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EFFECTS OF CHEMICAL ENVIRONMENT ON THE DURABILITY PERFORMANCES OF GLASS FIBER/EPOXY COMPOSITES

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Abstract: Glass fiber reinforced composites are widely used all over the world. In this paper, the durability performances of glass fiber/epoxy composites in chemical environment were investigated. The response of composites was characterized by measuring moisture uptake, dynamic properties and mechanical properties including short beam shear and 3-point flexure tests over a 100-week period. Optical microscope was applied to characterize the microstructure of the composites. Also, Fourier transform infrared spectroscopy (FTIR) method was used to detect the existence of reactant of composites and chemicals. The experimental results showed that the degradation of composites was strongly affected by the chemical corrosion on the constituents. The destruction of fiber and matrix both made contribution to weakening the interlaminar properties.

Keywords: glass fiber; polymer matrix composites; durability; chemical environmental; degradation; mechanical properties

1 Introduction
Glass fiber reinforced polymer composites have been widely used in diverse applications for many years. They were often used in hostile environments, such as marine circumstance, alkaline conditions. Polymeric composites are susceptible to moisture and chemical substances when operating in certain kinds of environmental conditions. The structural integrity and lifetime performance of fibrous polymeric composites are strongly dependent on the stability of the fibers, polymer, and also the interfacial region between them. It is well known that chemical substances may lead to the degradation of fibers, matrix and interface. For instance, sea water can cause both swelling and plasticization of the matrix and debonding of the interface [1]. The alkaline solution had very strong corrosion effect on the glass fibers [2]. The interaction of moisture with the metal oxides in E-glass fibers leads to corrosion induced damage and thus results in reduced mechanical strength [3].

It has been reported that when glass reinforced composites were immersed in water, the original voids or cracks in the material would be filled with water molecules gradually [4]. This would increase the weight of the specimen. Prolonged immersion would induce chemical reaction between the water molecules and the glass fibers as well as the matrix causing some elements to dissolve in water. This would cause the decline of the mass weight. The absorption studies on relative materials in literatures proved that diffusive and capillary processes were the main paths for composites to absorb moisture [5, 6]. In addition, different aging conditions may also change the fundamental diffusion mechanism in both neat resin and composites [7-9].

The performance of interfacial region is also an important part in the degradation mechanism of composites. Dynamic mechanical analysis method was applied to investigate properties changes of the composites, and also the nature of the interfacial adhesion [10]. Therefore the effect of different aging conditions on the characters of the interface could be determined.

In this paper, effects of chemical environment on the durability of glass fiber reinforced epoxy composites were investigated. Microstructures of glass fiber reinforced epoxy composites and moisture absorption were revealed. Interlaminar shear test and three point bending test were employed to study the effects of chemical environment on the durability of the composites. It was concluded that the durability of the composites were strongly dependent on the
chemical corrosion of glass fibers and moisture absorption of the composites.

2 Materials and experiment

The glass fiber textile and epoxy were used to fabricate the laminates in this study. Laminate composites were prepared by wet lay-up and autoclave processing technique. In order to assess the durability of this system, an investigation was undertaken considering a number of exposures including immersion in three different solutions: deionized water, salt water, and alkaline solution. The response of the composites was determined over a 100-week period through moisture uptake measurements, mechanical characterization, and dynamic mechanical analysis. In addition, microscopic photos of the composites were obtained before and after the immersion. The samples were also analyzed by means of Fourier transform infrared spectroscopy (FTIR). This method enables identification of degradation products and estimation of the influence of glass fiber and polymer on chemical degradation of the studied composites.

3 Results and discussion

3.1 Moisture absorption

The amount of water absorption by the composites was calculated according to the following equation.

\[ W\% = \frac{m_w - m_d}{m_d} \times 100\% \]  

where \( m_d \) and \( m_w \) is the dry and wet weight of the specimen, respectively. Absorbed water might have adverse effect on the performance of the materials. The moisture will cause progression of delamination between layers. The moisture absorption capability of the composite immersed in three different kinds of solutions is shown in Fig.1. In the first stage, the curve obeys the Fick’s law, weight gain went up gradually over time until reached the balance value. In the second stage, moisture absorption of the composite in all three conditions decreased due to the interaction of glass fiber and \( H_2O \), which gave the production of \( SiO_2 \), and the chemical reactions are shown in Equations (2) and (3). And also in the second stage, weight gain in the alkaline solution was higher compared to the other two. It’s due to glass fiber is sensitive to the alkaline environment, chemical reaction between glass fiber and \( NaOH \), is shown in Equation (3). The surface of the fiber was damaged, and the pits on the rough surface were filled with water. This could be a new pathway for water absorption. On one hand, the weight decrease of specimen was due to the \( Si-OH \) and \( SiO_2 \), which were separated out from the material; on the other hand, the weight increase of specimen was due to the water absorbed through the new pathway.

Compared to the weight changes of the pure matrix aging in the same conditions (Fig.2), corrosion of glass fibers by \( H_2O \) and alkali iron was an important factor resulted in the moisture uptake behavior of the composite, which can be proved using the Fourier transform infrared spectroscopy (FTIR). Three kinds of solutions which the specimens immersed in were collected as the FTIR samples. The spectra of three solutions were obtained using the attenuated total reflectance (ATR).
As the results shown in Fig.3, all these solutions contained Si-O-Si, and also Si-OH was detected in the alkaline solution. The fact could be easily observed by the appearance of the characteristic band at 1012 cm⁻¹ for Si-O-Si, and 1381 cm⁻¹ for Si-OH. Furthermore, the appearance of band at 871 cm⁻¹ for N-H demonstrated the degradation of the matrix. All these facts showed that the structure of glass fibers and epoxy were destroyed in these environments, especially in alkaline solution. It should be noted that the maximum moisture uptake levels for pure epoxy specimens immersed in all these solutions were about equal (Table 1), whereas for composite, the maximum moisture uptake percentage in alkaline solution was much higher than that in the other two environments. That might be primarily due to the reaction between glass fibers and the alkali substances. And the NaCl particles in the composite were trapped on the surface of glass fibers, becoming a coating, which played a role in separating the fibers from the moisture [11].

<table>
<thead>
<tr>
<th>Solution</th>
<th>Maximum moisture uptake(%)</th>
<th>Coefficient of diffusion (mm²/s)</th>
<th>Maximum moisture uptake(%)</th>
<th>Coefficient of diffusion (mm²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkali</td>
<td>5.75</td>
<td>9.06×10⁻⁴</td>
<td>4.72</td>
<td>8.7×10⁻⁴</td>
</tr>
<tr>
<td>Saline</td>
<td>3.18</td>
<td>5.63×10⁻⁴</td>
<td>4.30</td>
<td>6.72×10⁻⁴</td>
</tr>
<tr>
<td>Deionized water</td>
<td>3.95</td>
<td>7.57×10⁻⁴</td>
<td>4.66</td>
<td>7.24×10⁻⁴</td>
</tr>
</tbody>
</table>

The microstructures of the composites are shown in Fig.4, which indicated that alkali resulted in substantial corrosion on glass fibers. The surface of glass fiber aged in saline solution was rather intact compared to those in alkaline. The results showed that significant strength loss was due to the corrosion of glass fiber surface, especially in alkaline solution.

![Fig.4 Microstructure of glass fiber composites: (a) immersion in saline solution; (b) immersion in alkaline solution](image)

### 3.2 Interfacial performances

Interlaminar properties before and after aging were tested in this experiment, Fig.5 presents the results for the material. During the initial stage, the shear strength decreased rapidly after the water uptake, due to the existence of water molecules in the composites, which weakening the performance of interface between the fiber and matrix. It can be seen from the figure that there were differences based on the solution type, the alkaline solution immersion causing the maximum reduction and the salt water causing the minimum reduction in the interlaminar shear performance. In the second stage, no
significant differences were observed among the three solutions. At the end of the entire duration, the ILSS value in the saline solution was slightly higher than the others, as shown in Fig.5.

![Fig.5 Interlaminar shear strength of the composites](image)

Dynamic mechanical analysis can be used to study the performance of material over a wide range of temperatures. It is an effective method to measure the viscoelastic properties of FRP composites, most commonly the glass transition temperature $T_g$. In this study, the peak value of $\tan \delta$ is selected as $T_g$ of the material.

The glass transition temperature $T_g$ of glass fiber/epoxy composites had a slight drop at the beginning of the environmental exposure but increased gradually over time until it decreased again, as illustrated in Table 2.

Interface plays an important role in physical and mechanical properties of composites. Chemical degradation would lead to poor interfacial performances. According to Luis Ibarra’s theory [10], the interface factor is defined to describe the bonding level of the interface, as shown in Equation (4), where $(\tan \delta_{\max})_m$ and $(\tan \delta_{\max})_c$ represent the glass transition temperature ($T_g$) of the pure matrix and composite, respectively. $V_f$ represents the volume fraction of fibers in the composite, which is 65% in this study. The smaller of the value ‘A’ is, the stronger the interfacial bonding will be. The $T_g$ value of aged pure matrix and composites are listed in Table 2 and 3. According to Equation 4 and the values listed in Table 2 and 3, the changes of interface factor were revealed in Fig.6. The results showed that in the initial stage, interface property in the saline solution was stronger than others. After 72 weeks, the interface factor in the deionized water presented the lowest value. Also, the interlaminar performances were getting worse through time in all these solutions. The changes of interfacial properties did correspond with the interlaminar performances.

$$A = \frac{1}{1 - V_f} \times \frac{\tan \delta_c}{\tan \delta_m} - 1 \quad (4)$$

<table>
<thead>
<tr>
<th>Aging time[weeks]</th>
<th>Deionized water</th>
<th>Saline solution</th>
<th>Alkaline solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>70.31</td>
<td>70.31</td>
<td>70.31</td>
</tr>
<tr>
<td>24</td>
<td>77.98</td>
<td>60.78</td>
<td>73.96</td>
</tr>
<tr>
<td>72</td>
<td>84.95</td>
<td>80.03</td>
<td>72.90</td>
</tr>
<tr>
<td>104</td>
<td>62.00</td>
<td>65.24</td>
<td>57.05</td>
</tr>
</tbody>
</table>

Table 2 $T_g$ of composites changing with time [°C]

<table>
<thead>
<tr>
<th>Aging time[weeks]</th>
<th>Deionized water</th>
<th>Saline solution</th>
<th>Alkaline solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>98.39</td>
<td>98.39</td>
<td>98.39</td>
</tr>
<tr>
<td>24</td>
<td>83.80</td>
<td>67.16</td>
<td>76.15</td>
</tr>
<tr>
<td>72</td>
<td>84.23</td>
<td>84.08</td>
<td>75.26</td>
</tr>
<tr>
<td>104</td>
<td>61.55</td>
<td>63.63</td>
<td>56.00</td>
</tr>
</tbody>
</table>

Table 3 $T_g$ of pure matrix changing with time [°C]

3.3 Flexural properties

Changes of flexural properties are given in Fig.7 and 8. After 72-week duration, the failure mode changed from fiber damage to delamination. And flexural strength of specimen in the deionized water was much lower than that of the other two solutions. The chemical reaction of the alkali and glass fiber leded...
to the production of Si-OH, which may remained at the interfacial region, enhanced the friction of fiber and matrix. The relative motion was more difficult between fibers and matrix, thus, the interfacial performance was relatively better. Flexural modulus was slightly affected by the environment at the initial stage, according to the results obtained (Fig.8), after aging for 100 weeks, modulus in the saline environment almost reached to the original value.

Changes in the mechanical properties were correlated with the level of the moisture uptake and the extent of the corrosion. Corrosion of glass fiber at the initial stage of composites degradation was observed. Further degradation was dependent on the interlaminar performance of the composites.

4 Conclusions

Durability of glass fiber reinforced composites was strongly affected by the chemical corrosion of fibers and the moisture absorption of the material. The water absorption curves for composites were different from the pure epoxy primarily because of the reaction between glass fibers and the chemicals. The interfacial region was severely damaged after 100-week duration. The degradation of the fiber, the matrix and the interface caused by the chemical solution contributed to the deterioration of the mechanical performances of glass fiber reinforced composites.

5 Acknowledgements

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References

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