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Textiles - Textiles containing phase change materials - Determination of the heat storage and release capacity

纺织品 含相变材料的纺织品 蓄热和放热性能的测定

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1 Scope

The document describes the method for determining the heat storage and heat release performance of textiles containing phase change materials by differential scanning calorimetry (DSC).

It applies to textiles of every kind containing phase change materials.

2 Normative references

The content of the documents listed below constitutes indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies; for undated references, the latest edition, including all amendments, applies.

GB/T 6529 Textiles - Standard atmospheres for conditioning and testing; GB/T 4669-2008 Textiles - Woven fabrics - Determination of mass per unit length and mass per unit area.

3 Terms and definitions

The following terms and definitions apply to this document.

3.1 phase change material: material which, within the temperature range in which it changes state, is able to absorb or release heat energy in the form of latent heat and so buffer changes of the external temperature.

3.2 phase change temperature: peak temperature at which the phase change material

changes state, that is the melting phase change temperature or the crystallization phase change temperature. Note: in the ideal case the two temperatures are the same, but since the materials commonly used are mixtures, the two differ in an actual test.

3.3 extrapolated onset temperature: temperature corresponding to the intersection of the extrapolated baseline of the DSC curve with the tangent drawn at the point of greatest slope of the curve corresponding to the start of the transition.

3.4 peak temperature: temperature corresponding to the maximum value, or minimum value, reached by the peak on the DSC curve.

3.5 extrapolated end temperature: temperature corresponding to the intersection of the extrapolated baseline of the DSC curve with the tangent drawn at the point of greatest slope of the curve corresponding to the end of the transition.

3.6 enthalpy of phase change: quantity of heat absorbed or released by the phase change material during the change of state at constant pressure, in joules per gram. Note: the heat absorbed on melting is called the melting enthalpy and the heat released on crystallization is called the crystallization enthalpy.

4 Principle

Under a specified atmosphere and a programmed temperature control, the relation between the difference in heat flow rate input to the specimen and to the reference sample and the temperature, the time, or both, is measured; from it the melting temperature, the melting enthalpy, the crystallization temperature and the crystallization enthalpy of the specimen are obtained. The melting temperature and the melting enthalpy characterise the phase change heat storage performance of the specimen and the crystallization temperature and the crystallization enthalpy characterise its heat release performance.

5 Apparatus and materials

5.1 The differential scanning calorimeter and its accessories shall meet the following requirements. a) It shall be able to raise or lower the temperature at a constant rate between 0.5 °C/min and 20 °C/min. b) It shall be able to keep the test temperature constant, with a variation not exceeding +/- 0.5 °C. c) It shall be able to carry out a stepwise programmed temperature rise. d) The gas flow rate range shall be 10 mL/min to 50 mL/min, with the deviation controlled within +/- 10 %. e) The resolution of the temperature signal shall be within 0.1 °C and the noise interference below 0.5 °C. f) The instrument shall be able to record the DSC curve automatically and to integrate the area, with a deviation of less than 2 %. g) It shall be fitted with a sample holder assembly having one or more sample supports. h) Sample pans: used to hold the specimen and to act as reference sample, made of the same material of equal mass; under the measuring conditions the pan shall not undergo physical or chemical change with the specimen or with the atmosphere;

the pan shall have good thermal conductivity, shall be able to be covered and sealed and shall withstand the overpressure produced during the measurement. i) A press, used together with the sample pans, able to seal them. j) The temperature and the energy shall be calibrated periodically with a standard sample whose melting point is close to the test temperature range of the material to be measured, and the calibration shall be repeated when the heating rate or the gas flow rate changes.

5.2 Balance: precision 0.01 mg.

5.3 Gas supply: nitrogen of purity not less than 99.99 %, at a flow rate of 20 mL/min; other flow rates may also be used, the recommended flow range being 20 mL/min to 50 mL/min.

5.4 Scissors or Hardy microtome, able to cut the specimen.

6 Specimen preparation

6.1 Conditioning: the sample shall be conditioned to equilibrium under the standard atmosphere specified in GB/T 6529.

6.2.1 General: the sampling shall be representative and any contamination of the specimen shall be avoided. Sampling is carried out according to 6.2.2 to 6.2.4; after sampling, the specimen is placed in the sample pan, covered and sealed with the press. The bottom of the pan shall be flat and in good contact with the support. During the operation, tweezers or gloves are used to handle the specimen, avoiding direct contact of the hands with the specimen, the pan and their assembly.

6.2.2 Fibres or yarns: for fibres and yarns, specimens are cut at several random points in different parts of the sample, then cut up thoroughly and mixed; not less than 5 mg is taken as one specimen and three specimens are prepared in all. If the coefficient of variation of the results of the three specimens is too large, see 7.8, two further specimens shall be prepared and tested.

6.2.3 Fabrics: the mass per unit area is calculated according to method 5 of 3.3 of GB/T 4669-2008. Several specimens are cut at random points in different parts of the sample so that their total mass is not less than 5 mg, and this combined sample is taken as one specimen; three specimens are prepared in all. If the coefficient of variation of the results of the three specimens is too large, see 7.8, two further specimens shall be prepared and tested. For samples with a multilayer structure, a complex structure or large differences between areas, sampling and testing shall be carried out separately and the results reported separately.

6.2.4 Wadding: the mass per unit area is calculated according to method 5 of 3.3 of GB/T 4669-2008. Several specimens are cut at random points in different parts of the sample so that their total mass is not less than 5 mg, and this combined sample is taken as one specimen; three specimens are prepared in all. For thicker samples, the whole thickness is

cut through, then cut up and mixed, and a mass of not less than 5 mg is taken at random as one specimen; specimens are prepared in the same way at other positions, three specimens in all. If the coefficient of variation of the results of the three specimens is too large, see 7.8, two further specimens shall be prepared and tested.

7 Test procedure

7.1 The power of the differential scanning calorimeter is switched on and the instrument is preheated for at least 30 min.

7.2 With tweezers or another suitable tool, the sample pan holding the specimen and the reference sample are placed in the sample supports, ensuring good contact between the pan holding the specimen, the reference sample and the supports; the cover of the sample support is then put in place.

7.3 Before the programmed temperature operation starts, the instrument is purged with nitrogen for 5 min.

7.4 The instrument programme is set and the heating programme run, taking the differential scanning calorimeter from -20 °C, usually 50 °C below the expected crystallization temperature, and heating at a rate of 5 °C/min while recording, up to 60 °C, usually about 30 °C above the expected extrapolated end temperature of melting, and holding for 5 min.

7.5 The cooling programme is run, lowering the temperature at 5 °C/min to -20 °C while recording, and holding for 5 min.

7.6 A second heating programme is run, raising the temperature again to 60 °C at 5 °C/min while recording. The limit temperatures of heating and cooling may also be agreed between the parties concerned and shall then be stated in the report.

7.7 The instrument is cooled to room temperature.

7.8 Steps 7.2 to 7.7 are repeated for the two remaining specimens. If the coefficient of variation of the test results exceeds 20 %, two further specimens shall be tested.

8 Calculation and expression of results

8.1 From the DSC curve obtained in Clause 7, the enthalpy and the characteristic temperatures of each specimen are read, as shown in Figure 1 of the document. a) From the second heating curve, the melting enthalpy of the specimen is read, that is the area between the melting peak and the baseline, the result being accurate to 0.01 J/g, and the melting peak temperature T m1 is read, the result being accurate to 0.01 °C. If required, the extrapolated onset temperature of melting T m2 and the extrapolated end temperature of melting T m3 may also be read, accurate to 0.01 °C. b) From the cooling curve, the crystallization enthalpy of the specimen is read, that is the area between the crystallization peak and the baseline, the result being accurate to 0.01 J/g, and the crystallization peak