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Sodobna tehnična keramika – Metode za preskušanje keramičnih prevlek – 4. del: Ugotavljanje kemične sestave z elektronsko mikroanalizo (EPMA)

Advanced technical ceramics - Methods of test for ceramic coatings - Part 4: Determination of chemical composition by electron probe microanalysis (EPMA)

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 4: Bestimmung der chemischen Zusammensetzung durch Elektronenstrahl-Mikrobereichsanalyse (ESMA)

Céramiques techniques avancées - Méthodes d'essais pour revetements céramiques - Partie 4 : Détermination de la composition chimique avec analyse par microsonde électronique (EPMA)

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 4: Determination of chemical composition by electron probe microanalysis (EPMA)

Céramiques techniques avancées - Méthodes d'essais pour revêtements céramiques - Partie 4 : Détermination de la composition chimique avec analyse par microsonde électronique (EPMA)

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This European Standard was approved by CEN on 30 December 2005.

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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 Central Secretariat has the same status as the official versions.

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Contents

Foreword

This European Standard (EN 1071-4:2006) 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of an identical text or by endorsement, at the latest by August 2006, and conflicting national standards shall be withdrawn at the latest by August 2006.

EN 1071 *Advanced technical ceramics – Methods of test for ceramic coatings* consists of 11 Parts:

Part 1: *Determination of coating thickness by contact probe profilometer*

Part 2: *Determination of coating thickness by the crater grinding method*

Part 3: *Determination of adhesion and other mechanical failure modes by a scratch test*

Part 4: *Determination of chemical composition by electron probe microanalysis (EPMA)*

Part 5: *Determination of porosity*

Part 6: *Determination of the abrasion resistance of coatings by a micro-abrasion wear test*

Part 7: *Determination of hardness and Young's modulus by instrumented indentation testing*

Part 8: *Rockwell indentation test for evaluation of adhesion*

Part 9: *Determination of fracture strain*

Part 10: *Determination of coating thickness by cross sectioning*

Part 11: *Determination of internal stress by the Stoney formula*

Parts 7 to 11 are Technical Specifications.

This European Standard supersedes ENV 1071-4:1995.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, 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.

Introduction

Electron probe microanalysis (EPMA) is a commonly used analytical technique which is applicable to a wide range of materials in bulk form. While international standards for this procedure have been developed under ISO/TC 202 'Microbeam analysis', currently no European or international standard considers the application of EPMA to ceramic coating analysis.

Terms and definitions, including those used in this European Standard that are not specific to this standard are given in ISO 18115 and in ISO 23833.

The composition of a coating is a critical factor determining the performance of a product, so this analytical procedure can be used in quality control, coating development, characterisation and for design data acquisition purposes.

Reference works on electron probe microanalysis are listed in the Bibliography [1, 2].

1 Scope

This European Standard describes methods 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 can also be used for the analysis of bulk materials.

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 623-4, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 4: Determination of surface roughness*

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

ISO 14594, *Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy*

ISO 15632, *Microbeam analysis — Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

NOTE Definitions of further terms are given in ISO 23833 and the International Vocabulary of basic and general terms in Metrology. [3]

3.1 General

3.1.1
thick coating
coating with a thickness > 20 µm

3.1.2
thin coating
coating with a thickness < 20 µm

3.2 Terms used in electron probe microanalysis

3.2.1
absorption correction
A
matrix correction arising from the loss of X-ray intensity of element "A" while propagating through the specimen in the direction of the X-ray spectrometer due to photoelectric absorption by all elements within the specimen

3.2.2
atomic number correction
Z
matrix correction for the modification of the X-ray intensity for element "A" due to electron backscattering and stopping power, which are influenced by all elements in the analytical volume

3.2.3
backscattered electrons
incident beam electrons that have been re-emitted from the specimen surface due to multiple scattering

NOTE They are of a high energy (up to the energy of the beam) and can provide atomic number contrast.

3.2.4
beam current
electron beam current, in A (measured using a Faraday cup near the position of the specimen in the instrument)

3.2.5
Bragg angle
θ
angle, in degrees, between the diffracting crystal surface and the X-rays being analysed

3.2.6
count rate
number of X-ray counts per second

3.2.7
critical excitation energy
E_c
minimum energy required to ionise an atom from a specific electron shell, in eV

3.2.8
dead time
length of time, in a measurement system that processes one event at a time, during which the system is engaged in processing a photon event ("busy"), such that it is unavailable to process another photon which randomly appears in this time interval

3.2.9
electron beam energy
E_o
energy of the electrons of the beam at the sample, in eV

3.2.10
electron probe microanalysis (EPMA)
technique of spatially-resolved elemental analysis based upon electron-excited X-ray spectrometry with a focused electron probe and an electron interaction volume with micrometer to sub-micrometer dimensions

3.2.11

energy dispersive X-ray spectrometry (EDS)

method for examining the intensity of X-rays as a function of the photon energy

3.2.12

fluorescence correction

F

correction applied to account for characteristic X-ray excitation by X-ray photons of higher energy

3.2.13

overvoltage

U

ratio of the incident beam energy to the critical excitation energy for a particular shell

NOTE This factor should be greater than unity for characteristic X-ray production to occur from that atomic shell.

3.2.14

$\Phi(\rho z)$ matrix correction

method of quantitative electron probe X-ray microanalysis in which correction factors are calculated from empirical equations developed from fits to experimental data of X-ray production as a function of depth {the so-called $\phi(\rho z)$ function}

3.2.15

peak overlap

merging of peaks of nearly the same energy which cannot be resolved by the detector

3.2.16

secondary electron

electron of the specimen emitted as a result of inelastic scattering of the primary beam electron with loosely bound valence-level electrons of the specimen

NOTE They are of a low energy (< 50 eV) and provide information about surface topography.

3.2.17

take off angle

ψ

angle, in degrees, between the specimen surface and the line of sight to the centre of the detector

3.2.18

wavelength dispersive X-ray spectrometry (WDS)

device for determining X-ray intensity as a function of the wavelength of the radiation, where separation is based upon Bragg's law, $n\lambda = 2d\sin\theta$, where λ is the X-ray wavelength, d is the spacing of the atom planes of the crystal or the repeated layers of the synthetic diffractor, and θ is the angle at which constructive interference takes place

NOTE X-rays diffracted at a particular angle are directed to a gas counter operated in the proportional response regime where the charge produced is proportional to the photon energy.

3.2.19

working distance

distance, in metres, from the principal plane of the objective lens to the cross-over of the focused probe

4 Principle

Analysis is carried out by means of an electron beam striking the sample and characterization of the X-rays subsequently emitted. The incident electrons eject an electron from the K, L or M shell of an