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Advanced technical ceramics - Methods of test for ceramic coatings - Part 12: Reciprocating wear test

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 12: Schwingungs-Verschleißprüfung

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 12 : Essai d'usure en vaet-vient

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Foreword

This document (EN 1071-12:2010) 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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.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:

— *Part 1: 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— *Part 5: Determination of porosity* (withdrawn)

— *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— *Part 7: Determination of hardness and Young's modulus by instrumented indentation* (withdrawn)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*

Parts 8 and 11 are Technical Specifications. CEN/TS 1071-7:2003 was withdrawn on publication of EN ISO 14577-4:2007.

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

Proper evaluation of the wear performance of ceramic coatings is essential to understanding their behaviour and to increasing their use in applications where high performance and predictable life are critical requirements, for example in the aerospace, automotive and biomedical industries. This part of EN 1071 describes a technique for the determination of the wear behaviour of a ceramic coating by reciprocating, under load, a flat or spherically ended pin against a flat plate. Depending on the information required, either the plate 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 system under evaluation. Wear is determined by weight loss, by profilometry, by linear measurement or by a combination of these. Coatings may be tested 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 per se, 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 reciprocating under non-lubricated conditions is well described in [1].

The 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 13 [3], which describe techniques for micro-scale abrasion wear testing and pin-on-disc wear testing of ceramic coatings respectively.

1 Scope

1.1 This European Standard describes a method for evaluating the wear of ceramic coatings by use of a reciprocating wear test whereby a flat or spherically ended pin is reciprocated, under load, against a flat plate. Depending on the conditions being simulated, either the pin or plate 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. The method described is considered to be not suitable for evaluating fretting wear.

1.2 The method is intended for evaluating coatings with a thickness of more than 1 µm, though might also be used for testing thinner coatings.

1.3 The test may be carried out 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 reciprocating motion are given in references [4] – [6].

1.4 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

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

3 Terms and definitions

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

3.1
wear track
line of contact of pin on plate during reciprocation

3.2
wear scar
worn region on the test specimen

3.3
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

3.4
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

3.5
stroke length
total distance traversed by the leading or trailing edge of the pin over the surface of the plate between consecutive reversals in the direction of motion

3.6
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 applied load.

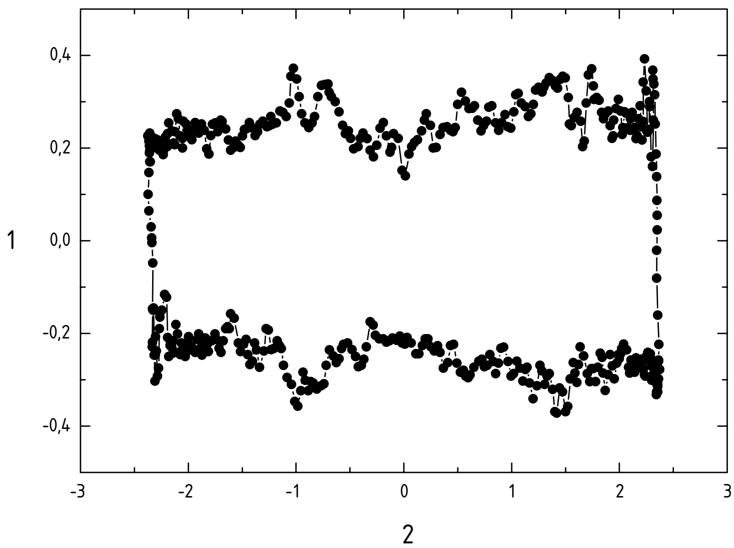
3.7
cycle average coefficient of friction
average value of the instantaneous coefficient of friction calculated for a complete cycle of reciprocation

NOTE Three possible ways of calculating the cycle average coefficient of friction are:

— to average the absolute magnitude of the friction, excluding the values towards the end of the stroke where the value is affected by the pin stopping, and to divide this by the applied load;

— to determine the area under the coefficient of friction-horizontal motion curve for a complete stroke and divide this by twice the stroke length – see Figure 1;

— to pass the instantaneous friction signal through a true analogue r.m.s. to d.c. converter amplifier, the output from which is then data logged at an appropriate sampling rate, which can be quite modest (typically 1 Hz to 10 Hz). Again the average friction signal obtained over a complete cycle should be divided by the applied load to obtain the cycle average coefficient of friction.



Key

1 Friction coefficient
2 Horizontal displacement, in millimetres (mm)

Figure 1 — Coefficient of friction – Horizontal displacement loop for reciprocating test with alumina ball rubbing against titanium nitride coated plate [11]

4 Significance and use

Reciprocating 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 draft 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 pin-on-disk 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 reciprocating 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.

5 Principle

The test consists in reciprocating a loaded pin against a flat plate and determining the wear of one or both components. Depending on the wearing system being simulated, either the pin or plate or both may be coated with the ceramic coating under test, and the test may be carried out 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 plate 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 and materials

6.1 Apparatus construction

6.1.1 General

The apparatus shall be rigid such that, during use under the maximum recommended load and reciprocating speed, the axis of the pin remains orthogonal to the plane of the test surface of the plate at all times.

NOTE 1 Whilst it might be more common for the pin and plate to be held rigidly during the test, additional rotary motion of the pin has been used, e.g. to simulate the conditions encountered in an artificial hip [7]. Where such an approach is adopted, care is necessary to ensure that the axis of the pin remains rigidly orthogonal to the plane of motion of the contacting surfaces. Modelling has shown that, depending on the pin geometry adopted, misalignments of as little as 0,5° can result in more than a tenfold increase in the actual contact stress [8].

Reciprocation may be achieved by means of a crank mechanism driven from an eccentric cam, a linear, electromagnetic actuator drive with sinusoidal motion, or other suitable arrangement, and either the pin or plate may be driven, whilst the other remains stationary. Whatever mechanism is chosen to produce the reciprocation, the motion produced shall be smooth and free from any erratic behaviour. The type of motion used in the test shall be reported, preferably by presenting a position versus time or velocity versus time plot for a full cycle of reciprocation.

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

NOTE 2 It should be appreciated that different test orientations might produce different results for apparently identical test conditions as a consequence of retention of, or loss of wear debris from the contact.

Loading between the pin and the plate may be achieved using dead loading with weights, and may be increased by use of a lever arrangement if necessary; with a calibrated spring, again using a lever arrangement if necessary; or using other suitable means. Whatever loading arrangement is used in the equipment selected it shall be such as to ensure that the load remains nominally constant throughout the duration of the test. The magnitude of the load applied can be determined by the use of suitably calibrated weights, by means of a suitably calibrated load cell, or by other suitable means. However, in all cases the load determined shall be the contact load between pin and plate.

NOTE 3 Dead weight loading systems produce more severe conditions than low mass loading systems (for example spring or pneumatic actuation). This is because, with dynamic conditions, inertia gives rise to shock loading. Dead weight loading systems should be avoided at anything other than very modest reciprocation frequencies, the magnitude of which will depend upon the precise test conditions.

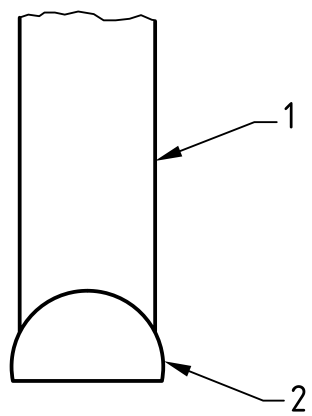
Where flat-ended pins are to be used, suitable means shall be provided to ensure conformance between the pin and the plate in order that the contact conditions are reproducible and so that the contact stress can be calculated, if required.

NOTE 4 One technique that has been used to ensure conformance between a flat ended pin and the surface of the plate 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 2. Contacting the flat on the ball with the surface of the plate will ensure that conformity is obtained. The use of a pin with a spherical cavity will also 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 4 is used, the centre of curvature of the cavity shall lie on the, extended, axis of the pin.

Equipment shall be provided with a suitable means to deliver appropriate lubricant to the contact zone and, where appropriate, means to collect used lubricant so as to enable, where required, the isolation and analysis of wear debris.

NOTE 5 Some investigators have immersed the contact zone of the plate and pin in a bath of fluid. This approach is appropriate for some situations, e.g. for simulating the wear of artificial joints [7].



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 2 — Diagram showing possible arrangement to obtain conforming contact between "pin" and plate – see 6.1.1, Note 4

6.1.2 Friction measurement

Equipment can be provided with a suitably calibrated force transducer to enable the measurement of the frictional force during the test. In view of the cyclic variation of speed and direction of motion, the output of the force transducer should be fed to a suitable recording device, such as a high-speed chart recorder or high-speed digital data recorder, for subsequent analysis. Where the friction measurement is recorded digitally, details of the sampling rate and any data processing shall be noted. Depending on the recording method used, care should be taken to ensure that the recorded signal is not influenced by any mechanical resonance of the test system. Annex A discusses the issue of equipment resonant frequencies and related issues concerning dynamic measurements in reciprocating wear testing.

NOTE 1 In view of the dynamics of reciprocating wear testing, the use of piezoelectric force transducer should be preferred for friction measurement. Although the use of other types of transducers is not prohibited by this standard, due consideration should be given to the relevance of the information obtained by their use.

NOTE 2 It is recommended that a sampling rate of at least 100 samples per reciprocation be used.

NOTE 3 The method of mechanical attachment of the friction force transducer to the apparatus can affect the results obtained. A common practice is to rest the pin holder against the measurement point of the transducer. In this case care is necessary to eliminate friction between the pin holder and the transducer as this might alter the nominal loading between pin and plate. It should be recognised that this arrangement gives different dynamic characteristics to the situation where the pin holder is clamped to the machine frame through the friction transducer.

6.2 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 an environmentally controlled 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.

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 required to help ensure the stable operation of the test equipment, and 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 required to help ensure the conditions approximate to those in the contact being simulated.

NOTE 3 It has been found for some ceramic materials, see [9] and [10], 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 precise conditions used during the test.

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 should 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, such 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 usable. The materials should be selected so as to minimize the influence of the subsequent coating operation on their final hardness.

In the absence of other requirements, the contact surfaces of both pin and plate shall be polished to a surface finish equal to or better than 0,02 µm Ra (see ISO 4288) using polishing materials compatible with subsequent coating operations. The surface of the plate shall have a flatness better that 0,01 mm (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 from embedded polishing material as this might significantly 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 1 Balls may be used in place of spherically-ended pins provided that they are held rigidly throughout the duration of the test.

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

NOTE 2 Although the dimensions of the test plate and pin are not prescribed in this standard, an example of dimensions that have been used for the plate is 80 mm long, 20 mm wide and 10 mm thick, and for the pin is 10 mm diameter and 20 mm long.

7.2 Coating deposition

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 should 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 B.

In cases where the test is being used to rank coatings without a specific application in mind, deposition of the coating should follow normal procedures for the material under investigation. If no normal procedures 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.

NOTE Care should be taken to minimise material removal during any polishing operation