SLOVENSKI STANDARD
SIST ENV 1159-3:2000

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

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

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

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

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English version

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

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CEN

European Committee for Standardization
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Foreword

This European Prestandard has been prepared by CEN/TC184 'Advanced technical ceramics', of which the Secretariat is held by BSI.

ENV 1159 consists of three Parts:

Part 1 : Determination of thermal expansion
Part 2 : Determination of thermal diffusivity
Part 3 : Determination of specific heat capacity

CEN/TC184 approved this European prestandard by resolution 1/12/1992 during its seventh meeting held in Berlin, 1992-12-08/09.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European prestandard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

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

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 2250 K, while method B is limited to 1900 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 Prestandard 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 Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to, applies.

EN 45001 :   General criteria for the operation of testing laboratories
HD 446.1 SI : 1992 : Thermocouples : Part 1 : Reference tables

3 Definitions

For the purposes of this Prestandard the following definitions apply:

3.1 Specific heat capacity
Amount of heat required to raise the temperature of a mass unit of material by 1 K at constant temperature and pressure.

3.2 Mean specific heat capacity
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 HD 446.1 shall be used for the measurement of temperature up to 1920 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 pieces

The test pieces shall be representative of the material.

> Note. This criterion is generally met by test pieces 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 pieces 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 pieces and a number of similar test pieces should be tested and an average value determined.

4.5 Calibration of calorimeter

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 piece with known specific heat capacity is dropped according to the procedure described in section 4.6.

4.5.1 Calibration using a resistor

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.

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Note 1. The method using power dissipation in a resistor is limited to temperature up to 1350 K.
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Note 2. This method can only be used if the sensitivity of the calorimeter is not is not affected by the filling.

4.5.2 Calibration using standard reference material

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

4.6 Test procedures

The avoidance of interaction between the specimen and the calorimetric chamber may require the use of a sealed crucible.

4.6.1 Test without a sealed 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 reference piece, and
     T = test of test piece

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 pieces.

Carry out each test as described in 4.6.3.

4.6.2 Test with crucible

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

4.6.2.1 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 = test of crucible
    C+R = test of crucible plus reference piece
    C+T = test of crucible plus test piece.

Carry out each test as described in 4.6.3.

4.6.2.2 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 = test of crucible
    C+T = test of crucible plus test piece.

Carry out each test as described in 4.6.3

4.6.3 Description of test

The test piece and reference piece shall be dried at (110 $\pm$ 5) °C until constant mass;

- measure the mass when a crucible is not used with an accuracy of $\pm$ 0,1 mg or $\pm$ 0,1 % whichever is the smaller
- when a crucible is used, measure the mass of each assembly dropped, (empty crucible, crucible and reference piece, crucible and test piece)

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

4.7 Calculations

The heat variation Q_i corresponding to the drop of the test piece (or reference piece or crucible) is related to S_i, calorimetric output signal. (See annex A).

$$Q_i = K S_i$$

where K is the calorimeter calibration factor.

4.7.1 Determination of the calorimetric calibration factor

4.7.1.1 Calibration using power dissipation in a resistor

$$K = \frac{Power\ dissipated}{Calorimetric\ output\ signal}$$

See paragraph 4.5.1.

4.7.1.2 With standard reference material

See annex B.

4.7.2 Determination of mean specific heat capacity Cp

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

$$\overline{Cp_{i_{(T_1,T_2)}}} = \frac{1}{m_i} \cdot \frac{Q_i(T_1 \rightarrow T_2)}{(T_2 - T_1)}$$

where T_1 is the initial temperature at which test pieces, reference pieces and crucibles are conditioned

T_2 is the calorimeter temperature

Q_i is the heat variation

m_i is mass (test piece, reference piece or crucible), and

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

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

$\quad$ i = c for a crucible
$\quad$ i = t for a test piece
$\quad$ i = t + c for a test piece and crucible

. **-Without crucible**

$$\overline{Cp}_t = \frac{K.A_t}{m_t\,(T_2-T_1)}$$

-With crucible

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

With :
$\quad A_t \quad$ = value of integration of calorimetric output signal of test piece
$\quad A_c \quad$ = value of integration of calorimetric output signal of crucible
$\quad A_{c+t} \quad$ = value of integration of calorimetric output signal of test piece plus crucible

5 Method B - Differential Scanning Calorimetry

5.1 Principle

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

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

5.1.1 Step method

The mean specific heat capacity $\overline{Cp}\,(T_1,T_2)$ is measured in a temperature interval defined by two isothermal levels T_1 and T_2. The heat quantity 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 quantity Q_E is :

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