SLOVENSKI STANDARD
SIST ENV 1071-6:2007

01-januar-2007

Sodobna tehnična keramika - Metode za preskušanje keramičnih prevlek - 6. del: Ugotavljanje odpornosti prevlek proti obrabi z mikroabrazivnim preskusom

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 des Abriebwiderstands von Schichten durch eine Mikroabriebprüfung

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

Ta slovenski standard je istoveten z: **ENV 1071-6:2002**

ICS:

25.220.99	Druge obdelave in prevleke	Other treatments and coatings
81.060.30	Sodobna keramika	Advanced ceramics

SIST ENV 1071-6:2007 **en**

EUROPEAN PRESTANDARD

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

ENV 1071-6

July 2002

ICS 81.060.30

English version

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

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 6: Bestimmung des Abriebwiderstands von Schichten durch eine Mikroabriebprüfung

This European Prestandard (ENV) was approved by CEN on 28 April 2002 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. ENV 1071-6:2002 E

Contents

Foreword

This document ENV 1071-6:2002 has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

EN 1071 'Advanced technical ceramics - Methods of test for ceramic coatings' consists of 7 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 depth sensing indentation

This standard includes an informative annex A and a bibliography.

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

1.1 This part of ENV 1071 describes a method of measuring the abrasive wear rate of ceramic coatings by means of a micro-abrasion wear test based on the well known crater grinding technique used for film thickness determination – see ENV 1071-2.

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

1.3 The test method may be applied to samples with planar or non-planar surfaces but the analysis described in clause 11 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

This European Prestandard 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 Prestandard 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).*

ENV 1071-2, *Advanced technical ceramics – Methods of test for ceramic coatings – Part 2: Determination of coating thickness by the crater grinding method.*

ISO GUM, *Guidance on the expression of uncertainty in measurement.*

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

3 Terms and definitions

For the purposes of this European Prestandard, the term and definition given below applies.

3.1
abrasive wear rate (also called 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 part in their degradation. Although techniques exist to measure the abrasive wear behaviour of bulk materials and thick films (see Bibliography), these techniques are not easily applied to thin films and are difficult to interpret when used on curved surfaces.

The purpose of this ENV is to provide a method for measuring the abrasion resistance of both thin and thick coatings and of bulk materials. The test can be carried out 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 11 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. By proper treatment of the results, as indicated in 11.2, where the test produces penetration of the coating it can provide abrasion coefficients for both coating and substrate from a single test series.

Although the test is designed to enable quantitative measurement of 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 whilst 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 these craters and measuring the size of the scar dimensions, the wear rate of both the coating and the substrate can be calculated.

6 Test parameters

6.1 The specific test parameters include:

a) load

b) speed

c) number of revolutions

d) diameter of ball

e) composition, morphology, and size of abrasive

f) concentration of abrasive

g) abrasive feed rate

h) surface condition of ball

i) composition of test fluid

6.2 The coating/substrate specific parameters include:

a) coating abrasion resistance

b) substrate abrasion resistance

c) coating thickness

d) radius of curvature of sample if any

e) adhesion of coating to the substrate

f) surface finish of coating and substrate

g) deposition process used

7 Apparatus

A ball or a profiled wheel which is rotated and pressed against the coated sample shall be used. When a wheel is used the sample shall also be rotated. 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.

In all cases, a slurry of SiC or other suitable abrasive in a suitable liquid, normally water, shall be drip fed into the contact zone between the ball and the sample, and wear is measured by measuring the size of the wear scar on the sample using a calibrated microscope.

NOTE 1 The abrasive is normally F1200 SiC, but F1200 silica or other fine abrasive can be used. The average size of the abrasive should not exceed 4 µm.

An homogeneous slurry shall be ensured during the test. This can be done by stirring the slurry continuously, or by adding stabilisers.

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

NOTE 2 Balls can be used in a polished condition but it has been found - see reference 3, Bibliography - 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 test ball for at least 50 revolutions on a non-critical part of the sample under normal test conditions before starting the testing.

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 depends critically on the abrasive concentration that is used, two concentrations are recommended. These are:

1) Dilute (promotes grooving wear)

Concentration of 2 % by volume

For SiC this is achieved by mixing 6,4 g to 98 ml of distilled water.

2) Concentrated (promotes rolling wear)

Concentration of 20 % by volume

For SiC this is achieved by mixing 6,4 g to 8 ml of distilled water.

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.

Measurement of the crater dimensions may be carried out 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 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.

8 Different types of test

8.1 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 can be necessary before the required conditions are obtained. Calculate the wear from the measured size of the wear crater.

8.2 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 wear rates for both the substrate and the coatings from an analysis of the measured crater sizes.

9 Sample preparation

9.1 Coated samples shall have a flat area large enough to perform the necessary series of experiments.

NOTE Samples with non-flat surfaces can also be tested but the analysis required to determine the wear rate of coating and substrate will be different to that given in this standard.

For apparatus that allows exact relocation of the ball in the crater after each measurement of crater diameter, use a single crater and measure it after each test. Otherwise, use a series of craters produced for increasing time intervals.

9.2 For tests using procedure B, where perforation of the coating occurs, evaluate the coating thickness as part of the test procedure. In all cases the coating thickness shall be larger than 1 μm.

The accuracy with which crater diameters can be measured is dependent upon the surface finish of the sample. 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 can be deposited onto polished substrates to ensure accurate location of the base of the coating. Where necessary, the surface of the coating may be polished to improve the surface finish.

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

a) ultrasonically clean in acetone for 15 min;

b) rinse in clean acetone;

c) dry in oven at 110°C for 10 min.

10 Test procedure

10.1 Test Type A: No perforation of coating

10.1.1 With the sample clamped firmly into position on the test system, adjust the motor speed to the correct value. Control the motor speed at a constant value throughout a set of tests. A recommended speed is 80 rpm. Report the sample rotation speed when a wheel test system is used.

10.1.2 Adjust the test system to give the correct normal loading between the ball and sample at a test point on the sample.

NOTE 1 If the load applied to the sample is too high the phenomenon of ridging occurs – see Figure 2. To prevent this, it is recommended that the load applied should not be greater than 0,4 N. The recommended load is 0,25 N.

NOTE 2 In free ball machines, the friction due to the ball rotation results in an error in the normal force acting on the sample. In these test systems a load cell should be employed to measure the true normal force.

10.1.3 Start the slurry drip feed, with the slurry continuously stirred by a magnetic stirrer. The feed rate 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

10.1.4 Stop the test (motor and slurry feed) after a predefined test duration.

NOTE The number of revolutions that is required will depend on the materials being tested and the test conditions employed, and will need to be defined using trials.

10.1.5 When the test has been completed, the sample should be removed, and cleaned using the same procedure as that used prior to testing – see clause 9.

10.1.6 Measure the size b of the wear scar(s) both parallel and perpendicular to the direction of ball rotation – see Figure 3. The average of these measurements shall be used as the size of the wear scar.

10.1.7 Calculate the wear rate of the coating using the method described in clause 11 below.

10.1.8 Repeat the test at least two times on each sample.