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Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of specific heat capacity

Céramiques techniques — Propriétés thermophysiques des composites céramiques — Détermination de la capacité thermique specifique

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of specific heat capacity

1 Scope

This document 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 can 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

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 19634, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Ceramic composites — Notations and symbols*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 19634 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

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 increase ($T_2 - T_1$) expressed in K

3.3
representative volume element
RVE
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 that 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 with IEC 60584-1 shall be used for the measurement of temperature up to 1 920 K.

For higher temperatures, 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.

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 cut into specimens; 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 of 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 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 of a standard reference material with a known specific heat capacity is dropped according to the test procedures described in 4.6. (See Annex B for standard reference material.) The calibration factor is determined according to Annex A.

4.6 Test procedures

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

where

R is the test of standard reference material;

T is the 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.

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

4.6.2 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

where

C is the test with the empty crucible;

C + R is the test of crucible plus standard reference material;

C + T is the 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

where

C is the test with the empty crucible;

C + T is the 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 (around 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 recording 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 $\overline{C_\text{p}}$

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

$$\overline{C_\text{p}}(T_1, T_2) = \frac{1}{m_\text{i}} \frac{Q_\text{i}(T_1, T_2)}{(T_2 - T_1)}$$

where

T_1	is the initial temperature at which test pieces are conditioned;
T_2	is the calorimeter temperature;
$Q_\text{i}(T_1, T_2)$	is the heat variation between T_1 and T_2;
m_i	is the mass of the test piece, determined by weighing;
$\overline{C_\text{p}}(T_1, T_2)$	is the 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

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

with crucible

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

where

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), whereas continuous heating allows determination of 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 baseline is performed using an identical imposed heating sequence with the empty crucible. The corresponding heat, Q_B, is given by:

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

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 to the system at every moment is:

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

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

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

The specific heat capacity can be calculated from:

$$C_p = \frac{K(S_{c+t} - S_c)}{m_t \beta}$$

where

K is the calibration factor;

S_c, S_{c+t} are the output signals;

$K \cdot S_c$ and $K \cdot S_{c+t}$ are the thermal powers supplied to the system.

5.2 Apparatus

5.2.1 Differential scanning calorimeter.

5.2.1.1 There are two types of differential scanning calorimeters operating on power compensation and heat flux principles, both designed to operate under adiabatic conditions.

Both comprise two measuring cells housed in a furnace which provides overall system heating. One cell contains the test specimen and its crucible, the other contains an empty crucible only.

5.2.1.2 Power compensation type: each cell has an additional heater to compensate for the temperature variations from the overall heating programme. The power which is supplied to either cell heater to maintain equal temperatures during heating is measured.

5.2.1.3 Heat flux type: power is exchanged between each cell and its respective surrounding during the heating programme. The difference in power exchange between the two cells is measured.

5.2.2 Balance, with an accuracy better than 0,1 mg.

5.2.3 Temperature detectors, thermocouples in accordance with IEC 60584-1 shall be used for the measurement of temperature.

5.2.4 Data acquisition system, the time duration between two successive measurements shall be less than 0,5 s.

5.3 Standard reference materials, SRM

Standard reference materials shall be used for calibration. An example is given in Annex B.

5.4 Test specimens

The test specimens shall be representative of the material.

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 cut into pieces, and a number of similar test pieces should be tested and an average value determined.

5.5 Temperature calibration

A temperature calibration curve for the furnace using the same heating rate as for the determination of the specific heat capacity is established by using the melting points of standard reference materials (see, for example, Annex C).

Thermocouples shall be calibrated in accordance with IEC 60584-1.

5.6 Test procedure for the determination of C_p

5.6.1 General

Depending on the necessity or not of using a calibration factor K for the calorimeter, two methods can be used:

