INTERNATIONAL STANDARD

ISO 15379-1

First edition
2004-06-15

Carbonaceous materials for the production of aluminium — Cathode block materials —

Part 1: Determination of the expansion due to sodium penetration with application of pressure

Produits carbonés utilisés pour la production de l'aluminium — Blocs cathodiques —

Partie 1: Détermination de l'expansion due à la pénétration du sodium avec application de pression

Reference number
ISO 15379-1:2004(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 15379-1 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

ISO 15379 consists of the following parts, under the general title *Carbonaceous materials for the production of aluminium — Cathode block materials*:

— *Part 1: Determination of the expansion due to sodium penetration with application of pressure*

— *Part 2: Determination of the expansion due to sodium penetration without application of pressure*

Introduction

Expansion due to sodium penetration is an important property of carbon cathode blocks. As soon as alumina electrolysis starts, sodium penetrates into the carbon cathode blocks causing swelling of these blocks. This increase in volume creates mechanical stresses within the blocks and/or bulging of the bottom block plate. This can lead to cracks through which liquid aluminium and/or liquid electrolyte can flow, reaching the thermal insulation beneath the blocks and destroying these ceramic materials. In such a case, the electrolysis cell has to be relined, resulting in loss of aluminium production and high expenses. Therefore, cathode blocks produced with materials allowing only low sodium penetration and having the lowest possible expansion due to sodium penetration are preferred.

The study can be supplemented by measuring electrical resistivity (see ISO 11713[1)]) before and after the test.

Due to thermal and sodium expansion when heating up the cathode blocks in the electrolysis cells to operational temperature, the block will be subject to pressure from the steel shell; therefore, in the present method 5 MPa is chosen as a realistic maximum pressure.

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1) ISO 11713, *Carbonaceous materials used in the production of aluminium — Cathode blocks and baked anodes — Determination of electrical resistivity at ambient temperature*

Carbonaceous materials for the production of aluminium — Cathode block materials —

Part 1: Determination of the expansion due to sodium penetration with application of pressure

1 Scope

This method covers the determination of linear expansion under external pressure due to sodium penetration in cathode-block materials used in the production of aluminium.

The linear expansion of the blocks depends on the sampling direction due to anisotropy.

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 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 8007-1, *Carbonaceous materials used in the production of aluminium — Sampling plans and sampling from individual units — Part 1: Cathode blocks*

ASTM E 220, *Standard Test Method for Calibration of Thermocouples By Comparison Techniques*

3 Principle

An anodic graphite crucible containing a cryolitic bath with an initial cryolite ratio of 4,0 and with a cathode carbon sample placed in the crucible, as shown in Figure 1, is used for this experiment. The whole sample is immersed in a cryolitic bath with a graphite cylinder fitted as an extension from the sample. The crucible is placed in a crucible support, which is connected to a hydraulic power cylinder. A constant pressure of 5 MPa is applied to the sample by pressing the graphite extension against a stop rod in the top of the furnace by the hydraulic power cylinder. The whole assembly is heated in a tubular furnace to (980 ± 5) °C and is then electrolyzed for 2 h with a current density of 0,7 A/cm^2. The expansion is measured by a probe, which is fastened to the frame of the furnace and measures the position of the crucible support.

4 Apparatus

The principle of the apparatus for measuring sodium expansion is shown in Figure 1.

4.1 Furnace, capable of maintaining a temperature of 980 °C, with a temperature gradient over the melt of less than 10 °C.

4.2 Furnace control device, suitable for holding the temperature at (980 ± 5) °C.

4.3 Temperature-measuring device, i.e. a thermocouple, preferably of type K or S, capable of determining the temperature to ± 5 °C at 980 °C.

The thermocouple should be calibrated in accordance with ASTM E 220.

4.4 Crucible, made of graphite, with an inner diameter of 90 mm and an inner height of 90 mm.

The crucible acts as an anode.

4.5 Lid, made of graphite, which shall have a hole in the centre to allow the sample/graphite extension to reach above the lid.

4.6 Insulating ring, made of a ceramic material which resists the temperature and the fluoride environment.

The ring shall be placed in the hole of the graphite lid and act as an electrical insulation between the lid and the sample/graphite extension. The ring shall be large enough to allow the sample/graphite extension to move freely in the vertical direction.

4.7 Crucible support, made of heat-resistant steel to conduct anodic current from the power supply to the crucible.

4.8 Stop rod, made of heat-resistant steel to conduct cathodic current to the top of the sample.

The stop rod acts as a fixed reference point for the expansion measurements.

The material for the steel support and the steel stop rod should have minimal deformation at a pressure of 5 MPa.

NOTE The steel quality Sanicro 31HT[2)], X 10 NiCrAlTi32.20 has been shown suitable for the crucible support rod and the stop rod. If other steel qualities are used it should be verified that they do not deform in use.

4.9 Alumina disk, capable of covering the bottom of the crucible and capable of acting as electrical insulation between the crucible and the sample. The disk should preferably have a centring slot to help position the sample in the centre of the crucible.

4.10 Graphite cylinder, which shall be used as an extension to the sample and shall have a diameter of (30,0 ± 0,1) mm and a length of (40 ± 1) mm.

4.11 Hydraulic power cylinder, capable of applying a constant pressure of 5 MPa ± 0,1 MPa during the whole experiment independent of the expansion of the sample.

4.12 Extensometer, connected to a computer or data recorder, with a measuring range of 10 mm and an accuracy of 1 µm over the whole range, in order to observe the expansion due to sodium penetration.

4.13 Power supply, capable of supplying 39,6 A dc, with a current density of the cathode that shall be 0,7 A/cm^2.

5 Reagents

5.1 Argon, welding-grade quality.

5.2 Cryolite, Na_3AlF_6, natural, 99,7 % by mass or synthetic, > 97 % by mass.

2) Sanicro 31HT is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 15379 and does not constitute an endorsement by ISO of this product.

5.3 Sodium fluoride, NaF, purum, > 99 % by mass.

5.4 Calcium fluoride, CaF_2, precipitated pure, > 97 % by mass.

5.5 Alumina, Al_2O_3, extra pure, > 98 %by mass.

5.6 Bath composition, which shall have a cryolite ratio of 4,0 and consist of the following: 71,5 % Na_3AlF_6, 14,5 % NaF, 5,0 % CaF_2, 9,0 % Al_2O_3. The bath is crushed to < 2 mm using a jaw crusher and shall have a mass of 765 g.

6 Samples

Sample the material in accordance with ISO 8007-1. The diameter of the sample shall be (30,0 ± 0,1) mm. The length shall be (60 ± 1) mm.

7 Procedure

Assemble the crucible support (4.7) with the crucible (4.4) and the sample so they can be pressed together with a hydraulic power cylinder against a heat-resistant steel stop rod (4.8) conducting the cathodic current to the top of the sample (see Figure 1). The stop rod (4.8) acts as a fixed reference point for the expansion measurements. Place the alumina disk (4.9) in the bottom of the crucible (4.4). Measure the length, l_0, of the sample at room temperature with an accuracy of 0,1 mm. Place the sample on the alumina disk in the centre of the crucible. Place the graphite cylinder (4.10) on top of the sample. Fill the crucible with the bath prepared in accordance with 5.6. Place the lid (4.5), with an insulating ring (4.6) around the graphite extension, on top of the crucible.

Lift the crucible by the hydraulic power cylinder (4.11) until the sample touches the stop rod (4.8) inside the furnace (4.1). Adjust the hydraulic pressure to 5 MPa.

Place the thermocouple close to the crucible near the centre of the melt height.

Place the extensometer (4.12) underneath the base of the crucible holder outside the furnace.

Heat the furnace to (980 ± 5) °C with an argon (5.1) flush, and measure the change in the length of the sample and the apparatus. Wait until no further thermally induced movement can be detected in the sample and apparatus. Determine the position of the crucible support and take this reading as the zero reference for subsequent measurements of the change in length, $\Delta l_{\text{meas}}(t)$.

Connect the power supply (4.13) to the crucible support and the stop rod. Electrolyze the system for 2 h with a constant current of 39,6 A. Record the change in length, $\Delta l_{\text{meas}}(t)$, every minute. Then turn off the electrolysis.

Release the pressure, but keep the crucible inside the furnace. Allow the furnace to cool down to room temperature.

8 Results

8.1 Calculation

Calculate the relative expansion of the sample for each recording using Equation (1):

$$\Delta L(t) = \frac{\Delta l_{\text{meas}}(t)}{l_0} \times 100 \qquad (1)$$

where

$\Delta L(t)$ is the relative expansion, in percent, at time, t;

$\Delta l_{\text{meas}}(t)$ is the measured change in length, in millimetres, at time, t;

l_0 is the initial sample length, in millimetres.

Plot the relative expansion, $\Delta L(t)$, versus time. Determine the maximum relative expansion, ΔL_{max}. Round the results to the second decimal place.

8.2 Precision

The precision is calculated in accordance with ISO 5725-2.

The repeatability is calculated from Equation (2):

$$r = 0{,}20 \times \Delta\bar{L}_{\text{max}} + 0{,}02 \quad (2)$$

where

$\Delta\bar{L}_{\text{max}}$ is the maximum average expansion;

0,02 is in absolute %.

EXAMPLE Material A has a maximum average expansion equal to 0,56 %. The repeatability is then $0{,}20 \times 0{,}56 + 0{,}03 = 0{,}15$, in % absolute.

The reproducibility is calculated from Equation (3):

$$R = 0{,}24 \times \Delta\bar{L}_{\text{max}} + 0{,}03 \quad (3)$$

where the parameters are the same as for Equation (2).

The degrees of freedom, i.e. number of laboratories (e.g. 4) times the number of samples (e.g. 3), is equal to 12.

9 Test report

The report shall include the following information:

a) reference to this part of this International Standard (e.g. ISO 15379-1:2004);

b) all details necessary for identification of the sample;

c) direction of the sample relative to the extrusion or vibration direction;

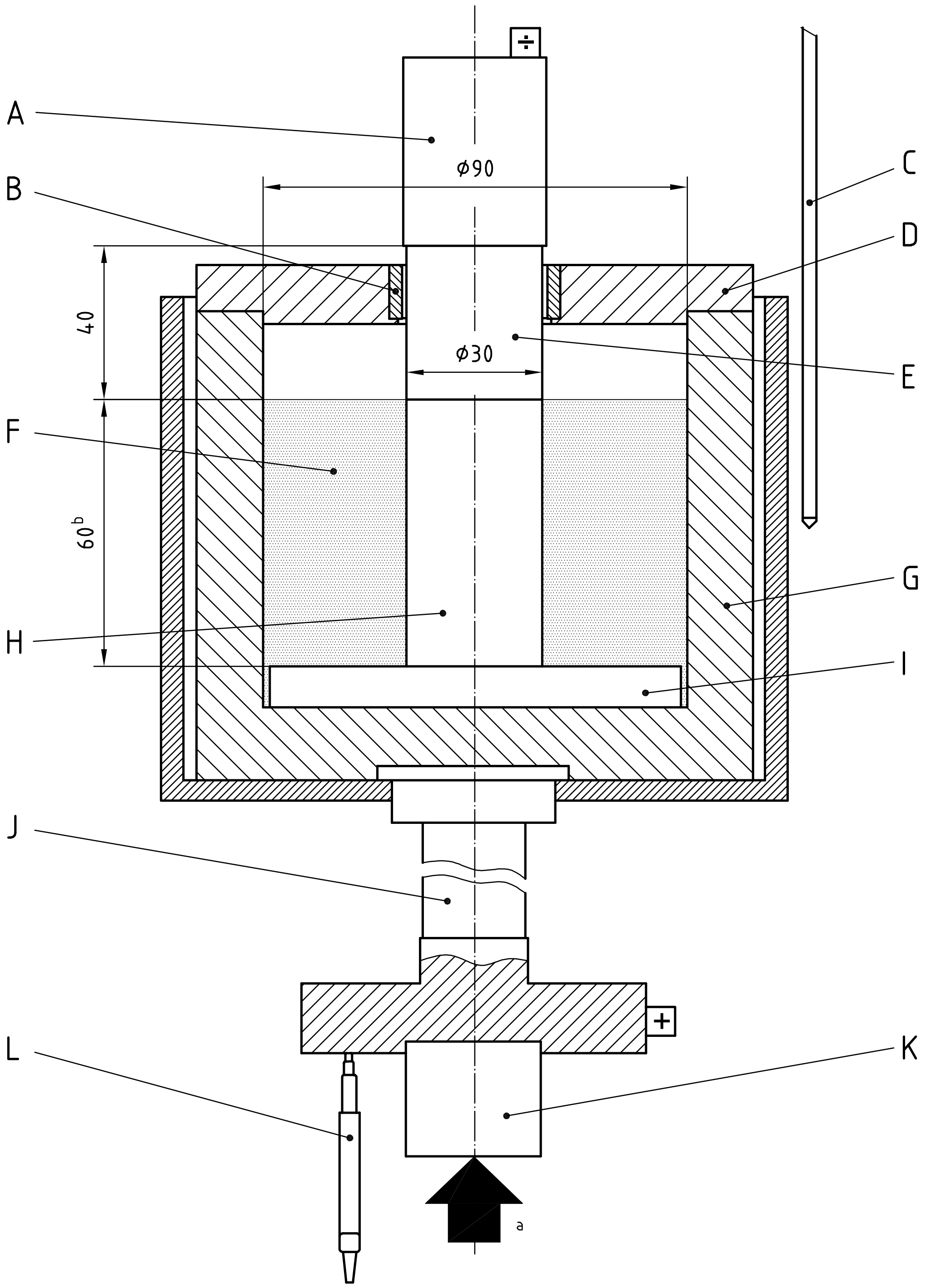
d) results of the test, including the maximum expansion, ΔL_{max}, in percent, due to sodium penetration and a plot of the relative expansion, $\Delta L(t)$, due to sodium penetration versus time, t;

e) date of the test;

f) details of any unusual features noted during the determination;

g) details of any operation not included in this part of this International Standard or regarded as optional.

Dimensions in millimetres



Key

A heat-resistant steel stop rod

B insulating ring

C thermocouple (Type K or S)

D graphite lid

E graphite cylinder (sample extension)

F cryolite melt

G graphite crucible

H sample

I alumina disk

J heat resistant steel crucible support

K hydraulic power cylinder

L extensometer

[a] Pressure.

[b] Initial sample length (= l_0).

Figure 1 — Apparatus for measuring expansion due to sodium penetration

ICS 71.100.10

Price based on 5 pages