

**Advanced technical
ceramics —
Ceramic composites,
thermophysical properties —**

**Part 3. Determination of specific heat
capacity**

Committees responsible for this Draft for Development

The preparation of this Draft for Development was entrusted to Technical Committee RPI/13, Advanced technical ceramics, upon which the following bodies were represented:

- British Ceramic Research Ltd.
- British Industrial Ceramic Manufacturers' Association
- British Steel Industry
- Bureau of Analysed Samples Limited
- China Clay Association
- Ministry of Defence
- Refractories Association of Great Britain
- Society of Glass Technology

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National foreword

This Draft for Development has been prepared under the direction of the Sector Board for Materials and Chemicals and is the English language version of ENV 1159-3 : 1995 *Advanced technical ceramics — Ceramic composites, thermophysical properties — Part 3 Determination of specific heat capacity* published as a European Prestandard by the European Committee for Standardization (CEN).

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature because it is considered that further experience is required in its application before it is converted into a British Standard. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained for reporting to the CEN committee responsible for converting the prestandard into a European Standard.

The UK did not support European adoption of this prestandard.

A review of the prestandard will be carried out 2 years after its publication.

Submission of comments from users of this publication is requested.

According to the replies received, the responsible BSI committee will judge whether to support the conversion into a European Standard or what other action should be taken.

Comments should be sent in writing to the Secretary of RPI/13 BSI, 389 Chiswick High Road, London, W4 4AL quoting the document reference (DD ENV 1159-3), the relevant clause and proposing, where possible, an appropriate revision of the text.

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English version

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

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Détermination de la capacité thermique
spécifique

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

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European Committee for Standardization
Comité Européen de Normalisation
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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.

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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 .

Note 1. The method using power dissipation in a resistor is limited to temperature up to 1350 K.

Note 2. This method can only be used if the sensitivity of the calorimeter 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.