
**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 spécifique*

Sample Document

get full document from standards.iteh.ai



Sample Document

get full document from standards.iteh.ai



COPYRIGHT PROTECTED DOCUMENT

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Method A – drop calorimetry	2
4.1 Principle	2
4.2 Apparatus	2
4.3 Standard reference materials	2
4.4 Test specimens	2
4.5 Calibration of calorimeter	3
4.5.1 General	3
4.5.2 Electrical calibration	3
4.5.3 Calibration using standard reference material	3
4.6 Test procedures	3
4.6.1 Test without a crucible	3
4.6.2 Test with a crucible	4
4.6.3 Description of test	4
4.7 Calculations	5
4.7.1 General	5
4.7.2 Determination of the calorimetric calibration factor	5
4.7.3 Determination of mean specific heat capacity $\overline{C_p}$	5
5 Method B – differential scanning calorimetry	6
5.1 Principle	6
5.1.1 General	6
5.1.2 Stepwise heating method	6
5.1.3 Continuous heating method	7
5.2 Apparatus	8
5.3 Standard reference materials, SRM	8
5.4 Test specimens	8
5.5 Temperature calibration	8
5.6 Test procedure for the determination of C_p	8
5.6.1 General	8
5.6.2 Method 1: Measurements requiring the knowledge of the K factor	9
5.6.3 Method 2: measurements requiring the use of a reference standard material (SRM)	11
5.7 Calculation of results	14
5.7.1 Method requiring the knowledge of the K factor	14
5.7.2 Method using an SRM	16
6 Test report	17
Annex A (normative) Drop calorimetry – determination of the calibration factor using standard reference material	18
Annex B (informative) Standard reference material	20
Annex C (informative) Materials for calorimeter calibrations	25
Bibliography	26

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

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

get full document from standards.iteh.ai

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 $(110 \pm 5) ^\circ\text{C}$ 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 $\pm 0,1$ 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 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 $\overline{C_p}$

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;

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