Effects of seawater temperature and NaCl concentration on interlaminar shear behavior of CFRP laminates

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Keywords: CFRP, marine environment, moisture absorption, interlaminar shear, degradation

Abstract

The aim of this paper is to study the interlaminar shear behavior of CFRP in critical service conditions. The specimens were immersed in the prepared artificial seawater for 7 months to investigate the ageing time, ambient temperature and salt concentration effects. Analytical balance was used to weight the samples for analyzing the dynamic moisture absorption behavior. Short beam shear tests were carried out to evaluate the interlaminar shear properties. Scanning electron microscopy (SEM) was used to observe the ageing damage and failure morphology. The results show that moisture absorption behavior of composites follows Fickian diffusion law. Moisture absorption rate and the maximum absorption content are mainly affected by ambient temperature but not by NaCl concentration. Compared with ageing time and NaCl concentration, the shear stiffness and shear strength are more sensitive to aging environmental temperature. After exposed in 3.5%–70°C seawater environment for 7 months, the retention rate of shear strength is only 68.8% and the failure displacement decreases by 20.5%. The shear strength increases first and then decreases with the increase of the maximum moisture absorption, which is mainly related to the release of curing shrinking stress and the damage of interface properties. The shear properties degradation after aging is mainly related to the damage of materials induced by environment (delamination, fiber/matrix interface debonding and matrix cracking).

1. Introduction

Due to their excellent physical/mechanical properties, superior performance of environmental resistance and fatigue life, FRPs have been widely used in shipbuilding industry [1–4], offshore oil industry [5–8] and marine infrastructure industry [9–11], such as recreational craft, workboats, risers for oil exploration and marine renewable energy device components. The current interest in marine renewable energy (MRE) creates a significant new market for composites in MRE device components. Since FRP composites can be molded to complex shapes, their products have been successfully introduced to the blades of tidal or underwater turbines [12–14]. The main challenges for composites used in marine environment are the long term exposure time to moisture, the ambient temperature, the ionic species and the microorganisms. In marine environment, the service life of mechanical structures is usually designed to be several decades. Therefore, the degradation of mechanical properties of composites is becoming the key issue for engineers to consider in the design of marine structures.

Many studies have been carried out in the mechanical degradation of FRPs serving in harsh and extreme environments. Degradation process is usually accompanied by physical damage and chemical damage of composites. Physical damage [15–17] includes micro-cracks in matrix, de-bonding of fiber-matrix interface and delamination between layers, which are all related to the moisture/thermal swelling stress. Chemical damage [18–20] consists of matrix hydrolysis, interface hydrolysis and fiber hydrolysis, which is an accelerator for...
physical damage, such as interface de-bonding. Rhee et al [21] investigated the compressive behavior of CFRP in deep-sea environment, and pointed out that the compressive elastic modulus increased about 10% when the hydrostatic pressure increased from 0.1 to 200 MPa. Kootsookos et al [22] studied the degradation of flexural properties of composites with different fiber types in seawater environment, they found that the flexural strength of polyester-based composites and ester-based composites at the saturation time have degraded by 20%–40% and 40%–50% respectively. In addition, Yan et al [23] analyzed the effect of ultraviolet radiation on the tensile and bending properties of flax fabric composites. The test results show that the tensile strength/modulus of the weathered composites decreased 29.9% and 34.9%, respectively. The flexural strength/modulus reduced 10.0% and 10.2%, respectively. However, the degradation of interlaminar shear performance in seawater environment is less studied.

In practical engineering application, the interlaminar shear performance is usually adopted to evaluate the interfacial property of composites using the typical short beam shear test method [24, 25]. The interlaminar shear strength (ILSS) is one of the most important influencing factors to determine the basic mechanical properties of CFRP composites, like tensile strength, compressive strength and flexural strength. Bulut et al [26] examined the effects of particle content and type of the fillers on ILSS properties with analyzing interfacial bonding and agglomeration aspects. These findings have great reference significance for the study of interlaminar properties of composites. However, due to its heterogeneity and anisotropy, the interlaminar interface of CFRP is easier to be destroyed by the aggressive environments (e.g. seawater, acidic solution), which is commonly to be considered as the weakest location in composites. There are several reports on the durability of interlaminar shear behavior of FRP composites in hygrothermal environment. First, Gautier et al [15] analyzed the interface damage of GFRP in different temperature water conditions, and found that the decrease of ILSS is mainly attributed to the interfacial debonding, induced by differential swelling and osmotic cracking at the interface. Then, the deleterious effects of temperature, thermal shock and freezing on the ILSS of GFRP were systematically studied by Ray et al [27–30]. Recently, Guo et al [31] investigated the water uptake and interface shear strength of two types of pultruded carbon/glass fiber reinforced epoxy hybrid composite rods, they found that long-term hygrothermal exposure led to a remarkable degradation in the interfacial shear strength of the rods, up to 17.5%–42.1%. However, there are limited reported studies on FRP composites in seawater environment, especially for CFRP composites. Those researches included the different factors, such as resin types [32], fiber types [33], fiber volume fractions of GFRP [34] and hybrid composites [35]. Zhao et al [33] compared the degradation behavior of basalt-, glass- and carbon- fiber composites bars in seawater environment, and pointed out that the durability of carbon composites was the best. To the best of the author’s knowledge, there are a little reported studies on the interlaminar shear behavior of CFRP in seawater environment, which greatly hinders the application of CFRP in marine field.

In this paper, we aims to explore the effects of ambient temperature and NaCl concentration on the interlaminar shear behavior of CFRP in seawater environment. Short beam shear specimens were immersed in the prepared artificial seawater with a period of 7 months. The weight of specimens were measured regularly to analyze the dynamic moisture absorption behavior. Then, short beam shear tests were carried out to obtain the interlaminar shear strength (ILSS). On this basis, the relationship between moisture content and ILSS was established to predict the long-term performance of CFRP in seawater environment. Finally, SEM tests were performed to explore the degradation mechanism and failure mode.

2. Experimental process

2.1. Material
The material used in this study is the carbon fiber reinforced epoxy resin matrix composites, which were manufactured by WEIHAI GUANGWEI COMPOSITES Co., Ltd using vacuum bag molding process. Firstly, the pre impregnated clothes were stacked by manual according the specified sequence of [0/±45/90]_4s in which the carbon fiber was T700 with a diameter of 7 µm and the matrix was 7901 fire-retardant unsaturated polyester resin. Then, the blends were firstly heated up in the vacuum oven form room temperature to 80 °C in 0.5 h and kept at this temperature for 1 h. After that, the temperature was further rose to 125 °C in 1.5 h. Finally, the blends were maintained for 3 h at this temperature for thorough cure. The thickness of the cured laminates was controlled at about 6.4 mm, and the density and fiber volume fraction were 1.6 g cm⁻³ and 67%, respectively.

2.2. Seawater aging test
In this study, there kinds of seawater ageing condition were designed. In the first case, the specimens were immersed in 50 °C-3.5% NaCl solution for 1, 3, 5 and 7 months, respectively, to study the ageing time effect. 50 °C was chosen to accelerate such aging process.
The second case was to investigate the ambient temperature effect. The specimens were immersed in 3.5% NaCl solutions with a temperature of 30 °C, 50 °C and 70 °C, respectively. 30 °C is the average temperature of marine in summer. In addition, the high temperatures (50°C and 70°C) can also be found in many places, such as the flight decks of aircraft carriers and engine area.

The third case was to evaluate the sensitivity of composites to NaCl concentration. The specimens were immersed in 0.0%, 3.5% and 5.0% NaCl solutions, respectively, and the temperature was kept at 30 °C. The 3.5% NaCl solution was almost the same as the average concentration in ocean. Extending the concentration up to 5.0% and down to 0.0% was to evaluate the concentration effect.

The samples were immersed in above prepared solutions for 7 months and regularly weighed at different intervals. For each condition, five specimens were prepared and tested in order to reduce the experimental error.

The average moisture absorption content in composites is calculated from the measured mass $m$ according to the ASTM D5229/D5229M-92 standard [36].

$$M_t, \% = \frac{m_t - m_0}{m_0} \times 100\%$$ (1)

where $M_t$ is the average moisture content at time $t$, %; $m_0$ and $m_t$ are the oven-dried and current mass, respectively, mg.

### 2.3. Short beam shear test

According to ASTM D2344/D 2344M M-00 specifications [37], samples with dimension of 36 × 11 × 6.4 mm$^3$ were tested under three-point bending load with a span length of 24 mm, loading nose diameter of 6 mm, support diameter of 3 mm, by an Electronic universal Testing Machine model INSTRON 5966 (America) with a maximum loading of 10 KN, as shown in figure 1. The crosshead speed was set to 1 mm min$^{-1}$. 

![Figure 1. Short beam shear test schema: (a) testing device; (b) fixture; (c) specimen.](image)
The interlaminar shear strength (ILSS) of composite laminates is calculated as follows.

\[ F_{\text{ILSS}} = 0.75 \times \frac{P_m}{b \cdot t} \]  

(2)

where \( F_{\text{ILSS}} \) is the ILSS, MPa; \( P_m \) is the critical load, N; \( b \) and \( t \) are the width and thickness of specimen, respectively.

3. Results and discussion

3.1. Moisture absorption behavior

Figure 2 shows the moisture absorption curves of composite laminates in 3.5% NaCl solutions with a certain temperature of 30 °C, 50 °C and 70 °C, respectively. It is obvious that there was an apparent separation among the moisture absorption curves. The effect of ambient temperature on moisture absorption behavior of composites is mainly reflected on the moisture absorption rate and the maximum absorption content. The higher the temperature was, the faster the rate was, and the larger the content was. With the prolongation of aging time, the moisture absorption rate decreased gradually, and the moisture content showed saturate tendency finally. It can be found that the moisture absorption process can be well expressed by Fickian diffusion model.

Figure 3 shows the moisture absorption curves of composite laminates in 30 °C-0.0%, 3.5% and 5.0% NaCl solutions, respectively. It can be seen that there was a small difference in the moisture absorption behavior of composites. At the initial stage (1 month), the moisture absorption rates are faster and have little difference. The
water molecules would penetrate into composites and be stored in voids and crevices, which may attribute to the rapid increase in moisture content and such behavior does not be affect by NaCl concentration. However, with the extension of ageing time, differences began to emerge among curves. The material in 3.5% NaCl solution showed the smallest moisture content, followed by that in 5.0% NaCl, and that in 0.0% NaCl solution had the largest content. Such phenomenon may be related to the osmotic pressure of water molecular, and chemical changes of matrix.

The moisture absorption coefficients of composites in different conditions are collected and listed in table 1. It can be found the diffusion coefficient and saturated content of composites in different NaCl concentration solutions have little difference, while they all increases sharply with the increasing of ambient temperature.

![Table 1. Moisture absorption coefficients of composites in different conditions.](image)

| Condition       | $D_{[mm^2\ day^{-1}]}$ | $M_{s\%}^{\text{}}$ |
|-----------------|------------------------|---------------------|
| 30°C-0.0%       | 0.046                  | 0.58                |
| 30°C-3.5%       | 0.044                  | 0.51                |
| 30°C-5.0%       | 0.046                  | 0.56                |
| 50°C-3.5%       | 0.078                  | 1.05                |
| 70°C-3.5%       | 0.170                  | 1.50                |

3.2. Mechanical degradation behavior

3.2.1. Load-displacement relationship

Figure 4 shows the load-displacement curves of specimens under different ageing conditions. Figure 4(a) shows the effect of ageing time on the mechanical behavior of composites. During the initial stage, the load increased linearly as the displacement increased, and there was no significant difference in stiffness (slope) for different aging times. However, the first peak load and the corresponding displacement showed a high sensitivity to aging time, which had a general decrease with the prolongation of aging time. After that, the load all suffered a sharp decrease.
drop, which is attributed to the horizontal crack at the interlayer. Then, the specimens experienced a second loading, and the load reached a smaller second peak.

Figure 4(b) shows the effect of environmental temperature on the mechanical performance of specimens after 7 months ageing in 3.5% NaCl solution. It can be noted that the higher the environment temperature was, the lower the ultimate bearing capacity was, and the smaller the failure displacement was. In addition, the difference in bearing stiffness can be well distinguished. In 30°C and 50°C conditions, the stiffness remained almost stable during loading. However, it is obvious that the stiffness of specimen in 70°C condition was subjected to a nonlinear degradation during loading. Moreover, the bearing load of specimens in 30°C and 50°C conditions showed a sharp drop due to the horizontal crack at the interlayer, while that in 70°C condition experienced a buffer due to local failure in material. Such differences may be related to the micro-cracks in matrix and debonding at fiber/matrix interface. The higher the temperature is, the higher amount of thermal stress and moisture stress are, which may promote crack initiation and propagation [28].

![Table 2. Interlaminar shear strength of specimen in different ageing conditions.](image)

| Condition | Ageing time [months] | Interlaminar Shear strength [MPa] | Error | Retention rate [%] |
|-----------|----------------------|-----------------------------------|-------|-------------------|
| Original  | —                    | 48.01                             | 0.7   | 100.0             |
| 30°C-0.0% | 7                    | 50.66                             | 1.7   | 105.5             |
| 30°C-3.5% | 7                    | 51.92                             | 0.52  | 108.1             |
| 30°C-5.0% | 7                    | 47.36                             | 1.1   | 98.6              |
| 50°C-3.5% | 1                    | 49.45                             | 0.9   | 103.0             |
|           | 3                    | 45.39                             | 0.54  | 94.5              |
|           | 5                    | 42.13                             | 0.59  | 87.8              |
|           | 7                    | 41.34                             | 0.52  | 86.1              |
| 70°C-3.5% | 7                    | 32.92                             | 0.48  | 68.6              |

Figure 5. Interlaminar shear strength of SBS specimens: (a) effect of ageing time; (b) effect of environmental temperature and (c) effect of NaCl concentration.
Figure 4 (c) shows the effect of NaCl concentration on the mechanical response of specimens after 7 months ageing in 30 °C environment. It can be noticed that NaCl solution had little effect on mechanical properties. There was almost no difference in stiffness. However, the load-bearing capacity of specimens in 5.0% NaCl solution was poorer than that in 0.0% and 3.5% NaCl solutions.

3.2.2. Interlaminar shear strength

Figure 5 illustrates the variation in ILSS of composite laminates under different ageing conditions, and such data are listed in table 2.

The effect of ageing time on the ILSS is shown in figure 5 (a). It is clear that the shear strength did not always degrade along with aging time. After 1 month aging, the shear strength increased from 48.01 MPa to 49.45 MPa, about 3.0%. However, with the further extension of aging time, the strength began to decrease exponentially. After 3, 5 and 7 months ageing, there was a reduction of 5.5%, 12.2% and 13.9%, respectively. The main reason for this rise in ILSS may be due to the hygroscopic swelling stress during the initial stage of moisture absorption, which releases the curing shrinkage stress and leads to a strain-free state in the laminates [27]. It can be noticed that the degradation level showed a tendency to be stable after long term ageing, which may be related to the saturated moisture content of composites.

This phenomenon can be explained by the two-stage evolution model. During the ageing process, there is a combined effect of strengthening and weakening on the mechanical property. At the first stage (I), the strengthening due to the release of curing shrinkage stress is greater than the weakening due to environmental ageing, so the mechanical property shows an improvement. With the prolongation of aging time, the weakening effect is aggravated. When the weakening is greater than the strengthening, the material enters the second stage (II), in which the mechanical property degrades.

Figure 5 (b) shows the effect of environmental temperature on the ILSS of specimens. Compared with unaged specimens, the reduction of shear strength of specimens aged in 30 °C, 50 °C and 70 °C-3.5% NaCl solutions for 7 months was 8.1%, 13.9% and 31.4%, respectively. It can be found that there was a strong linear relationship between environmental temperature and ILSS. The higher the temperature is, the higher the degradation level of shear strength is. Temperature does not change the ageing mechanism of composites, only accelerates the ageing process. In 30 °C condition, the strengthening rate and weakening rate of material were all

Figure 6. Failure displacement of SBS specimens: (a) effect of ageing time; (b) effect of environmental temperature and (c) effect of NaCl concentration.
Figure 7. Variation in retention of shear strength with moisture content in composite laminates.

Table 3. Comparison of experimental data and predicted data.

| Condition | Ageing time [months] | Moisture content [%] | Retention rate [%] |
|-----------|----------------------|----------------------|--------------------|
|           |                      |                      | Experiment | Model | error |
| Original  | —                    | 0                    | 100.0      | 100.0 | 0.0   |
| 30 °C-0.0%| 7                    | 0.54                 | 105.5      | 102.5 | −2.9  |
| 30 °C-3.5%| 7                    | 0.49                 | 108.1      | 103.9 | −3.9  |
| 30 °C-5.0%| 7                    | 0.53                 | 98.6       | 102.8 | 4.2   |
| 50 °C-3.5%| 1                    | 0.43                 | 103.0      | 105.2 | 2.2   |
|           | 3                    | 0.73                 | 94.5       | 95.4  | 0.9   |
|           | 5                    | 0.88                 | 87.8       | 88.6  | 0.9   |
|           | 7                    | 0.96                 | 86.1       | 84.9  | −1.4  |
| 70 °C-3.5%| 7                    | 1.49                 | 68.6       | 68.4  | −0.3  |

Figure 8. Typical ageing damages in specimens after seawater ageing.
slow, resulting in being in stage-I for a long time. So, it still showed a slight improvement even after 7 months ageing. However, in 50 °C and 70 °C conditions, the rates were greatly improved, so the period of stage-I was shortened and the material entered stage-II in a shorter aging time. So, after 7 months aging in 50 °C and 70 °C, the shear strength showed a serious degradation.

Figure 5(c) shows the effect of NaCl concentration on the ILSS of specimens. After 7 months ageing in 30 °C-0.0%, 3.5% and 5.0% NaCl solutions, the shear strength of specimens was 50.66 MPa, 51.92 MPa and 47.36 MPa, with a reduction of −5.5%, −8.1% and 1.4%, respectively. In 30 °C condition, the strengthening rate and weakening rate are pretty low. When in dissolved state, NaCl exists as cations and anions. Ions would penetrate along with the water molecules into the composites, causing damage to the matrix, fiber and interface [38].

Similar as environmental temperature, NaCl concentration can also accelerate this ageing process. According to the two-stage evolution model, the specimens in 0.0% NaCl solution for 7 months may be still in stage-I, while the specimens in 5.0% NaCl solution may be in stage-II. As for the specimens in 3.5% NaCl solution, it may be in the transition region of the two stages after 7 months aging because of the larger improvement. The same improvement can be found in Ray’s research, which was reported as about 10% [28].

3.2.3. Failure displacement
As shown in figure 6, the failure displacements (first peak) were compared for each ageing conditions since it is important to understand the ductility of composites. It can noticed that the evolution law of failure displacement is consistent with that of ILSS. During the initial 3 months, the failure displacement suffered a significant
decrease. After that, it showed a slight decrease with ageing time, illustrated in figure 6(a). However, the failure displacement decreased linearly with the increasing of environmental temperature, as shown in figure 6(b). It can be concluded that environmental temperature has a great influence on the ductility of composites. Figure 6(c) shows the failure displacement of specimens aged in different NaCl concentration solutions. It can be noted that the specimens aged in 5.0% NaCl solution had the smallest failure displacement, followed by that in distilled water and 3.5% NaCl solution.

3.3. Relationship between moisture and mechanical property

In marine environment, the mechanical property of composites is influenced by temperature, concentration and time simultaneously. It is difficult to directly establish the coupling relationship between those factors and the mechanical performance of composites, and the needed amount of experiment is also large. However, as one of the most important indicators of materials in humid environment, the moisture content can be used to bridge the environmental factors and the mechanical performance. The variation in retention of shear strength with moisture content in composite laminates is shown in figure 7. It can be noted that there was an apparent dependent relationship between the ILSS and the moisture content. The best fitting curve for experimental data was expressed as followed.

\[ R = R(M) = 39.8M^3 - 107.9M^2 + 51.2M + 100 \]  

where \( R \) is the retention of shear strength, %; \( M \) is the moisture content, %.

At the initial moisture absorption period, the shear strength was improved. This may be related to the moisture absorption stress, releasing the curing shrinking stress in composites. With the further increasing of moisture content, the shear strength showed a gradual degradation, which may be attributed to the ageing damage, such as matrix plasticization and fiber/matrix interface debonding.

The predicted retention rates of composites in different environmental conditions are listed in table 3, and compared with experimental data. The maximum deviation between the predicted results and experimental data is 4.2%, which means that the empirical formula can be well used to predict the degradation of ILSS of composites in marine application.
3.4. Ageing damage and failure mode

Figure 8 shows the typical ageing damages observed in specimens after long term seawater immersion. Delamination, fiber/matrix interface debonding and matrix cracking are the three typical physical damages caused by seawater environment. Such damages are mainly related to the moisture absorption of matrix, which causes the swelling of composites. Since the swelling is restrained in the fiber direction, significant residual stresses are induced in the multidirectional laminate by moisture absorption [39]. When the moisture stress is larger than the adhesion strength between layers, delamination will occur. Due to the different moisture expansion coefficients of matrix and fiber, the absorbed moisture may cause different expansion of the two, resulting in debonding at the interface. In addition, NaCl exists in the solution as cations and anions, which would penetrate together with water molecules into the composite, damaging the matrix, fiber and the fiber/matrix interface [40].

Figure 9 shows the failure morphology of short beam specimens. It is found that the main failure modes are interlaminar shearing cracking and transverse cracking. The number of such cracking increases and its damage becomes more serious with the increasing of ageing time and environmental temperature, as shown in figures 9(a) and (b). The increase of crack density is due to the degradation of interlaminar properties. However, the failure damage of specimen aged in 0.0% NaCl solution seems to be more serious than that aged in 3.5% and 5.0% NaCl solutions, as shown in figure 9(c). In addition, it can be noted that the interlaminar shear cracking usually appears at the edge of the specimens.

The fracture morphologies of unaged and 7 months aged CFRP specimens after short beam shear test were observed by SEM, as shown in figure 10. The SEM images are chosen from the horizontal crack area. It can be seen from the unaged specimen, figure 10(a), that the fibers were embedded well in matrix and covered tightly, only a little resin on the top of the fibers was removed, which suggests that the fiber/matrix interface bonding behavior is strong. However, after 7 months ageing in 30 °C seawater condition, the fibers were still embedded well in matrix, but the resin covered on the fibers became less, shown in figure 10(b). Moreover, for 50 °C and 70 °C conditions, there were almost no residues on the fibers, and the fibers separated from the matrix, shown in
It is clear that the higher the exposure temperature is, the weaker the bonding behavior between fibers and resin, the less the resin left on the fibers surface. Such SEM observation results are consistent with that of short beam shear test, and can explain the degradation of ILSS of CFRP after ageing in seawater environments.

4. Conclusions

The effects of ageing time, temperature, and NaCl concentration on moisture absorption behavior and ILSS of CFRP were investigated. In addition, the relationship between ILSS retention and moisture content was established. The following conclusions may be drawn:

(1) The moisture absorption behavior of composites in seawater environment follows the Fickian diffusion law. The moisture diffusion coefficient and saturated content are mainly affected by ambient temperature but not by NaCl concentration.
(2) The interlaminar shear stiffness and shear strength are more sensitive to aging environmental temperature than seawater ageing time and NaCl concentration. After exposed in 3.5%-70 °C seawater environment for 7 months, the retention rate of shear strength is only 68.8% and the failure displacement decreases by 20.5%.

(3) The shear strength increases first and then decreases with the increase of the maximum moisture absorption, which is mainly related to the release of curing shrinking stress and the damage of interface properties.

(4) The main failure modes of short beam specimens are interlaminar shearing cracking and transverse cracking. Moreover, the interlaminar shearing cracking usually appears at the edge of the specimens. The degradation of ILSS is related to the destroying of fiber/matrix interface.

Declarations of interest

None.

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