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Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Determination of rammability of unbaked pastes

Produits carbonés utilisés pour la production de l'aluminium — Pâtes de brasquage froides et tièdes — Détermination de l'aptitude au brasquage des pâtes non cuites

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Foreword

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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.

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ISO 17544 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*.

Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Determination of rammability of unbaked pastes

1 Scope

This International Standard describes a method of producing a compaction curve indicating the rammability of carbonaceous ramming pastes used to line cathodes utilized in the production of aluminium.

Determination of the rammability enables a ramming paste with the optimum density to be selected.

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

3 Symbols

h measured height of test specimen, in cm

m mass of paste placed in cylinder, in g

N number of impacts

$N2$ minimum value of second derivative of $\rho(N)$

r inner radius of rammer cylinder, in cm

α scaling or normalization parameter describing the steepness of the compaction curve (which decreases as α increases)

γ location parameter which shifts the origin of the distribution

ρ_{max} maximum value of density, in g·cm^{-3}

ρ_0 initial value of density, representative of loose compaction of the paste under its own weight, in g·cm^{-3}

$\rho(N)$ density after N impacts, in g·cm^{-3}

$\rho(N)'$ first derivative of $\rho(N)$

$\rho(N)''$ second derivative of $\rho(N)$

$\rho(N)'''$ third derivative of $\rho(N)$

$\rho(50)$ density after 50 impacts, in $g \cdot cm^{-3}$

$\rho(100)$ density after 100 impacts, in $g \cdot cm^{-3}$

$\Delta\rho$ pre-exponential factor, in $g \cdot cm^{-3}$: $\Delta\rho = \rho_{max} - \rho_0$

4 Principle

Ramming paste is compacted in a laboratory rammer. The paste is subjected to a total of 350 impacts. The density of the paste is measured as a function of the number of impacts. The data is fitted to a three-parameter cumulative Weibull distribution function given by the following equation:

$$\rho(N) = \rho_{max} - \Delta\rho \times \exp\left[-\left(\frac{\log_{10} N - \gamma}{\alpha}\right)^6\right] \tag{1}$$

A ramming parameter $N2$, corresponding to the number of impacts for which the second derivative of the Weibull function has a minimum value, is then calculated.

An alternative method of evaluation is to determine the ratio of the density after 50 impacts to the density after 100 impacts, as shown by the following formula:

$$\frac{\rho(50)}{\rho(100)} \tag{2}$$

5 Apparatus

An example of a rammer mould is shown in Figure 1.

5.1 Rammer cylinder, made of steel, with an inner diameter of 50 mm.

5.2 Ram base, made of steel. The ram base shall fit into the rammer cylinder and act as the bottom of the cylinder.

5.3 Rammer, consisting of a steel piston, a falling weight mounted so that it can be dropped onto the top of the piston, and a height-measuring device accurate to 0,1 mm. The mass of the falling weight shall be 6,35 kg and the weight shall travel a constant distance to impact of (50 ± 0,1) mm.

5.4 Box (with lid), capable of containing the amount of ramming paste needed to produce one test specimen. The free space over the paste shall not be more than 50 % of the volume of the paste.

5.5 Heating/cooling cabinet, capable of being maintained at a constant temperature ± 0,5 °C in the range 5 °C to 100 °C.

5.6 Insulation layer (for example, 1-cm-thick polystyrene), for insulation of the rammer cylinder, or **thermostatically controlled surround** (see Figure 1).

5.7 Balance, accurate to 0,1 g.

6 Sampling

Sample the paste in accordance with ISO 14422.

NOTE Experience has shown that 150 g to 200 g of ramming paste is necessary for each test specimen.

7 Procedure

Ensure the cylinder, the ram base and the piston are clean.

Weigh the necessary amount of ramming paste (to the nearest 0,1 g) into a suitable box (5.4). If the amount of ramming paste to be used is not known, determine the required mass by preliminary tests [the height of the test specimen after compaction has to be (50 ± 5) mm (see below)]. Seal the box. Heat or cool the box together with the rammer cylinder (5.1), the ram base (5.2) and the insulation/surround (5.6) to the test temperature (agreed between the interested parties) in the heating/cooling cabinet (5.5) for 2 h to 3 h.

Transfer the paste to the cylinder. Protect the cylinder with the insulation or surround (5.6). Place the cylinder in the rammer (5.3) and lower the piston until it is resting on the paste. Compact the ramming paste with a total of 350 impacts, reading the height h of the test specimen (to the nearest 0,1 mm) from the scale on the ramming apparatus, with the piston head resting on the specimen, after 1, 2, 3, 4, 5, 7, 10, 15, 20, 25, 30, 35, 40, 50, 60, 80, 100, 120, 150, 180, 230, 280 and 350 impacts. Where several impacts are made between height measurements, compact the test specimen at a rate of about 1 impact per second.

If the final height of the compacted test specimen is not equal to (50 ± 5) mm, discard the results and repeat the test with another sample mass.

Remove the test specimen. Clean the cylinder, the ram base and the piston carefully after each test.

8 Expression of results

8.1 Calculation of the density

Calculate the density of the paste $\rho(N)$ corresponding to each specimen height measured (i.e. to each number of impacts N at which a height measurement was made) from Equation (3):

$$\rho(N) = \frac{m}{\pi r^2 h} \qquad (3)$$

where

m is the mass, in g, of the ramming paste placed in the cylinder, accurate to 0,1 g;

r is the inner radius of the ramming cylinder, in cm, accurate to 0,01 cm;

h is the height of the unbaked test specimen as read from the rammer scale, in cm, accurate to 0,01 cm.

Round the results to the nearest 0,001 $g \cdot cm^{-3}$.

Plot $\rho(N)$ against N to give a compaction curve.

8.2 Interpretation of the curve

A paste showing a slow asymptotic approach towards its maximum density is said to be a dry paste, whereas a paste showing a rapid asymptotic approach towards its maximum density is said to be a wet paste.

8.3 Determination of $N2$

8.3.1 Fitting the data to a Weibull function

The data fit is based on the similarities between compaction curves presented with a logarithmic abscissa and statistical cumulative distribution functions. A general three-parameter cumulative Weibull distribution function is chosen.

Determine the parameters ρ_{max}, $\Delta\rho$, α and γ by fitting the values of $\rho(N)$ calculated in 8.1 to the Weibull function:

$$\rho(N) = \rho_{max} - \Delta\rho \times \exp\left[-\left(\frac{\log_{10} N - \gamma}{\alpha}\right)^6\right] \quad (4)$$

The fitting can be done by the least-squares method, for which various commercial computer programmes are available. Normally, a set of starting values for the four parameters is needed. The starting values in Table 1 can be used.

Table 1 — Starting values for Equation (5)

ρ_{max}	1,500
$\Delta\rho$	0,500
α	3
γ	−2

8.3.2 Calculation of $N2$

Putting the third derivative of $\rho(N)$ equal to zero [i.e. $\rho(N)''' = 0$] enables the turning point of the density distribution curve to be determined. The minimum value of the second derivative $\rho(N)''$ is used as the evaluation criterion for the rammability.

The number of impacts ($N2$) which gives the minimum value of the second derivative of $\rho(N)$ (with respect to $\log_{10}N$), when the third derivative is equal to zero, is given by:

$$\rho(N)''' = \frac{12\Delta\rho}{\alpha^3} \times e^{-\left(\frac{\log_{10} N - \gamma}{\alpha}\right)} \times \left[18\left(\frac{\log_{10} N - \gamma}{\alpha}\right)^{15} - 45\left(\frac{\log_{10} N - \gamma}{\alpha}\right)^{9} + 10\left(\frac{\log_{10} N - \gamma}{\alpha}\right)^{3}\right] = 0 \quad (5)$$

It is possible to solve Equation (5) exactly:

$$N2 = 10^{(1{,}145\alpha + \gamma)} \quad (6)$$

Calculate the number of impacts $N2$ which gives the minimum value of the second derivative of $\rho(N)$ (third derivative = 0) using Equation (6) and the parameters from the curve-fitting operation carried out in 8.3.1. Round the value calculated for $N2$ to the nearest whole number.

NOTE Typically, values lie in the range $65 < N2 < 130$. Such values were found to give good rammability when compared with practical work at one smelter. The range may, however, depend on the ramming technology used.

8.4 Determination of $\rho(50)/\rho(100)$

Alternatively, calculate the density after 50 impacts divided by the density after 100 impacts, $\rho(50)/\rho(100)$, using the values of $\rho(50)$ and $\rho(100)$ determined in 8.1.

NOTE Typically, values lie in the range $0{,}960 < \rho(50)/\rho(100) < 0{,}975$. Such values were found to give good rammability when compared with practical work at one smelter. The range may, however, depend on the ramming technology used.

9 Precision (determined in accordance with ISO 5725-2)

9.1 $N2$

Repeatability $(r) = 0{,}18 \times$ material average $-$ 3,71

Reproducibility $(R) = 0{,}34 \times$ material average $+$ 2,65

The number of degrees of freedom, i.e. number of laboratories (5) × number of samples (4), was 20.

9.2 $\rho(50)/\rho(100)$

Repeatability $(r) = 0{,}004\ 9$

Reproducibility $(R) = 0{,}496 \times$ material average $-$ 0,473 1

The number of degrees of freedom, i.e. number of laboratories (6) × number of samples (4), was 24.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for identification of the sample tested;

c) the temperature used for the test;

d) the results of the test:

   1) the curve of density versus number of impacts,

   2) the values of the parameters ρ_{max}, $\Delta\rho$, α and γ determined by fitting the curve to the Weibull function,

   3) the value obtained for $N2$ or $\rho(50)/\rho(100)$;

e) the date of the test;

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

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