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Hygrothermal Degradation Studies on E-Glass Woven Rovings-Epoxy Composite

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Abstract

In the present work, the property degradation of the selected material (i.e. E-glass & epoxy resin composite) manufactured by compression molding was investigated as a function of temperature after direct wetting in saline medium for varying periods. For the preparation of composite specimen, ARADUR HY951 used as hardener with ARALDITE LY556. From the experiment, moisture gain trends for different temperatures which vary with time. In that, hot setting laminate having good strength when compared to cold setting laminate. It is hoped that generation of such data will help in determining the active service life of products, beyond which, they need to be discarded to prevent catastrophic failures.

Keywords: Hygrothermal degradation, Compression molding, Composite

Introduction

Composite material is a material composed of two or more distinct phases (matrix phase and dispersed phase) and having bulk properties significantly different from that of its constituents. These composite applications touch several engineering fields like marine, land transportation (automobiles, railways) construction, structural elements in machinery, electrical, wind energy, material transportation piping systems, leisure & sports goods, aircraft interiors, military aerospace etc. Yasushi Miyano and Masayuki Nakada [1] are deals with the prediction of long-term fatigue life of various FRP laminates combined with resins, fibers and fabrics for marine use under temperature and water environments based on the time-temperature superposition principle (TTSP). Autar K. Kaw [2] introduced the first edition of Mechanics of Composite Materials, the ground breaking PROMAL software, a valuable tool for designing and analyzing structures made of composite materials. R.O. Ochola, K. Marcus and T. Franz [3] are presented the choice of composite materials as a substitute for metallic materials in technological applications is becoming more pronounced especially due to the great weight savings these materials offer. J.Tong [4] studied multiple fatigue crack growth behaviour has in quasi-isotropic GFRP laminates under constant amplitude fatigue loading conditions. Characteristics of fatigue crack growth in off-axis plies have been described and comparisons have been made between quasi-static and fatigue crack growth behaviour. Y. Miyano and M.Nakada [5] are presented the accelerated testing methodology has been proposed for the long-term durability of polymer composites based on the time–temperature superposition principle to be held for the viscoelasticity of polymer matrix. V.K. Srivastava [6] investigated the effects of water immersion on mechanical properties as flexural strength, interlaminar shear strength and impact energy of aluminium tri-hydrate and polyethylene filled and unfilled quasi-isotropic glass fibre reinforced epoxy vinylester resin composites (GFRP). G. Mishra, S.R. Mohapatra and P.R. Behera [7] are investigated the effect of thermal and cryogenic treatment on hygrothermally conditioned glass fibre reinforced epoxy matrix composites, and the impact on its mechanical properties with change in percentage of individual constituents of the laminates.J.L.V Coelho and J.M.L.Reis [8] are presented the mechanical response of a composite material based on glass fibers embedded in an epoxy resin was experimentally studied as a function of strain rate and temperature. Ichsan setya putra and Djoko Suharto are analyzed a manufacturing process for glass fiber reinforced plastics (GFRP) to improve volume fraction of fibers and mechanical properties. S.B.Singh and Himanshu Chawla are presented an experimental investigation of the effect of cutouts on the natural frequency and damping of the plate composite laminates were made from unidirectional glass fiber with stacking sequence of $(0/90)_s$. P.Sampath Rao, M. Manzoor Husain and D.V. Ravi Shankar [11] are studied the properties of the reinforcement materials which are highly hygroscopic, the matrix material which provides protection to the reinforcement.

Experimental Investigation

I.Preparation of composite laminate

Epoxy resins are low temperature curing resins, normally between 200 to 900°C, but some

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formulations are made for high temperature curing. The epoxy used in making of laminates was ARALDITE LY556, which is a liquid with medium viscosity. Aradur HY 951 is a low viscosity, unmodified, aliphatic polyamine. Resin and hardener should be mixed uniformly until they form a homogenous mixture. ARALDITE LY 556 with ARADUR HY951 provides a low viscosity, solvent free room temperature curing laminate system. By varying the contents of resin from 0 to 10 parts and hardener from 10 to 12 parts, the reactivity of the system can be adapted to suit the processing & cutting condition. The E-glass fiber woven roving used as matrix and mixture of resin and hardener used as reinforcement by preparing composite laminate, and the laminate placed in the mould and curing in hydraulic press. After the making of the laminates, the laminates are cut into small specimens, making them suitable for the hygrothermal testing purpose. The specifications of the laminate are almost equal to the dimensions of the mould. All the laminates thus prepared were of the dimension (after removal of the edges): length of the laminate is 380mm, Height (width) is 340mm and average thickness is 3.5mm for hot setting. The other specifications of the specimens were (250*25*3.75) mm in case of cold setting according to the standards. Immense care has been taken while cutting as to get all the specifications isotropically. As many as 36 specimens have been manufactured to facilitate the testing.

II.Testing Parameters

The testing takes into consideration many factors that seriously brings out a change in the mechanical properties of the composite specimen. There are two kinds of testing parameters:

1) Fixed parameters
   - Relative Humidity
   - Cross head speed of the UTM machine (Strain rate of the specimen).
2) Variable parameters
   - Concentration of the bath.
   - 5% salinity solution Temperature of the environment.
   - ✓ Ambient room temperature at 32°C
   - ✓ Elevated temperature of 50°C
   - ✓ Elevated temperature of 85°C
   - Time duration of exposure to degrading environment
   - ✓ 1hr, 5hr, 10hr for ambient temperature
   - ✓ 30mins, 60mins, 90mins for elevated temperatures

III.Calculations of cold setting

Weight of the total composite (fiber+matrix)=0.700gm
Weight of the fiber left after heating=0.500gm
Weight of the resin in composite=0.200gm
Density of the fiber(woven roving)=2540gm/cm^3
Density of the resin(epoxy-LY556)=960gm/cm^3
Hence, calculating the volume fraction:
Volume of fiber in the composite specimen (v_f) = (0.500/2540)=0.00019685cm^3
Volume of matrix in the composite specimen (v_m) = (0.200/960)=0.000208333cm^3
According to rule of mixtures
For a two component composite
Total volume (V)=(v_f+v_m) =0.00040518cm^3
Hence volume fraction of fiber =

$$\frac{Volume\ of\ fiber}{Volume\ of\ composite}$$

$$= \frac{0.00019685}{0.00040518}$$

$$= 0.48582996$$

Hence it shows that the volume fraction of fiber of cold setting laminate is around **48.582996%** in the total composite.

IV.Calculations of hot setting

Weight of the total composite (fiber + matrix) =580gm
Weight of the fiber left after heating=0.450gm
Weight of the resin in composite=0.130gm
Density of the fiber (woven roving) =2540gm/cm^3
Density of the resin (epoxy-LY556) =960gm/cm^3
Hence, calculating the volume fraction
Volume of fiber in the composite specimen
(V_f)= (0.450/2540) = 0.00017716cm^3
Volume of the matrix in the composite specimen (v_m)=(0.130/960)=0.00013541cm^3

According to rule of mixtures
For a two component composite,
Total volume (V)= (V_f+V_m)=0.00031258cm^3
Hence Volume fraction of Fiber =

$$\frac{Volume\ of\ fiber}{Volume\ of\ composite}$$

$$= \frac{0.00017716}{0.000312588}$$

$$= 0.566780373$$

Hence it shows that the volume fraction of fiber of cold setting laminate is around 56.6780373% in the total composite

V.***Exposing the specimens to Degrading Environment***

The specimens thus made, are exposed to degrading environment, i.e. Brine Solution of different concentrations. According to the literature, the sea water that has the highest salinity has 2% to 5% salinity in it. Hence, this research essentially throws light on the degradation in similar conditions. By simulating the real- time conditions; the research has been carried out in two different concentrations of brine:

Study of tensile stress degradation in 5% Brine solution.

5% Brine Solution Bath

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A 5% brine solution bath is made in the exposure chamber designed. This 5% brine solution is heated to required temperature using the heater element attached with the thermostat. The test rig is shown in figure1.

Figure1.Test Rig

A total of 36 specimens are used for analysing the hygrothermal degradation. The study is carried out at 3 different temperatures- Ambient temperature, 50ºC and 85ºC. For each of the temperatures 6 specimens (unsealed specimens) are used as shown in table1.

Table1. Number of specimens at different temperatures

Bath temperature	Holding time	Specimens
Ambient temperature (32°c)	1 hour	2
	5 hours	2
	10 hours	2
50°c	30 minutes	2
	60 minutes	2
	90 minutes	2
85°c	30 minutes	2
	60 minutes	2
	90 minutes	2
Total number of specimens		18

Results and Discussions

Moisture Gain calculations

Moisture gain plays a pivotal role in the degradation of mechanical properties. Gravimetric trends monitoring the weight change of a material over time allow for the interpretation of diffusion phenomena through the application of diffusion models. Knowledge of the process of water sorption in a polymer composite provides for an understanding of physical processes which occur as the water and constituent elements interact. When considering the uptake of water in material exposed to humid air and liquid water, it is assumed that the only absorbing substance is water molecules. The apparent moisture content at some time t, Mt, is calculated using the initial weight after pre-conditioning Wo and the "wet" weight after environmental exposure Ww

$$Mt = \frac{(Ww - Wo)}{Wo}$$

The weights of all specimens before and after the dipping are tabulated in tables 2 - 13. The following shows the moisture gain in grams and percentage w.r.t the original weight of the specimen.

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Table2. 5% brine solution at ambient temperature of hot setting tensile test specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 1 hour	23.9360	23.9369	0.0037%
Dipping time 5 hours	23.008	23.009	0.0043%
Dipping time 10 hours	23.691	23.699	0.0337%

Table3. 5% brine solution at 50°C of hot setting tensile test specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	22.848	22.854	0.0262%
Dipping time 60minutes	23.731	23.741	0.0421%
Dipping time 90 minutes	24.386	24.398	0.0492%

Table4. 5% brine solution at 85°C of hot setting tensile specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	23.311	23.328	0.0729%
Dipping time 60minutes	23.933	23.960	0.1128%
Dipping time 90 minutes	23.669	23.709	0.1689%

Table5. 5% brine solution at ambient temperature of hot setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 1 hour	5.922	5.923	0.01685%
Dipping time 5 hours	5.386	5.388	0.0371%
Dipping time 10 hours	5.440	5.444	0.0735%

Table6. 5% brine solution at 50°C of hot setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	5.437	5.439	0.0367%
Dipping time 60 minutes	5.446	5.449	0.0550%
Dipping time 90 minutes	6.192	6.196	0.0645%

Table7. 5% brine solution at 85°C of hot setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	5.367	5.373	0.1117%
Dipping time 60 minutes	6.051	6.060	0.1487%
Dipping time 90 minutes	5.397	5.411	0.2594%

Table8. 5% brine solution at ambient temperature of cold setting tensile specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 1 hour	25.429	25.427	-0.0078%
Dipping time 5 hours	25.476	25.472	-0.0235%
Dipping time 10 hours	25.637	25.630	-0.0273%

Table9. 5% brine solution at 50°C of cold setting tensile specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	24.949	24.947	-0.0080%
Dipping time 60 minutes	25.216	25.212	-0.0158%
Dipping time 90 minutes	25.715	25.710	-0.0194%

Table10. 5% brine solution at 85°C of cold setting tensile specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	25.501	25.492	-0.0353%
Dipping time 60 minutes	25.297	25.281	-0.0632%
Dipping time 90 minutes	24.582	24.565	-0.0691%

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Table11. 5% brine solution at ambient temperature of cold setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 1 hour	6.311	6.311	0%
Dipping time 5 hours	6.076	6.075	-0.0164%
Dipping time 10 hours	6.295	6.293	-0.0317%

Table12. 5% brine solution at 50°C of cold setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	6.366	6.364	-0.0314%
Dipping time 60 minutes	6.006	6.003	-0.0499%
Dipping time 90 minutes	6.313	6.309	-0.0633%

Table13. 5% brine solution at 85°C of cold setting short beam shear strength specimen

Dipping time of specimen	Weight of specimen before dipping (W)	Weight of specimen after dipping(w)	Moisture gain %
Dipping time 30 minutes	6.085	6.081	-0.0657%
Dipping time 60 minutes	6.116	6.110	-0.0981%
Dipping time 90 minutes	5.981	5.973	-0.1337%

Behaviour of Strength with lapse of time under hygrothermal conditions:

Results for the tensile strength deterioration for specimens dipped in 5% brine solution

The specimens are subjected to **direct dipping** in a 5% brine solution bath for different time durations. Bath maintained at 500°C and 850°C were held for 30mins, 60mins and 90mins and are then cleaned with a blotting paper and then tested in a UTM. For all the varying parameters, it was observed that, there is a considerable, though not uniform, decrease in the tensile strength of the material after exposition. The following tables 14 - 17 show the trend of decrease of tensile strength for unsealed specimens according to the change in the parameters like, time and temperature.

Table14. Hot-setting Laminate Tensile Test

Bath temperature	Holding time	Control value	Tensile strength (Mpa) of specimen
Ambient temperature	1 hour	476.19	438.09
	5 hours	476.19	419.05
	10 hours	476.19	400.00
50°C	30 minutes	476.19	413.14
	60 minutes	476.19	392.15
	90 minutes	476.19	371.40
85°C	30 minutes	476.19	389.43
	60 minutes	476.19	370.37
	90 minutes	476.19	352.83

Table15. Cold-setting Laminate Tensile Test

Bath temperature	Holding time	Control value	Tensile strength of specimen
Ambient temperature	1 hour	424.77	391.11
	5 hours	424.77	373.33
	10 hours	424.77	355.55
50°C	30 minutes	424.77	371.35
	60 minutes	424.77	353.67
	90 minutes	424.77	335.98
85°C	30 minutes	424.77	351.80
	60 minutes	424.77	334.21
	90 minutes	424.77	316.62

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Table16. Hot-setting short beam shear test

Bath temperature	Holding time	Control value	Short beam shear strength (Mpa) specimen
Ambient temperature	1 hour	31.42	30.14
	5 hours	31.42	29.22
	10 hours	31.42	28.07
50°C	30 minutes	31.42	29.76
	60 minutes	31.42	28.65
	90 minutes	31.42	27.43
85°C	30 minutes	31.42	28.76
	60 minutes	31.42	27.43
	90 minutes	31.42	26.15

Table17. Cold setting short beam shear test

Bath temperature	Holding time	Control value	Short beam shear strength specimen
Ambient temperature	1 hour	26.76	24.62
	5 hours	26.76	23.47
	10 hours	26.76	22.09
50°C	30 minutes	26.76	23.52
	60 minutes	26.76	22.25
	90 minutes	26.76	20.79
85°C	30 minutes	26.76	22.37
	60 minutes	26.76	21.01
	90 minutes	26.76	19.89

The percentage degradation in tensile strength of the hot and cold specimen results are shown in tables 18-21.

Table18. Hot setting Tensile strength

Holding time	% gain in weight after exposure			Tensile Strength after exposure. (Mpa)			% Degradation in tensile strength		
	32°c	50°c	85°c	32°c	50°c	85°c	32°c	50°c	85°c
After first holding time	0.0037	0.026	0.0729	438.09	413.14	389.43	8.001	13.24	18.219
After second holding time	0.0043	0.042	0.1128	419.05	392.15	370.37	11.99	17.64	22.222
After third holding time	0.0337	0.049	0.1689	400.00	371.40	352.83	15.99	22.00	25.905

Table19. Cold setting tensile strength

Holding time	% gain in weight after exposure			Tensile Strength after exposure. (Mpa)			% Degradation in tensile strength		
	32°c	50°c	85°c	32°c	50°c	85°c	32°c	50°c	85°c
After first holding time	-0.007	-0.008	-0.035	391.11	371.35	351.80	7.924	12.576	17.178
After second holding time	-0.023	-0.015	-0.063	373.33	353.67	334.21	12.110	16.738	21.319
After third holding time	-0.027	-0.019	-0.069	355.55	335.98	316.62	16.295	20.903	25.460

Table20. Hot setting Short beam shear strength

Holding time	% gain in weight after exposure			Tensile Strength after exposure. (Mpa)			% Degradation in tensile strength		
	32°c	50°c	85°c	32°c	50°c	85°c	32°c	50°c	85°c

After first holding time	0.0168	0.036	0.1117	30.14	29.76	28.76	4.0738	5.2832	8.4659
After second holding time	0.0371	0.055	0.1487	29.22	28.65	27.43	7.0019	8.8160	12.6989
After third holding time	0.0735	0.064	0.2594	28.07	27.43	26.15	10.6619	12.6989	16.7727

Table21. Cold setting Short beam shear strength

Holding time	% gain in weight after exposure			Tensile Strength after exposure.			% Degradation in tensile strength		
	32°c	50°c	85°c	32°c	50°c	85°c	32°c	50°c	85°c
After first holding time	0	-0.031	-0.0657	24.62	23.52	22.37	7.997	12.10	16.405
After second holding time	-0.0164	-0.049	-0.0981	23.47	22.25	21.01	12.29	16.85	21.487
After third holding time	-0.0317	-0.063	-0.1337	22.09	20.79	19.89	17.45	22.30	25.672

Conclusion

The main aim of this dissertation lies in finding the durable life of the composite structure under degrading environment. From this experiment conducted, Moisture gain trends for different temperatures which vary with time. Hot setting laminate is proved experimentally to have good strength when compared to cold setting laminate. Actually I used cold setting hardener for preparing laminate. so, this laminate cured at room temperature within 24 hours, but this curing at 200^{0}c in 1 hour. so, the percentage gain values in negative form. In this project % of degradation is high at 85^{0}c because of at high temperature the specimen gain more moisture compare to below temperatures.

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