it is seen that for both water and NaOH environments with increasing time of exposure the ultimate tensile strength of specimen decrease. The graphs (Fig.3.9 - Fig.3.12) show the effect of temperature on degradation in ultimate tensile strength for water and NaOH baths. It is observed that for water bath the decrease in ultimate tensile strength is similar in both the baths at 45 and 55 degree Celsius for all specimen, whereas in case of NaOH baths the decrease in ultimate tensile strength is much more pronounced in the bath at 55 degree Celsius. Graphs (Fig. 3.13 - Fig. 3.20) show the effect of varying pre-stretch loads on the ultimate tensile strength of the specimen immersed in water and NaOH baths. It is observed that in water baths with the variation pre-stretch loads a decrease in ultimate tensile strength is observed for all specimen, Also for NaOH baths (Fig 3.18-Fig 3.20) it is observed that with increasing pre-stretch loads there is a decrease in ultimate tensile strength for all specimen.



Fig. 3.6 Comparison of ultimate tensile strength of specimen in NaOH and Water Baths



Fig. 3.7 Ultimate tensile strength of specimen in Water Bath



Fig. 3.9 Ultimate tensile strength of specimen in water bath at $45^{\circ}C$



Fig. 3.11 Ultimate tensile strength of specimen in NaOH bath at 45^oC



Fig. 3.8 Ultimate tensile strength of specimen in NaOH Bath



Fig. 3.10 Ultimate tensile strength of specimen in water bath at 55°C



Fig. 3.12 Ultimate tensile strength of specimen in NaOH bath at $55^{\circ}C$



Fig. 3.13 Ultimate tensile strength of specimen in water bath at 45[°]C for one month

Fig. 3.14 Ultimate tensile strength of specimen in water bath at 45°C for two month





Fig. 3.15 Ultimate tensile strength of specimen in water bath at 55[°]C for one month in water bath at 55[°]C for two month

Fig. 3.16 Ultimate tensile strength of specimen





in NaOH bath at 45°C for one month

Fig. 3.17 Ultimate tensile strength of specimen Fig. 3.18 Ultimate tensile strength of specimen in NaOH bath at 45°C for two month



in NaOH bath at 55[°]C for one month

Fig. 3.19 Ultimate tensile strength of specimen Fig. 3.20 Ultimate tensile strength of specimen in NaOH bath at 55°C for two month

3.1.3 Percentage Decrease in Tensile Strength

The trend of decrease in ultimate tensile strength is shown in Fig.3.21 - Fig.3.24. The percentage decrease in tensile strength is much higher in both NaOH baths as compared to water baths The tensile strength reduction is higher for two months than in one month in both water and NaOH baths. The percentage reduction in ultimate tensile strength is increasing with increasing the pre-stretch loads in both water and NaOH baths. There appears a slight increase in percentage reduction of ultimate tensile strength with increasing temperature in both NaOH and water bath. The effect of temperature change doesn't seem to have much effect on the tensile strength reduction.

The reason for decrease in ultimate tensile strength and change in percentage tensile strength seems to be the continuous degradation due to hygrothermal load which affected the epoxy and fibre strength. Further in NaOH baths the reason for such large reduction is that fibre has been subjected to chemical attack as well which had made it loose strength and further supporting with SEM which also shows that fibre has been eaten up and its circularity has changed.



Fig. 3.21 Percentage decrease in UTS of specimen in water at 45°C

Fig. 3.22 Percentage decrease in UTS of specimen in water at 55°C



Fig. 3.23 Percentage decrease in UTS of specimen in NaOH at 45°C in

Fig. 3.24 Percentage decrease in UTS of specimen in NaOH at 55° C

3.1.4 Percentage Weight Gain In Specimen:

* The time variation of percentage weight gain (wt) can be measured as: wt = $\frac{(W_{(t)} - W_0)}{W_0} \times 100$

Here $W_{(t)}$ is the total weight after time t, W_0 is the reference dry weight of the specimen before immersion in medium. The percentage weight gain (of moisture) was compared for various baths in Fig.3.25 - Fig.3.28. It could be easily noticed that in water baths the percentage weight gain is increasing with respect to time indicating that more moisture is absorbed after two months compared to first month) in all the specimen at various loads . There was marginal increase in weight gain of specimen at 55°C temperature as compared to specimen at 45°C). In the case of NaOH baths the percentage weight gain was also much higher in second month and also it was much higher with respect to water baths in both the months. The average weight gain in water baths was 3.53% at 45 degree Celsius and 4.0% at 55 degree celsius. But in NaOH baths average values were 11.38% at 45 degree Celsius and 13.67% at 55 degree Celsius. The reason for the increase in percentage weight gain in both baths is obvious as with time the pores of epoxy will loosen due to given hygrothermal load giving way to moisture. But in case of NaOH baths the weight gain was much higher due to the chemical attack on the fibre periphery as could be seen in SEM images, creating more voids to hold more moisture. The graphical comparisons of the percentage weight gain by various specimen are:



Fig. 3.25 Percentage weight gain of specimen in water at 45°C



Fig. 3.27 Percentage weight gain of specimen in NaOH at 45°C



Fig. 3.26 Percentage weight gain of specimen in water at 55°C



Fig. 3.28 Percentage weight gain of specimen in NaOH at 55°C

3.2 Microscopic Behaviour

3.2.1 Mode of Failure (Microscopic SEM observations)

The SEM images (Fig.3.29 and Fig 3.30) for the GFRP composite specimen taken at 1000 X magnification from their cut-cross-section and cut-longitudinal-section at the start of testing, before exposure to hygrothermal loads show a fibre epoxy composite with fibres densely embedded in epoxy matrix with epoxy surrounding completely the fibres which maintain their circularity and suggest good strength. The SEM image (Fig. 3.30) of cut-longitudinal-section show cylindrical unbroken long fibres with no damage.

SEM AND EDS BEFORE EXPOSURE:



Fig. 3.29 SEM of cross-section



Fig. 3.30 SEM of longitudinal section

S.E.M. RESULTS OF WATER BATH T1 (after 1 month)

Holding Parameters: Plain water bath Time: 1 month

Temperature: 45°C

1) The SEM images of composite specimen which were subjected to 20% loading (T1, 1 month) shown below in Fig 3.31 and Fig 3.32 show undamaged circular fibres and epoxy smearing the surface of the fibres completely in small irregular lumps probably due to absorption of water by epoxy.



Fig.3.31 SEM image of specimen 1

Fig.3.32 SEM image of specimen 1 for EDS

The SEM images of specimen which were subjected to 40% loading (T1, 1month) are shown below, the undamaged fibre and lumping of the epoxy is clearly visible, the lumping of epoxy is due to hardening by absorption of water:



Fig.3.33 SEM image of specimen 3



Fig.3.34 SEM image of specimen 3 for EDS

The SEM images of specimen which were subjected to 60% loading (T1, 1month) shown below indicate no damage to the circular fibre, whereas lumping of epoxy is clearly evident due to its hardening by absorption of water, also voids are evident in the epoxy matrix indicating a loss of strength in the composite. :





Fig. 3.35 SEM image of specimen 5 Fig. 3.36 SEM image of specimen 5 for EDS The SEM images of specimen which were subjected to 80% loading (T1, 1month) are shown below, they indicate no damage to the circular fibre, whereas lumping of epoxy is clearly evident due to its hardening by absorption of water, also large number of voids are evident in the epoxy matrix surrounding the fibres indicating loss of strength.



Fig.3.37 SEM image of specimen 7



Fig.3.38 SEM image of specimen 7 for EDS

SEM RESULTS OF NaOH Bath

BATH T2 (after 1 month)

Holding Parameters: NaOH bath Time: 1 month Temperature: 45°C The SEM images of speci

The SEM images of specimen which were subjected to 20% loading (T2, 1month) shown below indicate damaged fibre with change in circularity of the fibre, small amount of lumping of epoxy is also visible due to its hardening by absorption of water.



Fig.3.39 SEM image of specimen 33



Fig.3.40 SEM image of specimen 33 for EDS

The SEM images of specimen which were subjected to 40% loading (T2, 1month) shown below clearly show fractured fibre surface, lumping of epoxy to a greater degree is clearly visible.



Fig.3.41 SEM image of specimen 35



Fig.3.42 SEM image of specimen 35 for EDS

Fibre Broken in Half

The SEM images of specimen which were subjected to 60% loading (T2, 1month) shown below indicate damaged fibre (changed circularity of fibre) and lumping of epoxy over fibre surface.





Fig.3.43 SEM image of specimen 37

Fig.3.44 SEM image of specimen 37 for EDS

The SEM images of specimen which were subjected to 80% loading (T2, 1month) shown below indicate circumferential damage to fibre, and some lumping of epoxy over the fibre surface.



Fig.3.45 SEM image of specimen 39



Fig.3.46 SEM image of specimen 39 for EDS

SEM RESULTS OF NaOH BATH T3 (after 1 month)

Holding Parameters: NaOH bath Time: 1 month Temperature: 55oC

The SEM images of specimen which were subjected to 20% loading (T3, 1month) shown below clearly indicate delamination of the fibres from the epoxy matrix, some amount of lumping of matrix is also observed.





Fig.3.47 SEM image of specimen 49

Fig.3.48 SEM image of specimen 49 for EDS

The SEM images of specimen which were subjected to 40% loading (T3, 1month) shown below indicate fibre being eaten up at the circumference and voids being formed, the fracturing of the fibre surface is also visible in fibre surface.



Fig.3.49 SEM image of specimen 51



Fig.3.50 SEM image of specimen 51 for EDS

The SEM images of specimen which were subjected to 60% loading (T3, 1month) shown below clearly indicate delamination of the fibres from the epoxy matrix in form of fibre pullout, some amount of lumping of matrix is also observed, though epoxy appears as a cohesive matrix.





Fig.3.51 SEM image of specimen 53

Fig.3.52 SEM image of specimen 53 for EDS

The results of specimen which were subjected to 80% loading (T3, 1month) shown below clearly indicate fibre damage (change in circularity), fibre fracture, voids between fibres in epoxy matrix.



Fig.3.53 SEM image of specimen 55



Fig.3.54 SEM image of specimen 55 for EDS

SEM RESULTS OF WATER BATH T4 (after 1 month)

Holding Parameters: Water bath Time: 1 month Temperature: 55oC

The SEM images of specimen which were subjected to 20% loading (T4, 1month) shown below show undamaged circular fibres and epoxy smearing the surface of the fibres completely in small irregular lumps probably due to absorption of water by epoxy and its flaking due to it.





Fig.3.55 SEM image of specimen 17

Fig.3.56 SEM image of specimen 17 for EDS

The SEM images of specimen which were subjected to 40% loading (T4, 1month) shown below indicate undamaged circular fibres and epoxy smearing the surface of the fibres completely due to absorption of water by epoxy and its flaking due to it.



Fig.3.57 SEM image of specimen 19



Fig.3.58 SEM image of specimen 19 for EDS

The SEM images of specimen which were subjected to 60% loading (T4, 1month) shown below indicate an absence of cohesive epoxy matrix with lumps epoxy covering the undamaged fibres.



Fig.3.59 SEM image of specimen 21

Fig.3.60 SEM image of specimen 21 for EDS

The SEM images of specimen which were subjected to 80% loading (T4, 1month) shown below indicate complete smearing of fibre surface by the epoxy, though fibre remains in undamaged state, the flaking of epoxy has occurred by absorption of water. :



Fig.3.61 SEM image of specimen 23



Fig.3.62 SEM image of specimen 23 for EDS

SEM IMAGES AFTER TWO MONTHS SEM AND EDS RESULTS OF WATER BATH T1 (after 2 months):

Holding Parameters: Water bath Time: 2 month Temperature: 45°C

The SEM images of specimen which were subjected to 20% loading (T1, 2months) shown below indicate degeneration of epoxy matrix, extensive void formation between fibres, flaking of epoxy is clearly evident, it appears as though epoxy has dissolved away from the surface, as protruding undamaged fibres are visible. :





Fig.3.63 SEM image of specimen 9 Fig.3.64 SEM image of specimen 9 for EDS The SEM images of specimen which were subjected to 40% loading (T1, 2months) shown below clearly show flaking of epoxy, which smears the fibre surface forming lumps, the degeneration of epoxy matrix is clearly visible, though no damage to fibres (no change in circularity, or fibre fracture) is observed.



Fig.3.65 SEM image of specimen 11



Fig.3.66 SEM image of specimen 11 for EDS

The SEM images of specimen which were subjected to 60% loading (T1, 2months) shown below indicate degeneration of epoxy matrix, void formation between fibres, the flaking of epoxy is clearly evident, though no fibre damage is seen.



Fig.3.67 SEM image of specimen 13

Fig.3.68 SEM image of specimen 13 for EDS

The SEM images of specimen which were subjected to 80% loading (T1, 2months) shown below indicate protruding undamaged fibres and degeneration of epoxy matrix, extensive void formation between fibres has taken place, with lumps of epoxy and smearing the fibre surface being clearly evident.



Fig.3.69 SEM image of specimen 15



Fig.3.70 SEM image of specimen 15 for EDS

SEM RESULTS OF NaOH BATH T2 (after 2 months)

Holding Parameters: NaOH bath Time: 2 month Temperature: 45°C

The SEM images of specimen which were subjected to 20% loading (T2, 2months) shown below clearly show the cracked fibre, fibre delamination and degradation of epoxy matrix , lumps of epoxy covering the fibre surface.



Fig.3.71 SEM image of specimen 41



Fig.3.72 SEM image of specimen 41 for EDS

The SEM images of specimen which were subjected to 40% loading (T2, 2months) shown below show extensive degradation of epoxy matrix with fibre pullout, damage of fibres, appearance of voids between fibres.



Fig.3.73 SEM image of specimen 43



Fig.3.74 SEM image of specimen 43 for EDS

The SEM images of specimen which were subjected to 60% loading (T2, 2months) shown below show damaged fibres, voids in matrix between fibres, degradation of epoxy matrix with epoxy forming small lumps over fibre surface.





Fig.3.75 SEM image of specimen 45

Fig.3.76 SEM image of specimen 45 for EDS

The results of specimen which were subjected to 80% loading (T2, 2months) shown below clearly show protruding fibres, degraded epoxy matrix, voids between fibres resulting in fibre pullout and fibre delamination besides fibre damage is also observed. :



Fig.3.77 SEM image of specimen 47



Fig.3.78 SEM image of specimen 47 for EDS

SEM RESULTS OF NaOH BATH T3 (after 2 months)

Holding Parameters: NaOH bath Time: 2 month Temperature: 55°C

The SEM images of specimen which were subjected to 20% loading (T3, 2months) shown below show the degraded epoxy matrix with epoxy flakes covering the protruding damaged glass fibres, voids between fibres in the epoxy matrix.



Fig.3.79 SEM image of specimen 57



Fig.3.80 SEM image of specimen 57 for EDS

The results of specimen which were subjected to 40% loading (T3, 2months) shown below show fibre delamination and voids between fibres. It also shows fibre damage (change in circularity) and lumps of epoxy being formed over the fibre surface.



Fig.3.81 SEM image of specimen 59



Fig.3.82 SEM image of specimen 59 for EDS

The results of specimen which were subjected to 60% loading (T3, 2months) shown below shows fibre pullout, formation of voids between fibres, fibre damage (change in circularity of fibres):





Fig.3.83 SEM image of specimen 61 Fig.3.84 SEM image of specimen 61 for EDS The SEM images of specimen which were subjected to 80% loading (T3, 2months) shown below show fibre cracking, change in fibre circularity, formation of voids, fibre delamination, degradation of epoxy matrix, smearing of epoxy over fibre surface.



Fig.3.85 SEM image of specimen 63



Fig.3.86 SEM image of specimen 63 for EDS

SEM RESULTS OF WATER BATH T4 (after 2 months)

Holding Parameters: Water bath Time: 2 month Temperature: 55°C

The SEM images of specimen which were subjected to 20% loading (T4, 2months) shown below show undamaged fibre, degradation of epoxy matrix forming flakes around the fibre surface.





Fig.3.87 SEM image of specimen 25

Fig.3.88 SEM image of specimen 25 for EDS

The SEM images of specimen which were subjected to 40% loading (T4, 2months)shown below show mostly undamaged fibre with only a rare ones with changed circularity, extensive degradation of epoxy matrix is visible forming lumps of epoxy smearing the fibre surface.





Fig.3.89 SEM image of specimen 27 Fig.3.90 SEM image of specimen 27 for EDS The SEM images of specimen which were subjected to 60% loading (T4, 2months) are shown below. They show damaged fibres and degraded epoxy matrix, with epoxy smearing the fibre surface. No fibre delamination or fibre pullout is observed.



Fig.3.91 SEM image of specimen 29

Fig.3.92 SEM image of specimen 29 for EDS

Damage to fibre

The SEM images of specimen which were subjected to 80% loading (T4, 2months) shown below show changes in fibre shape (circularity), smearing of epoxy over the fibre surface resulting in complete degradation of epoxy matrix. Some voids are also seen in the epoxy matrix surrounding the fibres.



- Fig.3.93 SEM image of specimen 31

Discussion of SEM Image Results

Fig.3.94 SEM image of specimen 31 for EDS

(a) Variation of SEM Image Results of specimen in water and NaOH baths

It is seen that in SEM images of specimen immersed in water show smearing and swelling of epoxy, indicAting damage to epoxy matrix but with minimal change in fibre shape. Whereas in SEM images of specimen immersed in NaOH baths show both epoxy matrix degradation in form of flake formation in epoxy and fibre damage shown by changes in fibre circularity.

(b) Variation of SEM Image Results of specimen in water and NaOH baths with change in time

With increased exposure time more epoxy matrix damage with void formation is observed both in water and in aquous NaOH baths. However with regards to fibre, not much change in fibre is observed in water baths, whereas increased fibre damage is observed in NaOH baths with increased exposure to the environment.

(c) Variation of SEM Image Results of specimen in water and NaOH baths with change in temperature

The change in temperature results in further degradation of the epoxy matrix in both the water and NaOH baths. There is not much change observed in fibre in specimen immersed in water baths, however fibre delamination is observed in NaOH bath at 55 degree Celsius.

(d) Variation of SEM Image Results of specimen in water and NaOH baths with change in pre-stretch loads

The effect of pre-stretch load variation is visible in water baths in terms of the degradation of epoxy matrix, its increased lumping, increase in formation of voids surrounding the fibres in the matrix. No effect is seen on the fibre. Similar is case with NaOH baths where with increasing pre-stretch loads increased matrix degradation is observed. Here though fibre degradation is observed but it is difficult to correlate it with the increase in pre-stretch loads.

3.2.2 Area fraction and Circularity using Image-J

The image analysis of all the SEM images was done in order to compare the area fraction of both epoxy as well as of the fibre and the circularity changes in the fibre. The commercially available software Image J was used for analysis of images. Image J is open source software developed by National Institute of Health (USA) and is considered as a powerful tool for image analysis. Few representative images after analysis by software are shown from Fig.3.95 to Fig.3.97. The red area represents the fibre, the green is taken as epoxy, and some black region observed is taken as voids. The yellow line represents the diameter of fibre.

After 1 month:





After 2 months:



Fig.3.96 (Left) SEM image of specimen 47, (Right) same SEM image by Image Analyser



Fig. 3.97 (Left) SEM image of specimen 63, (Right) same SEM image by Image Analyser

Circularity ratio = Minimum fibre diameter Maximum fibre diameter

The graphical comparison of the percentage area fraction results obtained from the image analysis of the SEM images using Image-J:



Fig.3.98 Comparison of Epoxy area fraction with respect to time



Fig.3.99 Comparison of Fibre area fraction with respect to time



Fig.3.100 Comparison of Circularity Ratio of fibre with respect to time

The area fraction of specimen is compared in Fig.3.98 for epoxy and Fig.3.99 for fibres. The T1,T2,T3 and T4 represent the water bath (45 degree Celsius), NaOH bath (45 degree Celsius), NaOH bath (55 degree Celsius), water bath (55 degree Celsius) respectively. The 20%, 40%, 60% and 80% are the pre-stretch loads given to specimen.

From comparison of epoxy area fractions it is clearly seen that epoxy area fraction is increasing with time as there is considerable increase after two month compared to one month. But the trend is seen to be opposite in fibre fraction comparison. Here the fibre fraction is increasing in first month and decreases in the second month. All the specimen loaded at different value show the same trend when compared for one and two month periods. The specimen in NaOH baths have shown more increase in epoxy area fraction after two months compared to one month and similarly more decrease in fibre area fraction when comparing to that of water baths.

The reason for such a trend seems to be that the fibre in total area is degrading with heat and moisture attack and epoxy seems to have expanded with above effect which obviously leaves

more area for epoxy as compared to fibre. In NaOH bath as seen in SEM images the fibre is also been eaten up at periphery which further supports more loss of fibre leaving more area for epoxy in the composite cross section.

The Circularity ratio (Fig.3.100) values have not changed in both the water baths for first month at all loads. For second month also there is no change in fibre circularity in water baths. Whereas circularity change is observed in NaOH baths in first month itself followed by further decrease in circularity in second month. The reason for such a trend is eating up of fibre by the NaOH attack leading to change in shape with most damage on circumference of fibres.

3.2.3 Composition on fibre surface (SEM-EDS):

The SEM+EDS measurements (shown in Fig 3.101) which are used generally for qualitative assessment were taken for the various specimen to assess the change in chemical composition of composite specimen with environmental degradation. The percentage elemental composition for specimen in various baths and with various pre-stretched loads was plotted in graphs shown in Figure 3.102 to Figure 3.112.



Fig.3.101 (a) Energy vs Electron volt graph at start for initial specimen, (b) Element percentage taken by EDS



Graphical comparison of the Composition (SEM-EDS) results for various baths:









Fig. 3.104 Composition on fibre surface (SEM-EDS) for specimen immersed in aq NaOH bath



Fig. 3.105 Composition on fibre surface (SEM-EDS) of specimen immersed in water bath (45 degree)



Fig. 3.106 Composition on fibre surface (SEM-EDS) for specimen immersed in water bath (55 degree)





Fig. 3.107 Composition on fibre surface (SEM-EDS) of specimen immersed in aq NaOH bath (45 degree)

Fig. 3.108 Composition on fibre surface (SEM-EDS) of specimen immersed in aq NaOH bath (55 degree)

Fig. 3.109 Composition of fibre surface (SEM-EDS) on water immersed specimen (one month)

Fig. 3.110 Composition of fibre surface (SEM-EDS) on water immersed specimen (two month)

Fig. 3.111 Composition of fibre (SEM-EDS) in aq NaOH immersed Specimen (one month)

Fig. 3.112 Composition on fibre suface (SEM-EDS) of specimen immersed in aq NaOH (two months)

From the analysis of the graphs shown in Fig.3.102 to Fig.3.112, following observations were made: The Fig 3.102 shows the elemental composition on the fibre surface of all the specimen immersed in water and aquous NaOH baths and of the unexposed specimen (specimen 0). It is easily seen that in the specimen immersed in aquous NaOH the percentage composition of silicon is generally higher as compared to that of specimen immersed in water baths and is also higher than the unexposed specimen. Whereas oxygen percentage values in water immersed specimen are generally higher than those observed for specimen in aquous NaOH baths. For the calcium percentage values similar trend is observed. The aluminium percentage values are nearly the same in aquous NaOH immersed specimen as compared to water immersed specimen and unexposed specimen. There is not any change in percentage magnesium composition for specimen immersed in both water and aquous NaOH baths and unexposed specimen, whereas titanium composition is decreasing in both water and aquous NaOH baths,

with titanium being reduced to traces in aquous NaOH bath. The element compositions observed are on the fibre surface and are basically either due to glass fibre composition, or due to moisture absorption by the fibre or due to fibre degradation and associated elemental diffusion process. It is observed that for specimen immersed in water baths the silicon content value is increasing rapidly with exposure time in water bath at temperature 55 degree celsius. This is not observed for specimen immersed in water bath at temperature 45 degree Celsius where the trend is reverse and percentage silicon values are decreasing with increasing time. NaOH bath. A reverse trend is also observed with calcium percentage values which decrease rapidly in water bath at 55 degree Celsius. It is also observed from Fig 3.109 and Fig 3.110 that with increasing temperature percentage silicon values at fibre surface are decreasing in specimen exposed to water for one month. This trend is also observed in second month. Similarly for percentage oxygen values at the fibre surface, with increasing temperature these values are increasing in specimen exposed to water for one month and after one month this trend is again observed. This trend is reversed in NaOH baths Fig 3.111 and Fig 3.112. The change in elemental composition of Si, O and Ca in water filled baths is of fluctuating nature attributed to different level of moisture diffusion and associated element diffusion in these specimen as no fibre degradation is observed in these specimen SEM images. For the case of NaOH specimen it is observed that the values of Ca, Si and O are stable in both months for nearly all loading conditions with slight change. This suggests a stable reaction equilibrium in the NaOH environment. In SEM images it is also seen that fibre is being eaten up in NaOH baths, this would suggest rather lower value for elements associated with fibre like Mg, Al, Ca and Si, to the contrary however rather stable values are observed for all specimen. This could suggest diffusion of these elements across the epoxy matrix between the fibres.

4. Conclusion

From the experimentation conducted following major conclusions have been obtained:

1) The reduction in tensile strength is considerably large (about 85 to 90%) in the almost all the specimen immersed in the aqueous NaOH bath as compared to the initial specimen tensile strength. This trend of reduction in tensile strength is on higher side in specimen loaded at 60% and 80% of U.T.L. While there is a marginal reduction in the tensile strength of the specimen immersed in water as compared to initial specimen. The trend of reduction in tensile strength in water bath is as per expectation, which shows 10-20% reduction in first month and 30-40% reduction in second month. The SEM images of these specimen also backup these results showing damage done to fibre especially in specimen immersed in aqueous NaOH baths, while there is no fibre damage in specimen immersed in water baths.

2) To further investigate the reason for reduction in strength a compositional change is taken into consideration by performing E.D.S. on damaged specimen. The trend shows very little change in almost all the elements like Silicon, Oxygen, Magnesium, Aluminium and Calcium in both water and aquous NaOH baths with few exceptions in water bath with regards to silicon, oxygen and calcium compositions. The analysis of chemical composition points out to the fact that there is moisture and element diffusion taking place between across the epoxy matrix and the fibres which result in the compositional changes observed.

3) The area fraction of the fibre and epoxy are analysed using image analysis. The results show an increase in area fraction of the epoxy with increasing time showing a marked increase in second month in all the specimen of both water and aqueous NaOH bath. Similarly the result of fibre area fraction shows a decreasing trend with time with marked decrease in second month. This indicates that epoxy is expanding (swelling) covering more area with fibre degradation and pullout leading to decrease in fibre in same area.

4) The change in circularity ratio is seen more pronounced in the fibres of specimen immersed in aqueous NaOH which can be noticed easily in SEM image of specimen. The chemical attack on periphery of fibres by NaOH leads to change in shape. Thus change in circularity ratio is evident in case of aqueous NaOH specimen but some specimen immersed in water bath had also shown chipping of an edge completely with rest of periphery in quite a circular shape.

5) The percentage weight gain showed an increasing trend with time as expected both in specimen immersed in water and aqueous NaOH bath. The weight gain is slightly more in specimen at 55°C compared to specimen at 45°C. But the trend in aqueous NaOH bath showed a larger weight gain than that in the water bath.

5. Direction of furthur work in future

1) The composite material with induced crack can be taken to study the effect of environmental exposure on pre-damaged composite materials.

2) The duration of current experiment can be increased to see the long term degradation effect.

4) The strength of alkaline aqueous solution can be varied to see the change in chemical attack on composite.

5) By changing the fibre orientation and number of plies we can compare the change in results with respect to current experimentation.

6) The use of cyclic loads can be made instead of static pre-stretching to see its effect on hygrothermal degradation of GFRP composites.

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