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Carbon fibre reinforced composites - Method for accelerated moisture absorption and supersaturated conditioning by moisture using sealed pressure vessel

碳纤维增强复合材料 密封压力容器 加速吸湿和过饱和调节方法

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WARNING: This document involves the operation of sealed pressure vessels. Personnel using this document shall operate in accordance with the product safety operating procedures for sealed pressure vessels. This document does not identify all possible security issues. Users are responsible for taking appropriate safety and protective measures and ensuring compliance with the conditions stipulated in relevant national regulations.

1 Scope

This document describes methods for accelerated moisture absorption and supersaturation conditioning of carbon fiber reinforced composites. This method is carried out in a saturated water vapor sealed pressure vessel at a temperature higher than 100°C but lower than the glass transition temperature (T_g) of the test material.

This document applies to thermosetting carbon fiber reinforced composite materials with a glass transition temperature greater than 150°C. For thermoplastic carbon fiber reinforced composite materials, this document can also be used as a reference.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 40396, *Test method for glass transition temperature of polymer matrix composites -- Dynamic mechanical analysis (DMA)*

GB/T 40724, *Terminology for carbon fiber and carbon fiber composites*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 40724 as well as the followings apply.

3.1 additional moisture content

The percentage of the additional moisture absorption mass of the test material to its mass before accelerating moisture absorption.

NOTE: See formula (1).

3.2 supersaturated conditioning by moisture

The process of cooling the test material from a saturated hygroscopic state at high temperature and high humidity (saturated water vapor pressure exists at high temperature) to a hygroscopic state at room temperature.

3.3 accelerated moisture absorption

The process of enabling the test material to obtain the saturated moisture content (3.4) in a short period of time under a certain high temperature (such as 120°C) and high-water vapor pressure (such as 0.2 MPa).

3.4 saturated moisture content

The moisture content of the specimen in the supersaturated moisture state when it is cooled from a higher temperature with saturated moisture content (such as 120°C) to a lower temperature (such as room temperature).

NOTE: See formula (2).

4 Principle

The test material is placed under high temperature and high pressure saturated water vapor conditions for moisture absorption treatment. Because high temperature can accelerate the diffusion rate of water molecules in the material, and high pressure can increase the rate at which the material surface absorbs water molecules from the outside, the material can reach a saturated hygroscopic state in a short time. When the test material that has reached the saturated hygroscopic state is cooled to room temperature, due to the slow diffusion rate of water molecules inside the test material and the slow release rate on the surface at room temperature, the material will be in a supersaturated hygroscopic state. This method can quickly obtain the supersaturated moisture absorption state of carbon fiber reinforced composite materials.

5 Equipment and material

5.1 Supersaturated moisture absorption device. As shown in Figure 1, the supersaturated moisture absorption device consists of the following parts.

a) Sealed pressure vessels. The maximum pressure is not less than 2.0 MPa. It has heating function. The temperature can be controlled within ±2°C of the set shall be controlled at (105±2)°C under decompression conditions.

5.3 Analytical balance. The graduation value is 0.1 mg.

5.4 Desiccator. 10 L glass desiccator with built-in silica gel.

5.5 Specimen bag. It is made of high temperature resistant nylon material; sealable.

5.6 Dust-free absorbent paper.

6 Specimens

6.1 The specimens shall comply with the requirements of the relevant test method to be carried out as given in 8.7.

6.2 The glass transition temperature of the specimen shall be determined according to the provisions of GB/T 40396 before accelerated moisture absorption begins.

7 Conditioning and laboratory environment

7.1 Conditioning

Before the test, the specimen shall be placed at a temperature of (23±2)°C and a relative humidity of (50±10)% for at least 24 h.

7.2 Laboratory environment

The temperature is (23±2)°C. The relative humidity is (50±10)%.

8 Operating procedures

8.1 Preparation before accelerated moisture absorption

8.1.1 Check the appearance of the specimen. Number each specimen.

8.1.2 Adjust the specimen condition according to the provisions of 7.1.

8.1.3 Use an analytical balance (5.3) to weigh the mass of each specimen, recorded as M_0, accurate to 1 mg.

8.1.4 The specimens are not dried before moisture absorption.

8.2 Supersaturated conditioning by moisture

8.2.1 Set the sealed pressure vessel temperature to the test temperature. The test temperature shall be greater than 100°C. The maximum test temperature shall not exceed the material's glass transition temperature (T_g) minus 25°C, that is, T_g-25°C.

8.2.2 Place the specimen on the specimen holder [5.1 b)]. Then put the specimen holder into the sealed pressure vessel [5.1 a)]. Try to avoid contact between specimen surfaces. Minimize contact between the specimen and the holder and sealed pressure vessel. Pour deionized water into the sealed pressure vessel. Allow the specimen to be completely immersed in water.

8.2.3 Close the sealing pressure vessel lid. Close the safety valve [5.1 c)]. Start the heating system.

8.2.4 When the test temperature is reached, open the safety valve for about 2 min. Remove the air from inside the sealed pressure vessel. Then close the safety valve. Use the pressure gauge [5.1 d)] to confirm that the pressure in the sealed pressure vessel reaches the saturated water vapor pressure corresponding to the test temperature.

8.2.5 Keep heating. The continuous heating time can be set based on experience. In the first few cycles of supersaturated moisture absorption adjustment, the continuous heating time can be shorter, such as (24±1) h or (48±1) h. When approaching the saturated hygroscopic state, the continuous heating time shall be increased, such as (72±1) h.

8.2.6 When heating reaches the set heating time, stop heating. Record the actual continuous heating time in hours (h). Open the lid of the sealed pressure vessel. Remove the specimen. Put it into the specimen bag (5.5) and seal it. Cool to room temperature.

8.2.7 Weigh the mass of each specimen according to the provisions of 8.3.1.

8.2.8 After weighing, immediately re-place the specimen on the original specimen holder. Repeat steps 8.2.2 to 8.2.7. The test temperature remains unchanged.

8.2.9 Repeat 8.2.8 until the specimen reaches the saturated moisture absorption state specified in 8.5.

8.2.10 Place the specimen that has reached the saturated moisture absorption state in the vacuum oven (5.2). Dry under reduced pressure at a temperature of 105°C until the mass is constant. That is, the difference in mass between two consecutive weighings does not exceed 1 mg. Cool the specimen dried under reduced pressure to room temperature in the desiccator (5.4). Weigh the mass of the specimen according to the provisions of 8.3.2.

8.3 Specimen mass weighing

8.3.1 Specimen mass weighing after accelerated moisture absorption

After each set heating time is over, weigh the specimen mass after accelerated moisture absorption according to the following regulations.

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