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Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of crystallite size of calcined petroleum coke by X-ray diffraction

Produits carbonés utilisés pour la production de l'aluminium — Coke calciné — Détermination de la taille de la cristallite du coke calciné de pétrole par diffraction aux rayons X

Reference number
ISO 20203:2005(E)

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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 20203 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

This International Standard is based on ASTM D5187-91(2002)[6] published by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

ASTM D5187-91(2002) was developed under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants as the direct responsibility of Subcommittee D02.05.0D on Petroleum Coke Sampling and Procedures, and was published in December 1991.

The crystallinity of petroleum coke, as reflected by the L_c value, is a general measure of quality affecting suitability for end use and is a function of the heat treatment used.

The crystallite height is used to determine the extent of such heat treatment, for example, during calcination. The value of the L_c determined is not affected by coke microporosity or the presence of foreign, non-crystalline materials such as dedust oil.

Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of crystallite size of calcined petroleum coke by X-ray diffraction

SAFETY PRECAUTIONS — This International Standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard describes a test method for the determination of the mean crystallite height of a representative, pulverized sample of calcined petroleum coke by interpretation of an X-ray diffraction pattern produced through conventional X-ray scanning techniques.

It applies to carbonaceous materials used in the production of aluminium.

Calcined petroleum coke contains crystallites of different heights. This test method covers the determination of the average height of all crystallites in the sample by empirical interpretation of the X-ray diffraction pattern. The crystallite diameter (L_a) is not determined by this test method.

2 Terms and definitions

For the purposes of this document, the following terms, abbreviated terms and definitions apply.

2.1
crystallites
stacks of graphitic carbon platelets located parallel to one another

2.2
L_C
mean or average height of crystallites in a sample

NOTE It is expressed as a linear dimension, in nanometres.

2.3
***hkl*(002)**
Miller indices of the crystalline planes of graphite corresponding to a lattice spacing (d) of 0,335 nm

2.4
Θ
glancing angle produced when a parallel beam of uniform X-rays impinges upon a crystalline lattice and measured by the X-ray goniometer

NOTE It is usually expressed in ° 2Θ.

3 Principle

A packed sample pulverized to less than 75 µm is subjected to a monochromatic X-ray beam and rotated to produce a diffraction pattern under specific conditions. The location and shape of the peak with hkl (002) at $d = 0{,}335$ nm is used to calculate L_c by manual interpretation of the peak or by computer simulation.

4 Apparatus

4.1 X-ray powder diffractometer, equipped with an X-ray source set for Cu-Kα radiation, a monochromator or filter for restricting the wavelength range, a sample holder, a radiation detector, a signal processor, and readout (chart or computer memory). The diffractometer shall be capable of rate scanning at 1°/min or incrementally step scanning at 0,2°/step.

4.2 Sample holders, as specified by the manufacturer of the diffractometer, that enable packing of a pulverized sample of sufficient height to expose a level, smooth surface to the X-ray beam.

4.3 Briquetting press, capable of generating pressures up to 70 MPa.

4.4 Compressible aluminum caps, used as a support for producing a briquetted sample.

4.5 Silicon or quartz sample, of reference material quality suitable for calibrating the diffractometer.

NOTE These materials are usually available from national reference organizations e.g. the National Institute of Standards and Technology in USA.

5 Reagents and materials

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity (see [1], [2] and [3] in the Bibliography).

5.1 Acetone.

5.2 Polyethylene glycol, approximate molecular weight of 200.

5.3 Binding agent, prepare a solution of polyethylene glycol and acetone, so that the proportion of polyethylene glycol in the solution, expressed as a mass fraction (w) by percentage of the total composition. is 15 %, by adding 15 g of polyethylene glycol to 85 g of acetone.

6 Sample preparation

6.1 General

For recommended practices for obtaining, handling and preparing coke samples, refer to ISO 6375 [5].

Reduce and divide the gross sample to obtain a laboratory analysis sample.

Divide, by riffling, a minimum of 100 g from the laboratory analysis sample.

Crush 100 g of the test sample such that 98 % will pass through a 75 µm (No. 200) sieve.

6.1.1 Any of the packing techniques listed in 6.2 may be used for packing the sample into the X-ray diffractometer specimen holder:

6.2 Packing techniques for X-ray diffraction specimen holder

6.2.1 Back-fill technique

Put the window on a glass slide (Slide 1) and transfer sufficient quantities of sample into the window. Work the sample towards the corners of the holder using a glass slide or spatula. Press down using a flat glass slide and scrape off any excess material. Place a glass slide (Slide 2) on top of the sample and secure with tape. Remove Slide 1 to expose a flat, smooth surface before inserting into the diffractometer for analysis.

6.2.2 Front-fill technique

Place a confining ring over the round sample holder and fill the holder cavity and ring with sample. The ring will initially overfill the sample holder. Work the sample into the entire cavity and ring. Scrape the excess off with a glass plate or spatula. Press down using a flat glass slide. Remove any excess material on the front face of the holder. Repress the sample with the glass slide while turning clockwise and anticlockwise. Continue until the sample is level with the holder face. Place the sample in the diffractometer holder.

6.2.3 Side-loading technique

Pack the sample. Clamp a glass slide over the top face to form a temporary cavity wall. With the holder in a vertical position, drift the powdered sample into the end opening. If necessary, use a cardboard pusher cut to fit the cavity, to lightly compress the sample so it will remain in the cavity. Return the holder to a horizontal position and carefully remove the glass slide. Place the sample in the diffractometer holder.

6.2.4 Briquetting technique

Weigh out 4,0 g of the sample onto a watch glass and pipette exactly 3 ml of the binding agent onto the sample and mix thoroughly with a spatula. Place the sample under an infrared-heat lamp and allow the acetone to evaporate. Typically, about 1 or 2 min will be required to eliminate the acetone odour from the sample. Break up the caked sample with a spatula and transfer to an aluminum cap whose diameter is compatible with the sample holder of the diffractometer. Place the cap in a briquetting press and press at 48 MPa. Transfer the pelletized sample to the sample holder and insert into the diffractometer for analysis.

7 Calibration

7.1 Ensure that the diffractometer is in correct mechanical and optical alignment and that intensities have been maximized through the procedures described in the instrument manufacturer's documentation. A service engineer or in-house technician who has been well instructed in the correct alignment procedures suggested by the manufacturer best accomplishes this.

7.2 Monitor proper angles and intensities with the reference silicon or quartz sample and take corrective actions if necessary.

8 Procedure

8.1 Prepare and operate the diffractometer in accordance with the manufacturer's instructions. Once established, closely control all instrumental parameters to ensure repeatable analyses.

8.2 Place the packed sample in the instrument's sample holder and energize the X-ray source.

8.3 Obtain a diffraction pattern rate scanned at 1°/min or step scanned at 0,2°/step over the range of 14 to 34° 2Θ. Record the data either on a strip-chart recorder scanning at 1°/min or through computer software designed to read and store the angular and intensity measurements. The measurement time can be significantly reduced by selecting suitable segments of the range 14 to $34° 2\Theta$. Segments are chosen to cover the required points in 9.1.

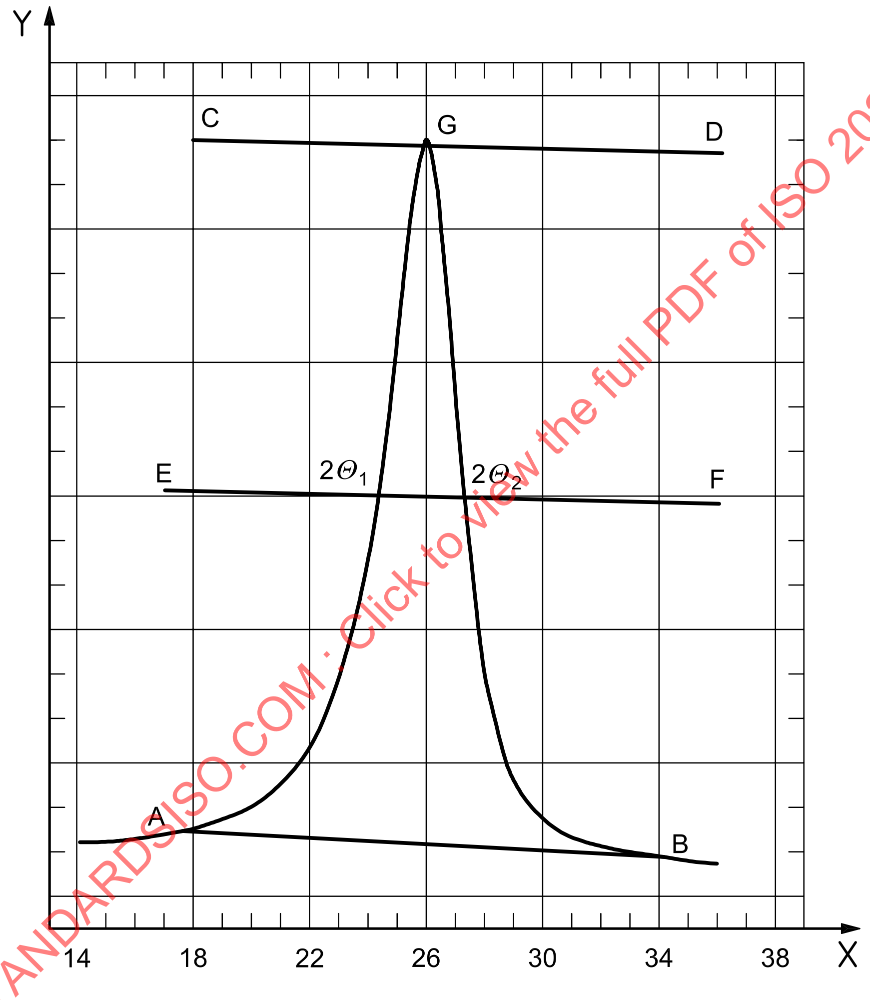
9 Calculation

9.1 Manual interpretation

9.1.1 For manual interpretation from a strip-chart recording, refer to Figure 1 and 9.1.2 to 9.1.5.

9.1.2 Determine the average low and high backgrounds (Points A and B, respectively) on the diffraction scan and connect them with a straight line.

9.1.3 Construct line CD parallel to line AB, and going through the apex of the peak at point G [(*hkl* (002) at 0,335 nm]. Draw the line such that, if the peak is irregular, it will pass through the average of the irregularities.



Key

X degree two theta (°2Θ)

Y intensity (arbitrary unit)

Figure 1 — Typical diffraction scan of petroleum coke

9.1.4 Determine the full-width half maximum (FWHM) of line AB. Construct line EF such that it intersects the peak at half of its maximum value. The points at which EF intersects the peak are $2\Theta_1$ and $2\Theta_2$, respectively.

9.1.5 For computer simulation based on the intensities recorded at 0,2° intervals, produce a mathematical representation of the diffraction curve. Determine the baseline, peak, peak height, and half-peak height to produce the half-peak height angles, $2\Theta_1$ and $2\Theta_2$, as above.

9.2 Determine the mean crystallite height $\overline{L}_c$

$$\overline{L}_c = \frac{0{,}89\lambda}{2\left(\sin\Theta_2 - \sin\Theta_1\right)} \qquad (1)$$

where

λ is the wavelength of the target material of the X-ray tube, expressed in nanometres;

Θ_1 is the angle at the half-peak intensity $(2\Theta_1/2)$ width on the low side;

Θ_2 is the angle at the half-peak intensity $(2\Theta_2/2)$ width on the high side;

0,89 is an arbitrary constant that is equivalent to 0,89 for L_C[4].

NOTE Equation 1 was derived from the Scherrer equation (Equation 2).

$$Lc = \frac{(0{,}89\lambda)}{(\beta\cos\Theta)} \qquad (2)$$

where

0,89 is an arbitrary constant that is equivalent to 0,89 for L_C;

λ is the wavelength of the source radiation, expressed in nanometres;

β is the line breadth of the pure diffraction peak, expressed in radians;

Θ is the angular location of the peak maximum $(2\Theta/2)$ expressed in degrees.

The above equations make the assumption that the true line width is equal to the measured width, the contribution of instrumental line broadening is negligible.

10 Reporting of results

Report the mean crystallite height to the first decimal i.e. L_C = x,xx nm.

11 Precision and bias

11.1 General

The precision of this test method, as determined by the statistical examination of inter-laboratory test results in which one operator in ten laboratories made a duplicate analysis on each of six materials, is as given in 11.2 to 11.4.