Study on properties of composite laminate with a hole in the hygrothermal environment

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Abstract. The influence of hygrothermal environment on compressive property of woven carbon fiber epoxy resin composite was investigated via tensile and compress tests under different hygrothermal environment. Moisture absorption, tensile and compressive properties, failure modes, FTIR and dynamic mechanical property were analyzed. The results show that the moisture absorption of woven carbon fiber epoxy resin composite is not high. The hygrothermal environment has larger influence on the compressive property than the tensile property. The failure modes of composite laminate with holes are hole failure, the tensile fracture is flush, the compressive fracture has an obvious fiber push-into phenomenon of the hole sides. There is no new peak in FTIR after moisture absorption, representing that no chemical reaction occured of the epoxy resin. The glass transition temperature T_g of the composite after moisture absorption is 173˚C, which fells 14˚C than the dry counterpart.

Introduction

Carbon fiber and its composites have good mechanical properties, as a kind of excellent materials in the structure of the aviation and aerospace vehicle, it is becoming more and more widely used[1-3].Composite materials will be affected by the hygrothermal environment inevitably, the hygrothermal environment is one of the most serious conditions to make composite materials aging easily, it can affect the property directly, the design and use of departments is particularly concerned with the effects of the hygrothermal environment on mechanical properties of composite materials. There are always some holes in the composite structures, for example, bolt holes for bolts, the through holes for inspection, and assembly holes, etc. Carbon fiber is completely cut off by holes, which can also cause serious stress concentrations. The composite structure is very sensitive to the damage, and the residual strength will be reduced seriously, especially for the tensile and compressive residual strength. The properties of composites with holes under the hygrothermal environment are key aspects to assess the mechanical properties of composites, the influence of hygrothermal environment on structural safety has also become one of the key techniques for the verification of composite structures[4].

In this paper, the woven carbon fiber epoxy resin composite laminate with the center hole was saturated with moisture, the morphology change of the specimen were observed by SEM, tensile and compression tests were carried out under different conditions, the influence of tensile and compressive properties by hygrothermal environment was analysed. This provides a certain basis for the structural design and the use of the composite materials, and it has important engineering practical significance.

Experiment Procedure Program

Materials and specimens. The specimen was made of 3K woven carbon fiber by autoclave process, and the matrix material was BA9916-Ⅱ. The center hole diameter of the specimen was 6 mm. The size of tensile specimen was 250×36×2.76mm^3(length×width×thickness), and was 300×36×3.68 mm^3(length×width×thickness) for the compressive specimen. The layer of tensile specimen was $[(45/-45)/(0/90)]_{3s}$, and the layer of compressive specimen was $[(45/-45)/(0/90)]_{4s}$.

Experiment method. The moisture adsorption process of composites specimen was tested according to ASTM D 5229-2014 [5]. The specimens ware put into temperature and humidity test chamber(temperature of 71˚C and relative humidity of 85%) after get engineering dry state, the moisture content of specimen was measured by electronic balance at periodic intervals. The composite specimens were put into the chamber immediately after measurement, and the mass and time were recorded, every measurement should not more than 30mins.The percent moisture content M_t is defined as followed

$$M_t = \frac{W_t - W_0}{W_0} \times 100\% \tag{1}$$

Where, W_t is the weight of moist specimen and W_0 is the weight of dry specimen.

When the moisture content of the specimen changed by less than 0.01% over each of two consecutive reference time period spans, the specimen was defined to be in a state of effective moisture equilibrium .The morphology of the specimens before and after moisture absorption were observed by SEM and analyzed by SPECTRUM 100 Fourier transform infrared (FTIR) and ATR model was used, the spectrum range was from 400cm^{-1} to 4000cm^{-1} and the resolution was 4cm^{-1}.

The dynamic mechanical property tests were conducted based on ASTM D7028-2007 [6], it was carried out by Q800 DMA dynamic mechanical thermal analyzer, and the single cantilever beam loading mode was used, the size of specimen was 35×12×4mm^3(length×width×thickness)， and the frequency was 1Hz， the heating rate was 5˚C/min.

The tensile test and the compressive test were conducted based on ASTM D5766-2011[7] and ASTM D6484-2014 [8], all the tests were carried out on INSTRON 5982 electronic universal testing machine, the load speed was 1 mm/min and the test environment include 23˚C-dry, 70˚C-wet, 90˚C-wet, 110˚C- wet, 130˚C- wet. The results represented the average value of six tests.

Results and Discussion

Moisture absorption behavior. The moisture absorption behavior is shown in Fig.1, where the specimen moisture content (*M_t*) is plotted as function of the square root of the absorbing time ($t^{1/2}$) for tensile and compressive specimens. As can be seen from Fig.1, the initial stage of moisture absorption is rapid moisture absorption phase, it is due to defects in the composites and the water absorption of the resin. During this phase, the moisture absorption rate of the tensile and compression specimens are similar. With the increase of the absorption time, the absorption rate decrease gradually, and the moisture absorption curve tend to be gentle. Because of the epoxy resin in the initial phase of moisture absorption, it can be cured, and the polymer had chemically reaction at the same time. It can make a large number of hydrophilic groups through absorbed water until saturation. But the moisture absorption rate of the compression specimen is higher than that of tensile specimen, it may be due to the size of compression specimen is larger than the tensile specimen and the error is from processing technology and specimen measurements. After moisture absorption, the moisture absorption rate of the compression specimen is about 0.84%, and the moisture absorption rate of the tensile specimen is about 0.77%.

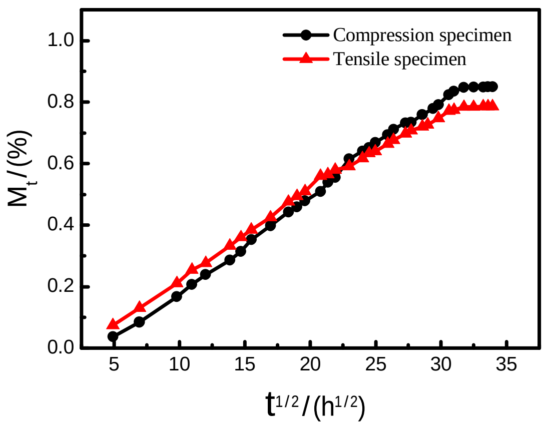
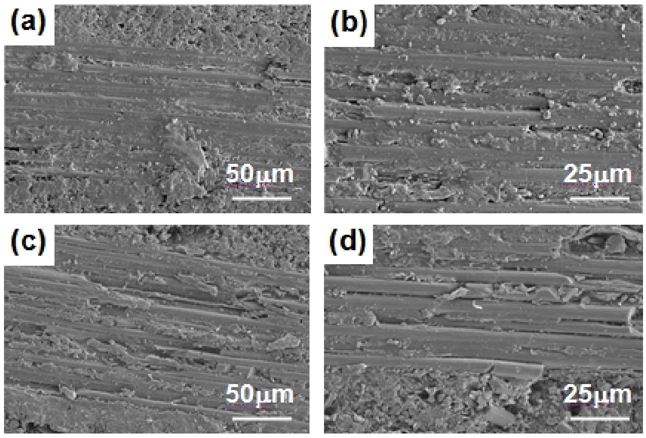


Fig.1 Moisture absorption curves of composite laminates



（a）low multiple of dry; (b) high multiple of dry;
（c）low multiple of wet; (d) high multiple of wet

Fig .2 The SEM photomicrographs of composite

Surface topography. The photomicrographs of specimens before and after moisture absorption were observed by SEM, the results are shown in Fig.2. As can be seen from Fig.2, the dry specimen has good adhesion property between fiber and matrix, and no fiber is pull-out. The photomicrographs of wet specimen is slightly changed, the specimen surface is more smooth after moisture absorption, the resin and the interface are damaged. Because the carbon fiber did not absorb moisture, the resin swelled after moisture absorption. The interfacial adhesion between fiber and resin matrix is damaged by the obvious difference of moisture expansion, thus causing the interface damage, and then the mechanical properties of the composite decrease.

Tensile property. The tensile property of specimen with a hole at different test environment is showed in Fig.3. With the increase of the temperature, the tensile strength curve shows a fluctuation, but the change is not very large. Because the tensile strength is controlled by the properties of carbon fiber, although the resin matrix properties will be reduced in hygrothermal environment, the resin matrix in which the role is not big, so the tensile strength of the specimen with a hole has a higher retention rate after moisture absorption. The edge of the hole is easy to produce a large stress concentration, the resin and the interface of the hole edge can absorb moisture in hygrothermal environment. The variation of stress concentration coefficient is caused by the different conditions of the hole edge moisture absorption, which leads to the fluctuation of the tensile strength. The effect of hygrothermal environment on the tensile property of composite with a hole is not large.

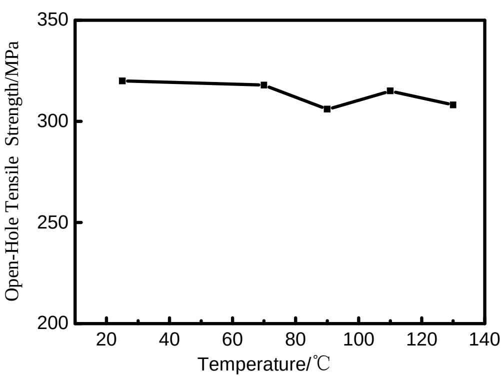


Fig.3 Relationship between tensile strength and temperature

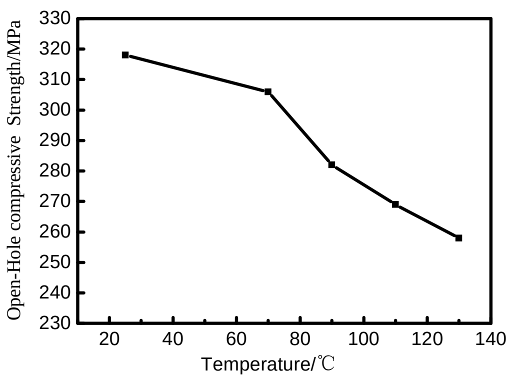


Fig.4 Relationship between compress strength and temperature

Compressive property. The compressive property of specimen with a hole at different test environment is showed in Fig.4. The compressive strength decreases with the increase of temperature, because the compressive strength of the composite is greatly influenced by properties of the resin matrix. The immersion of water molecules in the moisture absorption process of the wet specimen can cause the matrix to be swelled and plastic, reducing the strength of the matrix. In addition, water molecules can also enter the interior of the composite material, the fiber and resin occurs interfacial debonding by the difference of moisture expansion coefficient, which can reduce the interface ability of transfer load and lead to further decline of compressive property. After moisture absorption, when the temperature were 70˚C、90˚C、110˚C and 130˚C,the retention rate of compressive strength of specimens were about 90%, 85%, 75%, 70%. The influence of hygrothermal environment on the compressive property of the composite materials is obvious.

Fracture morphology. The failure mode of tensile and compressive specimens with a hole are over hole damage. Because the hole is the smallest net section of the specimen, it is the area of stress concentration, and the direction of the fracture propagates along the direction of maximum stress concentration. The fracture of specimen was observed by stereomicroscopy, the results are shown in Fig.5 and Fig.6. For the tensile specimens, the failure modes under different hygrothermal environment are basically the same, the fracture of dry and wet specimens are flush, the fracture sections are serrated, it has burr phenomenon and the outermost layer has an obvious characteristic of the ±45°fracture. There are part of the delamination and matrix cracking, but the fracture position of each layer is irregularity, the fiber of the middle layer is shortly pulled out, but no weft fibers are pulled out. For the compressive specimens, the failure modes under different hygrothermal environment are basically the same, there are obvious fiber pressing in the two sides of the hole, it has three types of failure modes, i.e. brittle fracture, fiber indentation and delamination. Sides of the fracture are mainly shear failure, which are characterized by delamination and buckling.

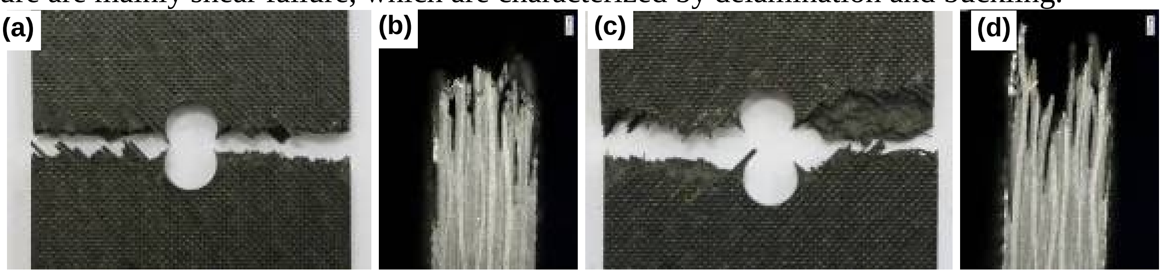


Fig.5 Typical failure modes of tensile test

（a）Front of 23˚C-dry;（b）Side of 23˚C-dry;（c）Front of 70˚C-wet;（d）Side of 70˚C-wet

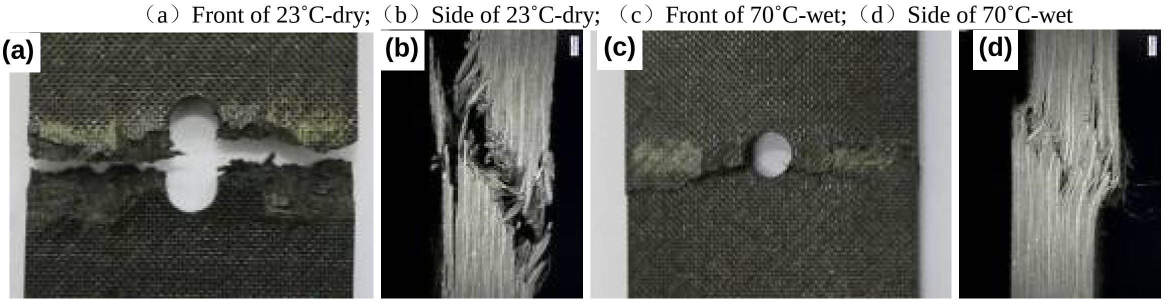


Fig.6 Typical failure modes of compressive test

（a）Front of 23˚C-dry;（b）Side of 23˚C-dry;（c）Front of 70˚C-wet;（d）Side of 70˚C-wet

Infrared spectrum. The infrared spectrums before and after moisture absorption are shown in Fig.7, the horizontal coordinate indicates the position of the absorption peak, and the vertical coordinate indicates the transmission rate. By comparing the infrared spectra of dry and wet specimens, it can be known that there is no new peak appeared in infrared spectra, but the intensity of the peak is partly changed, which shows that there is no chemical reaction of the epoxy resin and the hygrothermal environment has no effect on the chemical properties of the composites. Therefore, the composite material has better stability in hygrothermal environment.

Dynamic mechanical. Fig.8 showed the DMA curves of dry and wet specimens. The glass transition temperature T_g was measured from loss tangent peak in DMA curves, the loss peak is a comprehensive reflection of the mechanical loss of carbon fiber, resin matrix and interface. From Fig.8, it can be seen that the storage modulus of wet specimen are decreased compared with the dry specimens. The major reason is that resin matrix moisture plasticizing leads to stiffness reduction, and the moisture absorption of matrix and carbon fiber has great difference, a direct result of the fiber and the matrix volume expansion mismatch, leading to a decline in the composite modulus. The glass transition temperature corresponding to the loss peak of tanδ decreases, and the temperature of T_g decreases from 187˚C of the wet specimen to 173˚C of the dry one. The glass transition temperature

T_g is decreased, because the interfacial layer has an obvious interfacial debonding after moisture saturation.

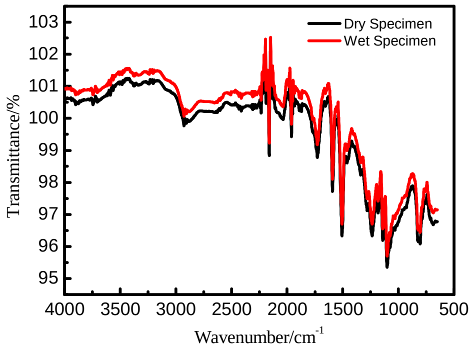


Fig.7 FTIR spectra of dry and wet specimen

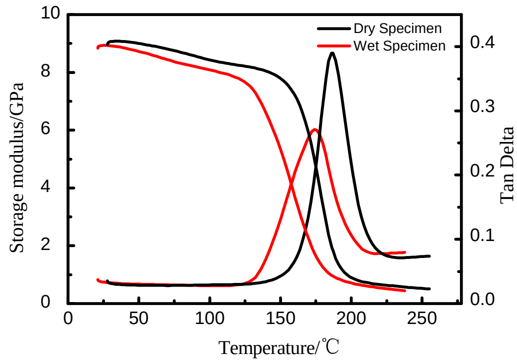


Fig .8 The DMA curves of dry and wet

Conclusions

（1）The woven carbon fiber epoxy composite material moisture absorption rate is low, the saturated moisture absorption of the tensile specimen is about 0.77%, and the compressive specimen is about 0.84%. The hygrothermal environment has little effect on the tensile properties of the composites, but the influence on the compressive properties is obvious.

（2）The hygrothermal environment has little effect on the failure modes of composites with a hole, the failure modes are all over the hole. The fracture of tensile specimens are flush, the sides of compressive fractures are mainly shear failure, which are characterized by delamination and buckling.

（3）There is no chemical reaction of the epoxy resin and the hygrothermal environment has no effect on the chemical properties of the composites. Therefore, the composite material has better stability in hygrothermal environment.

（4）The storage modulus of wet specimens decrease compared with the dry specimens. The temperature of T_g decreases from 187˚C of the wet specimen to 173˚C of the dry one.

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