Original Article

Influence of seawater absorption on vibrational and tensile characteristics of quasi-isotropic glass/epoxy composites

Acharya Pavan, Pai Dayananda, Kini M. Vijaya, Sriharsha Hegde, Padmaraj Narampady Hosagade

Article history:
Received 14 July 2018
Accepted 3 October 2018
Available online 28 November 2018

Keywords:
Damping
Quasi-isotropic
Stiffness
Seawater
Ageing
Moisture absorption

ABSTRACT

Glass fibre reinforced composites are common structural materials in civil engineering and marine applications. Exposure to moisture is the key challenge for using fibre-reinforced composites in marine structures. This study focuses on preparation of quasi-isotropic E-glass/epoxy laminates with stacking sequence [0°/90°/+45°/−45°], fabricated using vacuum bagging technique. Ageing studies in artificial seawater was conducted in sub-zero and ambient temperatures for a duration of 3600 h. Moisture absorption behaviour, variation of damping, stiffness properties, and microstructural failure analysis studies were carried out as per ASTM standards. Results indicate that the rate of moisture absorption depends on ageing temperature. In the current study, it is observed that composites aged in ambient temperature absorbed higher amount of moisture than sub-zero temperature aged specimen. Moisture absorption reduced stiffness and natural frequency of quasi-isotropic laminates.

© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Fibre-reinforced composites are materials consisting of fibres of high strength and modulus, embedded in or bonded to a matrix to form a new material. In this form, both fibres and matrix retain their physical and chemical identities, yet they produce a combination of properties of the constituent’s material [1]. Non-isotropic nature of composite material creates an opportunity of tailoring its physical and mechanical properties by modifying process parameters such as type of reinforcement and matrix material, volume fraction of reinforcements, and method of manufacturing. The outstanding mechanical properties such as high strength to weight ratio, corrosion resistance, good impact resistance, damping characteristics, chemical stability and improved fatigue properties enable their use in diverse fields. These materials have gathered great interest in marine sectors for applications in underwater construction, buoys, boat hulls, geological oceanography and many more [1]. To ensure the materials long-term...
durability, the effects of environmental conditions like moisture and temperature on the mechanical property of the composites requires comprehensive study and assessment.

The quality of the bonding at the interface between the reinforcement and the matrix material determines the strength of composite [2]. The degradation of fibre/matrix interface bonding is a dominant mechanism affecting the properties of fibre reinforced polymer composites (FRP). Exposure to moisture in marine environments is the major challenge for composites in marine applications. Ingression of moisture into polymeric material leads to plasticization, softening and swelling of composite material [3]. Formation of unfavourable stress concentration areas due to swelling of the matrix reduces the stiffness of the material [4]. Degradation of the stiffness reduces damping property and thus leads to loss of energy absorbing capacity of the material. Studies on the effects of moisture absorption on the properties of glass fibre reinforced polymers (GFRP) have demonstrated that the absorption of moisture is the primary reason for degradation of mechanical properties. Many scientific studies conducted on composites have explained the moisture absorption by marine structures based on Fick’s Law of diffusion. The model provides reasonable characteristics of moisture uptake by the structures. In addition, the presence of voids and cracks in the laminate tends to increase the uptake of moisture into the matrix material and the absorption behaviour will deviate from Fickian behaviour [1,5,6,7].

The present study focuses on understanding the effect of operational temperature and marine environment on tensile and damping properties of glass/epoxy quasi-isotropic composites. Ageing studies in artificial seawater was conducted in two different environmental conditions. The ageing of the material performed at ambient temperature and sub-zero temperature. Fracture and damping tests were conducted on dry and aged specimens. The fracture surface of aged specimens was studied using scanning electron microscope (SEM).

Fig. 1 – Stacking sequence of quasi-isotropic glass/epoxy laminate and vacuum bagging process.

2. Materials and methods

2.1. Laminate fabrication

Glass fibre reinforced epoxy polymer quasi-isotropic laminate were prepared using vacuum bagging technique as shown in Fig. 1. To obtain quasi-isotropic properties in the laminate, eight layers of unidirectional E-glass fabric were stacked.

E-glass unidirectional fibres (CFW Enterprises, New Delhi, India) with a density of 2.54 g/cm³ and an average thickness of 0.2 mm were used as reinforcement material. Epoxy L-12 (Atul Grade) having a density of 1.1 g/cm³ was used as thermoset matrix material to manufacture the laminates. The nomenclature used for fabricated laminate was [0°/90°/+45°/−45°], which was based on the stacking sequence of the plies. The fibre weight fraction of the laminates was 55% by weight and the average thickness of the laminates was 2.5 mm. Experimental density of the laminate was measured using Archimedes principle using 30 samples and the average density was found to be 1.54 ± 0.18 g/cm³. Theoretical density of the laminate was estimated using Eq. (1) [8] and was found to be 1.62 g/cm³.

\[
\rho_L = \left(\frac{\rho_f}{w_f}\right) + \left(\frac{\rho_m}{w_m}\right)
\]

(1)

Here; \(w_f\) is the weight fraction of fibre = 0.55, \(w_m\) is the weight fraction of matrix = 0.45, \(\rho_f\) is the density of E-glass unidirectional fabric = 2.54 g/cm³ and \(\rho_m\) is the density of matrix = 1.12 g/cm³.

The percentage of voids of four laminates before preparing the samples was estimated using as per Eq. (2) [8] and was found to be 4.93 ± 0.076%.

\[
V_v = \frac{\rho_L - \rho_t}{\rho_t} \times 100
\]

(2)

where \(V_v\) is the percentage of the void content of the laminate, \(\rho_t\) is the theoretical density, and \(\rho_e\) is the experimental density of the laminate.

2.2. Accelerated ageing test

GFRP composites are continuously exposed to saline environments and sub-zero temperatures during application. In this present study, saline (at room temperature, 30 °C) and sub-zero temperature (−10 °C) environments were artificially created to investigate the ageing effects on damping and tensile properties. The accelerated ageing in the artificial seawater in both
conditions were carried out for a period of 3600 h. Artificial seawater was prepared as per ASTM 1141 [9] and the salinity of seawater was maintained at a pH of 8.2–8.5. Appropriate ageing was ensured by properly submerging the specimen and maintaining the salinity of the medium. Moisture absorption rate of the laminate was measured periodically. To measure the moisture gain in the specimen, digital weighing scale with accuracy of 0.001 grams was used. The percentage of moisture absorption was determined through the gravimetric measure using Eq. (3) [4,6,10].

\[ M = \frac{W_f - W_i}{W_i} \times 100 \quad (3) \]

where \( M \) is the percentage of weight gain, \( W_f \) is the wet weight of specimen, \( W_i \) is the dry weight of the specimen.

From the results, the moisture gain curve was plotted and Fick’s law of diffusion was used to understand the moisture absorption behaviour of the polymer matrix composite. The diffusion coefficient, \( D \), was calculated using moisture absorption curve and Eq. (4) [4,6,11].

\[ D = \pi \left( \frac{h}{4M_{\infty}} \right)^2 \left( \frac{M_2 - M_1}{\sqrt{T_2} - \sqrt{T_1}} \right)^2 \quad (4) \]

where \( M_{\infty} \) is the percentage of the absorbed moisture at time \( t \) and at saturation state, \( h \) is the specimen thickness and \( D \) is the diffusion coefficient. In the above relation, \( T_1 \) and \( T_2 \) chosen from the initial linear region of the curve, \( M_1 \) and \( M_2 \) was the percentages of the absorbed moisture, related to times \( T_1 \) and \( T_2 \) respectively.

### 2.3 Static and vibration testing

The tensile properties of the glass/epoxy quasi-isotropic samples were tested based on ASTM 3039-09 standard [12] in a servo hydraulic universal testing machine (BSS Bengaluru, India) equipped with a 50 kN load cell. Five specimens were used for each test conditions as per ASTM standard [12]. The dimensions of the samples were 250 × 25 × 2.5 mm. The tensile test was carried out in load control mode with the loading rate of 2 kN/min. The damping property of the aged and pristine glass epoxy quasi-isotropic laminates were measured using vibration setup as shown in Fig. 2 as per ASTM E756-05 [13] using three specimens in each test condition. One end of the laminate was clamped to the setup to create fixed free boundary condition. The clamped specimen was excited using an impact hammer and the resulting displacement was measured at the free end of the laminate using an accelerometer. Data acquisition was accomplished using LabVIEW 2016 and NI 9234 data acquisition interface.

The damping ratio of the pristine and aged quasi-isotropic glass/epoxy laminates was determined using the logarithmic decrement from the time domain plot obtained from the experiment and by using Eq. (5) [14];

\[ \delta = \frac{2\pi \xi}{\sqrt{1 - \xi^2}} \quad (5) \]

where \( \delta \) is the logarithmic decay and \( \xi \) is the damping ratio.

![Fig. 2 – Experimental set up of free vibration measurement.](image)

Stiffness, \( k \) was estimated by considering natural frequency, \( \omega_n \) obtained by free vibrational test and mass, \( m \) of the specimen as per Eq. (6) [14].

\[ \omega_n = \sqrt{\frac{k}{m}} \quad (6) \]

### 3. Results

#### 3.1 Density and void content

Experimental density of quasi-isotropic laminates were estimated as per Archimedes principle using 30 specimens. The average density of the samples was found to be 1.54 ± 0.18 g/cm³. Quality of the fabricated laminates was quantified by estimating the percentage void content using Eq. (2). The average void content of the laminates was found to be 4.93 ± 0.076%. The estimated average density and void content of the samples were considered as a reference for further testing and for characterization of the material.

#### 3.2 Ageing test

Fig. 3 shows the moisture absorption profile of the sample immersed in seawater at room temperature and at sub-zero temperature. Ageing test carried out over a 5-month period (3600h) at ambient temperature and −10 °C in order to simulate the effect of marine environment on glass/epoxy quasi-isotropic composite.

The ageing study of both the samples followed Fick’s law of diffusion and during the initial ageing period i.e. rate of water absorption increased linearly and after a period of 600 h both samples reached a saturation point. The water absorption curve for ambient condition specimen exhibited two-stage absorption model [5]. In the initial stage, up to 600 h moisture diffusion curve followed Fick’s law of diffusion. In second stage; after the saturation point, curve followed gradual increment and decrements in water absorption rate. The specimens aged
under ambient temperature absorbed 13.22% of moisture up to equilibrium point; whereas in case of subzero temperature ageing condition, rate of moisture absorption was low and at equilibrium point the maximum moisture content was 2.61% by weight of the sample. Diffusion coefficient of aged specimens was evaluated from the linear portion of the moisture diffusion curve and using Eq. (4). The diffusion coefficient of ambient temperature aged specimen was $4.821 \times 10^{-10}$ m$^2$/s. Subzero temperature-aged specimen was $1.259 \times 10^{-12}$ m$^2$/s.

### 3.3. Tensile properties

The effect of ageing on glass/epoxy laminates was monitored by evaluating ultimate fracture stress and degradation of stiffness. The values obtained in the test compared with that of pristine laminate. Five specimens were used per test conditions as per ASTM standards [12]. Fig. 4(a)–(c) shows the variation of tensile properties of pristine, ambient and sub-zero temperature aged conditions laminates respectively. Immersion of glass/epoxy laminates in seawater at subzero and ambient temperature resulted in decrement of ultimate failure strength. Average tensile ultimate failure strength of pristine, ambient and sub-zero temperature aged specimens were found to be $200.83 \pm 10.85$ MPa, $146.42 \pm 7.32$ MPa and $185.27 \pm 9.26$ MPa respectively. Stress-strain curve of quasi-isotropic laminates exhibited two distinct slopes and it is approximated as bi-linear curve. Initial slope of the curve indicates failure of the fibres oriented perpendicular to the direction of loading. Fibres oriented along the direction of loading fails at maximum failure strain [15]. These phenomena were observed in pristine, ambient and sub-zero temperature aged specimens.

Reduction of stiffness, failure strain and ultimate fracture strength of the composite laminates subjected to marine environment was compared with pristine laminate and is shown in Table 1.

The degradation of stiffness due to moisture absorption can be attributed to internal stresses developed and plasticization of matrix during moisture uptake [16]. The specimen immersed in artificial seawater at ambient condition absorbed large amount of moisture and resulted in reduction of ultimate failure stress by 27% due to plasticization of matrix material. Ageing of specimen in sub-zero temperature condition reduced moisture absorption rate and showed reduction of only 8% of ultimate failure strength as compared to pristine specimen. The stiffness of pristine specimen was 6.76 GPa and degradation was 12.4% and 10.9% for ambient and sub-zero temperature aged condition respectively. The reduction of stiffness and failure strain shows brittle failure of the material. The stiffness of pristine samples in the current work is in agreement with the published literature [16].

The fibre matrix interface is one of the pathways to moisture diffusion into the composite laminates. Moisture diffusion rate is predominant in the composites in which wetting of the fibres by the matrix material is incomplete [8]. Presence of voids and incomplete wetting of fibres increases the diffusion coefficient and results in increment of moisture uptake in the composites. Moisture intake into polymer matrix composites leads to mechanical, chemical degradation and plasticization of matrix material [17]. Fig. 5(a), (b) and Fig. 6(a), (b) show the SEM micrograph of ambient and sub-zero temperature aged specimen respectively. Microscopic examination of failure surface revealed that failure of reinforcement material and fibre/matrix interface cracks is the major reason for the failure of laminates. Higher amount of moisture uptake reduced the interface bonding between fibre and matrix and resulted in failure of specimen at lower stress levels in case of ambient temperature aged specimen.

### 3.4. Vibrational characteristics

The vibration test conducted as per ASTM E756-05 and Table 2 shows the variation of vibrational characteristics of all the specimens subjected to different ageing conditions. Three samples were used in each testing conditions and
average values of the test results is used to understand the degradation of vibrational properties of the laminates.

The natural frequency of the dry specimen was 23.04 Hz; sub-zero temperature aged specimen was 22.84 Hz and was 21.76 Hz for ambient temperature aged specimen. The degradation in the damping properties of quasi-isotropic laminates can be better understood from Meirovich’s continuous beam model. In this theory, the natural frequency of the cantilever beam can be written as per Eq. (7) [3,18]  

$$f = \frac{1}{2\pi} \left( \frac{1.875}{L} \right)^2 \sqrt{\frac{EI}{\rho A}}$$  

(7)

where L is the length of the beam, E is stiffness of the material, \(\rho\) is density, and I is the moment of inertia and A is cross sectional area of the specimen. From Eq. (7), it can be understood that as the stiffness of the material decreases the natural frequency also decreases. The amount of moisture absorbed by the specimen plays an important role in varying the

<table>
<thead>
<tr>
<th>Ageing conditions</th>
<th>Density (g/cm³)</th>
<th>Void content (%)</th>
<th>Average tensile strength (MPa)</th>
<th>Stiffness (GPa)</th>
<th>Average failure strain (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pristine</td>
<td>1.54 ± 0.18</td>
<td>4.93 ± 0.076%</td>
<td>200.83 ± 10.85</td>
<td>6.76 ± 0.07</td>
<td>3.06 ± 0.001</td>
</tr>
<tr>
<td>Ambient</td>
<td>1.54 ± 0.18</td>
<td>4.93 ± 0.076%</td>
<td>146.42 ± 7.32</td>
<td>5.92 ± 0.04</td>
<td>2.3 ± 0.002</td>
</tr>
<tr>
<td>Sub-zero</td>
<td>1.54 ± 0.18</td>
<td>4.93 ± 0.076%</td>
<td>185.27 ± 9.26</td>
<td>6.02 ± 0.06</td>
<td>3.06 ± 0.002</td>
</tr>
</tbody>
</table>
natural frequency of the specimen. The increased presence of moisture in the composite causes plasticization of the matrix, resulting in the reduction of the stiffness of the specimen, in turn decreases the natural frequency of the specimen [19]. The moisture uptake in specimen exposed to sub-zero conditions was lower than the ambient condition. This caused the specimen to retain up to 99% of the natural frequency compared to the dry specimen. The stiffness coefficient of the laminates varied the values of natural frequency as per Eq. (6). The reduced stiffness coefficient of the composite laminate can be attributed to the plasticization and swelling of the matrix due to the ingressed moisture.

4. Conclusions

In this study, effect of artificial seawater environment on vibration and tensile properties of quasi-isotropic glass/epoxy composites laminates were investigated. Damping property and stiffness of the laminates were significantly affected due to moisture absorption. Natural frequency of the laminates was influenced by the amount of moisture gain. The ambient condition aged specimen showed 5.7% reduction in natural frequency and for sub-zero temperature aged specimen it decreased by less than 1% compared to pristine specimen. The stiffness of quasi-isotropic laminate degraded by 12.4% and by 10.9% for ambient and sub-zero temperature aged condition as compared to pristine specimens. Moisture absorption property of glass/epoxy composites largely depended on ageing temperature. Specimens aged in ambient condition absorbed higher amount of moisture than that of the specimens immersed in sub-zero conditions. Microstructure studies revealed that fibre failure is the major reason for the static failure of laminates.

Conflicts of interest

The authors would like to acknowledge Advanced Composite Research Lab and Vibration and Acoustics Lab, Department of Aeronautical and Automobile Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal for the support provided for the fabrication and testing facility of composite materials.

The authors declare no conflicts of interest.

REFERENCES


