SLOVENSKI STANDARD
oSIST prEN ISO 21068-4:2023

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Kemijska analiza surovin in ognjevzdržnih izdelkov, ki vsebujejo silicijev karbid, silicijev nitrid, silicijev oksinitrid in sialon - 4. del: Metode XRD (ISO/DIS 21068-4:2023)

Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon - Part 4: XRD methods (ISO/DIS 21068-4:2023)

Chemische Analyse von Rohstoffen und feuerfesten Erzeugnissen, die Siliciumcarbid, Siliciumnitrid, Siliciumoxinitrid und Sialon enthalten - Teil 4: XRD-Verfahren (ISO/DIS 21068-4:2023)

Analyse chimique des matières premières et des produits réfractaires contenant du carbure de silicium, du nitrure de silicium, de l'oxynitrure de silicium et du sialon - Partie 4: Méthodes de DRX (ISO/DIS 21068-4:2023)

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Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon —

Part 4:
XRD methods

ICS: 81.080

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Contents

Page

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.

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.

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 21068-4 was prepared by Technical Committee ISO/TC 33, *Refractories*

ISO 21068 consists of the following parts, under the general title *Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon*:

— *Part 1: General information and sample preparation*

— *Part 2: Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica*

— *Part 3: Determination of nitrogen, oxygen and metallic and oxide constituents*

— *Part 4: XRD methods*

Introduction

ISO 21068, Parts 1 to 4, have been developed from the combination of EN 12698:2007, Parts 1 [4] and 2 [5], and ISO 21068:2008, Parts 1 to 3 [6 to 8]. The latter has been originally developed from the combination of Japanese standard JIS R 2011 [1] and work items developed within CEN.

The new Part 4 is derived from the European standard EN 12698-2:2007 [5] describing XRD methods for the determination of mineralogical phases typically apparent in nitride and oxy-nitride bonded silicon carbide refractory products using a Bragg-Brentano diffractometer.

Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon —

Part 4: XRD methods

1 Scope

This standard describes methods for the determination of mineralogical phases typically apparent in nitride and oxy-nitride bonded silicon carbide refractory products using a Bragg-Brentano diffractometer.

It includes details of sample preparation and general principles for qualitative and quantitative analysis of mineralogical phase composition. Quantitative determination of α-Si_3N_4, β-Si_3N_4, Si_2ON_2, AlN, and SiAlON are described.

NOTE For the refinement procedures the total nitrogen content, analysed in accordance with ISO 21068-3 is needed.

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 21068-1, *Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon — Part 1: General information and sample preparation*

ISO 21068-2, *Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon — Part 2: Determination of volatile components, total carbon, free carbon, silicon carbide, total and free silicon, free and surface silica*

ISO 21068-3, *Chemical analysis of raw materials and refractory products containing silicon-carbide, silicon-nitride, silicon-oxynitride and sialon — Part 3: Determination of nitrogen, oxygen and metallic and oxidic constituents*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

ISO 10081-4, *Classification of dense shaped refractory products — Part 4: Special products*

3 Terms and definitions

For the purposes of this document the terms and definitions given in ISO 21068-1 and ISO 10081-4 apply.

4 Apparatus

The aim is to have a high resolution diffractogram with well defined peaks on a low background with minimal artefacts due to fluorescence and Kβ peaks. A Bragg-Brentano diffractometer with a copper X-ray tube operating at 40 kV and 20 mA to 45 mA. Primary beam monochromation is achieved using a suitable filter or primary beam monochromator or equivalent, secondary beam monochromation is achieved using a graphite monochromator, energy discriminator or equivalent and the following experimental setting for data collection are used:

— goniometer with a measurement uncertainty of ≤ 0,5° at a confidence level of 95 %;

— Suitable primary and secondary slit arrangements include variable slots or fixed slits with a primary soller slit with a divergence ≤ 2,5°;

— divergence slit 1°;

— receiving slit ≤ 0,2 mm;

— scatter slit ≤ 1°;

5 Sampling

Sample shaped and unshaped products using the procedures given in ISO 5022 and ISO 8656-1.

When sampling large fragments, take care to collect samples from different points of individual pieces.

Follow the sampling and grinding procedure as described in ISO 21068-1

6 Procedure

6.1 Sample preparation

Grind the sample using a mill so that the resultant powder can pass through a 150 µm sieve.

NOTE Care should be taken not to grind the sample excessively as this has been found to cause the silicon nitride, and silicon phases in particular, to reduce in intensity. This is believed to be due to a build-up of an amorphous layer on their particles due to damage induced by the silicon carbide.

Press the powder into the cavity holder from the reverse side of the cavity to that which is to be presented to the x-ray beam (to reduce preferred orientation). The depth of the cavity shall be sufficient to exceed the critical depth of CuKα radiation for the sample analysed.

6.2 Measuring parameters

Scan the sample on the instrument using the following parameters:

— start angle, 2θ 10°;

— end angle, 2θ 70°, 130° if β-SiAlON determination is required;

— step-spec, 2θ 0,02° or continuous;

— integration time 4 s.

An additional scan using the same conditions as above between 60° and 70° 2θ may be required if aluminium and/or iron is thought to be present.

NOTE Parameters for tube settings should be: voltage 40 kV, excitation current 20 mA to 45 mA.