INTERNATIONAL STANDARD

ISO 17947

First edition
2014-09-01

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of fine silicon nitride powders

Céramiques techniques — Méthodes pour l'analyse chimique de poudres de nitrure de silicium

Reference number
ISO 17947:2014(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Introduction

This International Standard has been developed from Japanese Industrial Standard JIS R 1603:2007 with reference to CEN ENV 14226:2002 and ASTM C1494-01:2007, and is applicable to the chemical analysis of silicon nitride raw powders for fine ceramics use. This International Standard covers both major and minor constituents such as total silicon, total nitrogen, and some of trace metallic and non-metallic elements.

INTERNATIONAL STANDARD **ISO 17947:2014(E)**

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of fine silicon nitride powders

1 Scope

This International Standard specifies the methods for the chemical analysis of fine silicon nitride powders used as the raw material for fine ceramics.

This International Standard stipulates the determination methods of total silicon, total nitrogen, aluminium, iron, calcium, oxygen, carbon, fluorine, and chlorine in fine silicon nitride powders.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2828, *Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

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

ISO 21068-2, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 2: Determination of loss on ignition, total carbon, free carbon and silicon carbide, total and free silica and total and free silicon*

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

ISO 21438-2, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 2: Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)*

ISO 21438-3, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 3: Hydrofluoric acid and particulate fluorides*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

EN 12698-1, *Chemical analysis of nitride bonded silicon carbide refractories - Part 1: Chemical methods*

EN 12698-2, *Chemical analysis of nitride bonded silicon carbide refractories - Part 2: XRD methods*

3 Analytes and ranges

Analytes and ranges specified in this International Standard shall be as follows.

a) Total silicon (T. Si), range of 30 % to 70 % (mass fraction)

b) Total nitrogen (T. N), range of 30 % to 45 % (mass fraction)

c) Aluminium (Al), range of 0,001 % to 0,6 % (mass fraction)

d) Iron (Fe), range of 0,001 % to 0,6 % (mass fraction)

e) Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction)

f) Oxygen (O), range of 0,05 % to 5 % (mass fraction)

g) Carbon (C), range of 0,01 % to 6 % (mass fraction)

h) Fluorine (F), range of 0,001 % to 0,2 % (mass fraction)

i) Chlorine (Cl), range of 0,001 % to 0,2 % (mass fraction)

4 Preparation of test sample

The method of preparing samples shall be in accordance with ISO 8656-1 unless otherwise mutually agreed upon between the analyser and the customer.

4.1 Sampling

Take the sample in accordance with ISO 8656-1.

4.2 Drying

Take about 10 g of the sample into a flat-type weighing bottle (60 mm × 30 mm) and spread it uniformly over the bottom of the bottle. Place the bottle in an air bath at 110 °C ± 5 °C for 2 h without a lid, and then cool in a desiccator (desiccant: magnesium perchlorate for drying) with a lid for 1 h.

4.3 Weighing

Weigh the sample of the required quantity to the nearest 0,1 mg using a balance.

5 Apparatus and reagents

Unless otherwise specified in each determination, use ordinary laboratory apparatus for chemical analysis listed in ISO 26845, Clause 4, as necessary. Reagents should conform to the requirements of ISO 6353-1, ISO 6353-2, and ISO 6353-3, as appropriate. Unless otherwise specified in each determination, use corresponding reagents of analytical grade listed in ISO 26845, Clause 5, as necessary.

6 Blank test

Blank test shall be carried out by using identical quantities of reagents, conditions, and procedures throughout each determination to correct the analytical values obtained.

7 Determination of total silicon

7.1 Classification of determination methods

Total silicon shall be determined by either of the following methods. If analytical results with four figures are required, use the method A. If analytical results with two or three figures are required, the method B can be used.

- Method A: Fusion–dehydration/insolubilization separation–gravimetry and ICP-OES
- Method B: XRF using fused cast-bead method

7.2 Fusion-dehydration/insolubilization separation-gravimetry and ICP-OES

7.2.1 Principle

A sample is fused with alkaline carbonate and the melt is treated with an acid to separate into two parts of silicon, insoluble silicon and soluble silicon, by filtration. Insoluble silicon is determined using gravimetry as silicon dioxide converted after ignition, whereas soluble silicon in the filtrate is determined using ICP-OES. The sum of them represents the total silicon.

7.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

7.2.2.1 Water, of grade 1 or superior specified in ISO 3696.

7.2.2.2 Sodium carbonate, anhydrous, specified in ISO 6353-3 or that of higher grade.

7.2.2.3 Hydrochloric acid (1+1), (1+4), (1+50), prepared by diluting hydrochloric acid with water, respectively.

7.2.2.4 Sulfuric acid (1+1), (1+4), prepared by diluting sulfuric acid with water, respectively.

7.2.2.5 Cellulose powder.

7.2.2.6 Polyethylene oxide solution [0,05 % (m/V)], prepared by dissolving polyethylene oxide with water.

7.2.2.7 Hydrofluoric acid, concentration of 48 %.

7.2.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for chemical analysis in accordance with ISO 26845, Clause 4.

7.2.3.1 Platinum dish.

7.2.3.2 Platinum crucible.

7.2.3.3 Burner, capable of heating at 1 100 °C.

7.2.3.4 Muffle furnace, capable of being operated at 1 100 °C.

7.2.3.5 Balance, readable to 0,1 mg.

7.2.3.6 Inductively coupled plasma optical emission spectrometer (ICP-OES).

7.2.4 Procedure

The procedure shall be as follows. The procedure described in ISO 21068-2, Clause 8 can be alternatively used.

7.2.4.1 Fusion of sample

Weigh 0,30 g of the sample and 2,0 g of sodium carbonate, anhydrous into a platinum dish and mix well. Start to heat carefully and increase the temperature gradually to 1 000 °C to completely fuse the sample using a burner or in a muffle furnace.

7.2.4.2 Separation of silicon

Add 20 ml of hydrochloric acid (1+1) to dissolve the melt on a hot plate. Silicon dioxide will appear to be jellified and precipitated at this stage. There are two methods to separate the precipitated silicon dioxide.

a) Dehydrate carefully the precipitate to dryness in order to prevent it from spattering and add 5 ml of hydrochloric acid and 20 ml of water to dissolve any salt mixed with the precipitate. Filtrate the precipitate with a filter paper and wash with hot hydrochloric acid (1+50) several times and then with hot water sufficiently until it contains no salt. Receive the filtrate and washings together in a volumetric flask and make constant volume. Preserve this precipitate for gravimetry of insoluble silicon and the solution for the ICP-OES determination of soluble silicon, respectively.

b) After eduction of jellified silicon dioxide, add 0,05 g of cellulose powder and 10 ml of polyethylene oxide solution to agglomerate silicon dioxide for easy filtration. Filtrate and wash in the same procedure, and then preserve this precipitate for gravimetry of insoluble silicon and the solution for the ICP-OES determination of soluble silicon, respectively.

7.2.4.3 Gravimetry for insoluble silicon

Transfer the precipitate embedded in the filter paper together into a platinum crucible and ignite at 1 100 °C after charring and ashing the paper. Weigh the crucible. Moisten the precipitate in the crucible with a few drops of water and sulfuric acid (1+1) and add 10 ml of hydrofluoric acid. Then evaporate to dryness on a hot plate to remove all of silicon dioxide, ignite it at 1 100 °C and weigh the crucible again. The loss of mass after hydrofluoric acid treatment shall be the amount of insoluble silicon dioxide.

7.2.4.4 ICP-OES for soluble silicon

Aspirate an aliquot of the preserved solution into an Ar plasma of ICP-OES to determine soluble silicon in the sample.

7.2.5 Blank test

Run blank determinations according to the operations of 7.2.4.1 to 7.2.4.4 without taking a sample.

7.2.6 Drawing calibration curve

For ICP-OES, prepare calibration solutions to span the range of concentration of silicon in the test solution. Each calibration solution shall have a similar matrix to the test solution.

With those calibration solutions, draw calibration curves for soluble silicon to establish the relation between the emission intensity and the amount of silicon.

7.2.7 Calculation

With the amount of insoluble silicon in 7.2.4.3, soluble silicon in 7.2.4.4 and the blank test in 7.2.5, calculate the content of total silicon according to Formula (1).

$$T.Si = [\{(m_1 - m_0) + (A_1 - A_0)\} / m\,] \times 0{,}4674 \times 100 \tag{1}$$

where

$T.Si$ is the content of total silicon in the sample, % (mass fraction);

m_1 is the amount of insoluble silicon dioxide in the sample, g;

m_0 is the amount of insoluble silicon dioxide in the blank test, g;

A_1 is the amount of soluble silicon dioxide in the sample, g;

A_0 is the amount of insoluble silicon dioxide in the blank test, g;

m is the weighed amount of the sample, g.

7.3 XRF using fused cast-bead method

The procedure shall be in accordance with EN 12698-2.

8 Determination of total nitrogen

8.1 Classification of determination methods

Total nitrogen shall be determined by either of the following methods. If analytical results with four figures are required, use the method A or C. If two figures are required, the method B can be used.

— Method A: Acid pressure decomposition–distillation separation–acidimetric titration method

— Method B: Inert gas fusion–thermal conductivity method

— Method C: Fusion–ammonia separation–acidimetric titration method

8.2 Acid pressure decomposition-distillation separation-acidimetric titration method

8.2.1 Principle

A sample is decomposed in a pressure decomposition vessel with a mixture of hydrofluoric acid and sulfuric acid to convert nitrogen into ammonia. Add boric acid and transfer the solution into a distillation flask. Add sodium hydroxide and perform steam distillation. React the distilled ammonia with a known amount of amidosulfuric acid and back-titrate the excess of amidosulfuric acid with a standardized sodium hydroxide solution.

8.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

8.2.2.1 Water, of grade 1 or superior specified in ISO 3696.

8.2.2.2 Hydrofluoric acid.

8.2.2.3 Sulfuric acid.

8.2.2.4 Sodium hydroxide, more than 97,0 % (mass fraction) of purity.

8.2.2.5 Sodium hydroxide solution (500 g/l), prepared by dissolving sodium hydroxide in water.

8.2.2.6 Amidosulfuric acid, more than 99,0 % (mass fraction) of purity.

8.2.2.7 0,1mol/l amidosulfuric acid solution, prepared by weighing 10,0 g of amidosulfuric acid and dissolving in water to make 1 000 ml. Calculate the factor of this solution according to Formula (2).

$$F = m \times P/(9{,}7095 \times 100) \tag{2}$$

where

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

m is the weighed amount of amidosulfuric acid, g;

P is the purity of amidosulfuric acid, % (mass fraction).

8.2.2.8 0,1 mol/l sodium hydroxide solution, prepared by dissolving sodium hydroxide in water in accordance with ISO 21068-3, 5.2.2.8. Take exactly 50 ml of the 0,1 mol/l amidosulfuric acid solution in a beaker (200 ml) and dilute with water to about 100 ml. Titrate this solution with the 0,1 mol/l sodium hydroxide solution using a pH meter. Take the end point as pH 5,5 and determine the volume of the titrant consumed. Calculate the factor of this solution according to Formula (3).

$$F_1 = F \times 50{,}00/V \tag{3}$$

where

F_1 is the factor of the 0,1 mol/l sodium hydroxide solution;

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

V is the titration volume of the 0,1 mol/l sodium hydroxide solution, ml.

8.2.2.9 Boric acid.

8.2.2.10 Ammonium sulfate, more than 99,9 % (mass fraction) of purity.

8.2.3 Apparatus

Use ordinary laboratory apparatus for chemical analysis and the following.

8.2.3.1 Platinum crucible.

8.2.3.2 Pressure decomposition vessel, on the market.

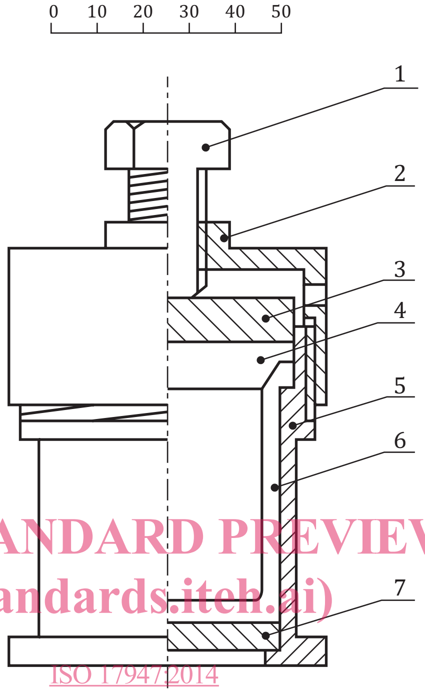
An example is shown in Figure 1. Use the vessels for the exclusive use in this analysis only to avoid cross-contamination by nitrogen. If the vessel which has ever contacted with nitric acid is used, the lower values of nitrogen can be obtained.

8.2.3.3 Air bath, capable of heating at 160 °C ± 5 °C.

8.2.3.4 Steam distillation apparatus, consisting of the components listed below.

An example of the apparatus is shown in Figure 2. Each component shall be made of hard glass coupled by common ground joints and fixed by springs or clamps just as per ISO 21068-3, Figure 1.

Dimensions in millimetres



Key

1	centre screw
2	screw cap
3	top plate
4	PTFE cap
5	cylinder
6	PTFE bottle
7	bottom plate

Figure 1 — An example of sealed decomposition vessel