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Sodobna tehnična keramika - Metode za preskušanje keramičnih prevlek – 7. del: Določanje trdote in modul elastičnosti (Youngov modul) z instrumentiranim vtiskanjem

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 revetements 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)

3.4

projected contact area, A_c

projected area (cross-sectional area) of the indenter at the contact depth

3.5

indenter area function

mathematical relationship or “look-up” table giving the projected area of an indenter as a function of distance from the tip

3.6

depth intercept of tangent to unloading curve, h_r

extrapolation of the tangent to the elastic unloading curve at maximum force to zero load (see Figure A.1)

3.7

residual penetration depth, h_p

depth of the residual indentation (see Figures A.1 and A.2)

3.8

frame stiffness, S_f

stiffness of the instrument frame (see clause A.3.3.2)

4 Apparatus and materials

Instrumented indentation allows the direct measurement of hardness and Young’s modulus from indentation experiments where the indentation process is continuously monitored with respect to force and displacement. The experiments can be performed under force or displacement control. A schematic diagram of typical equipment is shown in Figure 1. The principles of these instruments are described in detail in annex A, clause A.1.

A variety of instrumented indentation instrument types is commercially available which have a maximum force ranging between about 100 mN and 1000 mN; most of these can operate in either displacement or load control modes. Also many laboratory built instruments are in operation. For instruments complying with this Technical Specification it shall be possible to traceably measure force and displacement continuously during the loading and unloading cycle. It is also necessary to be able to determine the point of contact with the test piece surface.

Calibration procedures for instrumented indentation instruments are described in annex A, clause A.2.

While it is not a requirement, it is useful for an optical microscope to be incorporated in the instrument to allow selective positioning of the indents.

NOTE Instrumented indentation apparatus equipped with AFM to assess the indent shape allows the determination of possible pile-up or sink-in of the surface around the indent. These surface effects result in an increase (pile-up) or decrease (sink-in) of the contact area and hence may influence the measured results. Pile-up generally occurs for fully work hardened materials. Pile-up of soft, ductile materials is more important for thinner coatings due to the constraint of the stresses in the coating zone of plastic deformation. It has been reported that the piled up material results in an effective increase of the contact area for the determination of hardness, while the effect is less pronounced (about 50 %) for the determination of Young’s modulus, since the piled up material behaves less rigidly [1,2].

5 Preparation of test specimen

5.1 General

For reasons given below, provided the surface roughness criteria in 5.2 can be satisfied the best surface preparation is to do nothing other than to remove contaminants such as dust, fingerprints and preservative oils etc., as described in 5.3.

5.2 Surface roughness

The final surface finish shall be as smooth as available experience and facilities permit. Recommended R_a value shall be 5 % of the maximum penetration depth achieved whenever possible.

NOTE 1 Indentation into rough surfaces will lead to increased scatter in the results with decreasing indentation depth. Clearly when the roughness value, R_a , approaches the same value as the indentation depth the contact area will vary greatly from indent to indent depending on its position relative to peaks and valleys at the surface.

NOTE 2 It has been shown that for a Berkovich indenter when the angle that the surface presents to the axis of indentation is greater than 7 ° significant errors result [3].

NOTE 3 While R_a has been recommended as a practical and easily understood roughness parameter, it should be borne in mind that this is an average and thus single peaks and valleys may be greater than this as defined by the R_z value, although the likelihood of encountering the maximum peak, for example, on the surface is small. Modelling to investigate the roughness of the coating surface has concluded that there are two limiting situations for any R_a value. When the 'wavelength' of the roughness (in the plane of the coating surface) is much greater than the indenter tip radius, the force-penetration response is determined by the local coating surface curvature, but when the wavelength is much less than the tip radius, asperity contact occurs and the effect is similar to having an additional lower modulus coating on the surface.

NOTE 4 In cases where coatings are used in the as-received condition, nevertheless, random defects occur such as nodular growths or scratches and where an optical system is included in the IIT instrument, it is recommended that "flat" areas away from these defects are selected for measurement.

5.3 Polishing

Grinding and polishing shall be carried out such that any stress induced by the previous stage is removed by the subsequent stage and the final stage shall be with a grade of polishing medium appropriate to the displacement scale being used in the test.

NOTE 1 It should be appreciated that mechanical polishing of surfaces may result in a change of the residual stress state of the surface and consequently the measured hardness. For ceramics this is less of a concern than for metals although surface damage may occur.

NOTE 2 Many ceramic coatings replicate the surface finish of the substrate. If it is acceptable to do so, surface preparation problems can be reduced by ensuring that the substrate has an appropriate surface finish, thus eliminating the need to prepare the surface of the coating. In some cases, however, changing the substrate surface roughness may affect other coating properties therefore care should be taken when using this approach.

NOTE 3 During the deposition of coatings it is common for there to be relatively large residual stresses arising from thermal expansion coefficient mismatch between the coating and the substrate and / or stress induced by the coating growth process. Thus, a stress free surface would not normally be expected. Furthermore, stress gradients in coatings are not uncommon, so that removal of excessive material during a remedial surface preparation stage may result in a significant departure from the original surface state.

NOTE 4 Polishing reduces the coating thickness and so the effects of the substrate will be enhanced when indenting in-plan. The data analysis requires an accurate knowledge of the coating thickness indented and so polishing may require re-measurement of coating thickness. This again emphasises the need to carry out minimum preparation.

5.4 Surface cleanliness

Generally, provided the surface is free from obvious surface films, no special cleaning procedures are required. If cleaning is required the surface shall be wiped with a lintless tissue soaked in solvent to remove trapped dust particles and the surface shall be rinsed in a solvent which will remove contaminants picked up during exposure to the working environment, but which is chemically inert to the coating.

6 Test procedure

6.1 Calibration of instrument and indenters

6.1.1 The instrument shall be calibrated according to the procedures set out in annex A and all systems required for the test shall be operating correctly.

6.1.2 If not established in previous tests, the area function for the indenter to be used shall be measured - see clause A.2.3.3.

6.1.3 In all cases a reference material shall be introduced and initial indentation experiments shall be made with this material to ensure calibrations are valid and that no damage or contamination has occurred to the indenter tip. If the results of these initial indentations indicate the presence of contamination or damage then the indenter should be cleaned using the procedure recommended in annex B before further trial indents are made. If after cleaning, indentation into the reference material still indicates the presence of contamination or damage then inspection with an optical microscope at a magnification of 400x is recommended. Detection of submicroscopic damage or contamination is possible using scanned probe microscopy of indents or the indenter. Where damage is detected the indenter shall be replaced and appropriate calibration procedures implemented before the instrument is used.

6.2 Test piece

The test piece shall be mounted using the same methods as employed for determination of the instrument frame stiffness, and shall be such that the test surface is normal to the axis of the indenter.

NOTE 1 The surface of the test piece should be flat. A useful guideline is that the R_a value should be less than 5 % of the maximum displacement.

NOTE 2 Generally surface preparation of the test piece should be kept to a minimum, and if possible, the test piece should be used in the as-received state if surface flatness is consistent with the criteria given in 5.2 above.

6.3 Test conditions

6.3.1 Indenter geometry, maximum force and/or displacement and load displacement cycle (with suitable hold periods) shall be selected by the operator to be appropriate to the coating to be measured and the operating parameters of the instrument used.

6.3.2 Indentation cycles shall be selected and where multiple indentations are planned, each indent shall be separated from the next by at least 5 diameters of the neighbouring indent. In the case of indentation into a cross-section of the coating, the indent shall be placed such that it is more than three times the largest indent dimension from the coating-substrate and the coating-mountant interfaces.

NOTE It must be borne in mind that coatings can display a high degree of anisotropy, and thus the orientation of the indenter within the plane and the direction of indentation (plan or cross-section) can significantly alter the measured value of hardness and or modulus.

6.3.3 The specific test parameters include:

a) rate of force increase

b) rate of force decrease

c) maximum force

d) maximum displacement

e) hold times referenced to the force displacement cycle

f) indenter geometry and indenter area function

g) instrument frame compliance (stiffness)

h) distance between indents

6.3.4 The coating/substrate specific parameters include:

a) substrate hardness, Young's modulus and Poisson's ratio

b) coating thickness

c) surface roughness

d) adhesion of the coating to the substrate

e) coefficient of friction between coating and indenter

NOTE 1 Hardness and Young's modulus values may be affected by adhesion [4-8].

NOTE 2 Modelling suggests that friction between the coating and the indenter has a negligible effect [1].

6.3.5 All the parameters given in 6.3.3 and 6.3.4 shall be kept constant if a direct comparison is to be made between two or more test pieces.

NOTE Variations in test piece parameters other than hardness or modulus can affect measurement of these quantities. It is possible that if the indentation depth is a sufficiently small fraction of the coating thickness, (e.g. < 10 % for hardness or < 3 % - 5 % for modulus measurement) it may not be necessary to keep it constant for a direct comparison. The exact limits depend on the ratio of properties of coating and substrate. It is recommended that methods for normalising results to determine coating properties from coatings of different thickness be used if coating thickness is unknown or varies.

6.4 Measurement

6.4.1 Introduce the prepared sample and position it so that testing can be undertaken at the desired location.

6.4.2 Carry out the predetermined number of indentation cycles using the appropriate conditions.

6.4.3 Calculate the hardness and modulus of the test coating using the procedure described in 7 below.

7 Data analysis and evaluation of results

7.1 Composite properties

The hardness and indentation modulus of the test piece can be calculated using equations (1), (2) and (3) of annex A, clause A.1. However, before this can be done using the data obtained during the indentation experiments it is necessary to determine the values of A_c (projected contact area between the indenter and the test piece at maximum force) and S (the sample stiffness). Clause A.4 describes the determination of these parameters.

The properties thus calculated are composite properties for the coating/substrate combination. For indentation into a cross-section these properties can be considered to be those of the coating, provided that the recommendations in 6.3.2 have been followed. In the case of in-plan indentations, 7.2 provides guidelines for extracting the hardness and indentation modulus of the coating from the composite properties calculated.

7.2 Evaluation of coating hardness and modulus from in-plan indentation data:

7.2.1 Test parameters for ductile and brittle coatings need to be considered separately. However, in both cases, measurement of coating thickness, t_c, is highly recommended for reproducible measurement of coating properties. In both cases a set of trial indentations shall be performed (e.g. at two widely spaced forces) and analysed to enable estimates of force vs. indentation depth, h_c, and the radius of the contact area, a, to be determined. For indenters of different geometries (e.g. Berkovich, Vickers, spherical, cone, etc.), a is approximated by the radius of a circle having the same area as that in contact with the indenter,

$$a = \sqrt{\frac{A_c}{\pi}}$$

This value clearly has exact equivalence for a spherical or conical indenter but becomes increasingly less physically meaningful as the axial symmetry of the indenter reduces, i.e. Cone = Sphere > Berkovich > Vickers > Knoop. For in-plan indentation, elastic deformation of the substrate will always occur for all coatings, even though this could be negligibly small for a thick compliant coating on a stiff substrate. Thus the measured modulus will be the composite modulus of the coating and substrate and the value obtained will be a function of indentation depth. For hardness measurement it is recommended to use as small (i.e. as sharp) a radius indenter as possible and determine experimentally the onset of substrate plastic deformation and the substrate hardness. Then carry out indentation experiments such that the critical displacement is not exceeded.

NOTE 1 Empirical guidelines are given in BS 5411 Pt 6 for hardness measurement of electroplated coatings on steels, where it is recommended that the indentation depth does not exceed one tenth the thickness of the coating, while for paint films (ASTM D-1474) penetration of up to one third the coating thickness may be allowed. These approximations can be unsatisfactory in many cases.

NOTE 2 It is relatively easy to measure the hardness of ductile coatings or the elastic modulus of brittle coatings. It is more difficult to determine the hardness of brittle or hard coatings or the elastic modulus of ductile coatings.

NOTE 3 There is a compromise between: a) using a sufficiently high load (e.g.close to but not exceeding the limit corresponding to the onset of plastic deformation of the substrate) in order to obtain the maximum of force-depth data thereby improving the precision of the measurement; and b) indenting at a low enough displacement such that the plastic zone of the indentation does not interact with the substrate/coating interface – thus minimising the influence of the substrate on the measurement.

NOTE 4 Care should be taken when using the above approaches for multilayer or graded coatings.

NOTE 5 Where t_c is not measured nominal values of t_c may be used.

7.2.2 For modulus measurement of ductile coatings a maximum hold period shall be used that is sufficiently long to eliminate creep effects.

NOTE 1 Apparent rate sensitivity is often dominated by creep. A combination of a long hold at maximum load and a high removal rate of the test force is the best strategy for minimising the effect of creep on the measurement of contact compliance and subsequent calculation of modulus. The coating modulus may be obtained by taking a series of measurements at different indentation depths and extrapolating a linear fit to indentation elastic modulus vs. a / t_c to zero. This method provides a first approximation in the regime $a / t_c < 1.5$. Indentation load or displacement and indenter geometry shall be chosen such that data shall be obtained in the region where $a/t_c < 1.5$ (see Figure 2).