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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

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Foreword

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ISO 14420 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

This International Standard is based on DIN 51909:1984 prepared by the committee NMP 281 “Prüfverfahren für Kohlenstoff und Graphit” in DIN Deutsches Institut für Normung e.V., Berlin.

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

1 Scope

This International Standard 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 referenced documents are indispensable for the application 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 6906, *Vernier callipers reading to 0,02 mm*

ISO 3611, *Micrometer callipers for external measurement*

DIN 1333, *Presentation of numerical data*

3 Terms and definitions

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

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

NOTE The linear thermal expansion coefficient is calculated from the following formula.

$$a(\vartheta) = \frac{1}{l} \cdot \frac{dl}{d\vartheta} \quad (1)$$

where

$\alpha(\vartheta)$ is the linear expansion coefficient;

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

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

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

NOTE The average linear expansion coefficient is calculated from the following formula

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

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

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

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 at temperature ϑ_2;

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

4 Principle

The average linear thermal expansion coefficient 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 linear thermal expansion coefficient 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 max.

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 vernier calliper according to ISO 6906 or micrometer calliper according to ISO 3611;

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

A hole of minimum 1 mm depth may be drilled in the middle of a long side of the test specimen to hold the joint of the thermocouple.

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.

Insert the joint of the thermocouple into the hole in the side of the test specimen where required.

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 realize a point contact to 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 linear expansion coefficient, in units of 1/K, according to the following equation:

$$\alpha(\vartheta_1;\vartheta_2) = \frac{1}{l_1} \cdot \frac{l_2 - l_1}{\vartheta_2 - \vartheta_1} - \alpha_\mathrm{k} = \frac{1}{l_1} \cdot \frac{\Delta l}{\Delta \vartheta} - \alpha_\mathrm{k} \qquad (5)$$

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

Rounding to the last significant decimal place shall be done in accordance with DIN 1333.

9 Test report

The test report shall include the following information:

a) type and marking of specimens;

b) a reference to this International Standard;

c) pretreatment of the specimens, if relevant;

d) number of specimens;

e) temperature range of measurement;

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

g) agreed conditions deviating from this International Standard;

h) test date.

10 Precision

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

The repeatability:

r = 0,1 µm/mK

The reproducibility:

R = 0,17 µm/mK

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