BRITISH STANDARD

BS EN 1071-13:2010

Advanced technical ceramics — Methods of test for ceramic coatings

Part 13: Determination of wear rate by the pin-on-disk method

ICS 81.060.30

British Standards

National foreword

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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of an identical text or by endorsement, at the latest by September 2010, and conflicting national standards shall be withdrawn at the latest by September 2010.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— *Part 5: Determination of porosity* (withdrawn)

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— *Part 7: Determination of hardness and Young's modulus by instrumented indentation* (withdrawn)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

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.

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 µm 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

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)*

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.

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.

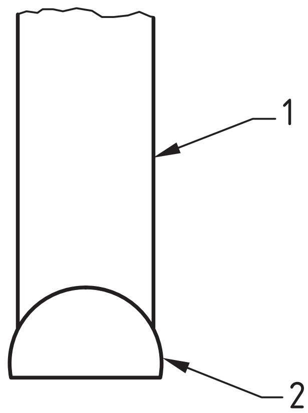
NOTE 2 One technique that has been used to ensure conformance between a flat ended pin and the surface of the disk has been to grind a flat onto a spherical ball and to mount the ball into a spherical cavity, of the same diameter as the ball, which has been machined into the end of the pin – see Figure 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.

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 µm 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.

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 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.

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

7.4 Cleaning

As the presence of surface contamination could have a significant effect upon the results obtained, in all cases the test surfaces of both pin and plate shall be thoroughly cleaned, for example by ultrasonic cleaning in a suitable clean solvent, e.g. high purity heptane, and dried, e.g. in hot air with a hot air blower or in an oven. Inspect the test pieces to ensure that the test surfaces are free from staining and other surface contamination, and if these are found to be present the cleaning process shall be repeated, possibly using different cleaning materials or procedures, until the surfaces are clean and dry. Unless they are to be tested immediately after cleaning, test pieces should be stored in a desiccator prior to testing.

The procedure used for cleaning test pieces should be detailed in the test report.

NOTE 1 Ferromagnetic materials having residual magnetism should be demagnetised before cleaning.

NOTE 2 In cases where a coated sample is tested against an uncoated steel sample, care is necessary to ensure the complete removal of any corrosion inhibitors used to protect the steel. A survey of tribological test procedures revealed the use of a variety of different cleaning solvents and found that the type of cleaning solvent ranks the friction and wear data [9].

8 Testing procedure

8.1 Preliminary setup

If suitable equipment is available, determine the masses of the pin and the disk to at least the nearest 0,01 mg, preferably 0,001 mg, using a calibrated balance. The mass determination shall be made after the components have been left to equilibrate in the same temperature and humidity controlled room or cabinet as the balance.

NOTE 1 Measurements have indicated that up to 24 h might be necessary for such equilibration.

Set the rotation speed of the disk or pin, if variable. Unless otherwise specified it is recommended that a linear speed of 0,1 m/s be used.

Mount the pin and disk in the normal way, ensuring that the test surface of the disk is level with respect to the plane of rotation. Ensure that the pin and plate are rigidly fixed by or in their clamping devices. A minimum diameter of the wear track of 30 mm is recommended.

NOTE 2 Levelling of the test surface of the disk can be checked by the use of a suitable clock gauge, or by ensuring that the force exerted between the pin and disk remains constant over a complete cycle of rotation by, for example, use of a calibrated load cell mounted on the disk.

Switch on the friction transducer, if fitted, and its related recording device and, with the pin and disk out of contact, zero the friction force after the equipment has warmed up.

Bring the pin into contact with the disk and, where a flat-ended pin is used ensure that the pin contacts the plate over the whole of its test surface.

If additional heating of the contact is to be used during the test the heater should be switched on and the power adjusted to give the appropriate contact temperature.

Allow the pin and plate to come into thermal equilibrium with the equipment. Record the temperature and humidity.

8.2 Producing the wear track and scar

8.2.1 After the testing atmosphere has stabilized at the set conditions, and, where additional heating is used, wait for the specimens to reach the set temperature; 30 min will usually be sufficient.

8.2.2 Apply the test load. Unless another value is specified it is recommended that a load of 10 N be used.

8.2.3 Begin the test by starting the sliding motion.

8.2.4 Where appropriate, measure the friction force continuously during the test and record it by using a data logger or other recording devices. A system for averaging the fluctuation with rotation period should be adopted. Before the test starts, the zero of the friction force measurement device should be checked with the specimens out of contact with one another.

8.2.5 When the test is completed, stop the sliding motion and remove the specimens from the holders carefully, in order not to scratch or damage them in anyway.

NOTE Cleaning the specimens after the test may remove wear debris from them, the observation of which can be of interest for a better understanding of the wear mechanisms involved.

It is strongly recommended that the wear test be repeated at least three times under the same testing conditions using new test pieces for each test.

8.3 Evaluation of wear and measurement of wear scars

8.3.1 General

Inspect the friction versus time trace, if available, for abnormal changes in behaviour that might indicate significant events, e.g. fracture or removal of the coating, or a change in the wear mechanism. Record all such observations.

Determine the volume of material lost from both the pin and the disk using one or more of the following procedures.

8.3.2 Mass loss

Determine the mass of the pin and disk to the nearest 0,01 mg, preferably 0,001 mg, and calculate the mass lost from each during the test by subtracting these values from those obtained prior to the test. The mass determinations shall be made after the components have been allowed to equilibrate in the same temperature and humidity controlled room or cabinet as the balance used for the measurement.

NOTE 1 Measurements have indicated that up to 24 h might be necessary for such equilibration.

Convert the mass loss to a volume loss, V_{pin} or V_{disk} for the pin and disk respectively, by dividing the mass lost by the density of the coating. If the density of the coating material is not known then either it will have to be determined using a suitable method or, if no alternative means is available to determine the wear volume, the wear rate will have to be given in terms of a mass loss per newton (N) per metre (m) of sliding.

NOTE 2 Methods that might be suitable for the determination of coating density are described in DIN 53217 Pt 1 [10] and ASTM B 767 [11].

8.3.3 Volume loss − Pin

8.3.3.1 Profilometry

The use of calibrated profilometry to determine the volume loss from the pin requires knowledge of the shape and position of the original surface. If optical profilometry is available then, in the case of spherically ended pins showing little wear, the wear volume can be computed by subtracting the original profile from the profile obtained after the test. In the case of flat ended pins, Annex B provides guidance on how the volume loss can be computed. If, however, the initial surface position or shape cannot be uniquely established, then one of the alternative methods for determining volume loss should be used.

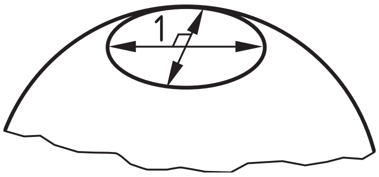
From the measurements calculate the wear volume V_{pin}.

8.3.3.2 Measurement

Ball-ended pin

The recommended procedure when using an optical microscope is described.

On the ball-ended specimen after the test, there will be a roughly circular scar as shown in Figure 2. Measure the minimum diameter and the diameter in the direction perpendicular.



Key

1 Minimum diameter and perpendicular diameter of scar

Figure 2 — Circular wear scar on ball-ended pin or ball specimen

Calculate the wear volume of the pin by the following Equation (1) from the minimum diameter of wear scar and the diameter in a direction perpendicular to it:

$$V_{pin} = \frac{\pi A^3 B}{32 D} \tag{1}$$

where

V_{pin} is the wear volume of ball specimen in cubic metres (m^3);

A is the minimum diameter of wear scar in metres (m);

B is the diameter in direction perpendicular to minimum diameter, in metres (m);

D is the diameter of ball specimen in metres (m).

NOTE When the shape of the wear scar is warped ($B > 1,5\ A$), this formula should not be used.

8.3.4 Flat-ended pin

8.3.4.1 General

The method described in Annex B can readily be adapted for use with a measuring microscope if a profilometer is not available.

Alternatively, provided that the original length of the pin is known then measurement of the length after the test will enable the volume lost to be calculated. The accuracy with which the length before and after the test needs to be known will depend upon the amount of wear that takes place. If in doubt these measurements should be made to an accuracy of better than ± 1 µm.

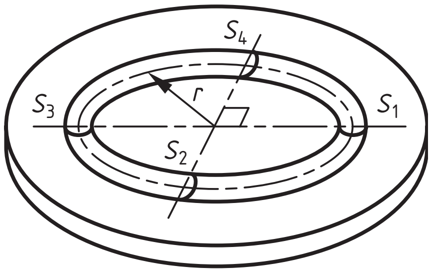
From the measurements calculate the wear volume V_{pin}.

8.3.4.2 Disk specimen

8.3.4.2.1 Profilometry

The procedure suggested when using a contact profilometer is described.

On the disk specimen after the test, there will be a wear track circle as shown in Figure 3. Measure its cross-sectional profile at four places at intervals of 90° using a contact stylus profilometer or similar instrument and obtain the cross-sectional area of the wear track circle at each position.



Key

r radius at the centre of the wear track circle

S_1 to S_4 cross-sectional areas

Figure 3 — Wear track circle on disk specimen

Calculate the wear volume of the disk specimen by the following Equation (2) from the cross-sectional area of wear track circle:

$$V_{disk} = \frac{\pi r (S_1 + S_2 + S_3 + S_4)}{2} \quad (2)$$

where

V_{disk} is the wear volume of disk specimen in cubic metres (m^3);

r is the radius at the centre of the wear track circle in metres (m);

S_1 to S_4 are the cross-sectional areas at four places, spaced uniformly around the wear track circle, in square metres (m^2).

NOTE For S_1 to S_4, if the ratio of the maximum value to the minimum value exceeds 1,5, this method should not be used.

8.3.4.2.2 Measurement

If a profilometer is not available, then the profile of the wear track at the four separate points can be obtained by use of a calibrated travelling microscope. Determine the width, w_1, w_2, …w_n, of the wear scar at four places, spaced uniformly around the wear scar circle. At each of these places measure the depth of the wear scar below the mean surface of the plate at a series of points, e.g. 10, across the scar in order to determine the profile of the scar at each position. From these profiles determine the area of the wear scar at each of the position and proceed as in 8.3.4.2.1 above.

NOTE Depending on the precise geometry of the wear scar, the depth of coating lost from a coated plate can also be determined by the use of the crater grinding (ball cratering) technique (see EN 1071-2). Further discussion on the use of this method is given in Annex C. Despite the destructive nature of this measurement method, it has the advantage over profilometry that it can readily distinguish between material removal and material deformation.

8.4 Calculation of test results

8.4.1 Specific volume wear rate of pin

The specific wear rate of the pin specimen shall be obtained from V_{pin}, obtained by weighing, profilometry or measurement − see Equation (1) − by using the following Equation (3):

$$W_{s(pin)} = \frac{V_{pin}}{PL} \tag{3}$$

where

$W_{s(pin)}$ is the specific wear rate of ball specimen, in square metres per newton (m^2/N);

P is the applied load in newtons (N);

L is the sliding distance in metres (m).

8.4.2 Specific volume wear rate of disk specimen

The specific wear rate of the disk specimen shall be obtained from V_{disk}, obtained by weighing, profilometry or measurement by using the following Equation (4):

$$W_{s(disk)} = \frac{V_{disk}}{PL} \tag{4}$$

where

$W_{s(disk)}$ is the specific wear rate of disk specimen, in square metres per newton (m^2/N);

P is the applied load in newtons (N);

L is the sliding distance in metres (m).

8.4.3 Coefficient of friction

Where the test system is equipped to measure the frictional force, calculate the coefficient of friction from the applied load and the average value of friction force using the following Equation (5):

$$\mu = \frac{F}{P} \tag{5}$$

where

μ is the coefficient of friction;

F is the average value of friction force, in newtons (N);

P is the applied load in newtons (N).

As the coefficient of friction usually changes with sliding distance, initial, steady-state, maximum and minimum values shall be determined and reported, when available.

8.4.4 Rounding off of numerical values

The specific wear rate and the coefficient of friction shall be stated to two places of the significant digits in accordance with ISO 31-0.

9 Repeatability and limits

Figure 4 [12] summarises the data obtained in the frame of a VAMAS (Versailles Advanced Materials and Standards) programme to develop wear test methodologies for inorganic coatings using the ball-on-disk configuration. For each couple (ball/disk) evaluated, the mean (dark line), standard deviation and spread of results are given. The conclusions of this study were:

— The steel ball/titanium nitride (ball/TiN) test couple showed unreproducible TiN wear particle transfer and embedding in the balls, with non-homogeneous wear of the TiN coatings, steel transfer and high scatter of wear measurements.

— The TiN/TiN test couple showed the disadvantage of possible wear through of the ball coatings and very low wear values for short distance runs.

— The Si_3N_4/TiN gave the highest reproducibility of friction and wear measurements.

The biggest difficulty with testing thin ceramic coatings is the small volumes of material that can typically be removed before exposing the substrate. For example, the change in mass resulting from the generation of a wear scar 2 mm wide, 30 mm in diameter and 4 µm deep in titanium nitride will be approximately 4 mg. Using a balance with an accuracy of ± 10 µg would result in an error of ± 0,25 %, which would be compounded by errors in the known value of the density of the film. If the volume of the wear scar was measured by direct measurement, with all dimension being measured to ± 1 µm, then the error in determining the volume would be around ± 25 %, i.e. 100 times the error when mass loss is used to determine the volume. Clearly both of these errors will increase as the depth of the wear scar reduces, becoming approximately ± 1 % and ± 100 % respectively when the depth becomes 1 µm. This demonstrates the extreme care needed when determining the volumes of wear scars by direct measurement. Use of, calibrated, profilometry or crater grinding will provide results that are subject to significantly smaller errors than can be achieved with direct measurement. Given the sensitivity and ease of use of the former, profilometry should be considered the preferred method for determining wear volumes.

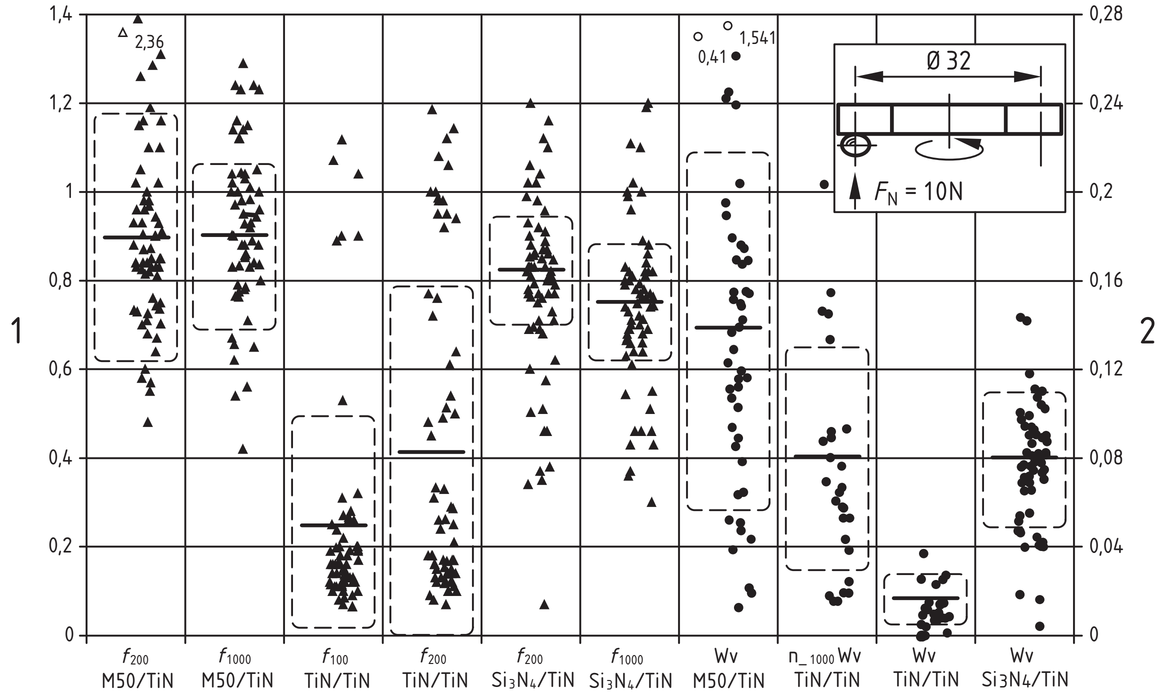


Figure 4 — Heuristic graph of individual results of coefficient of friction and wear volume under dry sliding of 23 laboratories (F_N = 10 N; v = 0,1 m/s, sliding distance = 1 000 m – except where indicated, e.g. f_{200} = 200 m, relative humidity = 50 %) [12]

10 Report

The test report shall be in accordance with EN ISO 17025 and shall contain the information listed below.

NOTE ASTM G118 [13] recommends a format and content for recording wear test data suitable for establishing data bases.

a) Name and address of testing establishment;

b) date of the test, unique identification of the report and of each page, customer name and address;

c) a reference to this standard, i.e. determined in accordance with EN 1071-13;

d) manufacturer, type and serial number of test equipment and description of any significant modifications made since manufacture;

e) manufacturer and type of equipment used for measuring mass loss and wear scar dimensions including details of calibration procedures;

f) description of the test material (material type, manufacturing code, batch number, date of receipt, coating process used, relevant deposition conditions, and any other relevant information);

g) test-piece condition (dimensions, coating thickness, coating type, test-piece preparation procedure including post coat treatments, surface roughness as *Ra* value, if known, and substrate composition and heat treatment);

h) details of the test procedure used including orientation of test surfaces, dimensions of pin, radius of wear scar, relative velocity of pin with respect to disk, normal force, total number of revolutions/total distance travelled by pin over the surface of the disk for each test, and composition and flow rate of lubricant if used;

i) results of the preliminary inspection of the wear scars, including removal or breakthrough of coating, uniformity of wear scars, material transfer and an assessment of the principal wear mechanism involved;

j) the value of the friction force at the start, middle and end of the test, together with changes in behaviour that might indicate significant events, e.g. fracture or removal of the coating, or a change in the wear mechanism. Provide a friction versus time plot for each test, if available;

k) all test results as wear rates for the coating on the pin and disk, as appropriate, and of the uncoated surfaces, where present, and, if appropriate, an uncertainty statement at the 95 % confidence limit which includes measurement and calibration errors (see ISO GUM);

l) any other relevant comments, noting, for example, where different procedures to those recommended have been used;

m) a warning that results from different tests should only be compared when the wear mechanisms and contact conditions are the same.

To obtain a representative value for the specific wear rate under the same testing condition, the arithmetic mean of all values obtained shall be used, and for the coefficient of friction, the arithmetic mean of all steady-state values obtained shall be used.

Annex A
(informative)

Methods for the determination of coating properties likely to be relevant to coating performance in a pin-on-disk wear test

A.1 Relevant properties

The coating properties most likely to be relevant to the performance of a ceramic coating in a reciprocating wear test include phase composition, preferred orientation and residual stress. Where composition, orientation and residual stress cannot be determined, either because the techniques are unavailable or because of coating morphology factors – see A.2 and A.3 – determination of the coating hardness will provide a useful, though less satisfactory, means of characterising the coating.

A.2 Phase composition and preferred orientation.

The simplest way to determine the phase composition and preferred orientation of a, polycrystalline, ceramic coating is to use x-ray diffractometry – see for example [14]. However, difficulties can arise where films display very strong preferred orientations, particularly if high levels of internal stress result in large shifts in peak positions, and where unconventional alloy, multilayer or graded structures are to be tested. In such cases, or where for other reasons coating materials cannot be identified by normal x-ray diffractometry, alternative methods of analysis, preferably offering chemical bonding information, should be used.

A.3 Residual stress

Two principal methods exist for determining residual stress – x-ray diffraction using the so-called $\sin^2$ psi method, and analysis of mechanical bowing using the Stoney Formula. The first of these offers a potential method to determine the stress in the test coating prior to wear testing, provided that it is deposited on the disk. However, it relies upon the presence of good quality, high angle peaks in the x-ray diffraction trace from the specimen, as well as knowledge of the Poisson's ratio of the coating material. The second method needs the coating to be deposited on a well-characterised substrate, which undergoes elastic deformation. This deformation is measured and related to the residual stress in the coating through the so-called Stoney formula.

The second of these techniques is described in CEN/TS 1071-11 – see [15].

A.4 Hardness

Where the other techniques are either unavailable or unusable, then measurement of the coating hardness can provide a means of characterising the coating. A technique for measuring the hardness of ceramic coatings is given in EN ISO 14577-4 [16]. However, it needs to be kept in mind that the hardness of a coating is not a unique material property and might have little or no bearing upon its performance in a pin-on-disk wear test.

Annex B
(informative)

Determination of volume loss from the pin using profilometry

The method uses a specially profiled pin that enables the position of the unworn surface to be uniquely identified. Figure B.1 shows the cross section of such a pin that should be carefully produced with two well defined steps, the size of which depends upon the amount of wear expected. The step closest to the wear surface – step A – is used to determine the axial wear that has taken place, whilst the second step – step B – provides a reference dimension to determine whether thermal or other effects experienced during the test have influenced the pin dimensions. Measuring the height of the two steps before (h_{iA} *and* h_{iB}) and after (h_{aA} *and* h_{aB}) the test enables the apparent axial wear ($h_{iA} - h_{aA}$) to be corrected for any thermal or other effects that might have affected the pin dimensions. The wear volume is then given by Equation (B.1):

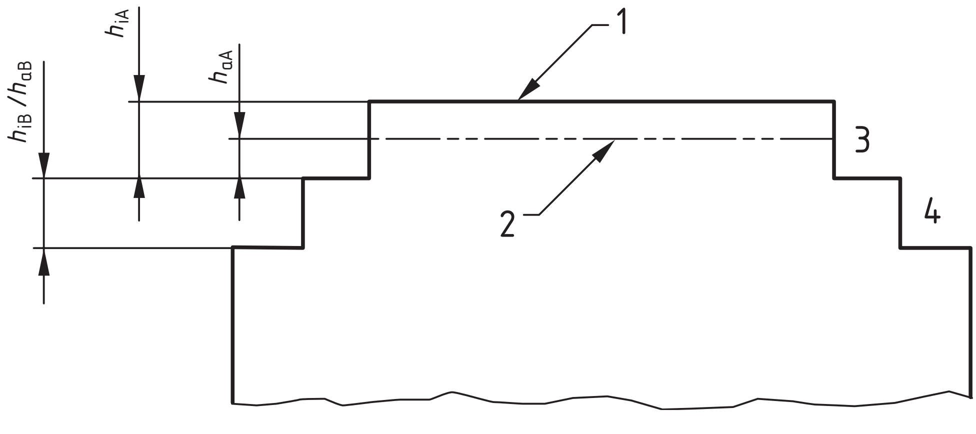
$$V = A^{*}(h_{iA} - h_{aA})\frac{h_{iB}}{h_{aB}} \tag{B.1}$$

where

*A** is the area of the wear face of the pin after the steps have been produced.

Typical dimensions for each of the two steps are 1 µm to ~ 100 µm high and ~ 100 µm wide, depending on the amount of wear expected and the initial diameter of the pin.

Measurement of the step heights can most conveniently be carried out using contact probe profilometry, although the use of a measuring microscope will provide a less accurate alternative.



Key

1 Wear surface before test

2 Wear surface after test

3 Step A

4 Step B (Reference step)

Figure B.1 — Cross section of profiled pin designed to simplify measurement of linear wear

Annex C
(informative)

Determination of wear of coated disk by ball crater technique

Depending on the precise geometry of the wear scar, the depth of coating lost from a coated disk can be determined by the use of the crater grinding (ball cratering) technique (see EN 1071-2). By placing craters in the worn and unworn regions of the coating then, provided that the craters penetrate the coating, the thickness of the coating present at each position can be determined by careful measurement of crater dimensions and use of the formulae in EN 1071-2. The depth of coating lost in the wear test can then be determined by subtracting the coating thickness remaining in the scar from the thickness of the unworn coating. Alternatively, if the crater intersects both edges of the scar, then the loss of coating within the scar can be determined without the need to penetrate the coating. Placing a ball crater in the correct position can readily be achieved with the aid of a travelling microscope. This should be focused on the centre of a crater, produced in any sample, with the sample still in position on the cratering instrument. Without moving the microscope, this sample is removed and replaced with the sample under investigation, and the position of the latter is adjusted until the area of interest is in the field of view of the microscope.

In most cases the wear scar will not have an ideal rectangular or circular cross section and it will be necessary to determine the actual scar profile. For narrow scars (< ~ 1 mm) a single crater can normally be produced which intersects both sides of the scar, and careful measurement of the crater dimensions will enable the scar profile to be determined, though great care is required in the measurement and interpretation of the craters produced. Figure C.1 shows an image of such a crater, and Figure C.2 shows the wear scar profile derived from this image. Figures C.3 and C.4 show the profile in Figure C.2 plotted onto the profiles obtained with a contact probe profilometer and a non-contacting optical profilometer respectively, and both show good agreement between the measured and calculated profiles. It should be noted that the three profiles will not be for exactly the same cross section of the scar and this might account for some of the differences seen. For wear scars greater than 1 mm or 2 mm in width, two or more craters will normally be required to span the scar. In all cases, it is recommended that measurements be made on photographic images taken at known magnifications.

NOTE Image analysis software packages are available that provide circle fitting routines and automatic layer thickness determination for video captured images of ball craters. Such packages can significantly reduce the work involved in determining loss of film thickness and scar profiles.

Depending on the size of the ball and the abrasive used to produce the craters, the ball crater method of film thickness determination offers an accurate and sensitive method of determining the amount of wear that has taken place on the disk in a pin-on-disk wear test. With care the method might also be applicable to evaluating the wear of coatings on pins. Although preferences vary, balls are typically around 25 mm diameter and are used with 1 µm diamond abrasive, although 0,25 µm diamond abrasive will increase the resolution available from the method. However, the technique is destructive and its use prevents further testing being carried out in areas where it has been used to measure wear scars. Despite this disadvantage, the technique allows wear and material deformation to be clearly differentiated, which is not the case when profilometry is used.

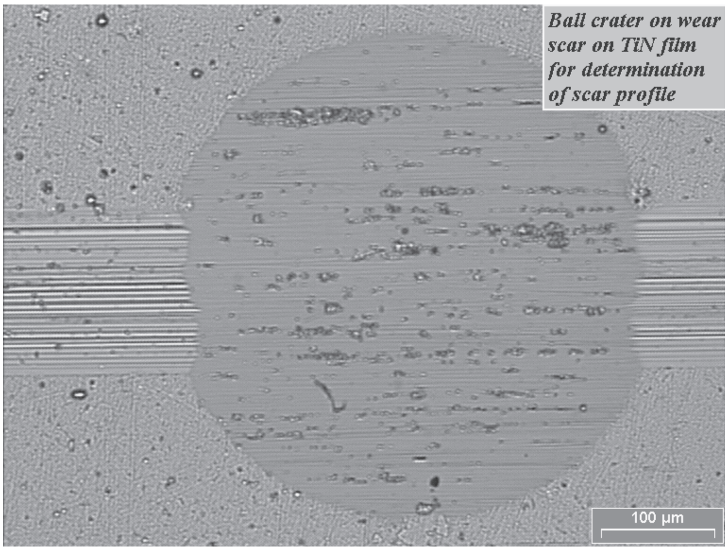
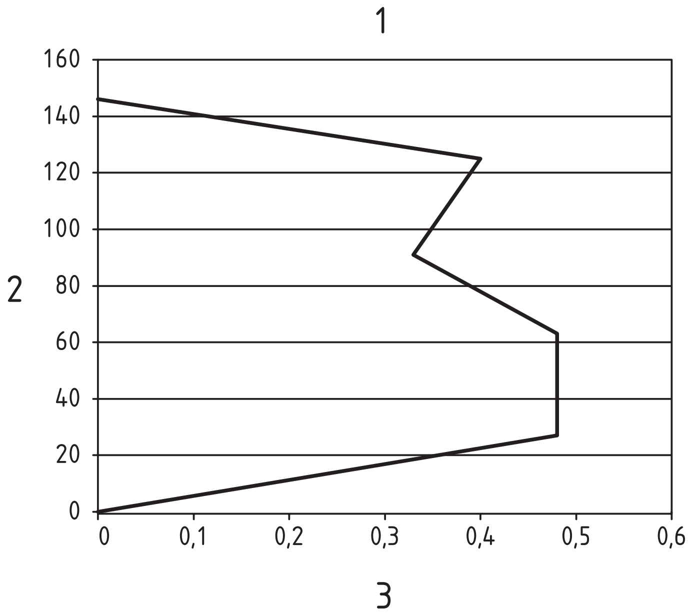


Figure C.1 — Image of ball crater on a wear scar produced by reciprocating a WC ball on a film of TiN deposited by arc evaporative PVD



Key

1 Profile of wear scar

2 Distance from lower edge of scar, in micrometres

3 Depth of scar, in micrometres

Figure C.2 — Profile of the wear scar immediately to the left of the ball crater in Figure C.1 and determined from the ball crater shown

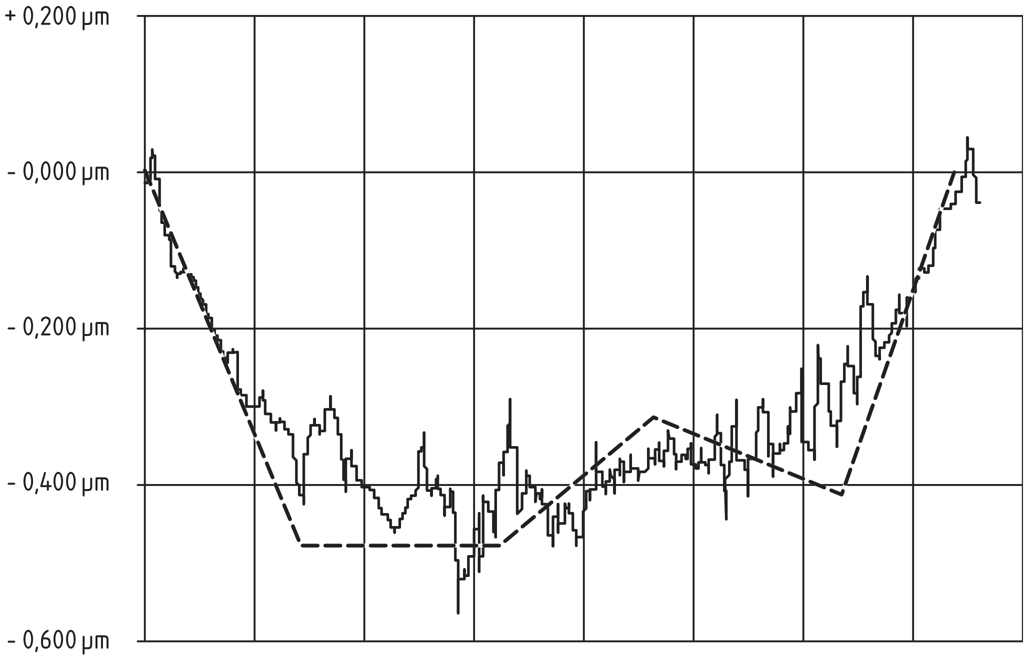
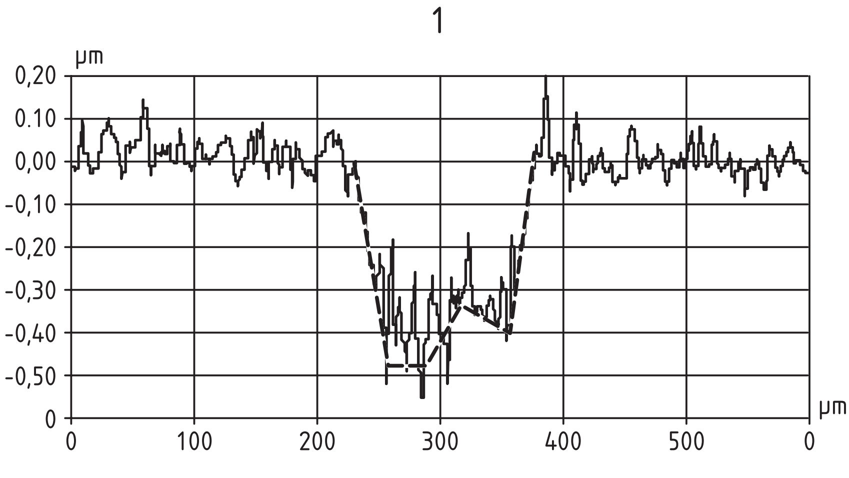


Figure C.3 — Profile of the wear scar shown in Figure C.1 determined using a contact probe profilometer overlaid with the profile from Figure C.2 rotated through 90°



Key

1 X profile

Figure C.4 — Profile of the wear scar shown in Figure C.1 determined using a non-contact, optical profilometer overlaid with the profile from Figure C.2 rotated through 90°

Bibliography

[1] ASTM G99, *Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus*

[2] EN 1071-6, *Advanced technical ceramics — Methods of test for ceramic coatings — Part 6: Determination of the abrasion resistance of coatings by a micro-abrasion wear test*

[3] EN 1071-12, *Advanced technical ceramics — Methods of test for ceramic coatings — Part 12: Reciprocating wear test*

[4] DIN 51834 Pt 2, *Testing of lubricants — Tribological test in the translatory oscillation apparatus — Determination of friction and wear data for lubricating oils*

[5] ASTM D6425-02, *Standard test method for measuring friction and wear properties of extreme pressure (EP) lubricating oils using SRV test machine*

[6] ASTM 6079-99, *Evaluating lubricity of diesel fuels by the high-frequency reciprocating rig (HFRR)*

[7] Gee, M G, The Formation of Aluminium Hydroxide in the Sliding Wear of Alumina, Wear, **153** (1992) 201-227

[8] Gee, M G and Butterfield, D, The Combined Effect of Speed and Humidity on the Wear and Friction of Silicon Nitride, Wear, **162-164** (1993) 234-245

[9] ASTM STP 1404, *Bench testing of industrial fluid lubrication and wear properties used in machinery applications*

[10] DIN 53217 Pt 1, *Determination of density of paints, varnishes and similar coating materials — Survey of test methods*

[11] ASTM B 767, *Standard guide for determining mass per unit area of electo-deposited and related coatings by gravimetric and other chemical analysis procedures*

[12] Santner, E and Koehler, N, Tribological testing of TiN-coatings in dry sliding contacts — Evaluation of an international multilaboratory project, WORLD Tribology Congress, ISBN 1-86058-109-9, 1997, p. 501

[13] ASTM G118, *Standard Guide for Recommended Format of Wear Test Data Suitable for Data Bases*

[14] EN 13925, *Non-destructive testing — X-ray diffraction from polycrystalline and amorphous materials*

[15] CEN/TS 1071-11, *Advanced technical ceramics — Methods of test for ceramic coatings — Part 11: Determination of internal stress by the Stoney formula*

[16] EN ISO 14577-4:2007, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 4: Test method for metallic and non-metallic coatings (ISO 14577-4:2007)*

[17] ISO 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

[18] ISO 1101, *Geometrical Product Specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out*

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