

SLOVENSKI STANDARD SIST EN 1071-13:2010

01-maj-2010

Sodobna tehnična keramika - Metode za preskušanje keramičnih prevlek - 13. del: Ugotavljanje stopnje obrabe (metoda pin-on-disk)

Advanced technical ceramics - Methods of test for ceramic coatings - Part 13: Determination of wear rate by the pin-on-disk method

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 13: Bestimmung der Verschleißrate mittels Stift-Scheibe-Prüfung III W

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques -Partie 13 : Détermination du taux d'usure selon la méthode pin-on-disk

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Ta slovenski standard je istoveten z: EN 1071-13-2010

ICS:

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

SIST EN 1071-13:2010

en,de

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SIST EN 1071-13:2010

EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 1071-13

March 2010

ICS 81.060.30

English Version

Advanced technical ceramics - Methods of test for ceramic coatings - Part 13: Determination of wear rate by the pin-on-disk method

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 13 : Détermination du taux d'usure selon la méthode pin-on-disk Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 13: Bestimmung der Verschleißrate mittels Stift-Scheibe-Prüfung

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

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Ref. No. EN 1071-13:2010: E

SIST EN 1071-13:2010

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Foreword

This document (EN 1071-13:2010) 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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.

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.

EN 1071, *Advanced technical ceramics — Methods of test for ceramic* coatings, consists of the following 13 parts:

- Part1: Determination of coating thickness by contact probe filometer
- 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 (withdrawn)
- <u>SIST EN 1071-13:2010</u>
- Part 6: Determination of the abrasion resistance of coatings by a micro-abrasion wear test f4fe3ddacd31/sist-en-1071-13-2010
- Part 7: Determination of hardness and Young's modulus by instrumented indentation (withdrawn)
- 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 12: Reciprocating wear test
- Part 13: Determination of wear rate by the pin-on-disk method

Part 7 was a Technical Specification and Parts 8 to 11 are 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, Croatia, 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.

Introduction

The determination of the wear resistance of thin ceramic coatings used in sliding contacts is of high importance in several industrial fields such as stamping, moulding, blanking and in many situations where two mechanical components slide on each other. This part of EN 1071 describes a method for evaluating the wear of ceramic coatings by use of a test in which a flat or spherically ended pin is brought, under load, into contact with a flat disk and the two are set in relative motion such that the pin describes a circular path on the flat surface of the disk. Depending on the information required, either the disk or pin or both may be coated with the material under test, with the other member of the couple being selected for its relevance to the tribosystem under evaluation. Wear is determined by weight loss, by profilometry, by linear measurement or by a combination of these.

Testing may be carried out under dry or lubricated conditions. Where suitable instrumentation is available, the test can provide important information about the friction generated in the system. In addition to providing data on the frictional interaction in the system, monitoring of the friction can, by detecting changes in the level or trend of the friction force, provide important information about changes occurring during the test, e.g. removal or fracture of the coating, changes in wear mechanisms, etc. The test for use with bulk materials sliding under non-lubricated conditions is well described in [1].

This standard identifies the basic equipment requirements and the test critical parameters for testing ceramic coatings, and provides for appropriate operating procedures and measurement protocols to ensure their proper control. In addition, it provides for consistency in the analysis of data and in the treatment of errors.

This part of EN 1071 complements parts 6 [2] and 12 [3] Which describe techniques for micro-scale abrasion wear testing and reciprocating wear testing of ceramic coatings respectively. 6c-47fa-a9ad-4fe3ddacd31/sist-en-1071-13-2010

1 Scope

1.1 This European Standard describes a method for evaluating the wear of ceramic coatings by use of a test in which a flat or spherically ended pin is brought, under load, into contact with the flat surface of a disk and the two are set in relative motion such that the pin describes a circular path on the disk. Depending on the conditions being simulated, either the pin or disk or both may be coated with the material under test, with the other member of the couple being selected for its relevance to the system under evaluation.

1.2 Where suitable equipment is available, the test may be used to determine the friction generated in the sliding contact.

1.3 The method is suitable for evaluating coatings in the thickness range from $1 \mu m$ to more than 100 μm , and with suitable choice of conditions might also be applicable to testing thinner coatings.

1.4 Testing may be under either dry or lubricated conditions. However, the test is not designed for evaluating the properties of lubricants except insofar as they affect the wear behaviour of the materials being tested. Related methods for testing lubricants using a reciprocating motion are given in references [4] - [6].

1.5 Testing a materials couple under a range of loading conditions might provide information about the adhesive and/or cohesive strength of the coating, in addition to its wear behaviour.

2 Normative references STANDARD PREVIEW

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. N 1071-13:2010

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

ISO 31-0, Quantities and units — Part 0: General principles

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

volume wear rate volume wear coefficient specific wear rate volume of material removed from a surface in a sliding distance of 1 m under a normal load of 1 N

3.2

mass wear rate

mass wear coefficient

mass of material removed from a surface in a sliding distance of 1 m under a normal load of 1 N

3.3

instantaneous coefficient of friction

instantaneous value of the friction force divided by the instantaneous value of the applied load

NOTE This is often approximated to the instantaneous value of the friction force divided by the mean applied load.

4 Significance and use

This standard gives guideline on conducting sliding wear and friction tests in a pin-on-disk configuration. It can be used to determine the wear resistance and friction generated in sliding contacts between a ceramic coating and a suitable counterpart (see below). Pin-on-disk wear testing can be used to simulate the operating conditions in different sliding contacts of technological significance. In the last few years there has been an increasing interest in the use of wear and friction reducing ceramic coatings for such contacts. This European Standard has been developed to provide guidance on the use and interpretation of the test method for evaluating the potential performance of ceramic coatings in these types of contacts and to provide complementary data to that obtained from other wear test methods, e.g. micro-scale abrasion wear testing [2] and reciprocating wear testing [3].

It should be noted that there are many parameters in sliding contacts that affect the magnitude of friction and wear. The aim of performing any wear test is to simulate, as closely as possible, the conditions that occur in the real application. As the deviation between the test conditions and the application conditions becomes larger, the test results will become less relevant. To add confidence to the test results, the appearance of the worn surfaces of the test samples should be compared with those of the worn surfaces of actual components to ensure that similar wear mechanisms have taken place in both cases.

NOTE Although it is relatively easy in a pin-on-disk wear test to reproduce the contact stress experienced in a specific tribological contact, it might be necessary to use additional heating to ensure that the contact temperature approximates to that of the contact being simulated.

The recommended test conditions in this standard should be used when the objective of the testing is to compare the performance of materials in the absence of well defined application conditions.

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5 Principle

The test consists in sliding a loaded pin against a flat disk-such that the pin describes a circular path on the disk, and determining the wear of one or both. Depending on the wearing system being simulated, either the pin or disk or both may be coated with the ceramic coating under test, and testing may be either with or without lubrication. The pin contact face may have either a flat or rounded geometry. If the former is chosen, great care is necessary in order to ensure that the contact faces of pin and disk both lie in the same plane, as any variation from this will produce substantially different contact conditions from those expected for a plane contact. The high contact stress generated by misalignment can be particularly damaging to brittle ceramic materials and can lead to spurious and un-reproducible results. If a rounded geometry is chosen, then the contact conditions will vary throughout the test and affect the analysis of the results.

6 Apparatus

6.1 Pin-on-disk method testing apparatus

The testing apparatus shall consist of:

- the holders for the disk and for the pin;
- the drive system for rotating the disk or for letting the pin slide in a circular path on the fixed disk surface;
- the loading mechanism for pushing the pin and the disk onto each other;
- means to determine the total sliding distance travelled by the pin over the surface of the disk during the test, e.g. rev counter, timer, etc.

Depending on the equipment selected, the test face of the disk may be oriented in either a horizontal or vertical plane and, for the former the pin may be positioned either above or below the disk. Report the orientation used for the test.

NOTE 1 Different test orientations might produce different results for apparently identical test conditions as a consequence of retention or loss of wear debris.

Where flat-ended pins are to be used, means shall be provided to ensure conformance between the pin and the disk so that the contact conditions are reproducible and the contact stress can be calculated, if required.

NOTE 2 One technique that has been used to ensure conformance between a flat ended pin and the surface of the disk has been to grind a flat onto a spherical ball and to mount the ball into a spherical cavity, of the same diameter as the ball, which has been machined into the end of the pin – see Figure 1. Contacting the flat on the ball with the surface of the disk will ensure that conformance is obtained. The use of a pin with a spherical cavity will enable a ball of the same radius of curvature to be accommodated and to act as a ball-ended pin.

In all cases where the geometry referred to in Note 2 is used, the centre of curvature of the cavity shall lie on the, extended, axis of the pin.

Equipment may be provided with a means to deliver lubricant to the contact zone and means to collect used lubricant to enable the isolation and analysis of wear debris.

The testing apparatus may also have a mechanism for measuring the friction force during the test. This can be done e.g. by means of a load cell, a distortion measurement of a leaf spring, or a measurement of rotational torque. The measurement method should not affect any of the test conditions including the frictional condition and the applied load. It is recommended that the accuracy of friction measurement be ± 1 %, or better, of the applied load.

The test system may also be fitted with a normal load cell to measure the applied normal load. The introduction of a normal load cell should not affect any of the test conditions.



Key

- 1 Cylindrical pin with spherical cavity of same radius as ball and centred on extended axis of pin
- 2 Ball with ground flat

Figure 1 — Diagram showing possible arrangement to obtain conformity between flat ended pin and disk

6.2 Test apparatus design requirements

The apparatus shall be rigid such that during operation, the axis of the pin remains orthogonal to the plane of the test surface of the disk at all times.

Any fluctuations in the position of the test surface of the disk relative to the mean position of the pin will result in fluctuations in the testing conditions and in particular in the applied load. Any such fluctuations in the applied load shall be less than 1 % of the nominal applied load.

The loading mechanism shall apply a constant load directly or, for example, through a lever-arm device with attached weight, or by a hydraulic or pneumatic system. The accuracy of the loading system and the limit for oscillations of the load shall be less than 3 % of the nominal load.

The rotation drive mechanism shall be such that the sliding speed remains constant to better than ± 1 % at all times during a test, including under the action of the frictional force that is generated. The friction force might rise considerably during the test. The eccentricity of the drive shaft shall result in an eccentricity of the circular path of no more than 3 % of the width of the wear track. The drive system should be fitted with a revolution counter or equivalent device.

6.3 Instruments for determining the volume loss

Any instrument or method suitable for determining the volume loss may be used. Possible instrument are, for example, contact profilometers and optical microscopes. Mass loss measurements may also be used but care is necessary to ensure that mass changes are the result of wear and not the result of material transfer. Values for the density of the materials under test will be necessary to convert mass loss to volume loss.

6.4 Operating environment

The equipment shall be maintained in a constant environment, fixed temperature and humidity, e.g. by enclosing it in a suitable cabinet or by operating it in a controlled environment laboratory. Care should be taken to avoid accidental contamination of the apparatus with lubricants or other materials that might affect the wear process, e.g. from spray mist from adjacent equipment. Where fresh lubricant is delivered continuously to the contact zone it shall be stored and delivered in such a way as to ensure that its temperature at delivery is nominally constant.

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NOTE 1 Although precise temperature and humidity conditions are not prescribed in this standard, typical ambient operating conditions are: temperature = (23 ± 2) °C; and humidity = (50 ± 10) %.

NOTE 2 The use of a constant environment is necessary to help ensure the stable operation of the test equipment. It should not be considered a requirement of this standard that the tribological contact itself should be maintained under the same fixed conditions. Indeed, there are many situations where additional heating of the contact will be necessary to help ensure the conditions approximate to those in the contact being simulated.

NOTE 3 It has been found for some ceramic materials, see [7] and [8], that variations in humidity, particularly at low levels of humidity (< 40 %), can have far greater influence on the test results than variations in temperature.

Report the conditions used during the test in the test report.

7 Preparation of test pieces

7.1 Substrate material and preparation

Where the test is used to simulate the working conditions in a mechanical device it is recommended that the substrate materials chosen for both pin and plate be representative of the couple in that device. The materials should, where practical, have the same heat-treatment and surface preparation as the components being simulated so as to ensure that they possess the same load bearing capacity and surface texture.

In cases where the test is being used to rank coatings without a specific application in mind, both pin and plate shall be made from materials that will have minimum elastic and zero plastic deformation under the test conditions used. Depending on the test conditions to be investigated, suitable materials might be cemented carbide, cermets, SiC, alumina (> 96 %), hardened and tempered high speed steel, e.g. UNS T 11302 (AISI M2), hardened and tempered bearing steel, e.g. UNS G52986 (AISI E 52100 or 100Cr6), or stainless steel, e.g. UNS S41000 (AISI 410) but this list is by no means exhaustive and other materials might also be suitable. The materials shall be selected with due consideration to the influence of the subsequent coating operation on the final hardness.

NOTE 1 The hardness of the base materials will affect the measurements: the higher this hardness the lower the elastic deformation of the pin and/or of the disk at the contact point and, as a consequence, the lower the stress imposed to the coating. At the disk surface these stresses are periodic in time, possibly leading to fatigue effects. The hardness of the ball and of the disk should be similar to that of the actual component(s). For basic studies on the coating material it is suggested that test piece hardness be as high as possible (i.e. HRc > 64), in order to better isolate the contribution of the coating material properties to wear. High hardness will also help prevent excessive debris formation in tests where either the ball or the disk is uncoated; this could lead to significant effects from three body wear.

Unless otherwise specified, the contact surfaces of both pin and disk shall be polished to a surface finish equal to or better than 0,02 μ m Ra (see ISO 4288) using polishing materials compatible with subsequent coating operations. This will help to better isolate the contribution of the coating material properties to wear The surface of the plate shall have a flatness better that 0,01mm (see ISO 1101) and this condition shall also apply to the contact face of flat ended pins, where used. Care should be taken to ensure that polished surfaces are free of embedded polishing material as this might affect the test results.

Where a flat-ended pin is used, the flat end shall be orthogonal to the axis of the pin.

Where a spherically-ended pin is used, the centre of curvature of the contact face shall lie on the axis of the pin.

NOTE 2 Where a spherically-ended (ball-ended) pin is used, the ball radius should be selected so that, at the nominal applied load, the necessary contact pressure is produced.

NOTE 3 Balls may be used in place of spherically-ended pins provided that they are held rigidly throughout the duration of the test.

Pin and disk dimensions shall be such that no bending of the pin or test surface of the disk occurs during the test.

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7.2 Coating deposition

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Where the test is being used to simulate the working conditions in a mechanical device, the substrate cleaning and deposition conditions selected for the coating shall be as near as practical the same as those that would be used for the components in that device. In particular, those process conditions that might influence the adhesion, chemical phase, preferred orientation or residual stress of the material being deposited shall be carefully monitored and controlled. In all cases, all relevant deposition conditions shall be recorded and this record shall form part of the test report.

NOTE 1 In view of the likely influence of the adhesion, chemical phase, preferred orientation and residual stress on the coating performance, it is recommended that determination of these properties of the coating be made and the results reported. Techniques for determining these additional properties of ceramic coatings are reviewed in Annex A.

In cases where the test is being used to rank coatings without a specific application in mind, deposition of the coating should follow procedures previously established for the material under investigation. If no established conditions exist, for example because the test forms part of a coatings development programme, then care should be taken to ensure that the conditions used are reproducible.

Coating thickness should, where practical, be the same as that used in the device being simulated.

NOTE 2 If no device is being simulated then, depending on the purpose of the test, it is recommended that a range of coating thickness be evaluated.

7.3 Post-coating preparation

In some instances, e.g. where the deposited coating is rough, the surface of the coating should be prepared in some way, e.g. by polishing, following its deposition. All procedures that modify the surface of the coating shall be documented in such a way that they are completely reproducible and this record shall form part of the test report. Where post-deposition preparation of the coating surface is used, care should be taken to remove all extraneous materials from the surface after treatment.