Research Mechanical Test of Accelerated Aging Method for Carbon Fiber Reinforced Polymer Composites

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Abstract—It is an important engineering problem that affected by the environmental factors, the performance of aircraft structure of carbon fiber reinforced polymer (CFRP) under the Marine environment has depredated; and it is an important means of analysis of CFRP material degradation by accelerating aging mechanics experiments under laboratory conditions. First of all, a laboratory accelerated aging spectrum of some sea area is compiled under the foundation of the sea temperature and humidity spectrum ultraviolet irradiation, and with the principle of damp and hot effection is equal to light endowment for CFRP sheet damage failure; Secondly, combining with this accelerated aging spectrum, the accelerated aging experiments have been done for the one-way and the single experiment conduct; Finally, the experimental results for hygroscopic rate and morphology have been analysis. It is showed that the accelerated aging 1a later, the moisture absorption rate is increasing, and the macro morphology changed obviously, the moisture absorption affection is most obvious by 0°, followed by 90°, the anti-aging performance is best by 45°.

Keywords-CFRP; accelerated aging experiment; Marine environment spectrum; analysis of moisture absorption rate; morphology analysis

I. INTRODUCTION

When the helicopter operating some mission in a certain sea area, stubborn Marine environment caused serious damage on the aircraft structure of CFRP materials, and aging speed is serious and more than the expected by contrasted of service period on the airport and offshore,. Natural aging experiment is the most real test method of researching composite material aging problem, but it is time consuming, and low efficiency which can not be ignored. This paper will carry out accelerated aging experiments for CFRP unidirectional single plate test piece under laboratory conditions by compiling CFRP laboratory accelerated aging spectrum. By the process of experiment,

the quality variation of test pieces is recorded real-timely, and macro and micro photos of test pieces are shouted and analysis in stages. By analyzing the aging test results, theoretical basis of engineering application is provided for reliability analysis of this type of material.

II. THE COMPLEMENT OF CFRP LABORATORY ACCELERATED SPECTRUM

A. Compiling principle of Acceleration spectrum

It was pointed out that the most important environmental factors affecting the aging of CFRP in the literature [1,2] were the temperature, humidity and ultraviolet radiation, respectively. The acceleration method of ultraviolet radiation spectrum is based on the total amount of ultraviolet radiation, so it is important to discuss the acceleration method of temperature and humidity spectrum. A lot of research results show that the aging effect of the composite material is more on the quality of the resin matrix. Because of the hydrolysis of the resin material in the moisture absorption, the damage of the material is caused by the material, which affects the life of the material.

And among moisture absorption and diffusion in the resin matrix, the Fick second law is satisfied:

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial^2 h} \qquad (1)$$

Where, t is the moisture absorption time, h is the composite material thickness, c is the diffusion concentration, D is the diffusion coefficient. The upper integral, then: $C_t = G(C_m - C_0) + C_0$.

Where, C_0 indicates the initial moisture absorption rate of composite materials, C_m is a function of the

equilibrium moisture absorption rate, G is a function of time [3].

$$G = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp[-(2j+1)^2 \pi(Dt/b^2)]}{(2j+1)^2} \quad (2)$$
$$\approx 1 - \exp[-0.73(Dt/b^2)^{0.75}]$$

Assuming that, when the temperature and moisture diffusion rate remain the same, the absorption affection occurs only on the up and down surface which have larger area, and the smaller side had no moisture phenomenon, then variation of relative mass by plate absorption process can be expressed as.

$$G = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp[-(2j+1)^2 \pi(Dt/b^2)]}{(2j+1)^2} \quad (3)$$
$$\approx 1 - \exp[-0.73(Dt/b^2)^{0.75}]$$

The initial default water content $C_0 = 0$, the moisture absorption rate is:

$$C_t = C_m \{1 - \exp[-0.73(Dt/b^2)^{0.75}]\} \quad (4)$$

Based on the fact that the initial value of the moisture absorption and diffusion of the composite material is linear, the diffusion coefficient is obtained by the initial value of the moisture absorption curve:

$$D = \pi (\frac{b}{4C_m})^2 (\frac{C_2 - C_1}{\sqrt{t_2} - \sqrt{t_1}})^2 \quad (5)$$

TABLE I. CONVERSION COEFFICIENTS FOR CFRP BETWEEN TEMPERATURE-HUMIDITY AND EXPERIMENT CONDITION

ϕ (%) \ T(℃)	25	30	35	40
70	0.155	0.240	0.327	0.416
80	0.215	0.316	0.416	0.511
90	0.278	0.391	0.499	0.599
>90	0.341	0.464	0.578	0.681

Experts and scholars at home and abroad have summarized a large number of moisture absorption test results; composited materials moisture diffusion rate under different hot and humid environment is drawn. And combined with CFRP hydrothermal affection experiment under different hot and humid environment (including the mechanics performance experiment), the common accelerated hydrothermal aging experience formula for engineering's gotten as followed [4, 5]:

$$K = \frac{t_2}{t_1} = \frac{e^{-C/(T_2\phi_2)}}{e^{-C/(T_1\phi_1)}} \quad (6)$$

Among them, K is the accelerate coefficient, t_1, T_1 and ϕ_1 represent the actual exposure time, temperature and humidity respectively, t_2, T_2 and ϕ_2 represent accelerated experiment time, temperature and humidity respectively; C is the parameter, when $T_2 \leq 60°C$, $C = 46.1$.

The conversion coefficient of CFRP between different temperature and humidity and accelerated experiment condition can be obtained, shown in TABLE I

B. *CFRP laboratory acceleration spectrum*

After CFRP test pieces arrived in some sea area, it takes 1a per cycle with natural exposure, but the actual exposure experiment time is only half a year, the rest of the time is used to walk back and forth and recycle and measure the test pieces.

According to the conversion coefficient between different temperature and humidity and the experiment conditions combined with the sea temperature and humidity accelerated experiment spectrum, the total time can be obtained by simulating 1a in laboratory:

$$T_{humidity} = \frac{1}{2}T_{humidity}'$$
$$= \frac{1}{2} \times 2504.938 = 1252.5h \quad (7)$$

In terms of the ultraviolet radiation acceleration, UVA UV lamp imported from the PHILIPS Germany is used for laboratory accelerated experiment. After calibrated by UV irradiation meter, the radiation intensity near the lamp tube is $70w/m^2$. To further shorten the test time, this experiment placed accelerated test pieces near the tube, which can be considered that the intensity of accelerated irradiation is $70w/m^2$. The total simulation time of the 1a can be obtained by the ultraviolet radiation spectrum of the sea area, which can be calculated by the following formula[6]:

$$T_{radiation} = \frac{1}{2}T_{radiation}' = \frac{1}{2} \times (\frac{Q' \times 10^6}{70 \times 3600}) \quad (8)$$

Where, Q' is the total amount of ultraviolet radiation in a certain sea area throughout years. Therefore, the acceleration time of the ultraviolet radiation of the 1a cycle in the natural exposure test of a sea area is 569.29h, and the radiation temperature is $55°C$.

In summary, the laboratory accelerated aging experiment to simulate the 1a cycle of natural aging experiment required soaking 1252.5h distilled water, and irradiating 569.29h by ultraviolet. Setting: A represent the immersion test 156.56h; B represent the ultraviolet irradiation 71.16h; AB cycle test method is adopted in this experiment as shown in Fig. 1.
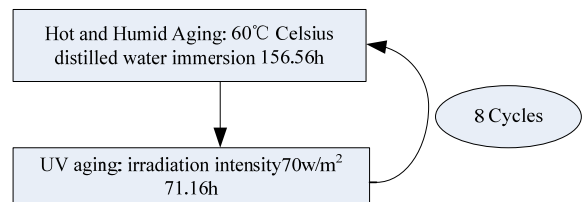

Figure 1. The experiment cycle plan

III. THE AGEING ACCELERATED EXPERIMENT IN LABORATORY

A. The ageing accelerated experiment

The CFRP plates used in the ageing accelerated experiment in laboratory is single one-way shop layer board specimens designed and made on basis of domestic carbon fiber to enhance CCF materials and BA9916-Ⅱresin matrix type. According to the design of the test pieces shape in GB3354-82 and GB3355-82, the laying angle is designed of 0°, 45° and 90° three types as shown in Fig. 2. To prevent its own damage from fixture device while doing mechanical properties test, we specifically equip pieces of alkali EW100 without fiber pads on both sides of the test pieces. All the test pieces are made by Beijing aviation manufacturing engineering research institute.
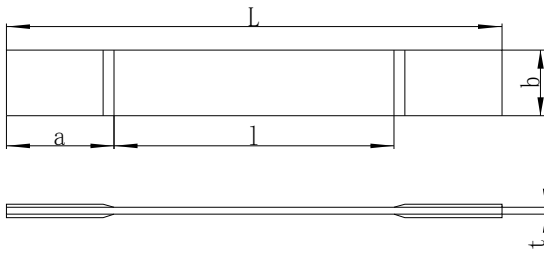

Figure 2. The design of the specimen

TABLE II.   TAB.2 DIMENSION OF SPECIMEN

| Layer angle	Strengthen gaskets	Size				
		L/mm	b/mm	t/mm	a/mm	Chamfering Φ
0°	EW100	230	12.5±0.5	3	50	≥15°
90°	——	170	25±0.5	4	50	——
45°	EW100	250	25±0.5	4	50	≥15°

The main testing device used in this experiment includes: SHHW21.600 AⅡ thermostatic water tank (made by Tianjin, LTD), PJZP (made by Qingdao sol test equipment co., LTD). In the process of test, the test pieces should be placed and weighed on high precision electronic balance (made by Shanghai precision scientific instrument co., LTD) in every 12 hours. According to the method ruled in GBT12626.8-1990, The balance should be performed under the condition of the relative humidity of 65±5% and temperature of 20±2℃ after the test piece has constant mass so that the testing data can be read. The entire test process is about 1882 h long, so the surface of test pieces would be filmed microscopically at the end of each test, for observing its changing law of morphologies.

B. The analysis of testing results

Define the primary mass as m0, after a period as long as t, the mass of test piece is m_t. Then the rate of mass changing (it is also called the moisture absorption rate in the following) can be expressed as:

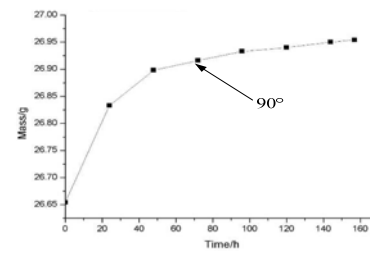$$r(t) = \frac{m_t - m_0}{m_0} \times 100\% \qquad (9)$$

In the process of the first soak test, the weighing of the experiment product is recorded. Find the test pieces with the same laying angle, take the average. Then the moisture absorption rate can be acquired. The moisture curve of different laying angle in the first soak cycle is draw and shown in Fig. 3.
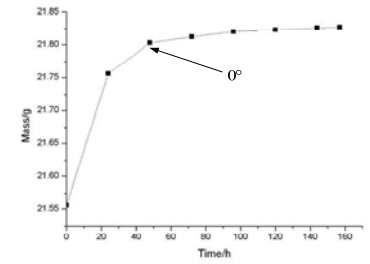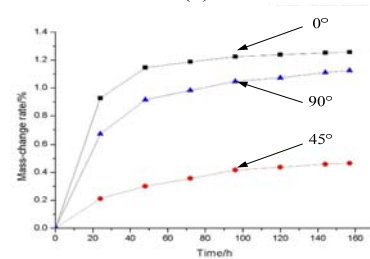

(a)
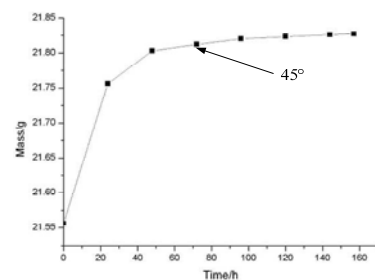
(b)

(c)

(d)

Figure 3. The mass-change for specimens when soaked during the first cycle

Comparing the different mass change curves of the test pieces of carbon fiber lying angle in the first soak cycle showing above, we can find that the test pieces' mass change curves have a similar overall trend. In the first soak cycle, 0° test piece has a greatly increase on mass during the first hours of the first soak cycle, although the volume is smallest, the added mass is greatest during the first hours; the increase during the last 40 hours is tiny, similar to a straight level line. At the

same time, the 90° test piece's mass increase also slows down after the same soaking period, the mass change curve is similar to a linear function with small slope, ranking first among the three test pieces. Though it does not has as strong effect of moisture absorption as 0° test piece, it may have some relation with the relative larger surface area. However, 45° test piece is different. Its' volume ranks first among the three pieces, but its' mass increase is much smaller than the 0o test piece and the 90° test piece during the first 24 hours. Meanwhile, we also find that along about 157 hours test progress, the mass data nearly obeys a quadratic function. Although the mass monotone decreases after the first 24 hours, it still keeps a stable increase by the end of soaking, and it's more obvious compared with the 0° test piece and the 45° test piece—increasing 0.01 gram during the last 13 hours, which is five times more than the 0° test piece.

Clearly, the former two have a much higher moisture absorption rate than the latter one, the 0° test piece has the strongest moisture absorption effect with the 45° test piece following. The gained mass amount decreases a lot because this two quickly reaches the range of the transient saturation moisture absorption. On the contrary, the 45° test piece is still far from the range of the range of the transient saturation moisture absorption, so it keeps a obvious increase.

During the test, the test pieces should be filmed macroscopically and microscopically regularly, observing the changing law of morphologies of single layer board surface caused by aging. The CFRP plates has not been damaged and surface resin is also in a good state at the beginning of the test, so it's almost the same macroscopically and microscopically, and the orientation of carbon fiber could not be found. In the first two aging cycle tests, composite materials surface morphologies just change a little, shown in Fig. 4.
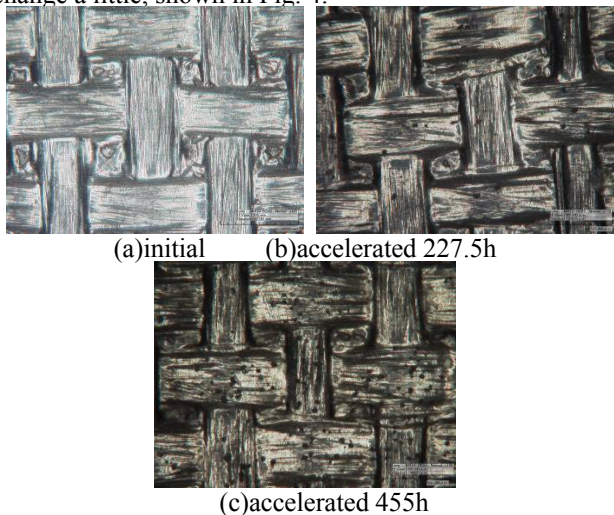

(a)initial   (b)accelerated 227.5h

(c)accelerated 455h

Figure 4.   Microscope photos of specimen for accelerated test during the first 2 cycles($\times$160)

Besides the test, we took photos of the panel both in macroscopic view and also in microcosmic, to find the changing laws of the pattern because of the age. In the prime test, because the CTRP pattern was not damaged, so the macroscopic photo and microcosmic photo were showing no differences. It's hard to find the carbon fiber arrange direction.

In the process of initial state, aging speed up 911h,to speed up the test at the end of the macro morphology photos. In the test before the start, CFRP board is black, in trials for four after cycle, the surface of the material color began to be turned into a pale black. Fiber soaked in direction when paper more clear. In speed up at the end of the test, the surface of the resin ash to black, the direction of the fiber arrangement has more clear. The surface of the resin consumption of serious damage. These phenomena in microscopic photo more intuitive in Fig. 5.
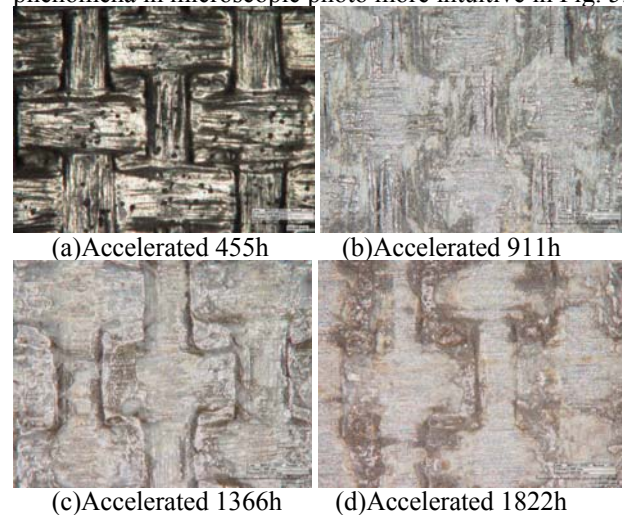

(a)Accelerated 455h   (b)Accelerated 911h

(c)Accelerated 1366h   (d)Accelerated 1822h

Figure 5.   Microscope photographs of specimen(0°) during accelerated test($\times$160)

(a)accelerated 455h   (b)accelerated 911h

(c)accelerated 1366h   (d)accelerated 1822h
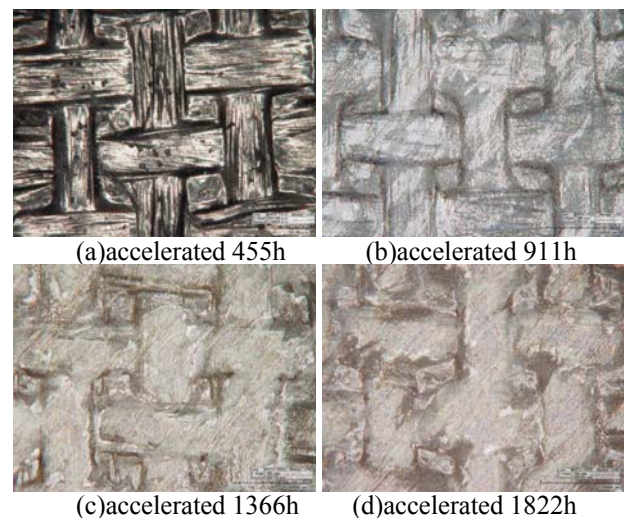
Figure 6.   Microscope photographs of specimen(45°) during accelerated test($\times$160)

(a)accelerated 455h   (b)accelerated 911h
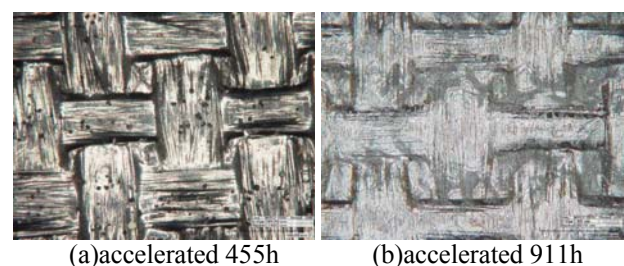
(c)accelerated 1366h   (d)accelerated 1822h

Figure 7. Microscope photographs of specimen（90°）during accelerated test(×160)

It is not hard to see that, after the accelerated aging test carries out 911 hours, namely, 4 cycles later, the surface of resin matrix has turned into the small flake off, from some black spot destruction, but the affection of resin package remains relatively intact, and it can be observed in the local scope that small amount of fiber bundle is exposed, and laying angle is visible. After accelerated aging 1366 h, fiber bare area is further expanded, the resin layer is missing lightly only within the scope of the local concentration, and a considerable amount of matrix has "crocodile" phenomenon. After the accelerated test, resin layer lost more seriously, carbon fiber laying angle is clearly visible, the area covered by a resin layer is further reduced.

By contrast of Fig. 5(d) , Fig. 6(d) and Fig. 7(d), we can find that during the same period of aging monolayer plate specimen in different laying angle, 0° and 90° specimen is much more serious. In the late test, the resin layer peeling phenomenon in great quantities appeared in the two groups of specimens, and the whole row of fiber is bare in the vertical angle direction of fiber laying, and surface microstructure is more smooth. And 45° specimen is not so: the resin layer covering affection is still relatively well until the end of the test, and strip shape resin covers along the fiber direction except the thick resin layer, which showed stronger anti-aging ability obviously. This phenomenon matches the results of section 3.2.2.1 which showed that specimen hygroscopic rateis different from different test pieces: the faster specimens aging rate is, the more serious moisture absorption rate of resin matrix damage.

IV. CONCLUSION

This paper carried out accelerated aging experiments for CFRP unidirectional single plate test piece by compiling CFRP laboratory accelerated aging spectrum.

The experimental results for hygroscopic rate and morphology have been analysis. It is showed that the accelerated aging 1a later, the moisture absorption rate is increasing, and the macro morphology changed obviously, the moisture absorption affection is most obvious by 0°, followed by 90°, the anti-aging performance is best by 45°.

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