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Fiber-reinforced Composites - Method for Accelerated Moisture Absorption and Supersaturated Conditioning by Moisture Using Sealed Pressure Vessel

纤维增强复合材料 密封压力容器 加速吸湿及过饱和状态调节方法 (ISO 22836:2020, IDT)

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Fiber-reinforced Composites - Method for Accelerated Moisture Absorption and Supersaturated Conditioning by Moisture Using Sealed Pressure Vessel

1 Scope

This document specifies the method for accelerated moisture absorption and supersaturated conditioning by moisture of fiber-reinforced composites using sealed pressure vessels at a heating temperature above 100 °C and saturated water vapor pressure conditions.

The purpose of this moisture absorption method is to screen materials by mechanical or thermal properties.

This document is applicable to fiber-reinforced composites with a glass transition temperature (T_g) greater than 150 °C, including carbon fiber-reinforced thermoset composites (CFRP), carbon fiber-reinforced thermoplastic composites (CFRTP) and glass fiber-reinforced composites, etc.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

ISO 472 Plastics - Vocabulary

NOTE: GB/T 2035-2008 Terms and Definitions for Plastics (ISO 472:1999, IDT)

3 Terms and Definitions

What is defined in ISO 472, and the following terms and definitions are applicable to this document.

Addresses of the terminology databased maintain by ISO and IEC for standardization are as follows:

---ISO online browsing platform: http://www.iso.org/obp;

---IEC electronic open platform: http://www.electropedia.org/.

3.1 additional moisture content

Additional moisture content refers to the additional moisture absorption mass of the test material as a percentage of the initial test material mass.

NOTE: see Formula (1).

3.2 initial moisture content

Initial moisture content refers to the moisture absorption mass percentage of the test material at the beginning of the moisture absorption test.

NOTE: see Formula (3).

3.3 supersaturated conditioning by moisture

Supersaturated conditioning by moisture refers to the conditioning process, in which, the test material is cooled from a moisture absorption saturated state of relatively high temperature and humidity (for example, a relatively high temperature and saturated water vapor pressure) to room temperature.

3.4 accelerated moisture absorption

In order to obtain the saturated moisture content (3.5) in a shorter time, moisture absorption is performed under specific conditions of a relatively high temperature (for example, 120 °C) and moisture absorption pressure (for example, 0.2 MPa).

3.5 saturated moisture content

Saturated moisture content refers to the moisture absorption rate obtained when the test material is cooled from a relatively high temperature (for example, 120 °C) to a relatively low temperature (room temperature) under supersaturated conditioning by moisture.

4 Principle

In order to facilitate the test material to obtain a moisture absorption saturated state that is almost similar to that at room temperature in a short period of time, the test material is placed under the conditions of a relatively high temperature and humidity [for example, temperature 120 °C and saturated water vapor pressure with air (oxygen) 0.2 MPa] for moisture absorption.

The higher the temperature, the faster the diffusion rate of water molecules inside the material; the higher the water vapor pressure, the higher the frequency of the material surface absorbing water molecules from the outside. Therefore, the moisture absorption saturated (or balanced) state of the material can be obtained in a relatively short period of time. However, if this moisture absorption saturated material is cooled to room temperature, due to the slow diffusion rate of water molecules inside the material and the slow-release rate on the material surface, the material will be in a supersaturated state by moisture absorption.

Through this mode, the supersaturated state by moisture absorption of thermoset and

During heating from room temperature to 120 °C, the exhaust system can discharge the air inside the autoclave when the temperature reaches 100 °C.

NOTE: autoclave is known as "high-pressure sterilizer" in the biological and medical industries. This high-pressure sterilizer differs from the stainless-steel sealed pressure vessel (5.2) in the chemical industry.

5.6 Vacuum Oven

The vacuum oven can be heated to 200 °C under reduced pressure, with a temperature control accuracy of \pm 1 °C.

5.7 Balance

The balance can weigh the specimens, and the resolution shall not be lower than 0.1 mg.

6 Test Procedures

6.1 Specimen Weighing

Use a balance (5.7) to weigh the individual specimens. Before weighing, the specimens should be conditioned for 24 h at 23 °C and 50% RH. Before moisture absorption, the specimens shall not be dried, so as to avoid any degradation.

6.2 Acquisition of Supersaturated State by Moisture Absorption

Use a support frame to place the specimens in the sealed pressure vessel. The surfaces of the specimens, the specimens and the sealed pressure vessel will not come into contact with each other. During moisture absorption, they can be completely immersed in water vapor.

Pour deionized water into the sealed pressure vessel (for example, pour $10 \text{ mL} \sim 20 \text{ mL}$ of deionized water into a 300 mL sealed pressure vessel).

Seal the sealed pressure vessel and place it in the oven as shown in Figure 1. Open Valve A and Valve B.

Set the oven temperature to above 100 °C, for example, 120 °C. When the temperature rises to about 100 °C, at Valve B, release water vapor for a period of time (for example, $1 \text{ min} \sim 2 \text{ min}$) to discharge the internal air. Then, close Valve B. At the set temperature, through the pressure gauge, confirm the water vapor pressure inside the sealed pressure vessel.

NOTE: set the appropriate oven temperature in combination with T_g of the test material and the temperature of the preparation process.

Continue heating for a determined period of time, for example, 72 h.

6.3 Sampling of Test Material under Supersaturated State by Moisture Absorption

If there is only one sealed pressure vessel in the oven, then stop heating, and cool the sealed pressure vessel to room temperature. Take the specimens out of the sealed pressure vessel.

If one of the multiple sealed pressure vessels is taken out from the oven, then, close Valve A and disconnect the connector. After taking the sealed pressure vessel out of the oven, cool it to room temperature. Open Valve A. Take the specimens out of the sealed pressure vessel.

This test method should use an autoclave (5.5) instead of a sealed pressure vessel and oven. When using the autoclave, the operating manual shall be followed.

6.4 Confirmation of Saturation by Moisture Absorption

Weigh the specimens of moisture absorption. Then, repeat the test steps 6.2 (if it is necessary to continue moisture absorption, determine the moisture absorption time, for example, 24 h) and 6.3. Repeat the weighing of the specimens of moisture absorption.

NOTE: in order to reduce the influence of the volatility of the specimens in the air, the weighing time shall be shortened as much as possible.

In accordance with Formula (1), calculate the additional moisture content (see Appendix A for example information of measured data of two types of test materials). Determine at least three specimens of moisture absorption and take the average value.

$$W_{\rm ad} = \frac{M_{\scriptscriptstyle n} - M_{\scriptscriptstyle i}}{M_{\scriptscriptstyle i}} \times 100 \qquad \qquad \cdots \qquad (1)$$

Where,

 $W_{\rm ad}$ ---the additional moisture content of the specimen in the saturated state by moisture absorption, expressed in (%);

 M_n —the mass of the specimen in the saturated state by moisture absorption, determined in accordance with Formula (2), expressed in (g);

 M_{i} --the mass of the specimen at the beginning of the moisture absorption test, expressed in (g).

In accordance with Formula (2), determine the saturated state by moisture absorption, record the mass of the specimen in the saturated state by moisture absorption and use it for the calculation of the saturated moisture content.

Where,

 W_{n+1} ---the additional moisture content of the specimen at the (n+1)th test point, expressed in (%);

 W_n ---the additional moisture content of the specimen at the n^{th} test point, expressed in (%).

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