Advanced technical ceramics — Ceramic powders — Determination of crystalline phases in zirconia

ICS 81.060.30

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National foreword

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It is being issued in the Draft for Development series of publications and is of a provisional nature because it is considered that further experience is required in its application before it is converted into a European and hence British Standard. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the European organization responsible for its conversion into a European Standard. A review of this publication will be initiated 2 years after its publication by the European organization so that a decision can be taken on its status at the end of its three-year life. The commencement of the review period will be notified by an announcement in *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into a European Standard, to extend the life of the prestandard or to withdraw it. Comments should be sent in writing to the Secretary of BSI Technical Committee RPI/13, Advanced technical ceramics, at 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

Summary of pages

This document comprises a front cover, an inside front cover, the ENV title page, pages 2 to 14, an inside back cover and a back cover.

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English version

Advanced technical ceramics - Ceramic powders - Determination of crystalline phases in zirconia

Céramiques techniques avancées - Poudres céramiques - Détermination des phases cristallines dans la zircone

Hochleistungskeramik - Keramische Pulver - Bestimmung der kristallinen Phasen in Zirconiumoxid

This European Prestandard (ENV) was approved by CEN on 11 April 2002 as a prospective standard for provisional application.

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Contents

Foreword

This document (ENV 14273:2002) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

Annex A is normative.

This Prestandard includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Prestandard specifies a method for the routine qualitative and quantitative determination of the crystalline phases (monoclinic, tetragonal and cubic) present in zirconia powders using X-ray powder diffraction.

2 Normative references

This European Prestandard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

[EN ISO/IEC 17025](http://dx.doi.org/10.3403/02033502U) General requirements for the competence of testing and calibration laboratories [\(ISO/IEC 17025:1999\)](http://dx.doi.org/10.3403/02033502).

3 Principle

The qualitative and quantitative determination of the crystalline phases present in a given zirconia powder is derived from the X-ray diffraction analysis of a representative sample of this powder.

The qualitative analysis relies on a comparison of the recorded spectrum with available reference data (e.g. ICDD-JCPDS data base).

For the quantitative analysis, two methods are used: the polymorph method and the full pattern method. The latter consists of determining scale factors by a least squares procedure involving all recorded diffraction peaks [1,2]. The former is a well-established method, details of which can be found in basic text books [3]; it relies on a comparison of the measured intensities of selected reflections from the previously identified phases.

NOTE A third method available for quantitative analysis is the Rietveld method. This method determines the phase constitution of the sample by fitting the whole X-ray diffraction spectrum through an iterative refinement approach. This refinement can be limited to the scale factors of each phase present (i.e. the volume fraction of the phases) or can address a whole set of parameters (e.g. cell parameters, atomic positions). Although it is

recognised that Rietveld refinement is probably the most reliable method and is more and more proposed in commercial XRD packages for quantitative analysis, this method is at present beyond the scope of this Prestandard.

4 Apparatus

Data acquisition is performed using a Bragg-Brentano diffractometer. The following experimental settings are recommended:

- 3⁄4 Copper X-ray tube;
- $\frac{3}{4}$ Precise goniometer (two-theta error 0,5°);
- $\frac{3}{4}$ Primary soller slit with a divergence 2,5°;
- $\frac{3}{4}$ 5 Divergence slit 1°;
- $\frac{3}{4}$ Receiving slit 2 mm;
- $\frac{3}{4}$ Scatter slit 1°:
- 3⁄4 Secondary monochromator:
- 3⁄4 Narrow line focus.

NOTE In those cases where the sample preparation leads inevitably to an inhomogeneous grain structure and/or composition (e.g. grains with an inhomogeneous radial phase distribution), the use of an X-ray tube with shorter wavelength, for instance, a molybdenum X-ray or silver X-ray tube, is recommended. The shorter X-ray wavelength provides a greater depth of penetration resulting in a more representative analysis in these cases.

5 Sample preparation

In order to avoid primary extinction and assure good statistics, the powder to be analysed shall have a homogeneous grain size less than 40 μ m.

By an adequate sampling procedure, a representative test sample of this powder is taken and pressed into the cavity of the sample holder. Use the backfill pressing technique to obtain a flat and smooth sample surface and to reduce preferred orientation. The dimensions of the sample holder will be such that X-rays irradiating outside of the sample volume can be avoided.

NOTE If the powder is obtained from a solid monolithic sample by comminution (e.g. using a crushing jaw in combination with a vibratory mill or alike) it is necessary to be aware that the sample preparation may affect the type and amount of the crystalline phases present and that, accordingly, the phase composition determined may be irrelevant.

6 Test procedure

Set the excitation voltage to at least 40 kV and the current intensity at the anode of the copper X-ray tube to at least 35 mA.

NOTE If a different X-ray tube is used, change the excitation parameters accordingly.

Data acquisition is preferably performed in the step-scanning mode, the step size being set between 0,01° and 0,05° two-theta. Alternatively record the spectrum under continuous mode with an integration width of 0,01 or 0,02.

6.1 Qualitative analysis

Set the scanning range from 10 to 80° two-theta and record the whole X-ray diffraction pattern. Select a counting time such that a good peak to background signal ratio is obtained. Practically, it should be sufficiently long to obtain a $_{rel}$ 0,02 for the maximum peak in the range scanned. The relative standard deviation, $_{rel}$, is given by

$$
\sigma_{\text{rel}} = \frac{\sqrt{N_{\text{t}} + N_{\text{b}}}}{N_{\text{t}} - N_{\text{b}}} \tag{1}
$$

with N_t the total counts and N_b the counts of the background.

6.2 Quantitative analysis – Method A: Polymorph method

For the polymorph method collecting the data under the following specific conditions is recommended.

Define two measurements programs:

- 34 The first, scan between 26,5 ° and 33,5 ° two-theta, with a counting time suitable for achieving a_{rel} 0,01 on the maximum peak;
- $\frac{3}{4}$ The second, scan between 70 ° and 77 ° two-theta, with a counting time suitable for achieving $_{rel}$ 0,02 on the maximum peak;

6.3 Quantitative analysis – Method B: Full pattern method

For the full pattern method the X-ray spectrum recorded for the preliminary qualitative analysis shall be used.

7 Qualitative and quantitative analysis of the recorded X-ray diffraction pattern

7.1 Qualitative analysis

Use an automatic or manual search and identify the crystalline phases present according to the ICDD-JCPDS PDF reference databank.

NOTE The following data sets are recommended, based on the present available information. However, as the ICDD-JCPDS PDF databank is regularly upgraded, for an exact analysis, updated data sets should be used.

7.2 Quantitative analysis – Method A: Polymorph method

7.2.1 Calculation

Calculation of the integrated intensities I(h k I)_{phase}

The intensities $I(h k I)_{phase}$ to be used in subsequent calculation are the integrated intensities computed from fitted and separated peaks.

NOTE 1 Due to overlapping of the peaks of different phases it is recommended for the determination of peak intensities to use a profile fitting method. The program used should incorporate a profile shape function sufficiently flexible (e.g. Pseudo-Voigt, Voigt or Pearson VII; not only Gaussian or Cauchy). Profile fitting programs are available in most of the instrumental software packages on the market. Alternatively they can be obtained from various sources [4].

NOTE 2 The use of graphical methods is in all cases discouraged.

Calculation of the intensity factors R(h k l) phase

A reliable determination of the crystalline phase composition of the powder sample by the polymorph method requires the use of correct intensity factors values. This in turn implies a precise knowledge of the crystallographic structure of the phases present.

The recommended intensity factor values are reported in Table 1 below. For any other zirconia/stabiliser system and/or reflection, derivation of the intensity factors should be done according to equation (2) using relevant crystallographic data available from the literature.

$$
R(hkl)_{\text{phase}} = \frac{LP(2\theta) \cdot p_{\text{hkl}} \cdot |F_{\text{hkl}}|^2}{N^2} \quad (2)
$$

with $LP(2)$ the Lorentz-Polarization factor, corrected if needed for the influence of the monochromator crystal, p_{hkl} the multiplicity factor of the specific reflection, F_{hkl} the structure factor and V, the volume of the unit cell. For a detailed explanation of these factors, one should refer to the "International Tables for X-ray Crystallography" [5].

Table 1 — Recommended intensity factors for selected reflections

NOTE 1 The above equations assume the stabiliser content is known, for example as a result of a preliminary chemical analysis of the powder (e.g. by X-ray fluorescence analysis).

NOTE 2 In the case in which the stabiliser amount is unknown, the stabiliser content in the tetragonal and cubic phases can be computed from the peak positions of selected reflections, provided that the latter are well determined by fitting and that any 2-theta errors (e.g. zero shift) are minimised.

Calculation of the volume fractions of the identified crystalline phases

The calculation of the crystalline phase composition requires a preliminary identification of the phases present in the specimen. Depending on the phases identified (monoclinic and cubic, monoclinic and tetragonal, cubic and tetragonal or monoclinic and cubic and tetragonal) use one of the set of equations described below.

Case 1: The mixture consists of two phases

Case 1A: Monoclinic and Cubic or Monoclinic and Tetragonal

Calculate the volume fraction of the monoclinic and of the quadratic (either cubic or tetragonal) phase using the following expressions respectively

$$
f_{\rm m} = \frac{k_1 X}{1 + (k_1 - 1)X} \tag{3}
$$

and

$$
f_{\text{quadra}} = 1 \quad f_{\text{m}} \tag{4}
$$

The integrated intensity ratio X is calculated from

$$
X = \frac{I(111)_{m} + I(111)_{m}}{I(111)_{m} + I(111)_{m} + I_{quadrat}} \tag{5}
$$

 I_{quad} being either $I(111)_c$ or $I(101)_t$, whereas the correction factor, k_1 , is obtained from the respective intensity factors by

$$
k_1 = \frac{R_{\text{quadra}}}{R(111)_{\text{m}} + R(111)_{\text{m}}}
$$
 (6)

with R_{quadra} being either $R(111)_{\text{c}}$ or $R(101)_{\text{t}}$.

Case 1B: Cubic and Tetragonal

Calculate the volume fraction of the tetragonal and cubic phases using the following expressions respectively

$$
f_{t} = \frac{k_{2}Y}{1 + (k_{2} - 1)Y}
$$
 (7)

and

$$
f_{\rm c} = 1 \quad f_{\rm t} \tag{8}
$$

The integrated intensity ratio Y is given by

$$
Y = \frac{I(004)_t + I(220)_t}{I(004)_t + I(220)_t + I(111)_c}
$$
\n(9)

The correction factor, k_2 , is calculated using the relevant intensity factors as

$$
k_2 = \frac{R(111)_c}{[R(004)_t + R(220)_t]}
$$
\n(10)

Case 2: The mixture consists of three phases

Calculate the respective volume fraction of the cubic, tetragonal and monoclinic phase using equations 11 to 13

$$
f_c = \frac{1}{1 + k_2 \frac{Y}{1 - Y} + k_{1c} + k_{1t} \cdot k \frac{Y}{1 - Y} \frac{X^*}{1 - X^*}}
$$
(11)

$$
f_{\rm t} = k_2 \frac{Y}{1 - Y} f_{\rm c} \tag{12}
$$

and

$$
f_{\rm m} = 1 \quad f_{\rm t} \quad f_{\rm c} \tag{13}
$$

with the integrated intensity ratio, X^{\dagger} and the correction factors k_{1t} and k_{1c} calculated using equations (14) and (15) respectively.

$$
X^* = \frac{I(111)_m + I(111)_m}{I(111)_m + I(111)_m + I(101)_t + I(111)_c}
$$
(14)

$$
k_{1t} = \frac{R(101)_t}{R(111)_m + R(111)_m} \text{ and } k_{1c} = \frac{R(111)_c}{R(111)_m + R(111)_m}
$$
(15)

The integrated intensity ratio Y and the correction factor k_2 are as previously defined (see Case 1B).

Calculation of the weight fractions of the identified crystalline phases

The weight fraction of a given phase is then calculated as follows

$$
w_{\text{phase}} = \frac{\rho_{\text{phase}} \cdot f_{\text{phase}}}{\hat{\mathbf{q}}_{i} \cdot f_{i}} \tag{16}
$$

with f_i , the calculated volume fraction of phase i, and \bar{f} the corresponding density value, the summation being done on all detected phases.

NOTE Density values are usually reported on the JCPDS-PDF card or can alternatively be computed from the known crystallographic data of the phase(s).

7.3 Quantitative analysis – Method B: Full pattern method

7.3.1 Symbols and definitions

 $I_{\text{mes,i}}$ measured intensity of the ith recorded peak;

- $I_{rel, i}$, relative intensity of the ith reflection of phase ;
- K correcting factor;
- X content of phase in weight percentage;
- μ ^{λ} mass extinction coefficient of sample;
- RIR reference intensity ratio of phase ;
- m number of phases present in sample;
- n number of measured peaks;
- S scaling factor of phase.

7.3.2 Calculation

In the full pattern method, the intensity of the ith recorded peak is considered as the sum of individual contributions of the m different phases present according to

$$
I_{\text{mes,i}} = \stackrel{m}{\overset{\mathbf{a}}{\mathbf{a}}} S \quad J_{\text{rel,i,}} \tag{17}
$$

with S, the scale factor of phase, defined as

$$
S = \frac{K.RIR \quad X}{\mu^*} \tag{18}
$$

NOTE The relative intensities $I_{rel,1}$ of the phase are known from the corresponding ICDD-JCPDS PDF reference data. As previously stated, updated data sets should be used for a reliable analysis.

Applying relation (17) for each recorded peak, a set of n linear equations is generated.

The scale factors corresponding to the crystalline phases present are then calculated using a least squares multivariate regression method i.e. by minimising the function

$$
\stackrel{\mathbf{n}}{\mathbf{a}} \mid I_{\text{mes},i} \quad \stackrel{\mathbf{m}}{\mathbf{a}} \quad S \quad \mathbf{X}_{\text{rel},i} \quad \mid^2 \qquad \text{min}
$$
 (19)

Finally, the individual weight fraction of the crystalline phases present can be derived from the calculated scale factors according to equation (19)

$$
X_{\text{phase}} = \frac{S_{\text{phase}}}{\text{RIR}_{\text{phase}}} \sum_{=1}^{m} \frac{\text{RIR}}{S}
$$
 (20)

The calculation relies on the precise knowledge of the Reference Intensity Ratio (RIR value) of the identified crystalline phases. These values can be found, quoted as I/I_{cor}, in the corresponding JCPDS PDF datasheet. Based on presently available data, the present European Prestandard recommends the use of the average RIR values reported in Table 2 below.

NOTE 1 For a more precise analysis and/or in the cases of other zirconia/stabiliser system, one should refer to the specific JCPDS PDF data.

NOTE 2 Alternatively the RIR values can be determined experimentally [8] provided pure samples of the different phases identified are available.

7.4 Limitations to the quantitative analysis

7.4.1 General limitations

The cubic and tetragonal phases cannot be separated if the two phases are strongly overlapped and the mean crystallite size is smaller than 10 nm. In the case of strong overlapping, cubic percentage or tetragonal percentage lower than 5 % to 10 % cannot be accurately estimated. If strong preferred orientations are present the results are not acceptable.

7.4.2 Method A: Polymorph method

If the monoclinic phase has a volume fraction larger than 20 %, the separation of cubic and tetragonal phases is not valid in the present formulation. The separation of the monoclinic phase with respect to the others is possible in this case using equations (3) and (4) and a correction factor calculated on the base of an estimated stabiliser concentration (the error in the monoclinic computation is very little).

When the crystallites are smaller than 20 nm and the cubic and tetragonal phases show strong overlapping, the determination of a cubic percentage lower than 10 % is difficult.

If other phases are present and in significant abundance with peaks in the measuring ranges, the analysis by this method is not possible.

7.4.3 Method B: Full pattern method

No additional limitations with respect to the general requirement are linked to this method. In general the accuracy by the full pattern method is better than that of the polymorph method.

8 Test report

The test report shall be prepared in accordance to [EN ISO/IEC 17025](http://dx.doi.org/10.3403/02033502U) and shall include the following information:

- a) name of the testing establishment;
- b) place and date of the test, report identification and signatory;
- c) a reference to this Prestandard, i.e. "determined in accordance with [ENV 14273](http://dx.doi.org/10.3403/02583578U)";
- d) powder identification, method of test specimen sampling and preparation;
- e) details of the apparatus and of the experimental parameters used when recording the diffraction pattern (radiation type used, voltage, current, 2 theta range, step size, accumulation time …) as well as a description of the software and databank used;

8.1 Qualitative analysis

In addition to the previous items, the test report shall include

f) the list of identified crystalline phases with the mention of the corresponding ICDD-JCPDS PDF numbers;

NOTE If possible a plot showing the recorded spectrum together with the identification of the crystalline phases detected should be provided. The phases present may be indicated by means of bars overlapping or separated from the spectrum, or by others graphical methods clearly readable.

8.2 Quantitative analysis

In the case of a quantitative phase analysis, the report shall in addition provide for each crystalline phase detected

g) the weight percentages or fractions, or the volume percentages or fractions for each crystalline phase identified with an estimation of the error or the standard deviation of the results, after a number of results have been obtained.

Thereupon, and although optional, the following data could be reported

h) the values of the integrated intensities, $I(hkI)_{phase}$, used in the calculation;

NOTE Reporting the profile shape function used and a list of parameters (e.g. peak position, peak height, FWHM) resulting from the peak fitting procedure with estimated standard deviations is advised.

- i) the known or determined stabiliser nature and content;
- j) the corresponding values of intensity factors, $R(hk)_{\text{phase}}$, used in the calculation;

Annex A

(normative)

Determination of stabiliser amount from cell parameters

In the case in which the stabiliser content is unknown, the stabiliser content in the tetragonal and cubic phases can be easily computed from the peak positions provided that they are well determined by fitting and minimising the 2 theta errors.

Yttria stabiliser [6]

Compute the cell parameters c and a for the tetragonal phase from the two peaks (004) and (220); then the stabiliser content in the tetragonal phase can be computed from the following formula:

$$
YO_{1,5} \text{mol.}\% = \frac{5,1892}{0,227 \frac{c}{a} + 0,256} \times 100
$$

NOTE The error on this determination is very low due to the fact that the ratio c/a minimises the goniometer and fitting errors.

For the stabiliser content in the cubic phase, compute the cell parameter a from the position of the peak (400). Compute the stabiliser content using the calculated parameter a in the formula:

$$
YO_{1,5} \text{mol.}
$$
% = $\frac{a}{0.0204} \times 0.001 \times 100$

Magnesia and Calcia stabiliser

In the case of magnesia and calcia stabilized zirconia, similar formula allowing the determination of the stabiliser content in the cubic phase can be found in [7].

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