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

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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'épaisseur du revetement par la méthode d'abrasion d'une calotte sphérique

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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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Foreword

This document (EN 1071-2:2002) 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of an identical text or by endorsement, at the latest by May 2003, and conflicting national standards shall be withdrawn at the latest by May 2003.

Annex A is informative.

This document supersedes ENV 1071-2:1993.

EN 1071 '*Advanced technical ceramics – Methods of test for ceramic coatings*' consists of seven 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 test

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, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, 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 a crater grinding method which includes the grinding of a spherical cavity and subsequent microscopic examination of the crater.

NOTE An alternative measurement of thickness, using a contact probe profilometer, is specified in EN 1071-1.

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

ISO 3290 *Rolling bearings – Balls – Dimensions and tolerances.*

3 Significance and use

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 crater grinding method and the step height method (see EN 1071-1) are methods that are easy to perform and applicable to most coated systems.

The method is simple and straightforward. A crater is ground into the coated part by means of a rotating ball wetted by an abrasive slurry. The thickness of the coating is derived from the ball and crater dimensional characteristics. Contrast between the different materials constituting the coating and substrate is a prerequisite for the method to enable the detection of the interface between the coating and the surface.

NOTE Test specimens should be either flat or cylindrical. Flatness can be considered as sufficient if the local specimen radius of curvature, R_s, satisfies the relation $R_s > 100 \times R$ (for error ≤ 1 %).

4 Sampling

A representative test specimen of the product under test shall be used. Test pieces shall be coated original items, or where this is not possible, made in the same way as the batch to be tested. For large parts, separate manufacturing of the test piece may be necessary.

5 Test procedure

5.1 General

A ball wetted by an abrasive suspension is rotated against the surface of the test piece. A spherical wear crater is thus produced, and the test is finished when the depth of penetration of the spherical crater is greater than the coating thickness. The coating thickness is then derived from the size of the wear scars (full crater and substrate crater widths) and the ball diameter.

NOTE 1 Different set-ups of test rigs can be used. The ball can be rotated freely on a drive shaft, whereby its mass is used to produce the contact load, or it can be clamped in the drive axis while the specimen is loaded by means of a lever system. Alternatively, a wheel instead of a ball can be used, in which case the sample must also be rotated (this is the same principle as dimple grinder used for the preparation of TEM specimens). A typical arrangement is shown in Figure 1.

NOTE 2 Different abrasives (e.g. diamond, alumina, silica) can be used, and commercially available suspensions based on alcohol, oil or water can be applied. The abrasive slurry can be smeared onto the ball surface prior to testing, but more repeatable measurements are achieved when the abrasive slurry is drip fed into the contact region, e.g. by peristaltic pumping of a stirred suspension. The grain size of the abrasive must be small to avoid roughening of the crater borders. As an example, 1 µm diamond paste suspended in ethanol is often used.

5.2 Preparation of the test

Make sure that the specimen and ball are clean. Ultrasonic cleaning for 5 min in fresh petroleum-ether followed by drying in ambient air is usually sufficient. The suitability of the ball shall be determined by measuring 10 diameters at random. A ball shall be rejected if the difference between any two measurements exceeds 5 µm (the maximum allowable value of V_{DWS} for grade G 200 balls as given in ISO 3290 (E)), or if scratches visible to the naked eye are present on the surface. Prepare a suspension of abrasive particles in a diluting agent. Position the test piece on a stable support.

NOTE 1 The abrasive suspension should be well stirred to ensure a uniform dispersion of abrasive particles.

NOTE 2 Hardened steel balls for rolling bearings can be used with a specified diameter and surface finish according to ISO 3290 (E).

5.3 Test parameters

The test specific parameters include:

a) ball diameter;

b) contact load;

c) sliding speed;

d) composition and concentration of the abrasive suspension;

e) slurry feed rate;

f) duration of the test.

The specimen specific parameters include:

a) surface quality (roughness, cleanliness);

b) optical contrast between coating(s) and substrate.

5.4 Example of test

Typical operating parameters are as follows:

a) ball diameter: 25 mm;

b) contact load: 0,25 N;

c) rotational speed: 100 rpm;

d) composition of the abrasive suspension: 1 µm grain size diamond paste suspended in ethanol;

e) abrasive suspension concentration of 1:4;

f) slurry feed rate: 20 drops/min;

g) test duration: 5 min.

NOTE Optimum test conditions will be different for different specimens. The above conditions are typical for thin (few µm in thickness) hard coatings on metallic substrates.

6 Microscopic examination and measurement

6.1 Examination

It is mandatory to clean the specimen prior to examination (cf. clause 5.2).

An imaging technique with calibrated size measurement shall be used. Examine the ground cavity at the highest magnification at which the complete worn crater is visible.

Focus the microscope on the concentric patterns and if necessary adjust the illumination to obtain maximum contrast.

NOTE Usually, an optical, reflected light microscope is used, but any other imaging technique such as scanning electron microscopy can be used, e.g. when it is not possible to discriminate between the coating and the substrate by other means. In the case of optical microscopy, etching can be used to enhance contrast between the substrate and coating.

6.2 Measurement

Measure the crater dimensions as appropriate using a calibrated measuring device.

For flat specimens measure the diameters D, d, or lengths X, Y, of the circular characteristics both parallel and perpendicular to the direction of ball rotation (Figure 2).

For cylindrical specimens measure only the largest dimensions of the circular characteristics, parallel with the cylinder axis (Figure 3).

At least 5 measurements shall be carried out to define the repeatability of the measurement.

NOTE 1 Size measurement can easily be achieved by preparing a micrograph of the cavity as well as of a traceably calibrated scale at the same magnification.

NOTE 2 Due to surface roughness effects, the boundaries of the layer(s) cannot be well defined and the best estimate of the centre line of a boundary should be used.

NOTE 3 The accuracy of the measurement is dependent on the roughness of the surfaces delineating the boundaries of the layer.

7 Definitions, symbols and calculation

7.1 Symbols and definitions

The following symbols and definitions shall be used:

R: radius of the ball, in micrometres (see Figure 2);

R_s: radius of curvature of specimen;

h: thickness of the coating, in micrometres (see Figure 2);

T: total penetration depth of the ball, in micrometres (see Figure 2);

t: penetration depth in the substrate, in micrometres (see Figure 2);

D: the best estimate of the diameter of the outer circle, at the surface of the coating, in micrometres (see Figure 2);

d: the best estimate of the diameter of the inner circle, defined by the bottom of the layer, in micrometres (see Figure 2);

X: distance between the outer circle periphery and that of the inner circle at the same side of the crater, in micrometres (see Figure 2);

Y: distance between the inner circle periphery and that of the outer circle at the opposing side of the crater, in micrometres (see Figure 2);

m: index for mean value (D_m, d_m, X_m, Y_m).

7.2 Calculations

The total penetration depth of the spherical crater is calculated as:

$$T = R - \sqrt{R^2 - D_m^2 / 4} \quad (1)$$

Whereas the penetration depth in the substrate below the coating-substrate interface is given by:

$$t = R - \sqrt{R^2 - d_m^2 / 4} \quad (2)$$

The coating thickness can thus be calculated from:

$$h = T - t \quad (3)$$

or,

$$h = \sqrt{R^2 - d_m^2 / 4} - \sqrt{R^2 - D_m^2 / 4} \quad (4)$$