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Advanced technical ceramics - Methods of test for ceramic coatings - Part 4: Determination of chemical composition

Advanced technical ceramics - Methods of test for ceramic coatings - Part 4: Determination of chemical composition

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 4: Bestimmung der chemischen Zusammensetzung

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Céramiques techniques avancées pour revetements céramiques - Méthodes d'essai - Partie 4: Détermination de la composition chimique

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European Committee for Standardization
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Foreword

This European pre-standard has been prepared by the Technical Committee CEN/TC 184 " Advanced technical ceramics" of which the secretariat is held by BSI.

ENV 1071 'Methods of test for ceramic coatings' consists of five Parts:

Part 1 : Determination of coating thickness by contact probe profilometer
Part 2 : Determination of coating thickness by cap grinding method
Part 3 : Determination of adhesion by a scratch test
Part 4 : Determination of chemical composition
Part 5 : Determination of porosity

CEN/TC184 approved this European pre-standard by resolution 3 during its fifth meeting held in Brussels on 1 April 1992.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European pre-standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

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1 Scope

This Part of ENV 1071 describes a method for chemical analysis of ceramic coatings by means of electron probe microanalysis (EPMA) using a scanning electron microscope (SEM) or an electron-probe microanalyser.

The methods described are limited to the examination of single layer coatings when the analysis is carried out normal to the sample surface, but graded and multilayer coatings may also be analysed in cross-section if the thickness of the individual layers or gradations are greater than the maximum width of the volume of material within which characteristic or fluorescent X-rays are generated.

NOTE : This method would also apply to the analysis of bulk materials.

2 Normative References

This European standard 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 listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

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EN 45001: General criteria for operation of testing laboratories.

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ENV 623-4: Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 4: Determination of surface roughness.

3 Definitions

For the purposes of this standard, the following definitions apply:

3.1 thin coatings: a coating with a thickness $<$ 20 mm.

3.2 thick coatings: a coating with a thickness $>$ 20 mm.

NOTE : Terminology used in this standard if not defined herein is according to the International vocabulary of basic and general terms in metrology.

4 Significance and use

EPMA is a commonly used analytical technique which is applicable to a wide range of materials in bulk form. Unfortunately no standard currently exists for this procedure so that the definitions, operating principles and procedures and calibration of the instruments used is described in Annex A; definition of terms, including those used in this standard that are not specific to coatings are also given in Annex A.

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The composition of a coating is a critical factor determining the performance of a product, thus this analytical procedure can be used in quality control, coating development, characterisation and for design data acquisition purposes. However there are many other analytical procedures that can be used to determine the composition of coatings. The particular method used will depend in large measure on the coating thickness and elemental atomic number of the coating constituents. Tables 1 and 2 list some of the electron, ion and X-ray beam analytical methods applicable to coatings and their characteristics.

The results obtained using EPMA can depend on the coating's conductivity, thickness, roughness and porosity, so that as detailed in clauses 6 and 7 care is required in interpretation and in preparation of the testpiece.

The size of the testpiece will vary depending on the geometry of the instrument used for the analysis. In general, however, modern instruments will allow analysis of testpieces of up to about 150 mm x 150 mm x 50 mm.

5 Test Instrument

Various commercial instruments are available which are based on a general purpose scanning electron microscope or a purpose built electron microprobe analyser; the former is more commonly used. The instruments deliver a finely focused beam of electrons to the testpiece which generates X-rays which are then analysed with either an energy or wavelength dispersive X-ray detector, and the operation of these detectors is described in detail in A2.2.

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6 Principles

6.1 Analysis of thin coatings

6.1.1 When analysing thin coatings with the electron beam normal to the sample surface, it is important to appreciate that for the lower thickness range under certain conditions X-rays could be generated from the substrate as well as the coating. The depth within the sample from which characteristic X-ray generation occurs is a function of the density of the material, Z, A, the electron beam energy and the critical excitation energy of the characteristic X-rays. Monte Carlo simulations have been most successful in calculating this depth, and various relationships have been proposed, one of which is given below:

$$z_r = 0,033(E_o^{1,7} - E_c^{1,7})\frac{1}{\rho}\frac{A}{Z}$$

where z_r is the depth, mm, from which characteristic X-rays are generated;
r is the density, g cm^{-3};
E_o is the electron beam energy, keV;
E_c is the critical excitation energy, keV, for production of characteristic X-rays;
A is the atomic weight,
Z is the atomic number.

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6.1.2 The above expressions do not include the effects of fluorescence excitation which may considerably enlarge the depth from which X-ray generation occurs. Fluorescence occurs when the energy of the characteristic peak is greater than E_c of an element of lower atomic number. For example Ni Ka would cause fluorescence of Fe Ka. In general, the effect is larger the greater the atomic number of the exciting element and the smaller the difference in atomic number between the exciting and the excited elements.

6.1.3 It is recommended that the highest electron beam energy be selected, up to an overvoltage of 3, consistent with the coating thickness in order to maintain maximum sensitivity and avoid errors to the presence of surface contaminants (eg carbon, oxide films, etc).

6.1.4 An alternative to reducing the beam energy might be to tilt the sample so that a greater volume of coating would be presented to the beam and, thus, changing interaction with the substrate. Such alterations to the operating conditions would need to be taken account of in quantitative analysis software - see sections A4.2.4.2 and A4.2.4.3.

6.2 Analysis multilayer coatings

6.2.1 Multilayer coatings may be analysed in cross-section provided that the thickness (width) of the individual layers is greater than the lateral resolution, d, which can be approximately defined by the following relationship:

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$$d = \frac{0,077}{\rho}(E_o^{1,5} - E_c^{1,5})$$

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where d, r, E_o and E_c are defined in 6.1.1.

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This relationship again only refers to characteristic X-rays and the effects of fluorescence would increase the lateral resolution.

6.3 Analysis of thick coatings

6.3.1 While analysis of thick coatings can be readily performed with the beam normal to the specimen surface, this procedure would not detect any change in composition with depth. Thus, where possible, cross-sections shall be prepared and then analysis shall be carried out at various points through the coating thickness. Sampling procedures shall be dictated by normal statistical methods.

6.3.2 Alternatively line scans can be employed to give semi-quantitative information on the variation of composition through the thickness of the coating.

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7 Specimen preparation

7.1 Surface roughness

7.1.1 The analytical results may vary markedly with specimen roughness due to variation of the local take-off angle (see section A3.1.1) and consequent different degrees of absorption of the characteristic X-ray as it leaves the sample (see figure 1). Thus, a smooth metallographically polished surface is required so that the take-off angle can be precisely defined. It is recommended that the final surface roughness should be as smooth as available experience and facilities permit, but at least to a 1 mm diamond finish (R_a values of better than about 0,1 μm).

7.1.2 It is recognised that some coatings have high hardness values which make the preparation of suitable surfaces difficult. In that event the surface roughness shall be quoted in the tabulation of the results as a R_a value.

7.2 Surface conduction

7.2.1 If the sample is an insulator, the sample will become negatively charged in the electron beam with consequent deflection of the beam and ill-defined analysis conditions will result. Specimen charging can also reduce the effective beam energy further contributing to errors in the quantitative analysis corrections (see section A4.2.4).

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7.2.2 These problems shall be overcome by coating the surface with a thin conducting layer. Commonly a layer of carbon of about 20 nm in thickness is used, but should this not be possible, for example, if the material to be analysed contains carbon, then it is suggested that aluminium or beryllium should be used. Thicknesses shall be similar to those used for carbon to prevent loss of X-ray signal by absorption in the conducting layer. For the most accurate quantitative analysis the same type and thickness of conducting layer shall be used for both the standard and the sample to be analysed.

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7.2.3 A conducting path shall be established between the surface and the metallic specimen holder in the SEM or microprobe. This is usually done by painting onto the sample a thin stripe of a conducting paint such that electrical contact is established directly with the sample holder, usually graphite or silver dag is used. These paints contain volatile solvents which shall be removed before placing the sample in the instrument, and it is recommended that the sample mounted on the holder is placed in a vacuum of at least 10^2 Pa (10^{-3} bar) for a period of at least 10 min.

7.2.4 Frequently conducting samples are mounted in non-conducting media and the same procedure using conducting paints shall be followed to establish electrical contact with the sample holder as described above.

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8 Test Procedure

Calibrate the instrument according to the procedures set out in annex A. Ensure that all systems are operating correctly, and introduce the testpiece after appropriate preparation as detailed in clause 7. In some cases, depending on the instrument manufacturer's recommendations, it will be necessary to have a suitable sample which can act as an internal standard to normalise data to the calibration. Analyse the testpiece in accordance with normal procedures developed for the instrument.

9 Accuracy and Interferences

9.1 Table 3 lists the limitations to the use of the different analytical methods described in detail in Annex A and the accuracy associated with each method.

9.2 Care is required when analysing some coatings using EDX where peak overlap can occur, for example, in the case of TiN the nitrogen Ka is very close to the titanium L series; this problem can be resolved by the use of WDX.

9.3 Another difficulty faced by the analyst would be the case of a ceramic coating which contains the same elements as the substrate, as for example aluminium oxide on aluminium.

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10 Test report

The test report shall be in accordance with EN 45001 and shall contain the following information:

a) name and address of the testing establishment;

b) date of the test, unique identification of the report and of each page, customer name and address and signatory;

c)a reference to this standard, ie determined in accordance with ENV 1071-4;

d) name of instrument used and the software package for analysis of results;

e) description of the test material (material type, manufacturing code, batch number, date of receipt and any other relevant information);

f) specimen condition (dimensions, coating type and number of layers, if known, sample preparation procedure and surface roughness in Ra values, if known, and substrate composition, if known);

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g) instrument conditions (beam energy - accelerating voltage, specimen tilt, detector take-off angle, detector azimuth, working distance, specimen to detector distance, calibration standard used, standards used in the analysis, spectrometer type, if wavelength, crystal(s) used, X-ray detector type, if gas proportional, gas composition and flow rate (for gas flow detectors).

h) any other relevant comments, noting, for example, where different procedures to those recommended have been used.

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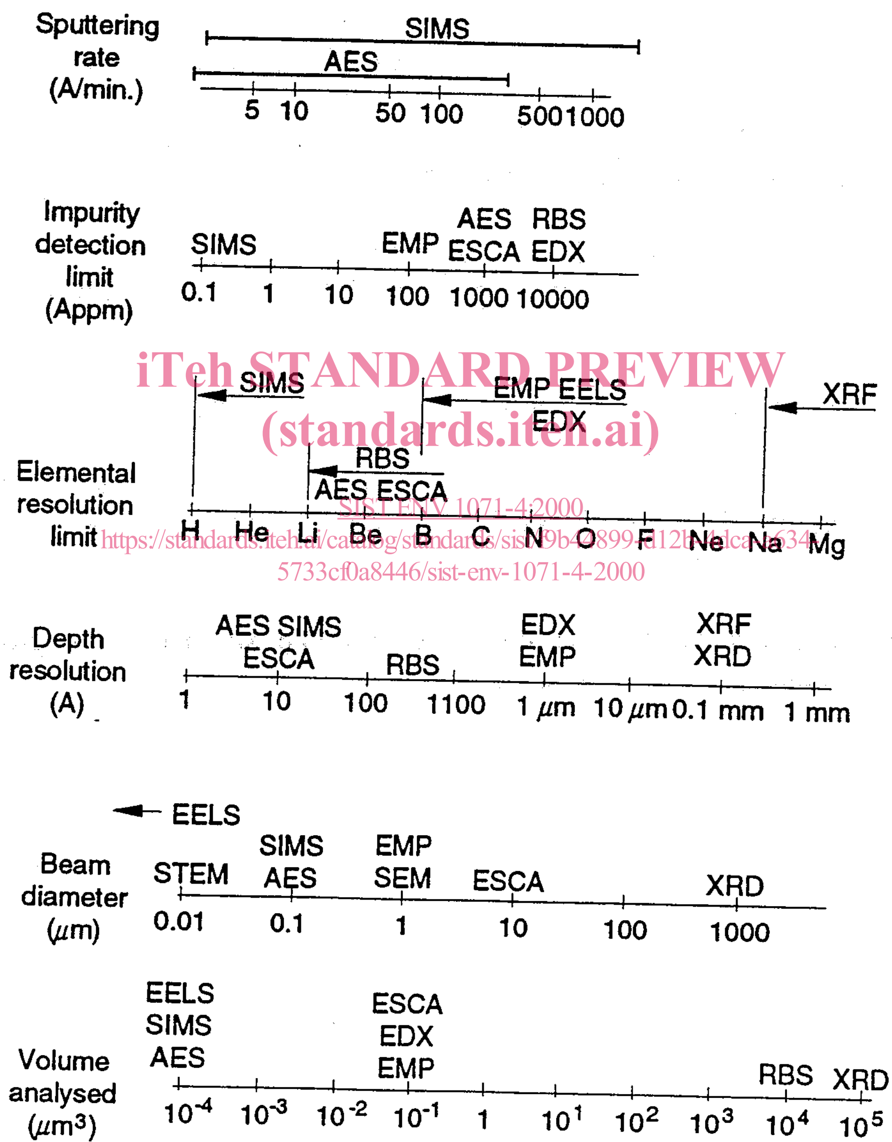
Table 1 : A comparison of various analysis methods



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AES Auger Electron Spectroscopy
DEX Energy Dispersive X-ray Analysis
EMP Electron Microprobe
ESCA X-ray phototelectron spectroscopy (XPS)
EELS Electron Energy Loss Spectroscopy
RBS Rutherford Backscatter Analysis
SIMS Secondary Ion Mass Spectroscopy
XRD X-ray Diffraction
XRF X-ray Fluorescence