INTERNATIONAL STANDARD

ISO 14420

Second edition
2020-03

Carbonaceous products for the production of aluminium — Baked anodes and shaped carbon products — Determination of the coefficient of linear thermal expansion

Produits carbonés utilisés pour la production de l'aluminium — Anodes cuites et produits carbonés formés — Détermination du coefficient de dilatation thermique

Reference number
ISO 14420:2020(E)

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 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 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 226, *Materials for the production of primary aluminium*.

This second edition cancels and replaces the first edition (ISO 14420:2005), of which it constitutes a minor revision.

The main changes to the previous edition are as follows:

— 5.4 has been revised to remove micrometer calliper;

— the formulae in Clause 9, previously Clause 10, have been revised;

— Clause 10, previously Clause 9, has been revised according to ISO/IEC Directives Part 2.

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

Introduction

This document is based on DIN 51909:2009.

Carbonaceous products for the production of aluminium — Baked anodes and shaped carbon products — Determination of the coefficient of linear thermal expansion

1 Scope

This document specifies a method to determine the coefficient of linear thermal expansion of carbonaceous or graphite materials (solid materials) for the production of aluminium between 20 °C and 300 °C. It applies to baked anodes and shaped carbon products.

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 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Design and metrological characteristics of callipers*

DIN 1333, *Presentation of numerical data*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
coefficient of linear thermal expansion
$\alpha(\vartheta)$
coefficient of thermal expansion correlated with the length change of a body with temperature

Note 1 to entry: The coefficient of linear thermal expansion, $\alpha(\vartheta)$, is calculated using Formula (1):

$$\alpha(\vartheta) = \frac{1}{l} \cdot \frac{\mathrm{d}l}{\mathrm{d}\vartheta} \tag{1}$$

where

l is the length of the test specimen at temperature ϑ;

d is the derivative;

$\frac{\mathrm{d}l}{\mathrm{d}\vartheta}$ is the length change with temperature.

3.2
average coefficient of linear thermal expansion
$\alpha(\vartheta_1;\vartheta_2)$
mean coefficient of thermal expansion correlated with the length change of a body with temperature

Note 1 to entry: The average coefficient of linear thermal expansion, $\alpha(\vartheta_1;\vartheta_2)$, is calculated using Formulae (2) to (4):

$$\alpha(\vartheta_1;\vartheta_2)=\frac{1}{l_1}\cdot\frac{l_2-l_1}{\vartheta_2-\vartheta_1}=\frac{1}{l_1}\cdot\frac{\Delta l}{\Delta\vartheta} \quad (2)$$

$$\Delta\vartheta=\vartheta_2-\vartheta_1 \quad (3)$$

$$\Delta l=l_2-l_1 \quad (4)$$

where

ϑ_1 is the lower limit of the temperature interval, in °C;

ϑ_2 is the upper limit of the temperature interval, in °C;

l_1 is the length of the test specimen, in mm, at temperature ϑ_1;

l_2 is the length of the test specimen, in mm, at temperature ϑ_2.

4 Principle

The average coefficient of linear thermal expansion is determined by means of a push-rod dilatometer. The test specimen is contained in a sample holder made from low-expansivity material (such as flint glass). It is heated in a furnace and the length change is transmitted to a mechanical, optical or electronic measuring system outside the furnace by a push-rod.

The average coefficient of linear thermal expansion is calculated from the measured length change, the original length and the temperature change of the test specimen, taking the expansion of the sample holder and the push-rod into account. Unless otherwise stated, the determination is performed between a lower limit for the temperature interval of 20 °C (i.e. room temperature) and an upper limit for the temperature interval of 300 °C.

5 Apparatus

5.1 Dilatometer, with sample holder and push-rod, for example, made from flint glass, as well as a mechanical, optical or electronic length-measurement device (error limits ± 0,5 µm), for temperatures above 300 °C in a vacuum or in a protective gas atmosphere.

5.2 Furnace, capable of holding the temperature constant to within ± 0,5 % over the length of the test specimen.

5.3 Temperature-measuring device, for example, a thermocouple with indicating instrument, accurate to within ± 0,5 %, to determine the average test-specimen temperature.

5.4 Instrument for measuring lengths, with error limit of ± 0,2 %, for example a vernier calliper according to ISO 13385-1.

5.5 Calibration samples, made from materials with known thermal expansion in the range of the material to be measured and made with similar geometry. The thermal expansion of calibration samples shall have been predetermined by the producer of the measuring equipment or by a recognized calibration authority.

6 Specimens

Prepare a test specimen of cylindrical or prismatic geometry. The cylinder diameter or the prism transverse edge length shall be at least twice the diameter of the largest structural constituent (for example maximum grain size) of the material to be examined, and in no case smaller than 4 mm (typically 30 mm to 50 mm). The length of the test specimens shall be at least 25 mm, but preferably should be 50 mm to 120 mm.

The test specimens shall be machined on all surfaces by turning or grinding, so that the surfaces in contact with the push-rod do not deviate from plane parallelism by more than 0,2 mm.

Remove existing stresses in a test specimen by annealing at 1 000 °C in a non-oxidizing atmosphere.

7 Procedure

7.1 Calibration

Calibrate the dilatometer according to 7.2 using calibrated samples.

7.2 Measurement

Measure the sample length l_1 of the test specimen at temperature ϑ_1.

Insert the test specimen into the dilatometer, taking care that the specimen ends are firmly in contact with the push-rod.

Measure the original length l_1 of the test specimen at the lower limit of the temperature interval ϑ_1. If the push-rod planes contacting the specimen end surfaces are not spherically or conically shaped, use connecting pieces to make a point of contact with the specimen end planes.

At the beginning of the measurement, set the measuring system to zero by either adjusting the zero point of the apparatus, or marking on the recording chart or the photosensitive paper. When using double dilatometers, with the two dilatometer motions recorded orthogonally, the assignment of the recording axes to the dilatometers shall also be determined and recorded.

Position the furnace (which may be preheated) around the sample holder. Allow the test specimen to attain the upper limit of the temperature interval ϑ_2. Then measure and record the length of the test specimen l_2.

If the upper limit of the temperature interval ϑ_2 is above 300 °C, avoid oxidation of the test specimen by applying a suitable protection gas or vacuum.

8 Evaluation

Calculate the average coefficient of linear thermal expansion, α, in units of 1/K, according to Formula 5):

$$\alpha(\vartheta_1;\vartheta_2)=\frac{1}{l_1}\cdot\frac{l_2-l_1}{\vartheta_2-\vartheta_1}-\alpha_k=\frac{1}{l_1}\cdot\frac{\Delta l}{\Delta\vartheta}-\alpha_k \quad (5)$$

where

ϑ_1 is the lower limit of the temperature interval, in °C;

ϑ_2 is the upper limit of the temperature interval, in °C;

l_1 is the length of the test specimen, in mm, at temperature ϑ_1;

l_2 is the length of the test specimen, in mm, at temperature ϑ_2;

α_k is the mean coefficient of linear thermal expansion, in 1/K, of the sample holder and the push-rod for the temperature range under consideration.

9 Precision

The precision of this method has been calculated according to ASTM E691, resulting in the following values.

Repeatability:

$r = 0{,}10 \times 10^{-6}\ K^{-1}$

Reproducibility:

$R = 0{,}17 \times 10^{-6}\ K^{-1}$

10 Test report

The test report shall include the following information:

a) an identification of the sample;

b) a reference to this document, i.e. ISO 14420:2020;

c) pre-treatment of the specimens, if relevant;

d) number of specimens;

e) temperature range of measurement;

f) the coefficient of linear thermal expansion, in units of $10^{-6}\ K^{-1}$, rounded to the nearest $0{,}01 \times 10^{-6}\ K^{-1}$, individual values;

g) any deviations from the procedure, including agreed conditions deviating from the procedure and any unusual features observed;

h) the date of the test.

Bibliography

[1] DIN 863-1, *Verification of geometrical parameters — Micrometers — Part 1: Standard design micrometer callipers for external measurement; concepts, requirements, testing*

[2] DIN 1319-3, *Fundamentals of metrology — Part 3: Evaluation of measurements of a single measurand, measurement uncertainty*

[3] DIN 51045-1, *Determination of the thermal expansion of solids — Part 1: Basic rules*

[4] DIN 51045-2, *Determination of the change of length of solids by thermal effect; testing of fired fine ceramic materials*

[5] DIN 51045-3, *Determination of the change of length of solids by thermal effect; testing of non-fired fine ceramic materials*

[6] DIN 51045-4, *Determination of the change of length of solids by thermal effect; testing of fired ordinary ceramic materials*

[7] DIN 51045-5, *Determination of the change of length of solids by thermal effect; testing of non-fired ordinary ceramic materials*

[8] DIN 51909:2009, *Testing of carbon materials — Determination of coefficient of linear thermal expansion — Solid materials*

[9] ASTM C372, *Standard method of test for linear thermal expansion of fired whiteware products by the dilatometer method (reapproved 1970)*

[10] ASTM E691, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*

[11] Kohlrausch F., Praktische Physik, Stuttgart 1968, Vol. 1, pp. 316 to 319, Chapter 4.3.2 "Wärmeausdehnung"

[12] Metz A., A new all-purpose dilatometer according to Bollenrath (in German), ATM 303 (1961) R 61 to R 72

[13] Thormann P., Investigations with a dilatometer in the field of ceramic raw materials and materials (in German), Ber. dt. Keram. Ges. 46 (1969), pp. 583 to 586

[14] Thormann P., Precision of dilatometric investigations and their significance in ceramic laboratories (in German), Ber. dt. Keram. Ges. 47 (1970), pp. 769 to 773

[15] Otto J., Thomas W., The thermal Expansion of Quartz Glass in the Temperature Range 0 to 1060°C (in German), Z. f. Phys. 175 (1963), pp. 334 to 337

ICS 71.100.10
Price based on 5 pages