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Sodobna tehniļ na keramika - Preskusne metode za keramiļ ne praġke - 3. del: Doloļ evanje kisika v neoksidnih praġkih s toplotno ekstrakcijo z nosilnim plinom

Advanced technical ceramics - Methods of test for ceramic powders - Part 3: Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 3: Bestimmung des Sauerstoffgehaltes in Nichtoxid-Pulvern mittels Trägergasheißextraktion

Céramiques techniques avancées - Méthodes d'essais pour poudres céramiques - Partie 3: Détermination de la teneur en oxygène de poudres non-oxydes par extraction a chaud sous gaz porteur

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This European Standard was approved by CEN on 2 December 2006.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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Foreword

This document (EN 725-3:2007) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2007, and conflicting national standards shall be withdrawn at the latest by July 2007.

This document supersedes EN 725-3:1994.

EN 725 *Advanced technical ceramics — Methods of test for ceramic powders* was prepared in Parts as follows:

Part 1: *Determination of impurities in alumina*

Part 2: *Determination of impurities in barium titanate*

Part 3: *Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas*

Part 4: *Determination of oxygen content in aluminium nitride by XRF analysis*

Part 5: *Determination of particle size distribution*

Part 6: *Determination of the specific surface area* [withdrawn]

Part 7: *Determination of the absolute density* [withdrawn]

Part 8: *Determination of tapped bulk density*

Part 9: *Determination of un-tapped bulk density*

Part 10: *Determination of compaction properties*

Part 11: *Determination of densification on natural sintering*

Part 12: *Chemical analysis of zirconia*

Parts 6 and 7 of the series were superseded in 2005 by EN ISO 18757 and EN ISO 18753 respectively.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This part of EN 725 describes a method for the determination of the oxygen content of non-oxide powders used for advanced technical ceramics, using an inert carrier gas thermal extraction method. The method described is suitable for oxygen contents of less than 3 %.

NOTE An indication of the limits of determination is usually given by the manufacturers of the gas analysis apparatus used. However, for a specific measurement procedure, such limits can be determined by experiments conducted by the user.

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.

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Principle

A test sample is heated in a graphite crucible at a high temperature in a flow of an inert carrier gas. Oxygen in the sample is converted to oxides of carbon, which are extracted and transformed to consist entirely of either carbon monoxide or carbon dioxide. This volume is then determined by a method of gas analysis.

NOTE Guidance on the selection of test conditions is given in Annex A.

4 Apparatus

4.1 ***Scoop***, for transferring the test sample.

4.2 ***Graphite crucible***, which is used as a carbon source. The crucible is capable of being electrically heated by two electrodes, or by induction.

4.3 ***Tin or nickel capsule (optional)***, to contain the ceramic powder sample.

4.4 ***Nickel wire basket (optional)***, for use as a fluxing agent with certain powders such as aluminium nitride.

4.5 ***Gas analysis apparatus***, based on one of the following techniques:

a) volumetric analysis, for measurement of carbon monoxide gas;

b) chromatography, for carbon monoxide;

c) thermal conductivity, for carbon monoxide and carbon dioxide;

d) coulometric analysis, for carbon dioxide;

e) infrared absorption, for carbon dioxide;

f) gravimetry, by absorption of carbon dioxide.

4.6 ***Furnace***, normally capable of reaching at least 2500 °C operating in an inert gas atmosphere (nitrogen, helium or argon).

NOTE A furnace which does not achieve 2500 °C may be used for lower test temperatures, but 2500 °C will be needed to give the correct result for certain types of material.

5 Sample preparation

Take a sample of the dry powder, the amount taken being based on the detection limit of the gas analysis apparatus (**4.5**) used and on the expected oxygen content of the powder.

EXAMPLE For infrared absorption (see **4.5** e)), samples of 100 mg to 300 mg should be used.

NOTE The measured (total) oxygen content consists of a contribution from surface and bulk.

Use a scoop (**4.1**) to transfer the powder and place the sample either directly in the crucible (**4.2**) or in the tin capsule (**4.3**). If the capsule is used, squeeze it to expel air and then fold over the open end several times, weighing again after squeezing.

6 Calibration

Calibration shall be carried out preferably by using a reference material of certified oxygen content to calibrate the furnace (**4.6**) and gas analysis apparatus (**4.5**), using the procedure given in Clause 8. An alternative method is to use a sample of pure carbon monoxide or carbon dioxide gas to calibrate the gas analysis apparatus, with a minimum of three results being recorded.

NOTE Since gas calibration only calibrates the detection and not the extraction efficiency, gas calibration does not automatically guarantee correct results. The same applies if sample and calibration material are not very similar.

7 Blank determination

Carry out a blank determination for each measurement run. Use the procedure given in Clause 8 and record the amount of gas evolved. Use this blank value to calculate the results for the sample analysis.

8 Procedure

8.1 Heat the empty graphite crucible (**4.2**) inside the furnace (**4.6**) to a temperature higher than the temperature for analysis. Record the flow of gas used in the furnace. Cool to ambient temperature.

NOTE 1 This is to prevent additional degassing of the crucible during the test run, which would produce errors.

NOTE 2 A recommendation for gas flow will normally be given by the manufacturer of the gas analysis apparatus (4.5).

8.2 Place the tin (or nickel) capsule (**4.3**) and sample in the crucible (**4.2**). Select a temperature which is high enough to dissociate the oxides to be determined (see Annex A). Use the nickel wire basket (**4.4**) if the preliminary tests indicate that it is necessary.

Heat the furnace to the test temperature and record the temperature and the length of time at temperature. Collect the gas evolved and measure the amount, using one of the analytical techniques listed in 4.5.

8.3 Repeat the procedure given in Clause 5 and 8.2 at least four times.

9 Test report

The test report shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall include at least the following information:

a) name and address of the testing establishment;

b) date of the test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this standard, i.e. determined in accordance with EN 725-3;

f) authorising signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) description of the test material (manufacturer, type, batch or code number);

i) type of analytical equipment used (see 4.5);

j) method of calibration (see Clause 6);

k) purity of the carrier gas and gas used for calibration; the calibration material used with its certified oxygen content;

l) mass of the samples and reference to optional use of the tin capsule (see 4.3);

m) test temperature;

n) individual values of oxygen content, mean value and standard deviation;

o) comments about the test or test results.