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

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

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

Partie 2: Détermination de l'expansion due à la pénétration du sodium sans application de pression

Reference number
ISO 15379-2:2004(E)

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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.

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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 15379-2 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 must 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.

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

1 Scope

This method covers the determination of linear expansion 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 for 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 suspended in the bath, as shown in Figure 1, is used for this experiment. The whole assembly is heated 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 inserted into a hole that is drilled into the sample.

4 Apparatus

A 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 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.

The crucible acts as an anode.

4.5 Copper pipe, with an outer diameter of 14 mm and an inner diameter of 12 mm.

The pipe acts as a sample holder and as a fixed reference for the expansion measurements. It also conducts current from the power supply to the sample. The pipe shall be threaded at one end and shall be long enough to extend out of the furnace when the sample is placed on the bottom of the crucible.

4.6 Inconel[2] rod, with a diameter of 5 mm, which is used to measure the expansion of the sample.

The rod shall be placed inside the hole in the sample and shall reach out of the copper pipe.

4.7 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, to observe the expansion due to sodium penetration.

4.8 Crucible holder or supporter, made of heat-resistant steel to conduct current from the power supply to the crucible.

4.9 Graphite powder, may be used to improve the electrical connection between the crucible and the crucible holder.

4.10 Power supply, capable of supplying (54 ± 1) 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.

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 1 300 g.

6 Sampling

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 100 mm. Drill a hole (6 mm ∅) centrally in the sample to a depth 10 mm above the bottom. The centred hole shall be threaded to a depth of (25 ± 5) mm, i.e. the distance between the measuring points shall be (65 ± 5) mm. The threaded hole shall have a diameter fitting the copper pipe.

2) Inconel 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.

7 Procedure

Mount the sample on the copper pipe (4.5). Determine the difference in position, l_0, between the bottom end of the copper pipe and the bottom of the hole in the sample at room temperature. Prepare the bath in accordance with the conditions given in 5.6. Place the cryolitic bath components in the crucible (4.4). Place the crucible in the crucible holder (4.8). Graphite powder (4.9) can be used to improve the electrical connection between the crucible and the crucible holder. Place the crucible and the holder in the furnace (4.1). Place the sample, which is attached to the copper pipe, above the bath allowing the sample to preheat together with the crucible. Insert the Inconel rod (4.6) through the copper pipe down to the bottom of the hole in the sample.

Place the thermocouple (4.3) close to the crucible near the centre of the melt height.

Heat the furnace to (980 ± 5) °C with an argon (5.1) flush. After reaching a stable temperature, insert the sample until the bottom end of the sample is 30 mm above the bottom of the crucible.

Place the extensometer holder on top of the copper pipe. The extensometer (4.7) shall rest on top of the Inconel rod (4.6). Detect the change in position between the copper pipe and the Inconel rod. Leave the sample to be heated by the bath until no further thermally induced movement can be detected in the sample and apparatus. Measure the difference in position between the copper pipe and the Inconel rod and take this reading as the zero reference for subsequent measurements of the change in length, $\Delta l_{meas}(t)$.

Connect the power supply (4.10) to the crucible holder and the copper pipe. Electrolyze the system for 2 h with a constant current of (54 ± 1) A. Record the change in length, $\Delta l_{meas}(t)$, every minute. Then turn off the electrolysis.

Remove the sample from the bath. 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;

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

l_0 is the difference in position, in millimetres, between the bottom end of the copper pipe and the bottom end of the Inconel rod[2] at room temperature.

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):

$$r = 0{,}22 \times \Delta \bar{L}_{\text{max}} + 0{,}03 \qquad (2)$$

where

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

0,03 is in absolute %.

EXAMPLE Material A has a maximum average expansion equal to 0,70 %. The repeatability is then $0{,}22 \times 0{,}70 + 0{,}03 = 0{,}18$, in % absolute.

The reproducibility is calculated from Equation (3):

$$R = 0{,}28 \times \Delta\bar{L}_{max} \quad (3)$$

where $\Delta\bar{L}_{max}$ is 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 15379-2:2004);

b) all details necessary for identification of the sample;

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

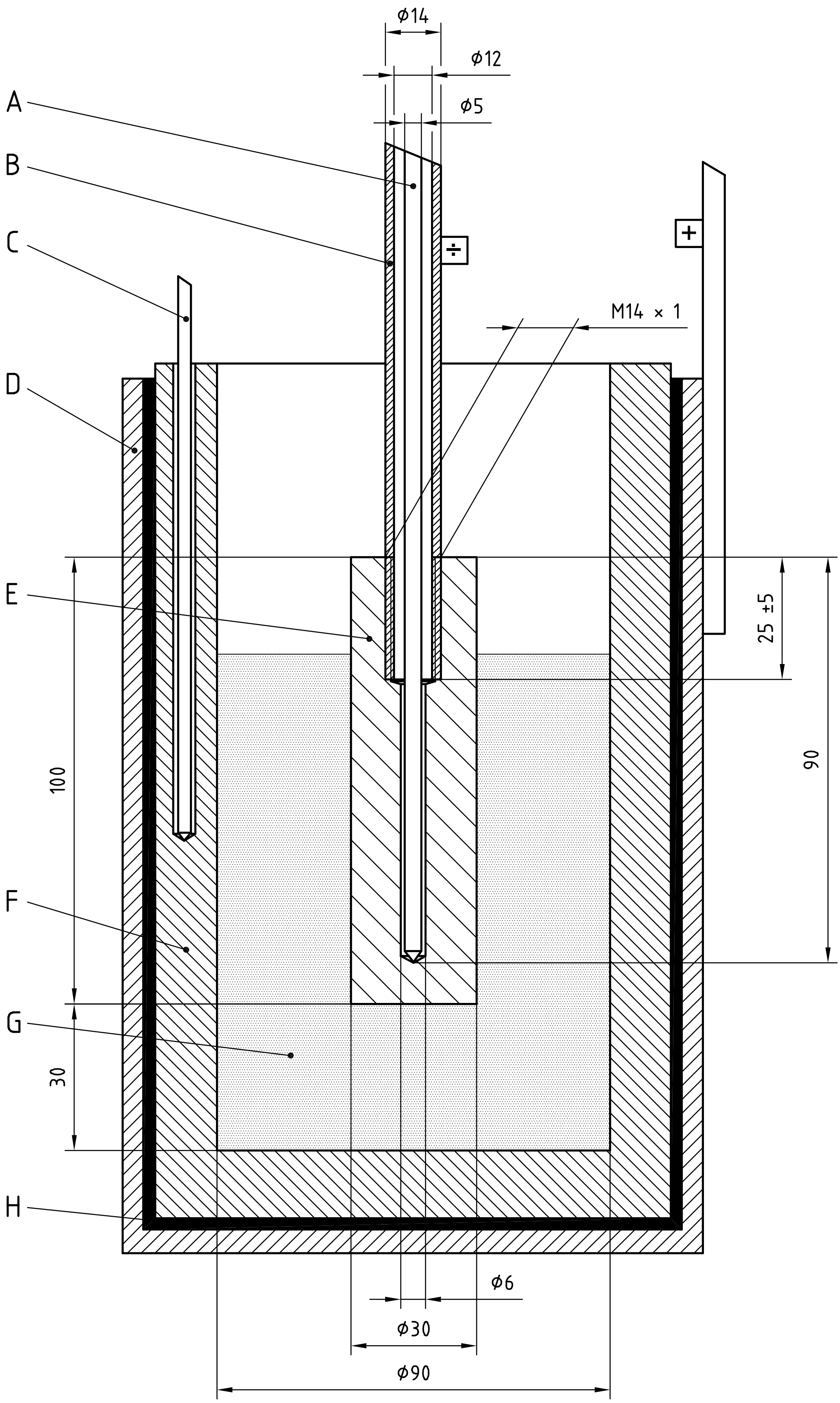
d) results of the test, including the maximum expansion, ΔL_{max}, in percent, due to sodium penetration and a plot of the relative expansion, $\Delta L(t)$, in percent, due to sodium penetration versus time, t;

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 Inconel[2)] rod
B sample holder (copper)
C thermocouple (Type K or S)
D steel container
E sample
F graphite crucible
G cryolite melt
H graphite powder

Figure 1 — Apparatus for measuring expansion due to sodium penetration

ICS 71.100.10

Price based on 6 pages