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Advanced technical ceramics - Methods of test for ceramic coatings - Part 6: Determination of the abrasion resistance of coatings by a micro-abrasion wear test

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 6: Bestimmung der Beständigkeit gegen Abriebverschleiß von Schichten mittels Mikroabriebprüfung

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 6 : Détermination de la résistance à l'abrasion des revêtements par essai de micro -usure

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 6: Determination of the abrasion resistance of coatings by a micro-abrasion wear test

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 6 : Détermination de la résistance à l'abrasion des revêtements par essai de micro-usure

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Foreword

This document (EN 1071-6:2007) 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of an identical text or by endorsement, at the latest by May 2008, and conflicting national standards shall be withdrawn at the latest by May 2008.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes ENV 1071-6:2002.

EN 1071 'Advanced technical ceramics - Methods of test for ceramic coatings' was prepared in 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 by electron probe microanalysis (EPMA)

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: Rockwell indentation test for evaluation of adhesion

Part 9: Determination of fracture strain

Part 10: Determination of coating thickness by cross sectioning

Part 11: Determination of internal stress by the Stoney formula

Part 5, a European pre-standard, was withdrawn in 2007. Part 7, a Technical Specification, was withdrawn in 2007, following publication of EN ISO 14577-4. At the time of publication of this document, Parts 8 to 11 were Technical Specifications.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 1071 specifies a method for measuring the abrasive wear rate of ceramic coatings by means of a micro-scale abrasion wear test, based on the well known crater grinding technique used for film thickness determination (see EN 1071-2).

This method can provide data on both film and substrate wear rates, either by performing two separate tests or by careful analysis of data from a single test series.

The test method can be applied to samples with planar or non-planar surfaces but the analysis described in clause 9 applies only to flat samples. For non-planar samples, a more complicated analysis, possibly requiring the use of numerical methods, is required.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 17025, *General requirements for the competence of testing and calibration laboratories* (ISO/IEC 17025:2005)

ISO 3290, *Rolling bearings — Balls — Dimensions and tolerances*

3 Terms and definitions

For the purposes of this European Standard, the following term and definition applies.

3.1
abrasive wear rate, *K*
abrasive wear coefficient
volume of material removed in unit sliding distance under a normal contact load of 1 N

4 Significance and use

Although few protective coatings are subject to single wear processes, the abrasive wear resistance of such coatings can play a decisive role in their performance. Hence knowledge of the abrasive wear resistance of ceramic coatings can help in the proper selection of coatings for applications where abrasion plays a major role in their degradation. Although techniques exist to measure the abrasive wear behaviour of bulk materials and thick films (see [1 – 3]), these techniques are not easily applied to thin films and are difficult to interpret when used on curved surfaces.

The purpose of this European Standard is to provide a method for measuring the abrasion resistance of both thin and thick coatings and of bulk materials. The test can be performed on flat surfaces or surfaces with a known radius of curvature, and requires only a few mm^2 of sample. However, the analysis described in clause 9 applies only to flat samples, and is applicable to homogeneous single layer coatings only; errors may occur if the test is used on in-homogeneous coatings. [4] and [5] give details of analytical treatments for determining the wear rate for coatings on curved surfaces.

By proper treatment of the results, as indicated in 9.2, where the test produces penetration of the coating it can provide abrasive wear coefficients for both the coating and substrate from a single test series.

Although the test is designed to enable quantitative measurement of abrasive wear coefficients, it can be adapted as a quality control test for use on real components.

5 Principle

In the test, a ball is rotated while being pressed against the sample and an abrasive slurry is fed into the contact zone. A spherical depression is produced and the size of this depression is measured. Where perforation of the coating has not occurred, the wear rate of the coating can be obtained from a single crater. When perforation of the coating occurs, by making a series of such craters and measuring their dimensions, the wear rate of both the coating and the substrate can be calculated.

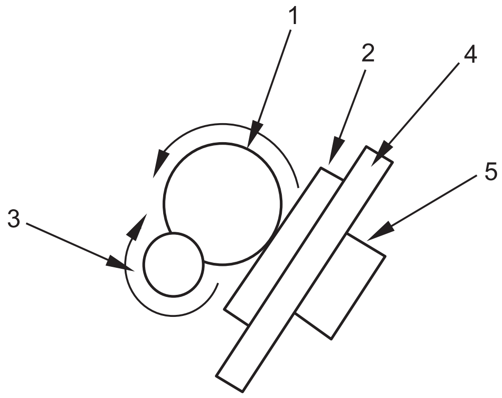
6 Apparatus and materials

6.1 Test system

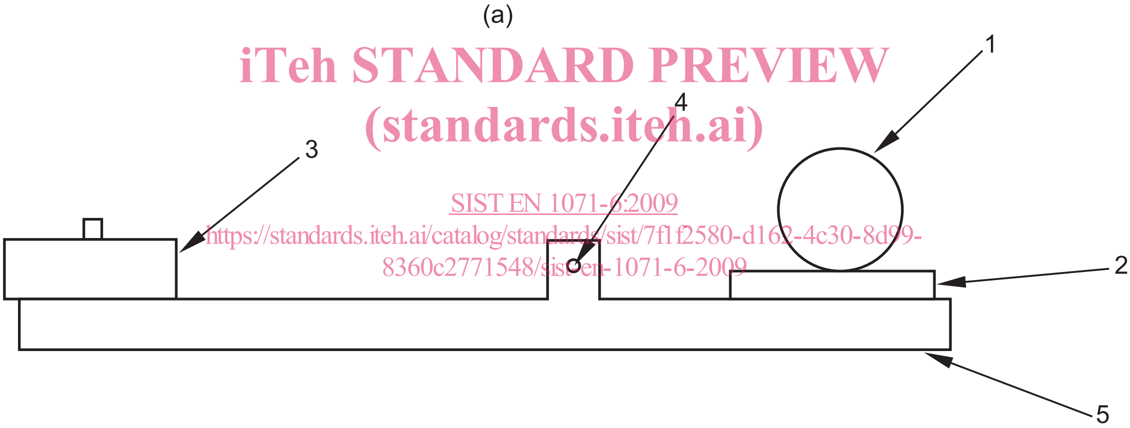
A ball that can be rotated and pressed against the coated sample shall be used. Two variants of the ball system are shown in Figure 1, where either the sample, mounted on a dead-weight loaded lever, is pressed against a directly driven ball, or the ball's own weight presses it against the sample.

NOTE The results obtained with free-ball systems [6] (see Figure 1a) - can vary depending on the precise system geometry. In particular, it has been found that the tilt angle of the sample holder and the width of the groove in the drive shaft that supports the ball can have an important influence on the results. A tilt angle of 60 ° to 75 ° and a shaft groove width of 10 mm have been found to result in the smallest variability under typical conditions [6].

The test system shall be constructed so that the rotational speed of the ball remains constant throughout any test and is reproducible to better than +/-10 % of the nominal value between tests. The drive shaft shall have a total run-out of less than 20 µm at its points of contact with the ball.



(a)



(b)

Key for a)
1 ball
2 sample
3 drive shaft
4 sample support
5 load cell

Key for b)
1 ball
2 sample
3 weight
4 pivot point
5 lever

Figure 1 — Two different types of a ball cratering system: a) free-ball, b) fixed-ball

6.2 Test balls

The balls used are typically 25 mm diameter hardened steel, e.g. UNS G52986 (SAE52100, [7]) and shall, prior to any conditioning, conform to the requirements of ISO 3290.

NOTE 1 Balls can be used in a polished condition but it has been found [8] that the test behaviour is erratic and poor results are obtained if balls are used without conditioning.

The recommended conditioning treatment consists of running the new test ball for at least 300 revolutions on a non-critical part of the sample, or other suitable surface, under normal test conditions and repeating this for at least 5 different orientations of the ball before starting the test programme.

NOTE 2 A flat, ground steel coupon with a hardness of between 200 and 800 HV 30 has been found to be suitable for conditioning the ball.

NOTE 3 Following conditioning, balls have been found to be usable for around 50 individual craters, depending on the precise conditions used.

The performance of balls shall be subject to regular performance checks to ensure that they continue to produce acceptable craters. Balls shall be replaced if such a check indicates any abnormal cratering behaviour.

NOTE 4 Performance checks can be made using any representative test piece, such as hardened and tempered high speed steel, or a well characterized titanium nitride or other coating deposited on a stable substrate material.

6.3 Test slurry

In all cases, a slurry of silicon carbide (SiC) or other suitable abrasive in a suitable liquid, normally water, shall be used. Other liquids may be used where their use is suggested by matching conditions to a particular application. In this respect the wetting properties and viscosity of the fluid are important parameters to consider.

NOTE 1 The abrasive is normally F1200 SiC, but F1200 alumina or other fine abrasive can be used. The average size of the abrasive should not exceed 5µm.

The use of different abrasive media will produce different wear rates, and only results produced from craters under identical conditions should be compared.

A homogeneous slurry shall be used throughout the test. This can be done by stirring the slurry continuously, or by adding stabilizers.

NOTE 2 If testing is to be on coatings deposited onto steel substrates that are susceptible to corrosion, it is recommended that sodium nitrite ($NaNO_2$) should be added to the slurry at the rate of 1 g for each 100 cm^3 of water to prevent corrosion of craters before they can be measured.

An abrasive slurry shall be made from the abrasive powder and the chosen liquid in the required proportions.

As the mode of wear that is observed can depend critically on the concentration of the abrasive slurry, two concentrations are recommended. These are:

1) *Dilute (promotes grooving wear)*

   Concentration of 2 % v/v.

   For SiC, for example, with a density of 3,2 gcm^{-3}, this is achieved by mixing 6,4 g of SiC to 98 ml of distilled or de-ionized water.

2) Concentrated (promotes rolling wear)

Concentration of 20 % v/v.

For SiC, for example, with a density of 3,2 gcm^{-3}, this is achieved by mixing 80 g of SiC in 100 cm^{-3} of distilled or de-ionized water.

NOTE 3 The type of wear promoted depends both on the concentration of the slurry and the type of abrasive as well as on the material being tested. For example, it has been found that micro-grain (submicron) rutile can promote rolling wear even at concentrations as low as 3 % by volume.

As an alternative to mixing slurries, ready mixed abrasive slurries can be used. If this is done all details of the supplier and makeup of the slurry shall be reported.

NOTE 4 Preliminary testing should be undertaken to ensure that the slurry concentration chosen produces the wear mode(s) of interest during the test.

6.4 Measurement of crater dimensions

Crater dimensions may be measured with any suitable equipment, e.g. a microscope with calibrated graticule, provided that the calibration used is traceable to national standards. Where measurements are made from photographically capture images, it is essential that fiducial (reference) marks of known dimensions are incorporated in the images to ensure that any shrinkage of the photographic film after development or during storage can be eliminated. Alternatively, automatic measurement using an electronically captured image may be used provided that the measurement system is fully calibrated, the procedure used being traceable to national standards.

NOTE 1 In some cases, e.g. rolling wear with relatively large abrasive particles, it may be difficult to identify the edge of craters, particularly at the outer surface of the coating. In such cases the use of profilometry, a change in illumination angle, or substrate etching (for craters that penetrate the coating) can help.

NOTE 2 Profilometry may lead to different results than optical microscopy evaluation of crater size, due to rounded crater edges. Results of tests evaluated by different measurement methods should not be compared to each other.

7 Preparation of test pieces

7.1 Coated samples shall have a flat area large enough to perform the necessary series of experiments. In all cases the coating thickness shall be larger than 1 µm.

NOTE Samples with non-flat surfaces can also be tested but the analysis required determining the wear rate of coating and substrate will be different to that given in this standard – see [4] and [5].

7.2 The accuracy with which crater diameters can be measured depends on the surface finish of the sample and the type of abrasive used. Although it is possible to improve the surface finish of the coating by polishing prior to testing, this is not the case with the substrate, and the surface finish of the substrate affects the accuracy with which the interface between coating and substrate can be located. Therefore, wherever possible, coatings should be deposited onto polished substrates to allow accurate location of the base of the coating. Where necessary, the surface of the coating may be polished to improve the surface finish.

NOTE To avoid damaging the surface of the coating or affecting its wear rate, it is recommended that any polishing should be done with the smallest diamond abrasive and lowest pressure commensurate with achieving the surface finish required. Polishing should therefore commence with, for example, 1µm diamond abrasive and this should only be increased if the required finish cannot be achieved.

7.3 Prior to the test, clean the sample to remove all traces of contaminants. A suitable preparation procedure is as follows:

a) ultrasonically clean in a suitable solvent;

b) rinse;

c) dry in an oven at 110 °C for 10 min.

8 Test procedure

8.1 Different types of test

8.1.1 Type A: no perforation of coating

In this type of test, control the duration of the test so that perforation of the coating does not occur. Some trials might be necessary before the required conditions are obtained. Measure the size of the crater and calculate the abrasive wear rate using the method described in 9.1.

8.1.2 Type B: perforation of coating

In this type of test perforate the coating. Produce a series of craters for different durations and measure the sizes of the craters in each case. Calculate the abrasive wear rates for both the substrate and the coating using the method described in 9.2.

For tests using procedure B, determine the coating thickness, t, as part of the test procedure – see 8.3.10 and Annex A.

8.2 Test Type A: No perforation of coating

8.2.1 Ensure that the ball and drive shaft, where a free-ball system is being used, are free from any deposits of slurry from previous tests. With the sample clamped firmly in position on the test system, adjust the motor speed. Control the motor speed at a constant value throughout a set of tests. A recommended surface speed for the ball is 0,1 ms^{-1}, which is equivalent to about 80 rpm for a 25 mm diameter ball.

NOTE For free-ball systems, the ball rotation speed will normally be different to the rotation speed of the shaft.

8.2.2 Adjust the test system to give a suitable normal loading between the ball and sample at the test point on the sample. The recommended load is 0,2 N.

NOTE 1 Poorly defined craters can be produced if the load applied to the sample is too high. To prevent this, it is recommended that the load applied should not be greater than 0,4 N.

NOTE 2 In free-ball systems, the friction due to the ball rotation causes the normal force acting on the sample to be different to when the ball is stationary - see [9] - these test systems a load cell should be employed to measure the true normal force.

8.2.3 Start the slurry feed and ball rotation, and ensure that the ball is completely coated in the contact zone during its first complete revolution. The feed rate of the slurry shall be sufficient that the contact between the ball and sample is always well wetted by the slurry. The slurry shall not be re-circulated. Report the ball rotation speed used.