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Advanced technical ceramics - Methods of test for ceramic coatings - Part 7: Determination of hardness and Young's modulus by instrumented indentation testing

Hochleistungskeramik – Verfahren zur Prüfung keramischer Schichten – Teil 7: Bestimmung der Härte und des Elastizitätsmoduls durch instrumentierte Eindringprüfung

Céramiques techniques avancées – Méthodes d'essai pour revêtements céramiques – Partie 7: Détermination de la dureté et du module de Young par essai de pénétration instrumenté

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Foreword

This document (CEN/TS 1071-7:2003) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document has been prepared under a mandate given to CEN by the European Commission.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, 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: Determination of adhesion by the Rockwell indentation test

Part 9: Determination of fracture strain

Part 10: Determination of coating thickness by cross section

Part 11: Determination of internal stress by the Stoney formula

Parts 7 to 11 are Technical Specifications.

This Technical Specification includes informative annexes A and B and a bibliography.

Introduction

The hardness and Young's modulus of a ceramic coating are critical factors determining the performance of the coated product. Indeed many coatings are specifically developed to provide wear resistance that is usually conferred by their high hardness. Measurement of coating hardness is often used as a quality control check. Young's modulus becomes important when calculation of the stress in a coating is required in the design of coated components. For example, the extent to which coated components can withstand external applied loads is an important property in the application of any coated system.

It is relatively straightforward to determine the hardness and Young's modulus of bulk materials using instrumented indentation, However, when measurements are made normal to a coated surface, depending on the load applied and the thickness of the coating, the substrate properties can influence the result. The purpose of this Technical Specification is to provide guidelines for conditions where there is no significant influence of the substrate, and, where such influence is detected, to provide possible analytical methods to enable the coating properties to be extracted from the composite measurement. In some cases the coating property can be determined directly from measurements on a cross-section.

Currently no standards exists to define usage of instrumented indentation testing of bulk materials, so that the operating principles and calibration of the instruments used is described in annex A. ISO 14577 Parts 1-3 are being drafted which cover instrumented indentation testing for the entire range from macro through micro- to nano-indentation experiments for bulk materials. The procedures detailed in Annex A complement those in the ISO standards, but place more emphasis on the nano/micro range applicable to thin coatings.

1 Scope

1.1 This part of EN 1071 describes a method of measuring hardness and Young's modulus of ceramic coatings by means of Instrumented Indentation Testing (IIT) using instruments capable of measuring force and displacement as a function of time during the indentation process. This class of instruments includes instruments previously known as "Depth Sensing Indenters, DSI" and "Mechanical Microprobes."

1.2 The method is limited to the examination of single layers when the indentation is carried out normal to the test piece surface, but graded and multilayer coatings can also be measured in cross-section if the thickness of the individual layers or gradations is greater than the spatial resolution of the indentation process. The latter is dependent upon instrument design and is determined by the displacement sensitivity and the precision of location of the indents.

2 Normative references

This Technical Specification 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 Technical Specification 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 17025: General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999).

ASTM D-1474 Indentation Hardness of Organic Coatings.

ASTM B578-87 Microhardness Testing of Electroplated Coatings (reapproved 1993). NR

ISO/DIS 14577-1 Instrumented Indentation Test for Hardness and Materials Parameters - Part 1: Test Method.

ISO/DIS 14577-2 Instrumented Indentation Test for Hardness and Materials Parameters - Part 2: Verification and Calibration of Test Machines.

ISO/DIS 14577-3 Instrumented Indentation Test for Hardness and Materials Parameters - Part 3: Calibration of Reference Blocks.

3 Terms and definitions

For the purposes of this Technical Specification, the following terms and definitions apply.

3.1
hardness, H
resistance to permanent deformation

EXAMPLE resistance to fracture damage is generally conferred *by higher toughness and ductility, or lower H*.

NOTE 1 With IIT, equation (1) in A.1.1 defines hardness as the maximum force, in Newtons, divided by the projected contact area (cross-section), in square metres, that the indenter makes with the test piece at maximum force and thus has the units of Pa. This definition is in accord with that generally agreed and first proposed by Meyer [27], and it should be observed that the projected contact area is assumed to remain constant during elastic unloading. (see Figures A.1 and A.2). This is an approximation and refinements to this approach are being developed [1].

NOTE 2 The term Martens Hardness, HM, (previously Universal Hardness) has been recently agreed to describe the total deformation during indentation and is the maximum force divided by the surface area of the indenter penetrating beyond the initial surface of the test piece at maximum force. Thus, this definition includes both plastic and elastic deformation of the test piece. (see Figures A.1 and A.2).

NOTE 3 It is important to use the correct area function. Indentation modulus, E_{IT} and indentation hardness, H_{IT}, both require calculation of the cross-sectional (Projected) area, $A_p(h_c)$, of the indenter that is in contact with the test-piece whilst under maximum load. HM uses a calculation of surface area, $A_s(h_{max})$, of the indenter but does not attempt to model the bowing of the surface and makes the simplifying assumption that all of the indenter penetrating below the original surface is involved. Vickers hardness, HV, measures the projected area of the residual indent and then calculates the surface area of a perfect Vickers pyramid with the same projected area. This is roughly equivalent in cases of nearly perfectly plastic materials (e.g. metals) to a function $A_s(h_c)$ and so there can be a scaling factor equivalence between HV and H_{IT} for certain materials. In practice, the blunt tip of real indenters means that, as indentation depths are reduced, there is a divergence - HV becomes infinite and H_{IT} measures the mean pressure of indentation but ceases to be a measure of plasticity as the pressure drops below the plastic yield stress.

3.2
stiffness, S
contact stiffness - resistance to elastic deformation, slope of the unloading curve at maximum force (see Figure A.1)

3.3
contact depth, h_c
depth of the indenter in contact with the test piece at maximum force

NOTE This is commonly approximated by the tangent depth adjusted for indenter geometry $h_c = h_{max} - \varepsilon\,(h_{max} - h_r)$ where ε is 1 for a flat indenter, 0.73 for a conical and 0.75 for paraboloid (see Figure A.1)