

# SLOVENSKI STANDARD SIST ENV 1071-2:2000

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# Advanced technical ceramics - Methods of test for ceramic coatings - Part 2: Determination of coating thickness by the cap grinding method

Advanced technical ceramics - Methods of test for ceramic coatings - Part 2: Determination of coating thickness by the cap grinding method

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 2: Bestimmung der Schichtdicke mit dem Kalottenschleifverfahren.

Céramiques techniques avancées - Méthodes d'essai pour revetements céramiques - Partie 2: Détermination de l'épaisseug du revetement par la méthode d'abrasion d'une calotte sphérique

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#### CEN

European Committee for Standardization Comité Européen de Normalisation Europäisches Komitee für Normung

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#### Foreword

This European pre-standard has been prepared by CEN/TC184 'Advanced technical ceramics'.

#### ENV 1071 has six Parts:

Part 1: Determination of coating thickness by contact probe

profilometer

Part 2: Determination of coating thickness by cap grinding method

Part 3: Determination of adhesion by a scratch test

Part 4: Determination of chemical composition

Part 5: Determination of the porosity
Part 6: Determination of the topography

CEN/TC184 approved this European pre-standard by resolution 3/1992 during its fifth meeting held in Brussels on 1992-03-31.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European pre-standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 2: Determination of coating thickness by the cap grinding method

#### 1 Scope

This Part of ENV 1071 describes a method for the determination of the thickness of ceramic coatings by a cap grinding method which includes spherical cap grinding and metallographical examination.

NOTE: An alternative measurement of thickness, using a contact probe profilometer, is described in ENV 1071-1.

#### 2 Normative references

This European Pre-Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and in the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Pre-Standard only when incorporated in it by amendment of revision. For undated references the latest edition of the publication referred to applies:

ENV 1071-1:

coatings Part 15 it Determination of coating thickness by contact probe profilometer

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ISO 468:1982

https://standards.ich.ai/catalog/standards/sist/44e05bd2-e112-4b2f9b55-Surface roughness - Parameters, their values and general rules for specifying requirements

ISO 4287-1:1984

Surface roughness - Terminology - Part 1 : Surface and its parameters  $\cdot$ 

#### 3 Principle

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 cap grinding method and the step height method (see ENV 1071-1 ) are the principal alternatives.

The method is intended as a simple metallographic test obtained with spherical cap grinding (see figure 1). A cavity is ground into the coated part. The test procedure is normally carried out with a steel ball of defined diameter wetted with a suspension of alcohol and diamond particles. The pattern of a plane monolayer shows two concentric circles due to the surface (D) and interface (d). The criterion is the difference of the diameters.

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#### 4 Sampling

Test pieces shall be sampled as whole items, or where this is not possible, made in accordance with the batch to be produced. For large parts, separate manufacturing of the test piece is necessary. A representative test specimen of the product under test shall be used.

#### 5 Procedure

#### 5.1 General

Mount the specimen on a flat support (see figure 3). Drop a suspension of alcohol and diamond paste (grain size 1 micron) on to a spherical tool, for example a steel ball bearing, material 1.3505, hardened. Measure the ball diameter (R) to 0.01 mm with the precision micrometer. The liquid drops on the sphere which is turned by a controlled shaft motion towards the sample surface so that the polishing film is dragged within the contact area.

Before testing, stir the suspension in a glass tube by means of air compression. (standards.iteh.ai)

### 5.2 Suspension concentration TENV 1071-22000

The mixing of commercial available diamond paste with alcohol reduces the relief polish. For general purposes a mixture of 1:1 is recommended, but for the metallographical examination of coated steel parts, a mixture of 1:4 (75 % alcohol) may be used. Report the concentration used.

#### 5.3 Peripheral limits

The limits of the procedure itself (see also clause 8) are:

- a) the angle of specimen adjustment which influences the surface formation of the ground cap:
- b) the ball diameter, which influences the penetration depth and the in depth resolution:
- c) the speed of shaft rotation which influences the polishing rate, and the surface formation;
- d) the suspension concentration (see 5.2).

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#### Example of test 5.4

Physically vapour deposited titanium nitride layers on HSS steel have been tested with the following peripheral limits:

- a) Ball diameter: 20 mm
- b) Ball mass: 35 g
- c) Inclination angle (A): approximately 60°
- d) Diamond paste, grain size: 1 µm
- e) Suspension concentration: 1:1
- f) Number of drops/min: 20
- g) Grinding time: 3 min
- Rotation speed: 300 rpm PREVEW

(standards.iteh.ai)

6 Microscopic examination

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**Calibration** https://standards.iteh.ai/catalog/standards/sist/44e05bd2-e112-4b2f-9b55-530228105dc6/sist-env-1071-2-2000

# 6.1.1 Magnification

Examine the pattern of the cap ground cavity microscopically at a magnification of 100:1.

### 6.1.2 Focusing and contract

Examine the test specimens as received. Sharp focusing of both of the concentric patterns and a maximum of contrast should be obtained at once. In some cases, etching of the substrate enhances the contract between substrate and layer.

#### 6.1.3 Micrographs

Calibrate, the photographic enlargement with respect to the magnification factor, and record in the test report (scaling mark) (see clause 8).

#### 6.2 Examination

Measure the diameters of the characteristics (D,d or X,Y) by means of a calibrated micrometer scale which is placed in the optical system of the microscope.

Measure the diameters (D, d) of circular characteristics in two rectangular directions and determine the mean values ( $D_{m^*}$ ,  $d_{m^*}$ ). If profile peaks are superimposed on the circular sections as a result of the surface and/or interface formation, the average values between profile peak and profile valley need to be chosen in accordance with ISO 468:1982 and ISO 4287-1:1984

#### 7 Expression of results

#### 7.1 General

The following symbols and definitions shall be used:

R: radius of the spherical tool (see figure 1)

h: thickness of the layer (see figure 1)

T: total penetration depth of the ball (see figure 1)

t: penetration depth in the substrate (see figure 1)

D: diameter of the circle due to the interface (see figure 2)

m : index for mean value (D<sub>m</sub>., d<sub>m</sub>.)

X: difference between the semiaxial lengths in the patterns obtained for coated cylindrical samples (see figure 2)

Y: sum of the semiaxial lengths in the patterns obtained for coated cylindrical samples (see figure 2)

ß: inclination angle Soft the Ospecimen support (see figure 3) https://standards.iteh.ai/catalog/standards/sist/44e05bd2-e112-4b2f-9b55-

## 7.2 Test of coated parts with plane surfaces 00

Obtain the total penetration depth of the spherical grinder from the equation (1):

(1)

$$T = R - \sqrt{R^2 - D^2/4}$$

The penetration depth in the bulk material below the interface of the monolayer is then given by equation (2):

(2)

$$t = R - \sqrt{R^2 - d^2/4}$$

The thickness is then given by equation (3) and equation (4):

(3)

$$h = T - t$$

(4)

$$h = \sqrt{R^2 - d^2/4} - \sqrt{R^2 - D^2/4}$$