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Carbonaceous materials for the production of aluminium — Cold and tepid ramming pastes — Expansion/ shrinkage during baking

Produits carbonés utilisés pour la production de l'aluminium — Pâtes de brasquage froides et tièdes — Expansion/rétrécissement durant la cuisson

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Foreword

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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).

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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 14428:2005), which has been technically revised.

The main changes to the previous edition are as follows:

— Clause 8: new calculation concept based on average expansion/shrinkage within newly defined temperature ranges

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

The expansion/shrinkage of ramming pastes used in the production of aluminium is an important property, because excessive shrinkage can cause cracks in the baked paste serving as a lining material in alumina electrolysis cells. These cracks can cause leakage of the liquid bath, destroying the sidewall lining and the steel shell and thus leading to shutdown of the cell. Ramming pastes change phase from plastic to non-plastic between 400 °C and 600 °C (200 °C and 300 °C for resin binders). The shrinkage which occurs between the temperature at which the paste becomes non-plastic and the operating temperature (950 °C) is an important factor. Apparent shrinkage in the viscous range is due to slumping rather than actual shrinkage.

Carbonaceous materials for the production of aluminium — Cold and tepid ramming pastes — Expansion/shrinkage during baking

1 Scope

This document specifies the determination of expansion/shrinkage during baking of cold and tepid ramming pastes used in the production of aluminium.

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 14422, *Carbonaceous materials used in the production of aluminium — Cold-ramming pastes — Methods of sampling*

ISO 14427, *Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Preparation of unbaked test specimens and determination of apparent density after compaction*

ISO 17034, *General requirements for the competence of reference material producers*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

The change in height of a rammed sample of paste is measured at temperatures increasing to 950 °C, with a holding period of 3 h at the highest temperature. The shrinkage of the paste between the temperature at which the paste has become non-plastic and at 950 °C (both before and after the holding period) is determined.

5 Apparatus and materials

5.1 Device for measuring the sample length, accurate to ± 0,1 mm.

5.2 Push-rod dilatometer, capable of determining changes in length of the specimen of $2 \times 10^{-5}\ l_0$ (i.e. 1 μm for 50 mm length).

The contact force of the extensometer shall not exceed 2 N. The expansion/shrinkage shall be measured vertically. An example of a vertical dilatometer is given in Figure 1. The dilatometer should preferably consist of a push rod and an outer tube made of the same material. Vitreous silica is recommended as the material for the push rod and the outer tube. The gap between the outer tube and the sample shall

not be less than 2 mm and not larger than 10 mm. If the sample-holding assembly is made of vitreous silica, see the precautions given in 7.2.

No irreversible changes should occur in the material in the temperature range 20 °C to 950 °C.
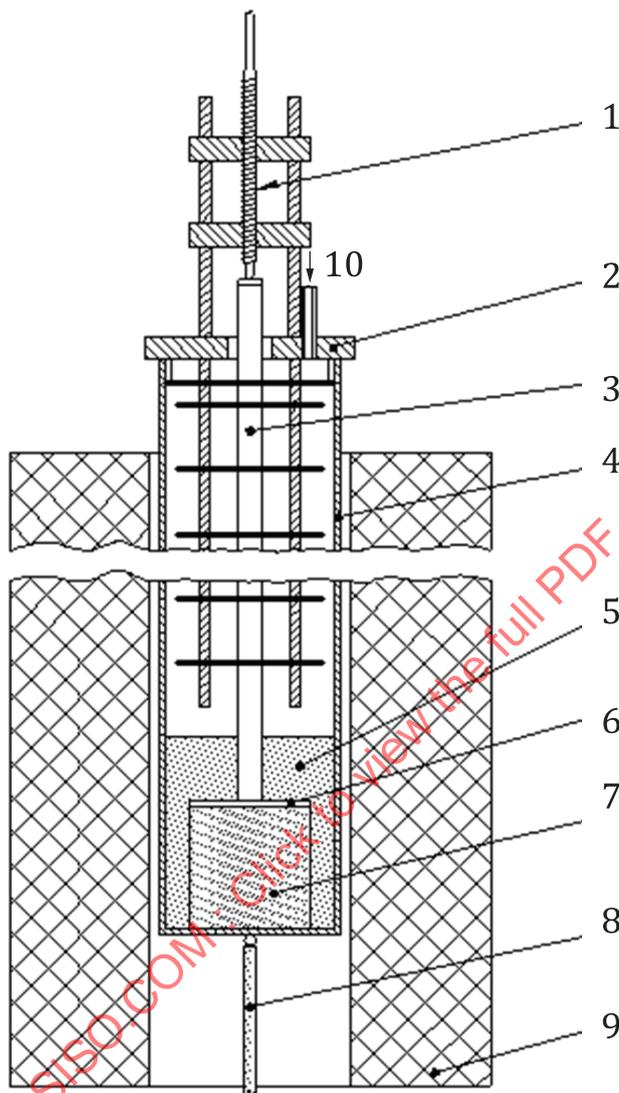
Key

1 extensometer
2 lid
3 push rod
4 outer tube
5 carbon particles
6 disk
7 test specimen, ⌀ 50 mm
8 thermocouple
9 furnace
10 inert gas inlet

Figure 1 — Example of quartz equipment for measuring thermal expansion/shrinkage of ramming pastes with 50 mm diameter

5.3   Disk, preferably made of the same material as the outer tube, with the same diameter as the sample ± 0,1 mm and (2,0 ± 0,5) mm thick, to be inserted on top of the sample.

If the disk is made of vitreous silica, see the precautions given in 7.2.

5.4   Carbon particles, used to support the sample during heating by filling the gap between the sample and the reference tube.

The size shall be < 1 mm. For example, the carbon material could be calcined anthracite or graphite.

5.5   Furnace, compatible with the dilatometer assembly, capable of attaining a temperature of 950 °C, with a temperature gradient over the sample height of less than 10 °C and with a furnace control device, suitable for the desired rate of increase in temperature of 3 K min^{-1}.

5.6   Temperature-measuring device, i.e. a thermocouple, preferably type S (Pt/Pt10 %Rh), capable of determining the temperature of the sample to ± 2 °C in the temperature range 20 °C to 950 °C.

5.7   Inert gas, for example nitrogen or argon 99,99 volume % pure.

5.8   Annealing furnace, capable of attaining a temperature of 1 100 °C and with a furnace control device, suitable for the desired rate of decrease in temperature of 0,2 K min^{-1} from 1 100 °C to 900 °C.

5.9   Reference sample, with shape and dimensions similar to those of samples usually tested in the test device, and certified by a national standards organization or laboratory meeting the requirements of ISO 17034.

Vitreous silica, annealed according to 7.2, is recommended for the reference material. Care shall be taken to ensure that the thermal expansion behaviour of the reference material is not altered by the test. If the reference material is vitreous silica, it shall be annealed (or re-annealed) in accordance with 7.2 before certifying its properties.

5.10   Fine abrasive material, for example silicon carbide on paper or fabric.

6   Samples

Sample the ramming paste in accordance with ISO 14422.

Prepare the sample in accordance with ISO 14427. Keep the sample in a closed box if it is not used within 1 h. The expansion/shrinkage measurement should preferably be started within 48 h of sample preparation.

7   Procedure

7.1   Cleaning

Clean all the surfaces (push rod, reference tube, disk, support and extensometer holder) by removing binder condensate and coked material.

If necessary, remove solid coating from the surface of the support with a fine abrasive material (5.10).

Ensure the end surfaces of the sample-holding assembly are clean and smooth. If the sample-holding assembly is made of vitreous silica, it will gradually devitrify and thus should be changed regularly.

7.2 Preparation of vitreous silica materials

Anneal the vitreous silica reference sample (5.9) and equipment made of vitreous silica (5.2, 5.3) before they are used for the first time, by first heating the vitreous silica to 1 100 °C for 7 h in a suitable furnace (5.8) and then cooling it from 1 100 °C to 900 °C at a constant rate of 0,2 K min^{-1}. Then allow the vitreous silica to cool to room temperature. For the reference sample, perform this procedure before determining and certifying its properties.

7.3 Calibration

In order to correct the measured length change, $\Delta l_{\mathrm{meas},T}$, with respect to irregular thermal expansion of the different parts of the dilatometer (5.2), the test procedure laid down in 7.4 shall be carried out on a reference material with measured thermal expansion (5.9).

Determine the measured change in length of the reference material by making at least three separate determinations in accordance with 5.4, and calculate the average change in length, $\Delta l_{\mathrm{ref},T}$. Perform the calibration at least each time any part of the equipment is changed.

7.4 Test procedure

Measure the length of the sample, l_0, at room temperature with an accuracy of 0,1 mm. Insert the sample into the dilatometer (5.2) and fill the gap between the sample and the outer tube with carbon particles (5.4). Place a disk (5.3) on top of the sample and mount the rest of the equipment. Wait until the signal from the extensometer is stable.

Ensure that all parts of the dilatometer are able to move freely in the vertical direction, because any undesired contact will influence the expansion/shrinkage.

Place the thermocouple (5.6) close to the sample.

Determine the position of the dilatometer at the initial temperature, t_0, and take this reading as zero for the uncorrected change in length, $\Delta l_{\mathrm{meas},T}$, which will be measured. Heat the furnace (5.5) at a rate of 3 K min^{-1} from room temperature to (950 ± 5) °C under an inert atmosphere (5.7). Keep the furnace at the highest temperature for 3 h (holding time) and then allow it to cool to room temperature. Record the temperature, T, and the related change in length, $\Delta l_{\mathrm{meas},T}$, at least every 5 min as shown in Figure 2.

Coke packing could hinder shrinkage, especially for wet paste. It is recommended that a gas-tight furnace chamber (Figure 1) with argon flushing is used. The use of carbon particles around the sample can then be avoided.

8 Results

8.1 Calculation of the correction term

Calculate the correction term for each temperature using Formula (1).

$$\Delta l_{\mathrm{corr},T} = \Delta l_{\mathrm{ref},T} - \Delta l_{\mathrm{real},T} \tag{1}$$

where

$\Delta l_{\mathrm{corr},T}$ is the correction term at temperature T, in mm;

$\Delta l_{\mathrm{ref},T}$ is the measured change in length of the reference material at temperature T, in mm;

$\Delta l_{\mathrm{real},T}$ is the actual measured change in length at temperature T, in mm.

8.2 Calculation of the expansion/shrinkage

8.2.1 Calculation of expansion or shrinkage for each temperature

Calculate the expansion or shrinkage for each temperature, in %, using Formula (2).

$$\Delta l_T = \frac{\Delta l_{\mathrm{meas},T} - \Delta l_{\mathrm{corr},T}}{l_0} \times 100 \tag{2}$$

where

Δl_T is the expansion/shrinkage at temperature T, in %;

$\Delta l_{\mathrm{meas},T}$ is the uncorrected change in length at temperature T, in mm;

$\Delta l_{\mathrm{corr},T}$ is the correction term at temperature T, in mm;

l_0 is the initial sample length, in mm.

8.2.2 Calculation of reference points

Calculate the reference points ΔL_A, ΔL_B and ΔL_C from Formulae (3) to (5) as shown in Figure 3:

a) Calculate the expansion before becoming non-plastic ΔL_A (%), as an average of the expansion between 450 °C and 500 °C, for pitch-based binders, using Formula (3).

$$\Delta L_A = \sum_{T=450\,°C}^{T=500\,°C} \frac{\Delta l_T}{n} \tag{3}$$

where n is the number of data points.

For non-pitch-based binders with different solidification temperature ranges, an adapted temperature interval to determine ΔL_A shall be used.

b) Calculate the expansion at maximum temperature, ΔL_B (%), as an average of the expansion between 900 °C and 950 °C, using Formula (4).

$$\Delta L_B = \sum_{T=900\,°C}^{T=950\,°C} \frac{\Delta l_T}{n} \tag{4}$$

where n is the number of data points.

c) Calculate the expansion after 3 h of holding at maximum temperature, ΔL_C (%), as an average of the expansion between 2,5 h and 3,0 h, using Formula (5).

$$\Delta L_C = \sum_{t_h=2,5\,h}^{t_h=3,0\,h} \frac{\Delta l_T}{n} \tag{5}$$

where

n is the number of data points;

t_h is the holding time at maximum temperature.

8.2.3 Calculation of shrinkage

a) Calculate the shrinkage between becoming non-plastic and maximum temperature, Δl_{heat}, in %, rounded up to the nearest 0,01 %, using Formula (6).