Study on the Moisture Absorption and Bending of Carbon Fiber Composite Material

Wen Cheng  
School of Mechatronic Engineering  
Xi'an Technological University  
Xi'an, 710021, PR China  
E-mail: 358968672@qq.com

Qingming Fan  
School of Mechatronic Engineering  
Xi'an Technological University  
Xi'an, 710021, PR China  
E-mail: fanqingming@xatu.edu.cn

Yan Cao  
School of Mechatronic Engineering  
Xi'an Technological University  
Xi'an 710021, PR China  
E-mail: jantonyz@163.com

Bing Shen  
School of Aeronautics  
Northwestern Polytechnical University  
Xi'an, 710072, PR China  
E-mail: 814052598@qq.com

Abstract—The research object of this paper is a kind of carbon fiber composite material that is used in a special UAV in a certain marine area. The purpose of the research is to understand the material properties of this composite in extreme hot and humid environments. First of all, this material was subjected to a 24-hour cycle damp heat test that lasted sixty days. The test temperature was 70°C and the relative humidity is 85%. The results of the damp heat test basically reflected the hygroscopic property of the composite material accorded with Fick's first law. Secondly, since the components of the UAV structure that use this material are primarily subjected to bending loads, three-point bending tests were conducted with the specimens subjected to different stages of damp heat tests. Finally, the decay of the bending properties of this composite material in the hygrothermal condition was obtained by the experimental data analysis, which provides technical support for its applications in engineering.

Keywords—Carbon Fiber Composite Material; Damp Heat Test; Hygroscopic Property; Three-Point Bending Test

I. INTRODUCTION

Carbon fiber composite materials have characteristics such as high specific strength and large specific modulus, and are therefore widely used in fields such as aviation and aerospace. The composite materials have effectively reduced the structural weight of the aircraft and improved the technical performance of the aircrafts. Today, usage of the composite materials has become one of the important indicators to measure whether the aircraft is advanced[1]. Therefore, the preparation, testing and evaluation of aging properties of composite materials have become the focus of research by scientists. Different accelerated aging test methods are commonly used to evaluate the performance of composites[2-5], that is, by performing mechanical tests on specimens that have undergone aging for different periods of time, the discipline of performance degradation could be found out. During The aging process, the plasticity, chemical structure, stress state, and even the interface between the fiber and the polymer of the composite may change[6-8]. Unfortunately, these changes are usually negative and can lead to a reduction of the mechanical properties. Therefore, it is necessary to study the aging behavior of composite materials in their service environment. Only by studying the aging laws and mechanisms of composite materials, can we have a more correct assessment of their performance in engineering applications.

During the commissioning process of a small UAV, members of the R&D team have discovered that the carbon fiber composite material was aging in a hot and humid environment. This UAV is mainly used in some certain marine area. The carbon fiber composite material used in the wing structure is degraded due to the long-term operation in a hot and humid environment. This is a major hidden danger to the structural reliability of the UAV. This prompted the research work of this paper.

In this research, we conducted a hygrothermal aging test on the carbon fiber composite material used in this UAV to understand the hygroscopicity characteristics of this type of composite material under a specific temperature and humidity condition. Meanwhile, since the application of the composite material is mainly the panel of the wing box structure, the material is mainly subjected to bending loads. Therefore, the three-point bending test is performed on the specimens after hygrothermal aging test, and the curve of the bending performance with the aging time was obtained, which provided the data basis for the subsequent structural analysis. In this study, a more precise test was designed, the main purpose of which is to analyze the changes in the mechanical properties of carbon fiber composite material affected by the heat and humidity environment. The bending performance of specimens subjected to the hot and humid environment test is compared to the specimens without damp heat test. Careful analysis of the experimental results helps in drawing useful conclusions on the specific influence of hot
and humid environment on the change of bending performance of this composite material.

II. EXPERIMENTAL PROCEDURES

A. Test specimens preparation

The carbon fiber composite laminates used in this study were made of 3K Twill Prepreg which is provided by Xi’an Carbon Materials Co., Ltd. The relevant parameters are shown in Table 1.

![Table 1. Mechanical properties of 3K Twill Prepreg in orthogonal directions.](image)

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Tensile strength</th>
<th>Tensile modulus</th>
<th>Compression strength</th>
<th>Compression modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value</td>
<td>600MPa</td>
<td>50GPa</td>
<td>200MPa</td>
<td>30GPa</td>
</tr>
</tbody>
</table>

According to the requirements of structural parameters of the composite material in the application of the UAV, and in accordance with the requirements of the standard ASTM D7264/D7264M, the dimensions of the carbon fiber laminates are 160mm*12.5mm*4mm. The test specimen is shown in Fig. 1.

![Figure 1. The shape and dimensions of the specimen (thickness=1.5mm)](image)

B. Damp heat test

In order to accurately test the water absorption rate of the composite material, all the specimens were first dried in a vacuum drying at 70℃. When the mass change due to evaporation of water in the specimens was less than 0.02% per day, the drying is completed. In this experiment, the drying process lasted for 15 days. All the dried specimens were weighted by a BSA124S electronic balance and the weight of the specimens in fully dry condition was recorded. In this research, a total of 65 specimens were prepared, of which 65 were evenly divided into thirteen groups. One group was used to directly test the bending properties of the material in dried condition, and the other twelve groups were used for the damp heat test.

The main control parameter for damp heat test was time of exposure of the test specimens to the hot and humid environment. All of the specimens were exposed to the same solution for varying lengths of time. The prepared test specimens were placed in a LHS-100CL constant temperature and humidity chamber for the damp heat test. The chamber is shown in Fig.2. The test temperature was set to 70℃ and the relative humidity was set to RH 85%. Damp heat test time lasted for a total of sixty days. Five groups of test specimens were taken out every 2 days, then four groups were taken out every 5 days, the last three groups were taken out every 10 days. Wipe the condensate that may appear on the surface of the specimen and weigh it to test its moisture absorption rate. Then, the moisture absorption rate of test specimens was calculated according to formula (1).

![Figure 2. LHS-100CL constant temperature and humidity test chamber](image)

\[
M_t = \frac{m_t - m_0}{m_0} \times 100\% 
\]  

(1)

Where,

- \(M_t\) = Moisture absorption rate of the specimen after a damp heat test with a duration of \(t\), %.
- \(m_t\) = Mass of the specimen after a damp heat test with a duration of \(t\), g.
- \(m_0\) = Mass of the specimen in dry condition, g.

C. Three-point bending test

Through the test, the load of the loading head and the displacement curve of the loaded part of the specimen were obtained. According to the formula provided in the standard ASTM D7264/D7264M, the bending stress and bending strain curve, force and displacement curve of the test were obtained, and the bending strength of the material was obtained at the same time.

The specimen size is 160mm*12.5mm*4mm, span ratio \(l/h=32\), specimen thickness \(h=4mm\), loading speed of the testing machine is 2mm/min. There are 5 specimens in each group. The average value of the collected data is used for data analysis. The stress can be calculated for any point on the load-deflection curve by the following equation:

\[
\sigma = \frac{3PL}{2bh^3}
\]  

(2)

where:

- \(\sigma\) = stress at the outer surface at mid-span, Mpa[psi],
- \(P\) = applied force, N[lbf],
- \(L\) = support span[in.],
- \(B\) = width of beam, mm[in.],
- \(h\) = thickness of beam, mm[in.].

III. RESULTS AND DISCUSSION

A. Moisture absorption rate measurement

After taking out the damp heat test specimens, the weight of the specimens was measured with a BSA124S electronic balance, and compared with the weight of the dried specimens. After calculating the data of each group, the
curve of the moisture absorption rate of the test specimen changes with the test time was obtained, as shown in Fig. 3 and Table 2.

![Moisture Absorption Rate - Time curve](image)

**Figure 3. Moisture Absorption Rate - Time curve**

<table>
<thead>
<tr>
<th>Specimen group</th>
<th>Moisture Absorption Rate (%)</th>
<th>Damp heat test time (Day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>A</td>
<td>0.16</td>
<td>2</td>
</tr>
<tr>
<td>B</td>
<td>0.3</td>
<td>4</td>
</tr>
<tr>
<td>C</td>
<td>0.47</td>
<td>6</td>
</tr>
<tr>
<td>D</td>
<td>0.56</td>
<td>8</td>
</tr>
<tr>
<td>E</td>
<td>0.63</td>
<td>10</td>
</tr>
<tr>
<td>F</td>
<td>0.71</td>
<td>15</td>
</tr>
<tr>
<td>G</td>
<td>0.73</td>
<td>20</td>
</tr>
<tr>
<td>H</td>
<td>0.75</td>
<td>25</td>
</tr>
<tr>
<td>I</td>
<td>0.78</td>
<td>30</td>
</tr>
<tr>
<td>J</td>
<td>0.81</td>
<td>40</td>
</tr>
<tr>
<td>K</td>
<td>0.82</td>
<td>50</td>
</tr>
<tr>
<td>L</td>
<td>0.82</td>
<td>60</td>
</tr>
</tbody>
</table>

**TABLE II. MOISTURE ABSORPTION RATE**

From the curve in the figure, it can be found that the process of moisture absorption of test specimens is mainly divided into three stages. The first stage is from the beginning of the test until 10 days. At this stage, the rate of moisture absorption grows quite fast, and its growth trend is close to linear. The second phase is between 10 days to 30 days. During this phase, the moisture absorption rate of the test specimens slowed down significantly. The third phase is 30 days until the end of the test. At this stage, the moisture absorption rate of the specimens tends to be saturated, with slight fluctuations around 0.8%. This curve shows that the composite material basically conforms to Fick’s law in the early stage of moisture absorption, but as the test continues, the moisture absorption curve gradually deviates from Fick’s law, showing that the moisture absorption rate increases slowly and tends to be saturated.

**B. Bending properties measurement**

In the three-point bending test, the bending load and the corresponding bending displacement data of each specimen were recorded by using the matching software of the testing machine. According to the formula given in the standard ASTM D7264/D7264M, the stress-strain curve of the loaded part of the specimen during the test is calculated, and the bending strength of the specimen is obtained. The curve of the average bending strength of specimens with the damp heat test time is shown in Fig. 4. The average bending strength of each group of specimens is shown in Table 3.

![Average Flexural Strength – Time curve](image)

**Figure 4. Average Flexural Strength – Time curve**

From the curve in the figure, it can be found that in the first two stages of the damp heat test, that is, within the first 10 days of the test, the bending strength of the specimens did not decrease significantly. When the damp heat test enters the third stage, the bending strength of the specimens appeared to decrease more rapidly than the previous specimens. At the same time, in the observation of the third-stage specimens, it was found that the phenomenon of fiber delamination at the fracture of the specimens increased. Although the moisture absorption of the specimens mainly occurred in the first two stages, the moisture absorption rate did not reach saturation. However, after the moisture absorption rate of the specimens reached saturation, the bending performance of the specimens has changed significantly. Due to the damp heat test, the specimens absorbed a large amount of water, resulting in the destruction of the microstructure of the composite material, and the interface strength between the fiber and the resin matrix is deteriorated, which lead to a decrease in the bending properties of the material. The average bending strength of the specimens subjected to the one-week damp heat test was about 9.45% lower than that of the original specimens in the dry state.

**TABLE III. AVERAGE BENDING STRENGTH OF EACH GROUP OF SPECIMENS**

<table>
<thead>
<tr>
<th>Specimen group</th>
<th>Average bending strength (Mpa)</th>
<th>Damp heat test time (day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original</td>
<td>478.99725</td>
<td>0</td>
</tr>
<tr>
<td>A</td>
<td>467.94111</td>
<td>2</td>
</tr>
<tr>
<td>B</td>
<td>465.47411</td>
<td>4</td>
</tr>
<tr>
<td>C</td>
<td>463.05186</td>
<td>6</td>
</tr>
<tr>
<td>D</td>
<td>462.2404</td>
<td>8</td>
</tr>
<tr>
<td>E</td>
<td>461.27626</td>
<td>10</td>
</tr>
<tr>
<td>F</td>
<td>454.10714</td>
<td>15</td>
</tr>
<tr>
<td>G</td>
<td>446.55</td>
<td>20</td>
</tr>
<tr>
<td>H</td>
<td>442.75551</td>
<td>25</td>
</tr>
<tr>
<td>I</td>
<td>439.89115</td>
<td>30</td>
</tr>
<tr>
<td>J</td>
<td>435.98568</td>
<td>40</td>
</tr>
<tr>
<td>K</td>
<td>437.99453</td>
<td>50</td>
</tr>
<tr>
<td>L</td>
<td>433.72798</td>
<td>60</td>
</tr>
</tbody>
</table>
C. Relation between mechanical properties degradation and moisture absorption rate

Fig. 5 shows the changes of mechanical properties and moisture absorption of the composites with aging time at 70 C and RH 85%. It can be seen from the figure that there is a certain corresponding relationship between the mechanical properties and moisture absorption of the composites. In the early stage of moisture absorption, when the moisture absorption rate of composites is fast, the performance of composites decreases obviously; in the later stage, the moisture absorption rate of composites tends to be balanced, and the mechanical properties of composites tend to be flat.

![Figure 5. Curve of relationship between mechanical properties and moisture absorption rate](image)

D. Microstructure of the specimen fracture

The observation of the carbon fiber at the fracture of the sample was performed by using a microscope, including the original specimens and the specimens subjected to the damp heat test. Due to the large number of specimens, except for the original specimens, only specimens that had been subjected to damp heat tests for 60 days were selected for microscopic observation.

On the original specimens, the fiber broke unevenly, and the fiber end still retained a large amount of epoxy resin, indicating that the carbon fiber and the epoxy resin bonded well to each other, and there was no obvious failure to the interface between them, as shown in Fig.6.

With the continuation of the damp heat test, the specimen fracture gradually became neat, and the epoxy retained on the fiber surface began to decrease, indicating that the bond strength between the fiber and the resin matrix gradually decreased. In the final stage of the damp heat test, the surface of the fractured fiber surface was smooth and showed a clear interface, indicating that the bond strength between the fiber and the resin matrix was seriously reduced, as shown in Fig.7.

![Figure 6. SEM micrographs showing the microstructure of fracture of original specimens](image)

![Figure 7. SEM micrographs showing the microstructure of fracture of damp heat test specimens after 60 days](image)

In the three-point bending test, when the specimen is loaded, the upper surface bears compressive stress and the lower surface bears tensile stress. In the early stage of damp heat test, the failure of the specimens are mainly caused by the tensile failure of the lower surface, while the upper surface remains intact, and only slight indentation occurs at the loading nose. With the continuation of the damp heat test, the failure mode of the sample changed obviously. Compressive failure of the upper surface of the specimen occurred many times during 10 to 30 days of damp heat test. For the specimens aged for more than 30 days, the failure modes are mainly mixture of compressive failure on the upper surface and tensile failure on the lower surface. The micro-structure of the fracture surface of the specimen with compressive damage is shown in Fig. 8. As shown in the figure, the traces of fibers drawn out from the matrix are clearly visible at the fracture of compression failure, indicating that the debonding phenomenon of fiber/matrix is more serious at this time.

![Figure 8. Microstructure of Compression Damaged Fracture of 60-days damp heat test specimens under SEM](image)
In the moisture absorption process of the test specimen, the water molecules enter the resin matrix in the form of free water, and also enter the voids and cracks produced by the processing of the composite material. As the damp heat test continues, on one hand, hydrogen bonds are formed between the water molecules and the resin, which weaken the intermolecular forces between the resin and the fibers; on the other hand, due to the non-absorbent nature of the carbon fiber, there is a difference in wet heat expansion between the carbon fibers and the resin matrix. Due to the increasing moisture absorption rate of the resin matrix, the differences are getting bigger and bigger, causing internal stress at the interface between carbon fiber and resin matrix. The continuous increase of internal stress will lead to the debonding and cracking of the interface, the cracks will promote the absorption of water, and the absorbed moisture will promote the increase of internal stress. This vicious cycle is the root cause of the gradual deterioration of the mechanical properties of composites [10].

IV. CONCLUSION

Based on the results obtained from a study aimed at understanding the influence of hot and humid environment on bending properties of carbon fiber composite material, following are the key findings:

1) After the damp heat test of the fully dried carbon fiber composite test specimen, the hygroscopic property of the material at a temperature of 70°C and a relative humidity of RH85% were obtained. Meanwhile, a moisture absorption curve was drawn at the same time. In the early 10 days of the test, the moisture absorption rate of this material increased linearly and quickly; in the next 20 days, the increasing rate of the moisture absorption rate gradually slowed down; within the next 30 days, the moisture absorption rate eventually got saturated.

2) Three-point bending test was performed on the specimens subjected to the damp heat test for various periods of time. The failure of the specimens subjected to different aging time was observed using an optical microscope. The fractures of the specimens were observed by using an optical microscope. Observations of the original specimens that had not been subjected to a damp heat test revealed that the fibers at the fracture still retained a large amount of resin matrix. The observations showed that the adherence of the fibers to the resin matrix can be maintained better when the conforming material is in a dry state. As the damp heat test continued, the interface between carbon fiber and the resin matrix at the fracture was getting more and more clear. This showed that under the dual effects of temperature and humidity, the physical structure of the composite was gradually changed.

3) As the moisture absorption rate continued to increase, the bending properties of the material continued to decrease. The average bending strength of the specimens subjected to the one-week damp heat test was about 9.45% lower than that of the original specimens in the dry state.

Therefore, in the application of carbon fiber composite materials, it is necessary to fully consider the effect of the hot and humid environment on its mechanical properties. The results obtained in this study provide important reference for engineering applications using this material.

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REFERENCES