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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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ISO 14428 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

This corrected version of ISO 14428:2005 incorporates the following correction:

⎯ The publication date has been changed to 2005.

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 shut-down 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.

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

1   Scope

This International Standard 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 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 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*

ASTM E220, *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*

3   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.

4   Apparatus and materials

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

4.2   **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 6.2.

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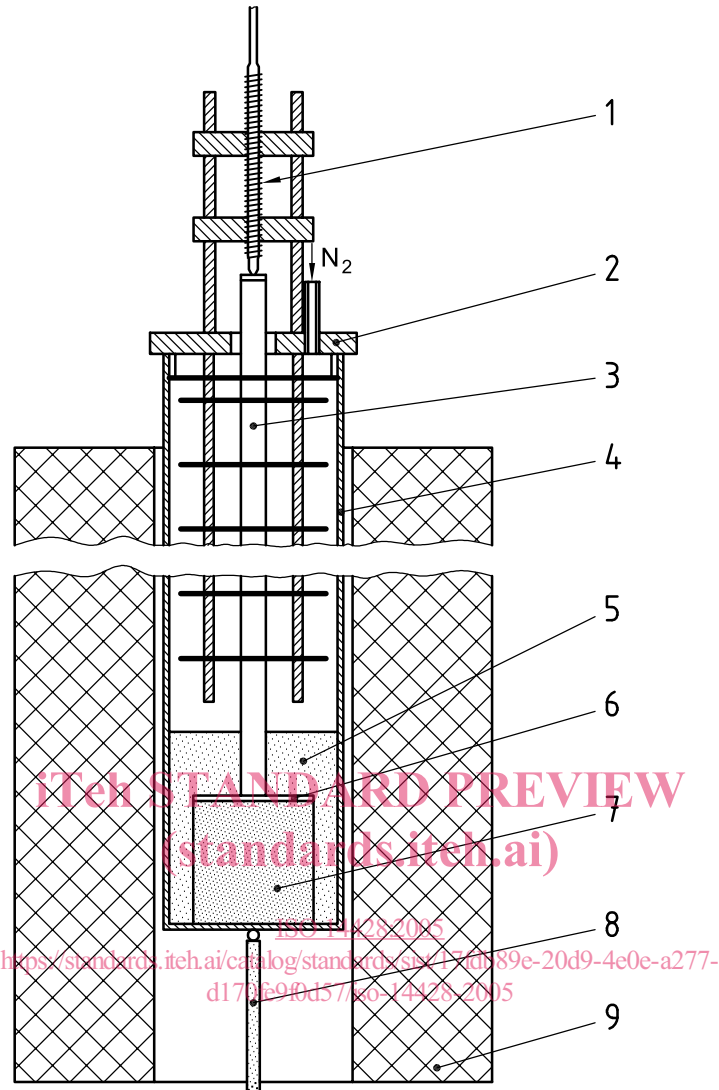
Key

1 extensometer
2 lid
3 push rod
4 outer tube
5 carbon particles
6 disk
7 test specimen, $\varnothing$ 50 mm
8 thermocouple
9 furnace

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

4.3 **Disk**, preferably made of the same material as the outer tube, with the same diameter as the sample $\pm$ 0,1 mm and (2,0 $\pm$ 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 6.2.

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

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

4.5   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 °C min^{-1}.

4.6   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 and calibrated in accordance with ASTM E220.

4.7   Inert gas, e.g. nitrogen 99,99 % pure.

4.8   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 °C min^{-1} from 1 100 °C to 900 °C.

4.9   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.

Vitreous silica, annealed according to 6.2, 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 6.2 before certifying its properties.

4.10   Fine abrasive material, e.g. silicon carbide on paper or fabric.

5   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 after sample preparation.

6   Procedure

6.1   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 (4.10).

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.

6.2   Preparation of vitreous silica materials

Anneal the vitreous silica reference sample (4.9) and equipment made of vitreous silica (4.2, 4.3) 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 (4.8) and then cooling it from 1 100 °C to 900 °C at a constant rate of 0,2 °C 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.

6.3   Calibration

Calibrate the equipment.

In order to correct the measured length, change, $\Delta l_{meas}(T)$, with respect to irregular thermal expansion of the different parts of the dilatometer (4.2), the test procedure laid down in 6.4 shall be carried out on a reference material with certified thermal expansion (4.9).

Determine the measured change in length of the reference material by making at least three separate determinations in accordance with 6.4, and calculate the average change in length, $\Delta l_{ref}(T)$. Perform the calibration at least each time any part of the equipment is changed.

6.4   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 (4.2) and fill the gap between the sample and the outer tube with carbon particles (4.4). Place a disk (4.3) 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 (4.6) 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, $\Delta l_{meas}(T)$, which will be measured. Heat the furnace (4.5) at a rate of 3 °C min^{-1} from room temperature to (950 ± 5) °C under an inert atmosphere (4.7). 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, $\Delta l_{meas}(T)$, at least every 5 min.

NOTE   Coke packing could hinder shrinkage, especially for wet paste. Coke packing could hinder measurement of CTE by cooling.

7   Results

7.1   Calculation of the correction term

Calculate the correction term for each temperature from the following equation:

$$\Delta l_{corr}(T) = \Delta l_{ref}(T) - \Delta l_{real}(T) \qquad (1)$$

where

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

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

$\Delta l_{real}(T)$ is the actual change in length at temperature T supplied by the certifier, in mm.

7.2   Calculation of the expansion/shrinkage

Calculate the expansion or shrinkage for each temperature from the following equation.

$$\Delta L(T) = \frac{\Delta l_{meas}(T) - \Delta l_{corr}(T)}{l_0} \times 100 \qquad (2)$$

where

$\Delta L(T)$ is the expansion/shrinkage at temperature T, in %, rounded off to the nearest 0,01 %;

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

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

l_0 is the initial sample length, in mm.

Plot a curve of the expansion, $\Delta L(T)$, versus the temperature, T (see Figure 2).

Using the curve and the calculated data, determine the following (see Figure 2):

a) the expansion, in %, (plateau or maximum), ΔL_A, at the temperature at which the paste has become non-plastic [usually between 400 °C and 600 °C (200 °C and 300 °C for resin binders)] with the corresponding temperature, T_A, in °C;

b) the expansion at the highest temperature before the holding period, ΔL_B, in %;

c) the expansion after 3 h of holding at top temperature, ΔL_C, in %.

Calculate the maximum thermal shrinkage before hold, ΔL_{before}, in %, from the following equation:

$$\Delta L_{before} = \Delta L_A - \Delta L_B \tag{3}$$

Calculate the maximum thermal shrinkage after the hold period, ΔL_{after}, in % from the following equation:

$$\Delta L_{after} = \Delta L_A - \Delta L_C \tag{4}$$
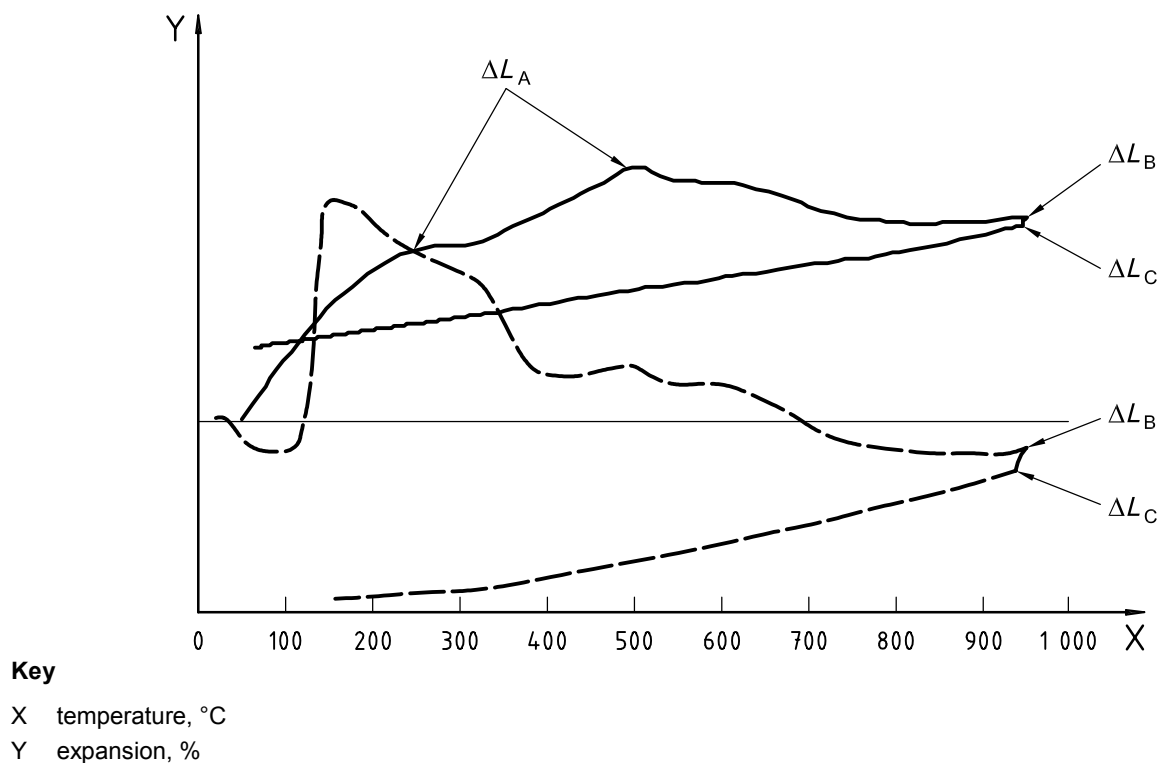
Key

X   temperature, °C

Y   expansion, %

Figure 2 — Example of a record of change in length versus temperature