Effect of Hygrothermal Loading on Laminate Composites

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Abstract

Objective: To find the effect of hygrothermal loading on CFRP and GFRP composite materials by finding the properties like capacitance, micro hardness, moisture diffusivity and area of fraction. Methods: Hand layup and vacuum bag methods were used for fabrication of composites. Finding: Three-point bending test was done on each specimen by varying load, i.e., zero load, with 30% load, 50% load and 70% load for different time periods. Micro hardness and capacitance were determined using Vickers Hardness tester and LCR meter respectively on both the composites before and after the thermal treatment. The capacitance increased with time whereas the percentage area of fraction of the fibre decreased. Also, the reduction in strength and damage to fibre were investigated using SEM. It was observed that with increase in %weight gain, micro hardness of specimen decreased. Application/Improvement: FRPs can substitute traditional steel reinforcements in reinforced concrete structures. The continuous loading under thermal actions due to natural environmental factors lead to degrading effect that needs to accurate investigations in evaluating durability of fibre reinforced polymers.

Keywords: Area of Fraction, Capacitance, Failure Modes, Hygrothermal Loading, Micro Hardness

1. Introduction

Composite materials are the engineered made up of two or more constituent materials with different mechanical properties. The final product has properties higher than that of individual components. It is a combination of two materials matrix phase and reinforcement phase, matrix is a continuous phase usually more ductile and holds the reinforced phase dispersed in it and both shares the load applied. Different materials are obtained by changing the matrix and reinforced phases. The material mentioned in this paper is made by FRP as reinforced phase and epoxy as matrix phase. The main benefits of composites is that, less weight, high strength-to-weight ratio, directional stiffness and surface properties like corrosion resistance etc. made to use composites in almost every field of engineering including aerospace applications. Delamination I the major type of failure¹². As the FRP material is concerned, the environmental factors like moisture, temperature, heat or radiation from surroundings has a great impact on the degradation³⁴ of the material. The present study deals with impact of hygrothermal loading⁵⁶ on FRP materials⁷⁸, this effect is calculated by using capacitance, impedance values and by calculating molecular diffusivity. The specimens are fabricated according to ASTM standards⁹¹⁰ and is kept in a water bath maintained at a temperature¹¹¹² and is taken out at regular interval of time and is tested, readings are tabulated. Firstly, the material is pre conditioned and ultimate load is determined using UTM and 30%, 50% and 70% of ultimate load is applied, for both hygrothermally placed material¹³ and normal material, results are compared in both the cases. The reasons behind the decrease or increase in properties are discussed.

2. Experimentation and Fabrication of Specimen

Specimens are made by using commercially available GFRP and CFRP sheets and placed along 0° orientation side for cutting purpose. In order to decide the specifica-
tions, ASTM standard D3037/3039 and D790 were used and mentioned in Table 1.

Table 1. Specimen specifications used for testing

| S. No | Specification of the Specimen | For tensile testing (mm) | For bending test (mm) |
|-------|-------------------------------|--------------------------|----------------------|
| 1.    | Length                        | 500                      | 250                  |
| 2.    | Width                         | 15                       | 15                   |
| 3.    | Thickness                     | 3 (for GFRP) & 4.32 (for CFRP) | 3 (for GFRP) & 4.32 (for CFRP) |

The specimens are placed in water bath and maintained at a temperature (45º) and at regular interval of time as mentioned in the preceding tabular columns, specimen is taken out and test are done accordingly.

3. Testing Methods Used

3.1 Tensile Testing

A UTM is used for the testing the tensile strength of the specimen and prepared according to ASTM-D-3039 standard. Initial pre stretching is done, (shown in Figure 1) peak load is calculated i.e., ultimate stress value that the specimen can with stand.

![Figure 1. Tensile laminate composite.](image)

3.2 Capacitance Test

In order to relate the moisture absorption rate with capacitance, the capacitance and impedance readings from RCL meter are used (shown in Figure 2). To create magnetic field in the specimen copper strips were used as terminals.

![Figure 2. LCR meter.](image)

3.3 Micro Hardness Test

To estimate the hardness of the specimen before and after thermal treatment micro hardness test is used. A load of 200gm is applied and VHN values were determined by applying this load and dwell time used while applying load is 20 sec. A diamond shaped indent is formed as shown in Figure 3.

![Figure 3. Indent of the specimen.](image)

3.4 SEM (Scanning Electron Microscopy)

To find the micro structure of the specimen scanning electron microscopeis used and polishing is done by gold coating equipment and microstructure is obtained at magnification factor of 2000X and 1500X.

4. Failure Modes in Composites

Delamination and fibre pull-out are the failure mechanisms in composite materials. Delamination is an insidious kind of failure and it develops inside of the material (Figure 4) and it may be detected in a material by its sound. Delaminated part sounds fewer dull compared to solid composite which give more sound. Non-destructive testing may also be used to determine the failure in composites which include ultrasonic, radiographic and infrared imaging.

5. Percentage Weight Gain

The pre conditioned sample is weighed periodically and ensure that moisture on the surface is wiped before weighing. Weight gain is determined as follows

\[ \%W = \frac{W_1 - W_2}{W_2} \]

where,

- \( \%W \) is weight gain percentage in specimen
- \( W_1 \) is weight of specimen
Figure 4. Failure of CFRP and GFRP laminates after 1 month.

6. Calculation of Apparent Moisture Diffusivity

Assuming that the moisture absorption follows Frick’s law, the diffusivity D can be calculated as,

\[ D = \pi \left( \frac{h}{4M_m} \right)^2 \left( \frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}} \right) \left[ 1 + \frac{h}{L_e} + \frac{h}{W} \right]^{-2} \]

where,
Le- length of the specimen
W- width of specimen
M1- moisture content at time t1
M2- moisture content at time t2
Mm- % weight gain from above equation

7. Results and Discussion

The initial specimen had a fine and shiny epoxy coating, when loaded under uniaxial tensile load showed a broom type failure due to instant release of stored energy.

8. Moisture Diffusivity Results of Bending Laminate

The moisture diffusivity is compared for various loads (Tables 2 and 3) and is noted that the specimens kept in water-bath (T2) for two months absorbed more moisture than that of specimens of first month in almost all the specimens. The reason behind the decrease in moisture diffusivity is that, Weight gain of specimen increases with time for a given hygrothermal load giving way to moisture. The moisture diffusivity in GFRP is almost same as shown in Figure 6. The range of moisture diffusivity values of CFRP (Figure 5) specimens were low compared to GFRP as shown in Figure 6.

Table 2. Moisture diffusivity of GFRP

| Time period | GFRP 30% loading | GFRP 50% loading | GFRP 70% loading | GFRP without load |
|-------------|------------------|------------------|------------------|------------------|
| 3           | 0.0113           | 0.0109           | 0.0110           | 0.0112           |
| 6           | 0.0055           | 0.0055           | 0.0055           | 0.0056           |
| 9           | 0.0036           | 0.0036           | 0.0037           | 0.0037           |
| 10          | 0.0063           | 0.0061           | 0.0059           | 0.0060           |
| 12          | 0.0027           | 0.0027           | 0.0027           | 0.0028           |
| 20          | 0.0018           | 0.0017           | 0.0016           | 0.0014           |
| 30          | 0.0011           | 0.0010           | 0.0010           | 0.0012           |
| 40          | 0.0009           | 0.0008           | 0.0009           | 0.0009           |
| 50          | 0.0014           | 0.0014           | 0.0013           | 0.0012           |

Table 3. Moisture diffusivity in CFRP laminated specimen

| Time period | CFRP 30% loading | CFRP 50% loading | CFRP 70% loading | CFRP without load |
|-------------|------------------|------------------|------------------|------------------|
| 3           | 0.007            | 0.020            | 0.018            | 0.020            |
| 6           | 0.022            | 0.010            | 0.009            | 0.010            |
| 9           | 0.011            | 0.007            | 0.006            | 0.007            |
| 10          | 0.006            | 0.006            | 0.008            | 0.008            |
| 12          | 0.005            | 0.005            | 0.005            | 0.005            |
| 20          | 0.001            | 0.001            | 0.001            | 0.001            |
| 30          | 0.001            | 0.001            | 0.001            | 0.001            |
| 40          | 0.001            | 0.001            | 0.001            | 0.001            |
| 50          | 0.002            | 0.001            | 0.001            | 0.001            |

Figure 5. Graph for moisture diffusivity of GFRP.

Figure 6. Graph for moisture diffusivity in CFRP.
9. LCR Meter Results of Bending Specimen

The capacitance of various loaded specimens (Tables 4, 5) are compared and is noted that capacitance increasing with respect to time indicating the degradation of the fibre. There is a marginal increase during early days but with course of time i.e. after 40days in GFRP and 50 days in CFRP there is a marginal increase. The reason behind the increase in capacitance is that with time the %weight gain increases for a given hygrothermal load giving way to moisture. The capacitance of 30%loaded GFRP specimen has attained saturation before other specimen as shown in Figure 7, whereas for CFRP specimen started increasing after one month as it started gaining weight as shown in Figure 8.

### Table 4: Capacitance results in GFRP laminated specimen

| Time period | GFRP 30% loading | GFRP 50% loading | GFRP 70% loading | GFRP Without load |
|-------------|------------------|-----------------|-----------------|------------------|
| 3           | 1.78E-11         | 2.90E-11        | 2.58E-11        | 2.08E-11         |
| 6           | 4.38E-11         | 3.35E-11        | 3.45E-11        | 2.59E-11         |
| 9           | 4.61E-11         | 3.60E-11        | 3.60E-11        | 2.63E-11         |
| 10          | 8.85E-11         | 3.73E-11        | 4.62E-11        | 3.42E-11         |
| 12          | 8.92E-11         | 3.99E-11        | 6.19E-11        | 3.58E-11         |
| 20          | 8.97E-11         | 5.31E-11        | 7.11E-11        | 3.69E-11         |
| 25          | 9.00E-11         | 6.12E-11        | 7.16E-11        | 4.79E-11         |
| 30          | 9.20E-11         | 7.04E-11        | 7.67E-11        | 6.95E-11         |
| 40          | 9.30E-11         | 7.74E-11        | 8.36E-11        | 7.52E-11         |
| 50          | 9.52E-11         | 8.51E-11        | 9.13E-11        | 9.13E-11         |

### Table 5: Capacitance results in CFRP laminate specimen

| Time period | CFRP 30% loading | CFRP 50% loading | CFRP 70% loading | CFRP Without load |
|-------------|------------------|-----------------|-----------------|------------------|
| 3           | 1.03E-12         | 1.86E-12        | 3.40E-12        | 4.20E-13         |
| 6           | 2.04E-12         | 2.28E-12        | 3.60E-12        | 1.20E-12         |
| 9           | 2.60E-12         | 4.20E-12        | 4.36E-12        | 6.20E-12         |
| 10          | 4.82E-12         | 4.98E-12        | 5.30E-12        | 1.32E-11         |
| 12          | 5.10E-12         | 5.70E-12        | 7.40E-12        | 1.40E-11         |
| 20          | 5.70E-12         | 7.90E-12        | 9.80E-12        | 1.54E-11         |
| 25          | 1.24E-11         | 9.00E-12        | 1.86E-11        | 1.93E-11         |

### Figure 7. Graph for capacitance results in GFRP

### Figure 8. Graph of capacitance results in CFRP

10. Percentage Weighgt Gain of Bending Specimen

The Percentage weight gain (of moisture) was compared for various loads (shown in Tables 6, 7). It is noted that the percentage weight gain is increasing with respect to time indicating more moisture is absorbed after two months compared to first month in almost all the cases. The moisture absorption of CFRP (Figure 10) specimen is comparatively low as that of GFRP (Figure 9), so more time to degrade. The reason for increasing the weight gain with time is that, the pores of epoxy will loosen due to given hygrothermal load giving way to moisture.

### Table 6: Percentage weight gain results of laminated bending composites

| Time period | GFRP 30% loading | GFRP without load | GFRP 70% loading | GFRP 50% loading |
|-------------|------------------|------------------|-----------------|-----------------|
| 3           | 9.58             | 9.78             | 9.48            | 9.25            |
| 6           | 10.51            | 10.57            | 9.26            | 10.00           |
| 9           | 11.13            | 10.60            | 11.97           | 9.88            |
| 10          | 9.14             | 8.88             | 8.95            | 10.29           |
| 12          | 12.17            | 12.97            | 13.43           | 11.50           |
| 20          | 14.37            | 16.81            | 14.22           | 14.69           |
| 30          | 14.01            | 16.34            | 17.20           | 17.65           |
| 40          | 16.58            | 16.85            | 19.35           | 18.11           |
| 50          | 14.15            | 20.06            | 21.62           | 18.22           |
**Table 7.** Percentage weight gain in CFRP laminated specimen

| Time period | CFRP 30% loading | CFRP 50% loading | CFRP 70% loading | CFRP without load |
|-------------|------------------|------------------|------------------|-------------------|
| 3           | 4.39             | 3.71             | 3.88             | 3.80              |
| 6           | 3.48             | 3.25             | 3.55             | 3.00              |
| 9           | 3.80             | 4.04             | 4.80             | 3.98              |
| 10          | 4.26             | 4.51             | 4.44             | 4.10              |
| 12          | 4.75             | 4.08             | 5.91             | 3.94              |
| 20          | 6.05             | 7.09             | 4.83             | 4.83              |
| 30          | 8.34             | 8.53             | 7.87             | 7.07              |
| 40          | 9.45             | 10.89            | 9.46             | 8.39              |
| 50          | 7.22             | 11.51            | 10.16            | 11.58             |

**Figure 9.** Graph of percentage weight gain in GFRP.

**Figure 10.** Graph of percentage weight gain in CFRP.

**11. Micro Hardness of Laminates**

The micro hardness readings of GFRP and CFRP (Tables 8, 9) laminates specimen decreased when compared to initial readings before hygrothermal treatment due to hardening of epoxy with absorption of water and micro hardness of GFRP (Figure 11) specimen is same at the end of exposure duration, whereas for CFRP (Figure 12) there is no appreciable change after some days.

**Table 8.** Micro-hardness results in GFRP laminated specimen

| Time period | GFRP 30% loading | GFRP without load | GFRP 70% loading | GFRP 50% loading |
|-------------|------------------|-------------------|------------------|------------------|
| 3           | 14.75            | 13.78             | 15.71            | 12.85            |
| 6           | 12.81            | 12.56             | 13.63            | 12.79            |

**Figure 11.** Graph of micro-hardness in GFRP.

**Figure 12.** Graph of micro-hardness in CFRP.

**12. % Area Fraction of Bending Laminate**

Graphs between %area of fraction and time period is drawn using values shown in Table 10. The GFRP bend-
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Moisture diffusivity of GFRP specimen is decreasing relatively with time for all specimens whereas for CFRP specimen decrease is almost same for all materials and after one-month moisture diffusivity of CFRP specimen has decreased.

Capacitance of CFRP specimen had not attained saturation as water absorption is slow and the capacitance was increasing with time.

The reduction in strength and damage in fibre is investigated using SEM. It was observed that with increase in % weight gain, micro hardness of specimen decreases. It was also observed that with time % area of fraction of fibre decreases.

### Table 10. Percentage area fraction for bending specimen

| Time period         | %area fraction of fibre (GFRP) | %area fraction of fibre (CFRP) | %area fraction of epoxy (GFRP) | %area fraction of epoxy (CFRP) |
|---------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| Initial             | 69.2                          | 68.8                          | 30.8                          | 31.2                          |
| 30% loading (month 1)| 67.3                          | 64.1                          | 32.7                          | 35.9                          |
| 30% loading (month 2)| 67.8                          | 57.8                          | 42.2                          | 42.2                          |
| 50% loading (month 1)| 67.3                          | 60.7                          | 32.7                          | 39.3                          |
| 50% loading (month 2)| 60.9                          | 51.3                          | 39.1                          | 48.7                          |
| 70% loading (month 1)| 62.6                          | 33.8                          | 37.4                          |                               |
| 70% loading (month 2)| 63                            | 34.2                          | 37                            |                               |

13. Conclusion

Moisture diffusivity of GFRP specimen is decreasing relatively with time for all specimens whereas for CFRP specimen decrease is almost same for all materials and after one-month moisture diffusivity of CFRP specimen has decreased.

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