

SLOVENSKI STANDARD SIST EN 1071-1:2004

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe filometer
Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe filometer
Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 1: Bestimmung der Schichtdicke mit einem Kontaktprofilometer
Céramiques techniques avancées - Méthodes d'essai pour revetements céramiques - Partie 1: Détermination de l'épaisseur du revetement par profilemetre a contact 835aa4da52f0/sist-en-1071-1-2004

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<u>ICS:</u>

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81.060.30	Sodobna keramika	Advanced ceramics

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en



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Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe filometer

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 1: Détermination de l'épaisseur du revêtement par profilomètre à contact Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 1: Bestimmung der Schichtdicke mit einem Kontaktprofilometer

This European Standard was approved by CEN on 28 November 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 1071-1:2003) 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 September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.

This document supersedes ENV 1071-1:1993.

EN 1071 'Advanced technical ceramics – Methods of test for ceramic coatings' consists of 10 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 ARD PREVIEW
- Part 5: Determination of porosity (standards.iteh.ai)
- 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

Annex A is informative.

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

1 Scope

This part of this European Standard specifies a method for the determination of the thickness of ceramic coatings by measurement of the step height using a contact probe profilometer.

NOTE An alternative measurement of thickness, using a crater grinding method, is specified in ENV 1071-2.

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 are 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 (including amendments).

EN ISO 3274 Geometrical product specifications (GPS) - Surface texture: Profile method - Nominal characteristics of contact (stylus) instruments (ISO 3274:1996).

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

3 Principle

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The coating thickness often plays a major role in the performance of coated tools and machine parts. Many different techniques have been developed for assessing the coating thickness. Among these, the step height measurement is a very convenient technique because of its ease, wide applicability and accuracy.

In order to create a step, part of the substrate shall remain uncoated. This can be done by covering part of the substrate during deposition, e. g. by means of a covering plate or tape or by scratching the coating substrate/-system until delamination of the coating occurs (see, for example, ENV 1071-3).

The thickness of the coating is then determined by scanning this step beneath the contact stylus of a contact probe profilometer (see Figure 1).

NOTE 1 When covering part of the substrate during deposition, it can happen that the deposition rate near the step is influenced by the covering medium. This results in a step which is not representative for the coating thickness. This can be prevented by using very thin covering plates, or it can be circumvented by etching away part of the coating after the coating process.

NOTE 2 In the case of line-of-sight processes (such as ion beam deposition methods), shadowing effects are to be avoided, in order to create a representative step.

4 Apparatus

The contact probe profilometer shall be in accordance with EN ISO 3274.

The stylus tip consists of a diamond and its tip radius may be $2 \mu m$, $5 \mu m$ or $10 \mu m$. The instrument shall be calibrated by using step height calibration standards in accordance with the limits given in clause 7.

NOTE 1 It is important to ensure that the profilometer tip is not damaged.

NOTE 2 In metrology, it is always very important to have calibration and checking conditions corresponding to the measurement conditions. Therefore, the calibration or check standards should be as similar as possible to the step height to be measured.

NOTE 3 The effect of the stylus tip radius on the lateral profile resolution, i. e. the step edge broadening due to the bluntness of the tip (see Figure 2), should be considered. However, due to the horizontal compression of the displayed picture (far greater vertical than horizontal magnification), this effect can be neglected for routine coating thickness measurements where definitive horizontal resolution is unimportant.

5 Sampling and preparation of test specimens

Select a representative test specimen from the coating to be tested. Clean the specimen, using an appropriate method for the coating, so that it is free from dust, from oil, moisture and any other surface films.

6 Procedure

Create a step using, for example, the principles outlined in section 3.

Level the sample so that the uncoated portion of the substrate is parallel to the x-axis datum of the measuring machine. Procedures for this will depend upon the machine being used. Errors in leveling will result in differences between the measured and the true value of the coating thickness – see annex A. As shown in annex A, the magnitude of the error depends on both the angular leveling error and the ratio (width of step): (coating thickness).

Set the stylus loading to its lowest value and scan the step. To avoid damage to the stylus tip or the profile of the step, scanning shall take place in the direction from the coating surface to the substrate surface, i.e. down the step. Using the highest possible magnification consistent with measuring the whole step (see Figure 3) inspect the scan, for example by light microscopy, in order to determine whether any damage to the sample has occurred.

NOTE 1 The most accurate measurements are obtained when the instrument is mounted on a vibration isolation table.

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Locate the x-position for the inflection point in the step. Calculate the two least-squares straight lines from representative points on either side of the step. (see Figure 3) 2 Calculate the difference in y-values of the two straight lines at the x-position previously determined for the inflection point. The obtained value is the step height thickness of the coating.

If the data are not digitized (chart record), the drawing of the two least-squares straight lines may be done visually, upon agreement between the parties.

NOTE 2 It is recommended that the plotted out step height be at least 10 mm to enable accurate measurement of the step.

NOTE 3 Only the representative data for the upper and lower level of the step should be considered to eliminate artifacts caused by the curvature of the step.

Because the surfaces on either side of the step are often not flat and not parallel, a single coating step height thickness measurement is not significant. Therefore, repeat the procedure to give five measurements for each sample tested.

Because surface roughness will affect the result, the Ra value of both substrate and coating shall be no greater than 1/5 of the step height, and the average wavelength of the roughness shall be < 10 % of AB for the substrate or CD for the coating (see Figure 3).

In order for the limits of the step width (points B and C in Figure 3) to be accurately identified, the travel for the probe on the uncoated and coated portions of the sample (distances AB and CD respectively – see Figure 3) shall each be a minimum of 10x BC.

7 Limits to step height

Using the method specified above, contact probe profilometers can repeatedly identify steps in the 2 nm to 5 nm range and repeatedly measure larger steps. Height calibrations of contact probe profilometers can be accurate to about 1 %, assuming that the standard is calibrated to an accuracy of better than 1 %.

NOTE in all cases the means of step production is important and should be chosen to ensure as sharp a step as possible without causing damage to the surface of either coating or substrate in the vicinity of the step.

8 Repeatability

The repeatability of the step height measurement depends on the electronic noise level of the instrument, the digitization increment of the signal and the mechanical stability of the stylus or sample motion. However, the major source of statistical variation in calculated step heights is the surface roughness R_a of the step specimen, which is the ultimate uncertainty in assigning a height value to a stylus step profile.

Systematic errors are related to non-parallel reference lines at both sides of the step either due to insufficiently flat substrates or to local coating thickness artefacts related to sample preparation.

9 Test report

The results shall be reported in accordance with EN ISO/IEC 17025, and the test report shall include the following information:

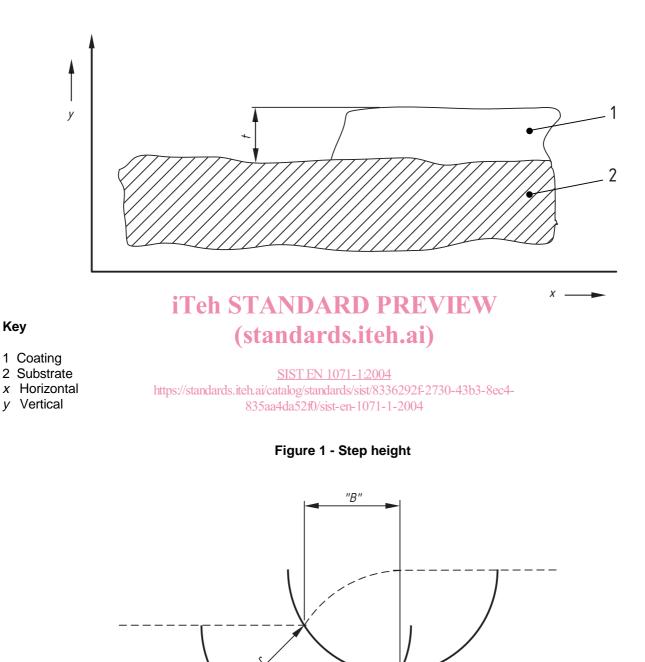
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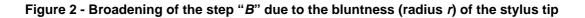
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a) name of the testing establishment;

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- b) date of the test, report identification and number, signatory;
- c) reference to this standard, the e. standard in accordance with EN 1071-173,0-43b3-8ec4-
- d) description of the test material; type of product, type of coating, date of receipt;
- e) method of test specimen sampling and preparation;
- f) method of calculation of step height (see clause 6);
- g) at least five values for the coating step height thickness;
- h) test results;
- i) comments about the test or the test results.





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