SLOVENSKI STANDARD
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Sodobna tehnična keramika – Metode za preskušanje keramičnih prevlek – 10. del: Ugotavljanje debeline prevleke s prečnim prerezom

Advanced technical ceramics - Methods of test for ceramic coatings - Part 10: Determination of coating thickness by cross sectioning

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 10: Bestimmung der Schichtdicke mittels Querschliff

Céramiques techniques avancées - Méthodes d'essai pour les revetements céramiques - Partie 10: Détermination de l'épaisseur du revetement par découpage transverse

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 10: Determination of coating thickness by cross sectioning

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Foreword

This document CEN/TS 1071-10:2004 has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

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

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*

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*[1)]

Part 9: *Determination of fracture strain*

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

Part 11: *Measurement of internal stress with the Stoney formula*[1)]

Parts 7 to 11 are Technical Specifications.

This document includes a bibliography.

1) In preparation at the time of publication of this document.

Introduction

The thickness of a coating is an important property that controls its functional behaviour. Thickness determinations are also used as part of quality control in the production of coatings. It is normal to specify a thickness when defining a coating, so that valid methods of measurement are required. The method described here is direct, but is destructive, requiring preparation of a metallographic cross-section. A number of other standard non-destructive methods exist and some of these are listed in the Bibliography (references [1] to [7]).

1 Scope

This document specifies a method of measuring the thickness of ceramic coatings by means of examination of a metallographically prepared cross-section of the coating in a calibrated optical or scanning electron microscope. It draws strongly on EN ISO 9220 [8], modifying and updating as required to be relevant to ceramic coatings and current best practice.

2 Normative references

The following referenced document is 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.

ENV 13005, *Guide to the expression of uncertainty in measurement*

3 Terms and definitions

For the purposes of this document, the following term and definition apply.

3.1
local thickness
mean of the thickness measurements, of which a specified number is made within a reference area [EN ISO 2064:2000, 3.4][5]

4 Principle

This test procedure covers the measurement of coating thickness by examination of a cross-section in an optical or scanning electron microscope. Preparation of the cross-section requires care to ensure that the total thickness is revealed and that when viewed it is normal to the axis of the microscope. After proper calibration of the microscope, it is a simple matter to determine the coating thickness from knowledge of the magnification used. This can be done directly using a modern measuring microscope, or indirectly from photographic images obtained from an optical or scanning electron microscope.

5 Apparatus

5.1 Scanning electron microscope (SEM)

The SEM shall have a spatial resolution of 50 nm or better. Suitable instruments are available commercially.

5.2 Optical microscope

The optical microscope shall have a spatial resolution of 500 nm or better. Suitable instruments are available commercially.

NOTE 1 Microscopes that incorporate a system to automatically record the XY coordinates are available and if the stage movement has been calibrated, can be used directly to measure coating thickness without the need to take photographs. This method is particularly useful where coating thickness variations around a component are likely.

NOTE 2 The choice of instrument will depend on the thickness of the coating to be measured and the accuracy required.

6 Sample preparation

6.1 Cross-section preparation

Prepare the cross-section so that:

a) it is perpendicular to the plane of the coating;

b) the surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for measurement;

c) all material damaged by cutting or cross-sectioning is removed;

d) the boundaries of the coating cross-section are sharply defined by no more than contrasting appearance, or by a narrow well defined line.

NOTE Further guidance is given in Annex A.

6.2 Surface roughness

If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating may be too irregular to permit accurate measurement of the average thickness in the field of view.

6.3 Taper of cross-section

If the plane of the cross-section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10 degrees to the perpendicular will contribute an error of 1,5 %.

NOTE It is recommended that a cross-section of a reference sample of known thickness be prepared using the same procedures as the test sample as a check on the accuracy of cutting, mounting and polishing procedures.

6.4 Specimen tilt

Any tilt of the specimen (plane of cross-section) with respect to the electron beam or optical axis will result in an inaccurate measurement. This error is compounded if the test specimen tilt is different from that used during calibration.

6.5 Coating damage

Ceramic coatings are generally brittle, and hence easily damaged during preparation of the metallographic cross-section.

6.6 Rounding of edges of the coating

If the edge of the coating cross-section is rounded, i.e. if the coating cross-section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing or etching (see 6.8 and Annex A).