Study on the hygrothermal aging behavior and diffusion mechanism of Ti/CF/PMR polyimide composite laminates

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Abstract
A series of hygrothermal aging experiments were performed on Ti/CF/PMR polyimide (TCPP) composite laminates in this work in order to verify the moisture diffusion characteristics and diffusion mechanism of the laminate. The specimens were subjected to various hygrothermal aging parameters, specifically 70 °C/95RH%, 85 °C/75RH%, 85 °C/85RH% and 90 °C/95RH%. The results illustrate that the temperature affected the diffusion rate of water molecules in the composite laminates, and the relative humidity determined the saturated moisture absorption rate. For the orthogonally laminated TCPP composite laminates, two equations have been obtained to predict the saturated moisture absorption and diffusion rate of water molecules of the prepreg layer. Additionally, the moisture absorption as a function of time has been expressed for the prepreg layer.

1. Introduction
Fiber metal laminates (FMLs) are a class of hybrid composites consisting of alternating layers of thin metal alloy sheets and fiber-reinforced polymer matrix composites [1]. FMLs utilize the high fracture toughness properties of composite laminates and superior impact resistance of metals [2, 3]. After the first generation ARALL (Aramid Reinforced ALuminium Laminates) materials from the 1970s, several grades of fiber metal laminates have been created to meet the critical requirements in aerospace applications [4]. Nevertheless, as the aerospace industry has developed, both commercial and military aircrafts are designed to fly faster and last longer than ever before. It is necessary for these materials to possess a high temperature resistance capability. To improve the temperature resistance of FMLs, it is obvious that high temperature resistant titanium alloys and polymer matrices are appropriate selections to manufacture novel FMLs [5]. Polyimide produced by in situ polymerization of monomeric reactants (PMR) have gained popularity in aerospace and aviation applications due to their excellent combination of melt processability, thermal stability, and mechanical properties. Thus, PMR polyimides have been chosen as the matrix to manufacture high temperature resistant FMLs. In our previous work, a novel FML based on carbon fiber-reinforced PMR polyimide was designed to meet the demands of high temperature applications [6].

Hygrothermal aging resistance is vital for laminated composites. The durability of the material affects the safety and economy of the aircraft. For a resin-matrix composite, the glass transition temperature or modulus of the matrix decreases due to the moisture absorption during the hygrothermal aging process, which affects the overall performance of the components. Therefore, research on the moisture absorption of laminated composites is necessary and helpful to predict the effect of moisture on the properties of such materials and has a
vital effect on the design of aircraft components and the selection of materials. Compared to traditional fiber-reinforced matrix composites, FMLs are covered with metal layers on the surface of the material. Therefore, it is difficult for moisture to diffuse into the composite layers (through the thickness direction, or in the direction of the alternating layers); consequently, FMLs exhibit excellent moisture and corrosion resistance. However, moisture can still penetrate the composite layers through the free edges of the laminate or holes after long-term high humidity and temperature exposure [7]. In practical service, FMLs are inevitably subjected to severe variation of high temperature and humidity [8]. Hygrothermal aging can result in matrix degradation and interfacial delamination, which lead to early structure failure [9, 10]. Hence, hygrothermal aging can still be a threat to FMLs. Hygrothermal aging behavior and the diffusion mechanism must be systematically investigated to understand the effects of temperature and humidity on the material properties.

In the past several years, Botelho, et al investigated the influence of elevated temperature and humidity on elastic properties and damping behavior for GLARE (Glass fiber Reinforced Epoxy laminate) laminates [11, 12]. The results showed that mechanical properties (tensile and compression strength) and viscoelastic properties (storage modulus and loss modulus) of glass fiber/epoxy composites were reduced more than GLARE laminates after hygrothermal aging. It was determined that moisture absorption through free edges of GLARE laminates was likely the primary cause. Zhong et al [13] showed that both tensile strength and fatigue life of GLARE 4A laminates decreased even though no structural defects were identifiable in the microstructures of the conditioned laminates. The results indicated a 64% decrease in strength of untreated GLARE laminates. They also observed that the moisture diffusion through the matrix of the composite layers resulted in degradation similar to conventional fiber-reinforced matrix composites.

Borgonje and Ypma introduced the long-term behavior of GLARE in their study [14]. They found that the GLARE possessed good corrosion resistance since the prepreg layers acted as corrosion barriers. They reported that the influence of moisture on GLARE was relatively minor; however, properties still presented significant degradation after hygrothermal exposure. Ypma et al [15] showed that the values of blunt notch strength and interlaminar shear strength decreased by 15% with hygrothermal treatment for 3000 h, at 80 °C and 85% RH. The impact of the porosity level on moisture absorption was remarkable, which resulted in an increased intensity of moisture absorption on the composite edges, according to the investigation by Lopes et al [16].

Many researchers have focused on the potential effects of hygrothermal aging on the mechanical properties of FMLs. However, hygrothermal aging behavior and the diffusion mechanism of FMLs have rarely been mentioned. In this work, the hygrothermal aging behavior and diffusion mechanism of Ti/CF/PMR polyimide (TCP) composite laminates were investigated systematically. These laminates were chosen because of their excellent properties, such as good mechanical properties and high damage tolerance. Herein, the specimens were subjected to various hygrothermal aging parameters, then the saturated moisture absorption and diffusion rate of the orthogonally laminated TCP composites was determined. Additionally, the relationship between moisture absorption and time variation of the prepreg layer has been evaluated.

2. Experimental procedure

2.1. Materials
PMR polyimide (KH-308, Institute of Chemistry, Chinese Academy of Science, China), carbon fibers (CF, TR505 6L, Mitsubishi Rayon Co. Ltd, Japan), and titanium plate (TA2, 0.3 mm in thickness, Baoji Titanium Industry Co. Ltd, China) were used as the starting materials. The PMR polyimide contained about 50 wt.% of polymerized reaction mixtures. 0.125 mm.

2.2. Preparation process of TCPP
Ti/CF/PMR polyimide (TCP) composite laminates with the configuration of [Ti/0°/90°/Ti/0°/90°/Ti/90°/0°/Ti] were fabricated according to the preparation process, as depicted in literature [6, 17]. Carbon fiber reinforced prepregs (CFRPs) composed of carbon fibers and PMR polyimide resin were self-prepared using numerical control automatic placement machine in the laboratory. Before laminating, the titanium was surface treated by sandblasting method in order to obtain a good bonding strength between titanium and prepreg. Then, titanium and prepreg layers were stacking in a picture-frame mold. The schematic diagram of TCPP composite laminates is presented in figure 1. The mold was then put into a hot press system, after curing, the TCPP samples were obtained.

The experimental parameters used in the hygrothermal aging experiment are illustrated in table 1. The dimensions of the testing specimens are 100 mm × 25 mm. Before starting the hygrothermal aging experiment, the specimens were placed in a vacuum oven at 60 °C for 24 h to remove the free water retained in the specimens until the quality of the specimens remained unchanged.
During the hygrothermal aging process, mass variations of the specimens were measured at certain intervals, and the moisture absorption rate of the material was calculated according to equation (1) [13].

\[
M = \frac{W_{\text{wet}} - W_{\text{dry}}}{W_{\text{dry}}}
\]

where \( W_{\text{wet}} \) and \( W_{\text{dry}} \) correspond to the mass of the material after moisture absorption and the mass of the material in the dry states, respectively.

### 3. Results and discussion

#### 3.1. Hygrothermal aging of TCPP composite laminates

In the TCPP composite laminates, titanium plates cannot absorb water, and the mass variations of the composite laminates during the hygrothermal aging process are primarily attributed to the moisture absorption of the prepreg layer and moisture absorption between interfaces. The moisture absorption rate of the prepreg layer is more representative than the moisture absorption rate of composite laminates in studying such composite materials. The moisture absorption rate of the prepreg layer can be calculated by equation (2).

\[
M' = \frac{W'_{\text{wet}} - W'_{\text{dry}}}{W'_{\text{dry}}}
\]

where \( W'_{\text{wet}} \) and \( W'_{\text{dry}} \) correspond to the mass of the prepreg layer in the wet and dry states, respectively.

The mass relationship between \( W'_{\text{dry}} \) and \( W_{\text{dry}} \) under the dry state satisfies equation (3), where \( Vf \) is the volume fraction of the components and \( \rho \) is the relative density.

\[
W_{\text{dry}} = Vf_{\text{Tl}} \rho_{\text{Tl}} + Vf_{\text{prepreg}} \rho_{\text{prepreg}}
\]

Figure 2 presents the relationship between the moisture absorption rate and time of the prepreg layer in TCPP composite laminates. It can be seen that the hygroscopicity curves under different conditions exhibit a similar hygroscopic tendency. In the initial stage of hygrothermal aging, the hygroscopicity of the material significantly increases with time, and the hygroscopic process is a typical Fickian diffusion. The mass increment rate of the prepreg layer decreased with further extension of the hygrothermal aging time, and finally, its mass reached a saturated state and achieved equilibrium. For the fiber-matrix composites, the moisture absorption
process can be divided into two stages [18, 19]. The first stage is mainly the diffusion and penetration of water in the resin matrix, where the accumulation of water causes defects such as microcracks and pores, which is a reversible physical adsorption process [20]. In the second stage, the growth of the moisture absorption rate is reduced, while the moisture absorption mechanism is more complicated. The moisture absorption can be caused by resin hydrolysis, crack growth as well as diffusion. However, the damage caused by both stages is irreversible.

3.2. The diffusion mechanism of TCPP composite laminates

According to Fick’s hygroscopic model, the moisture diffusion during the hygroscopic process can be calculated by equation (4):

$$\frac{\partial c}{\partial t} = D_X \frac{\partial^2 c}{\partial x^2} + D_Y \frac{\partial^2 c}{\partial y^2} + D_Z \frac{\partial^2 c}{\partial z^2}$$

For fiber-reinforced composite laminates, the dimensions in the thickness direction are significantly smaller than that in the length and width directions. The diffusion of moisture in the thickness direction (the X-direction as shown in figure 1) plays a leading role, while the diffusion in the Y-direction and Z-direction can be ignored. Therefore, for fiber-reinforced composite laminates, its water diffusion can be regarded as a one-dimensional diffusion mode. Formula 4 can be reduced to equation (5).

$$\frac{\partial c}{\partial t} = D_X \frac{\partial^2 c}{\partial x^2}$$

where \( c \) is the moisture concentration and \( x \) is the diffusion distance along the diffusion direction. When hygrothermal aging is not performed, that is, when the aging time is less than 0, \( c = c_i \) is constant. When the hygrothermal aging starts, that is, \( t = 0 \), the moisture concentration at the edge of the specimen is saturated, \( c(h) = c(0) = c_e \).

According to the above boundary conditions and equation (5), the following relationship can be obtained:

$$\frac{c}{c_e} = 1 - \frac{4}{\pi^2} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n + 1)^2} \exp\left[ -\frac{D_X t}{c_e} (2n + 1)^2 \right]$$

At the same time, the moisture absorption of the material in the hygrothermal aging process can be calculated by equation (7):

$$W = \int_0^h c \, dx$$

Simultaneously, equation (8) can be deduced via the above equation.

$$M_t = M_f \left\{ 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n + 1)^2} \exp\left[ -\frac{D_X t}{c_e} (2n + 1)^2 \right] \right\}$$

Figure 2. Relationship between moisture absorption rate and time of prepreg layer in TCPP composite laminates.
For fiber-reinforced composite laminates, the moisture absorption rate and diffusion coefficient at any time are in accordance with equation (8). Equation (8) is often approximated by the following expression in equation (9) [21].

\[
M_t = M_e \left\{ 1 - \exp \left[ -7.3 \left( \frac{D_{Xt}}{h^2} \right)^{0.75} \right] \right\}
\] (9)

where \(M_t\) is the saturated moisture absorption rate of the materials, \(D_{Xt}\) is the diffusion coefficient of moisture in the X-direction, that is, the thickness direction, and \(h\) is the thickness of the material. \(D_{Xt}\) can be obtained from equation (5), where \(M_1\) and \(M_2\) correspond to the moisture absorption of the material at \(t_1\) and \(t_2\) time, respectively.

\[
D_X = \pi (h/4M_e)^2 [(M_2 - M_1)/(\sqrt{t_2} - \sqrt{t_1})] 
\] (10)

However, for the TCPP composite laminates, moisture cannot diffuse into the material in the thickness direction in the hygrothermal aging process but can diffuse along the Y and Z directions. Therefore, equations (9) and (10) cannot apply to the TCPP composite laminates. It is obvious that the diffusion of moisture in the TCPP composite laminates belongs to a two-dimensional diffusion mode. According to Fick’s second law, equation (11) is obtained for the two-dimensional scenario.

\[
\frac{\partial c}{\partial t} = D_Y \frac{\partial^2 c}{\partial Y^2} + D_Z \frac{\partial^2 c}{\partial Z^2} 
\] (11)

For the moisture absorption process of TCPP composite laminates, the following boundary conditions exist:

\[
\begin{align*}
\c(Y, Z, 0) &= c_i \\
\c(0, Z, t) &= c_e \\
\c(l, Z, t) &= c_e \\
\c(Y, 0, t) &= c_e \\
\c(Y, b, t) &= c_e \\
\c(Y, Z, \infty) &= c_e 
\end{align*} 
\] (12)

where \(D_Y\) and \(D_Z\) are the diffusion coefficients in the Y and Z directions, respectively, \(c_i\) is the initial concentration, and \(c_e\) is the concentration at equilibrium. Under the above boundary conditions, the relationship between moisture concentration in the laminates and the diffusion coefficient (equation (13)) at any time, based on equation (11) can be obtained.

\[
\frac{c_e - c}{c_e - c_i} = \frac{16}{\pi^2} \sum_{n=0}^{\infty} \sum_{m=0}^{\infty} A^2 \sin \left( \frac{2m + 1}{2} \right) \sin \left( \frac{2n + 1}{2} \right) \frac{\pi b}{b} \times e^{-(2m + 1)(2n + 1) \pi b h r} 
\] (13)

The following equation can also be defined: \(A = 1/(2m + 1)(2n + 1)\), \(B = 1/A\).

The moisture absorption of composite laminates in the hygrothermal process can be calculated by equation (14).

\[
W = \int_0^l \int_0^b c(Y, Z, t) \, dy \, dz 
\] (14)

After simultaneous analysis and calculation, the following relationship can be obtained (equation (15)) [22].

\[
\frac{\sqrt{D_Y}}{l} + \frac{\sqrt{D_Z}}{b} = \frac{\pi}{4M_e} x \frac{M_t}{\sqrt{t}} 
\] (15)

In this section, the TCPP composite laminate is a 4/3 layup configuration with orthogonal ply, so \(D_Y = D_Z\). Therefore, \(D_{YZ} = D_Y = D_Z\), and \(D_{YZ}\) can be calculated by equation (16).

\[
D_{YZ} = \left[ \frac{M_e \times l \times b}{4M_e \times (l + b)} \times \frac{\pi}{\sqrt{t}} \right]^2 
\] (16)

Figure 3 illustrates the linear section and fitting curve of the moisture absorption curve of the TCPP composite laminates prepreg layer. The relationship between saturated moisture absorption and relative humidity of the prepreg layer of the TCPP composite laminate is shown in table 2.

Figure 4 exhibits the relationship between the diffusion rate and hygrothermal aging time. The diffusion rate of water molecules is greatly affected by the temperature during the hygrothermal aging process, and the variations in humidity have little effect on the diffusion rate. The results of the diffusion rates at different temperatures are shown in table 3.

In the process of moisture absorption, the diffusion rate of water in the material can be expressed by the Arrhenius equation [23], as shown in equation (17).
where $D$ is the diffusion coefficient, $D_0$ is the diffusion constant, $E_a$ is the diffusion activation energy, $R$ is the gas constant, and $T$ is the diffusion temperature. By taking the logarithm on both sides of equation (6) and plotting $\ln D - 1/T$, the slope and intercept will give the diffusion activation energy ($E_a$) and diffusion constant.
It can be seen from equation (17) that the diffusion rate of water is correlated with the temperature and diffusion activation energy but is unrelated to relative humidity.

Figure 5 shows the relationship between the diffusion coefficient and temperature during diffusion. It can be seen from figure 5 that the relationship between the diffusion coefficient of the moisture in the TCPP composite laminate prepreg layer and the temperature during the hygrothermal aging process can be explained according to equations (18) and (19).

\[
\ln D_{YZ} = -\frac{1532.22}{T} - 4.68821 \tag{18}
\]

\[
D_{YZ} = 9.2 \times 10^{-3} \times \exp\left(\frac{-1532.22}{T}\right) \text{mm}^2 \text{s}^{-1} \tag{19}
\]

Therefore, the diffusion constant and diffusion activation energy can be obtained.

In addition, the published [24] points out that the saturated moisture absorption rate of the materials is correlated to the relative humidity (RH) during hygrothermal aging, and the relationship between them satisfies equation (20).

\[
M_e = a \times (RH)^b \tag{20}
\]

where \( a \) and \( b \) are material constants determined by experimental data.

It can be seen from table 2 that when the relative humidity is 75% or 85%, the saturated moisture absorption of the prepreg layers of TCPP composite laminates correspond to 0.50779 and 0.65856, respectively, which can be substituted into equation (20).

\[
\begin{aligned}
0.55 &= a \times (75)^b \\
0.72 &= a \times (85)^b
\end{aligned}
\]

Simultaneous calculations above can be obtained to give:

\[
\begin{aligned}
b &= \frac{\ln(0.55/0.72)}{\ln(75/85)} = 2.15 \\
a &= \frac{0.55}{75^{2.15}} = 5.11 \times 10^{-5}
\end{aligned}
\]

Therefore, the relationship between the saturation moisture absorption rate and the relative humidity of the prepreg layer of TCPP composite laminates satisfies equation (21).

\[
M_e = 5.11 \times 10^{-5} \times (RH)^{2.15} \tag{23}
\]

In summary, for orthogonally laminated TCPP composite laminates, when they are subjected to the hygrothermal aging treatment, the moisture diffusion process in the prepreg layer can be analyzed by the following equation. The saturated moisture absorption rate of the prepreg layer is:

\[
M_e = 5.11 \times 10^{-5} \times (RH)^{2.15} \tag{24}
\]
The diffusion coefficient of water molecules in the prepreg layer of TCPP composite laminates is:

\[ D_{YZ} = 9.2 \times 10^{-3} \times \exp\left(-\frac{1532.22}{T}\right) \text{mm}^2 \text{s}^{-1} \]  

(25)

and the moisture absorption of the prepreg layer at any time is:

\[ M_t = 4M_r \times \frac{1 + b}{1 \times b} \times \sqrt{\frac{9.2 \times 10^{-3} \times \exp\left(-\frac{1532.22}{T}\right) \times t}{\pi}} \]  

(26)

4. Conclusions

In the hygrothermal aging process, the experimental temperature affects the diffusion rate of water molecules in the composite laminates, and the relative humidity determines the saturated moisture absorption rate of the TCPP composite laminates. For orthogonally laminated TCPP composite laminates, the saturated moisture absorption of the prepreg layer can be predicted by the equation:

\[ M_t = 5.11 \times 10^{-5} \times (RH)^{2.15}. \]

The diffusion rate of water molecules in the prepreg layer can be calculated by the equation:

\[ D_{YZ} = 9.2 \times 10^{-3} \times \exp\left(-\frac{1532.22}{T}\right) \text{mm}^2 \text{s}^{-1} \]

The moisture absorption of the prepreg layer at any time can be expressed as:

\[ M_t = 4M_r \times \frac{1 + b}{1 \times b} \times \sqrt{\frac{9.2 \times 10^{-3} \times \exp\left(-\frac{1532.22}{T}\right) \times t}{\pi}} \]

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Author contributions

Yubing Hu designed and performed the experiments, wrote the draft; Cheng Liu performed the experiment; Chen Wang and Xuelong Fu revised the draft; Yanan Zhang contributed to the theoretical analysis. All authors contributed to the general discussion.

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