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Advanced technical ceramics - Ceramic composites, thermophysical properties - Part 3: Determination of specific heat capacity

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Hochleistungskeramik - Keramische Verbundwerkstoffe, thermophisikalische Eigenschaften - Teil 3: Bestimmung der spezifischen Wärmekapazität

Céramiques techniques avancées - Composites céramiques, propriétés thermophysiques - Partie 3: Détermination de la capacité thermique spécifique

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword

This document (EN 1159-3:2003) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

This document supersedes ENV 1159-3:1995.

EN 1159 *Advanced technical ceramics – Ceramic composites, thermophysical properties* consists of three parts:

— *Part 1: Determination of thermal expansion*

— *Part 2: Determination of thermal diffusivity*

— *Part 3: Determination of specific heat capacity*

Annex A is normative. Annexes B and C are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 1159 describes two methods for the determination of the specific heat capacity of ceramic matrix composites with continuous reinforcements (1D, 2D, 3D).

Unidirectional (1D), bi-directional (2D) and tridirectional (XD, with $2 < x \leq 3$).

The two methods are:

— method A: drop calorimetry;

— method B: differential scanning calorimetry.

They are applicable from ambient temperature up to a maximum temperature depending on the method: method A may be used up to 2 250 K, while method B is limited to 1 900 K.

NOTE Method A is limited to the determination of an average value of the specific heat capacity over a given temperature range and can give a larger spread of results.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 60584-1, *Thermocouples - Part 1: Reference tables (IEC 60584-1:1995)*.

ENV 13233:1998, *Advanced technical ceramics – Ceramic composites – Notations and symbols*.

3 Terms and definitions

For the purposes of this European Standard, the following definitions and those given in ENV 13233:1998 apply.

3.1
specific heat capacity, C_p
amount of heat required to raise the temperature of a mass unit of material by 1 K at constant temperature and pressure

$$C_p = \frac{1}{m}\frac{dQ}{dT}$$

where

Q is the heat required for a test-piece of mass m

3.2
mean specific heat capacity, $\overline{C_p}$
amount of heat required to raise the temperature of a mass unit of a material from temperature T_1 to temperature T_2 at a constant pressure, divided by the temperature range $(T_2 - T_1)$ expressed in K

3.3
representative volume element (R.V.E.)
the minimum volume which is representative of the material considered

4 Method A - Drop calorimetry

4.1 Principle

A test piece is dropped from a conditioning chamber at a constant temperature T_1 to another chamber at a constant temperature T_2.

The mean specific heat capacity is determined from the measured amount of heat required to maintain the temperature constant in the second chamber. Transfer of the test piece shall be done under conditions as close as possible to adiabatic conditions.

4.2 Apparatus

4.2.1 Drop calorimeter, there are several types of drop calorimeters. They include one (or more) conditioning chambers and measuring chambers which can be operated under controlled atmosphere and which are all equipped with a temperature control system which allows a temperature stability of less than 1 K.

The conditioning chamber shall have a homogeneous temperature zone size greater than the test specimen size. The measuring chamber shall have a homogeneous temperature zone of a sufficient length to accept several specimens and a sufficient thermal inertia to limit the temperature disturbance, due to the drop.

Heat transfer by radiation during the drop shall be avoided as far as possible.

4.2.2 Balance, with an accuracy of 0,1 mg for test pieces over 10 mg and an accuracy of 0,01 mg for test pieces below 10 mg.

4.2.3 Temperature detectors, thermocouples in accordance to EN 60584-1 shall be used for the measurement of temperature up to 1 920 K.

For higher temperature, infrared detectors or any other suitable device may be used.

4.2.4 Data acquisition system, the sampling period during the test shall be less than 0,5 s.

4.3 Standard reference materials

Standard reference materials which can be used for calibration purposes are listed in annex B.

4.4 Test specimens

The test specimens shall be representative of the material.

NOTE This criterion is generally met by test specimens containing the maximum number of representative volume elements, compatible with the volume of the crucible, if this number is less than five, several solutions are possible:

a) the test specimens should have an exact number of representative volume elements;

b) the material should be ground to powder and a specimen taken from this powder. However this solution will lead to results which may differ from results obtained on solid test pieces and should be used only if no other solution is possible;

c) the material should be cut into specimens and a number of similar test specimens should be tested and an average value determined.

4.5 Calibration of calorimeter

4.5.1 General

Calibration of calorimeters, may be done according to two different methods. The first consists in dissipating a known amount of thermal power using a calibrated resistor introduced in the second chamber of the calorimeter. In

the second method a reference specimen with known specific heat capacity is dropped according to the procedure described in section 4.6.

4.5.2 Electrical Calibration

The calibration factor is the ratio of a known amount of thermal power dissipated in the resistor to the steady state calorimetric output signal and is measured at temperature T_2.

NOTE 1 The method using power dissipation in a resistor is limited to 1 350 K.

NOTE 2 This method can only be used if the sensitivity of the calorimeter is not affected by the filling of the measuring chamber.

4.5.3 Calibration using standard reference material

This calibration is called “drop calibration”. A specimen made from a standard reference material with a known specific heat capacity is dropped according to the test procedures described in section 4.6. (See annex B for standard reference material). This allows determination of the calibration factor (see annex A).

4.6 Test procedures

NOTE The avoidance of interaction between the test specimen and the calorimetric conditioning and measuring chambers can require the use of a sealed crucible.

4.6.1 Test without a crucible

4.6.1.1 Test with drop calibration

The test without a crucible and with drop calibration is done in the following order:

R, T, R, T, R, T, R

with

R = test of standard reference material, and;

T = test of test specimen.

Carry out each test as described in 4.6.3.

4.6.1.2 Test with electrical calibration

The test without a crucible and with calibration using power dissipation in a resistor is done in the following order:

— calibration of calorimeter;

— test on three test specimens.

Carry out each test as described in 4.6.3.

4.6.2 Test with a crucible

4.6.2.1 General

The mass of all empty crucibles used for the test shall not differ by more than 5 %.

4.6.2.2 Test with drop calibration

The test with a crucible and with drop calibration is carried out in the following order:

C, C + R, C + T, C, C + R, C + T, C, C + R, C + T, C

with

C is the test with the empty crucible;

C + R = test of crucible plus standard reference material;

C + T = test of crucible plus test specimen.

Carry out each test as described in 4.6.3.

4.6.2.3 Test with electrical calibration

The test with a crucible and with calibration using power dissipation in a resistor is done in the following order:

— calibration of calorimeter;

— carry out the following sequence:

C, C + T, C, C + T, C, C + T, C

with

C is the test with the empty crucible;

C + T = test with crucible plus test specimen.

Carry out each test as described in 4.6.3.

4.6.3 Description of test

The test piece (test specimen, standard material or empty crucible) and reference material shall be dried at (110 ± 5) °C until the difference in weight of two successive weighings is lower than 0,2 mg:

— measure the mass when a crucible is not used with an accuracy of ± 0,1 mg or ± 0,1 % whichever is the smaller;

— when a crucible is used, measure the mass of each assembly dropped, (empty crucible, crucible and standard reference material, crucible and test specimen);

— place the test piece (test specimen, standard material or empty crucible) in the conditioning chamber at temperature T_1 and wait for a sufficient period (in the order of 15 min), to reach thermal equilibrium of the test piece with its environment. Measure T_1 and T_2 start recording the calorimetric signal before the test piece is dropped. Drop the test piece. Stop the record when the steady state output signal is reached.

4.7 Calculations

4.7.1 General

The change in heat Q corresponding to the drop of the test piece is related to the area A under the calorimetric output signal by the following equation.

$$Q = K \cdot A$$

where

K is the calorimeter calibration factor.

4.7.2 Determination of the calorimetric calibration factor

4.7.2.1 Electrical calibration (see annex A)

$$K = \frac{\text{heat dissipated}}{\text{area under the calorimetric output signal}} = \frac{H}{A}$$

4.7.2.2 With standard reference material

See annex B.

4.7.3 Determination of mean specific heat capacity $\overline{C_p}$

The mean specific heat capacity is determined using the following formula:

$$\overline{C_p}\,(T_1, T_2) = \frac{1}{m_i}\,\frac{Q_i\,(T_1, T_2)}{(T_2 - T_1)}$$

where

T_1 is the initial temperature at which test pieces, are conditioned;

T_2 is the calorimeter temperature;

$Q_i\,(T_1, T_2)$ is the heat variation between T_1 and T_2;

m_i is the mass of the test piece, determined by weighing;

$\overline{C_p}\,(T_1, T_2)$ mean specific heat capacity between T_1 and T_2.

The subscript i has a different meaning depending on the type of test piece:

- i = c for an empty crucible;
- i = t for a test piece;
- i = t + c for a test piece and crucible.

without crucible

$$\overline{C_{pt}} = \frac{K \bullet A_t}{m_t\left(T_2 - T_1\right)}$$

with crucible

$$\overline{C_{pt}} = \frac{K\,(A_{c+t} - A_c)}{m_t\,(T_2 - T_1)}$$

with

A_t is the value of integration of calorimetric output signal of test specimen;

A_c is the value of integration of calorimetric output signal of crucible;

A_{c+t} is the value of integration of calorimetric output signal of test specimen plus crucible.

5 Method B - Differential scanning calorimetry

5.1 Principle

5.1.1 General

The method consists in measuring the difference in power needed to raise the temperature of the test specimen in its crucible and of an empty identical crucible using the same heating programme which may be stepwise heating or continuous heating.

Stepwise heating allows only the determination of the mean specific heat capacity $\overline{C_p}(T_1, T_2)$ over a temperature range (T_1, T_2) while continuous heating allows to determine the specific heat capacity C_p at a given temperature.

5.1.2 Stepwise heating method

The mean specific heat capacity $\overline{C_p}(T_1, T_2)$ is measured in a temperature interval defined by two isothermal levels T_1 and T_2. The heat Q_E which is necessary to change the temperature from T_1 to T_2 is determined by integrating the thermal power P_E with respect to time. The corresponding heat Q_E is:

$$Q_E = \int_o^t P_E \, dt = (m_t \, \overline{C_p}(T_1, T_2) + C_c + C_o)(T_2 - T_1)$$

where

m_t is the mass of the test specimen;

$\overline{C_p}(T_1, T_2)$ is the mean specific heat capacity of the test specimen;

C_o is the heat capacity of the calorimeter;

C_c is the heat capacity of the crucible.

Another experiment for the determination of the base line is performed using an identical imposed heating sequence with the empty crucible. The corresponding heat Q_B is given by:

$$Q_B = \int_o^t P_B \, dt = [C_c + C_o](T_2 - T_1)$$

From the above equations the mean specific heat capacity can be calculated as:

$$\overline{C_p}(T_1 - T_2) = \frac{Q_E - Q_B}{m_t (T_2 - T_1)}$$

5.1.3 Continuous heating method

Temperature is increased linearly versus time at a constant heating rate ß. Using the same notation as in 5.1.2 the thermal power P_E supplied at every moment to the system is:

$$K \cdot S_{c+t} = (m_t \, C_p + C_c + C_o)\beta$$

Another experiment for the determination of the base line is performed with the empty crucible. The corresponding thermal power is given by

$$K \cdot S_c = (C_c + C_o)\beta$$