International Standard

ISO 19628

Second edition
2024-11

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 massique

Reference number
ISO 19628:2024(en)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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

For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 184, *Advanced technical ceramics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 19268:2017), which has been technically revised.

The main changes are as follows:

— revised scope to extend the maximum temperature of use of Method A to 3 000 K;

— revised Clause 4 by introducing the possibility to apply the drop calorimetry method for temperatures T1 > T2 (conventional drop calorimetry);

— relevant specifications added concerning the containers and thermometers to be used;

— description of in-situ calibration methods of the calorimeter and thermometers;

— addition of a paragraph dealing with the determination of specific heat capacity at given temperatures from measurements performed by drop calorimetry;

— updated list of references in the Bibliography.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies 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.

The two methods are applicable from ambient temperature up to a maximum temperature that is method dependent: method A can be used up to 3 000 K, while method B is limited to 1 900 K.

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: EMF specifications and tolerances*

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 terminology databases for use in standardization at the following addresses:

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

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

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

$$C_p = \frac{1}{m}\frac{dQ}{dT} \quad (1)$$

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

3.2
mean specific heat capacity
$\overline{C_p}(T_1, T_2)$
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

In "conventional" drop calorimetry, the test piece is heated in a furnace at a constant temperature T_2 then dropped in a calorimeter at a constant temperature T_1. The quantity measured is the amount of heat Q released in cooling the test piece to the calorimeter temperature T_1.

In "inverse" drop calorimetry, the test piece is maintained in a conditioning chamber at a constant temperature T_1 near to room temperature and then dropped in a calorimeter heated at a constant temperature T_2. The quantity measured is the amount of heat Q absorbed in heating the test piece to the calorimeter temperature T_2.

Whatever the method, "conventional" drop calorimetry or "inverse" drop calorimetry, $T_2 > T_1$.

In both methods, the tested material must not undergo a phase transition in the temperature increment $(T_2 - T_1)$.

Transfer of the test piece shall be done under conditions as close as possible to adiabatic conditions.

Specific heat capacity and mean specific heat capacity are determined from the amount of heat absorbed or released by the test piece in the calorimeter depending on the drop calorimetry mode applied.

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. It is recommended that these control systems enable a temperature stability better than 1 K below 1 300 K, better than 2 K from 1 300 K to 2 300 K and better than 4 K above 2 300 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 temperatures higher than 1 273 K, radiation thermometers (also named pyrometers) can be used.

Thermocouples and radiation thermometers shall be periodically calibrated in their operating temperature ranges as they can be subjected to drift over time.

Thermocouples may be calibrated by measurement either at a series of fixed-point temperatures (e.g. melting/freezing points) or by comparison with reference thermometers in thermally stabilised baths or furnaces.

NOTE 1 Guidelines on the Calibration of Thermocouples[2] are available at the following address: https://www.euramet.org/publications-media-centre/calibration-guidelines/.

Pyrometers are usually calibrated in radiance temperature using reference blackbodies. In addition to these calibrations performed outside the drop calorimeter facility, it is recommended to perform in-situ calibrations of the pyrometers by using fixed-point temperatures[3].

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 Containers

When containers are used, care shall be taken to choose suitable containers to avoid any chemical reaction or contamination of the specimen from the container material, in particular at high temperature.

4.5 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.

4.6 Calibration of the calorimeter

4.6.1 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 4.7.

4.6.2 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. It is recommended to let the calibrated resistor in the calorimeter during the electrical calibration and the tests, so that the experimental conditions during both steps remain strictly unchanged[4,5].

NOTE 1 For the "inverse" drop calorimetry, the method using power dissipation in a resistor is limited to 1 350 K to avoid damaging the resistor at high temperature.

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

4.6.3 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 4.7. (See Annex B for standard reference material). The calibration factor is determined according to Annex A.

4.7 Test procedures

4.7.1 General

The test procedures described in sub-clauses 4.7.2 to 4.7.4 shall be applied depending on the experimental configuration (test performed with or without a container) and the calibration method (electrical calibration or calibration with standard reference material).

In the case of determination of the mean specific heat capacity $\overline{C_p}(T_1, T_2)$ (cf. sub-clause 4.8.3), the tests are performed for one couple of temperatures T_1 and T_2.

For performing specific heat capacity C_p measurement (cf. sub-clause 4.8.4), the tests shall be carried out for different couples of temperatures T_1 and T_2 covering the temperature range of investigation.

— In “conventional” drop calorimetry, the tests are repeated by varying the temperature of the furnace T_2, the temperature of the calorimeter T_1 being kept constant.

— In “inverse” drop calorimetry, the tests are repeated by varying the temperature of the calorimeter T_2, the temperature of the conditioning chamber T_1 being kept constant.

4.7.2 Test without a container

4.7.2.1 Test with drop calibration

The test without a container 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 4.7.4.

4.7.2.2 Test with electrical calibration

The test without a container 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 4.7.4.

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

4.7.3 Test with a container

4.7.3.1 General

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

4.7.3.2 Test with drop calibration

The test with a container 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 container;

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

C + T is the test of container plus test specimen.

Carry out each test as described in 4.7.4.

4.7.3.3 Test with electrical calibration

The test with a container 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 container;

C + T is the test with container plus test specimen.

Carry out each test as described in 4.7.4.

4.7.4 Description of test

The test piece (test specimen, standard material or empty container) 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 container is not used with an accuracy of ±0,1 mg or ±0,1 %, whichever is the smaller;

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

— place the test piece (test specimen, standard material or empty container) in the conditioning chamber at temperature T_1 and wait for a sufficient period 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.

4.8 Calculations

4.8.1 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 Formula (2).

$$Q = K \cdot A \tag{2}$$

where K is the calorimeter calibration factor.

4.8.2 Determination of the calorimetric calibration factor

4.8.2.1 Electrical calibration

The calibration factor K at a temperature T is determined by dividing the amount of heat Q dissipated by the calibrated resistor inside the calorimeter maintained at temperature T by the area A under the calorimetric output signal.

$$K = \frac{Q}{A} \tag{3}$$

where

Q is the amount of heat dissipated;

A is the area under the calorimetric output signal.

4.8.2.2 With standard reference material

See Annexes A and B.

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

The mean specific heat capacity is determined using Formula (4):

$$\overline{C_p}(T_1, T_2) = \frac{1}{m_i} \frac{Q_i(T_1, T_2)}{(T_2 - T_1)} \tag{4}$$

where

T_1 is the calorimeter temperature when using “conventional” drop calorimetry, or the initial temperature at which test pieces are conditioned in case of “inverse” drop calorimetry;

T_2 is the initial temperature at which test pieces are conditioned when using “conventional” drop calorimetry, or the calorimeter temperature in case of “inverse” drop calorimetry;

$Q_i(T_1,T_2)$ is the amount of heat to increase the temperature of the test specimen from T_1 to 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 at temperature $T = (T_1 + T_2)/2$.

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

— i = c for an empty container;

— i = t for a test piece;

— i = t + c for a test piece and container.

without container

$$\overline{C_{pt}} = \frac{K \cdot A_t}{m_t (T_2 - T_1)} \quad (5)$$

with container

$$\overline{C_{pt}} = \frac{K(A_{c+t} - A_c)}{m_t (T_2 - T_1)} \quad (6)$$

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 container;

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

4.8.4 Determination of the specific heat capacity C_p

The specific heat capacity is determined by applying the following procedure:

— Plot the amounts of heat $Q(T_1,T_2)$ obtained for different couples of temperatures T_1 and T_2 as a function of T_2;

In "conventional" drop calorimetry, the amounts of heat $Q(T_1,T_2)$ are equal to $K(T_1)\cdot(A_{c+t} - A_c)$ with a container or to $K(T_1)\cdot A_t$ without container;

In "inverse" drop calorimetry, the amounts of heat $Q(T_1,T_2)$ are equal to $K(T_2)\cdot(A_{c+t} - A_c)$ with a container or to $K(T_2)\cdot A_t$ without container.

— Determine the mathematical model giving the amounts of heat $Q(T_1,T_2)$ versus T_2, which best fits the obtained experimental data;

— Differentiating this mathematical model with respect to temperature and dividing the result by the mass of the test piece leads to the following relationship giving the specific heat capacity $C_p(T)$ as a function of temperature T.

$$C_p = \frac{1}{m}\frac{dQ}{dT} \quad (7)$$

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.