
**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Determination of the abrasion resistance
of coatings by a micro-scale abrasion
test**

*Céramiques techniques — Détermination de la résistance à l'abrasion
des revêtements par essai d'abrasion à micro-échelle*
(standards.iteh.ai)

ISO 26424:2008

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 26424 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of the abrasion resistance of coatings by a micro-scale abrasion test

1 Scope

This International Standard specifies a method for measuring the abrasive wear rate of ceramic coatings by means of a micro-scale abrasion wear test based on the well-known crater-grinding technique used for coating thickness determination in ISO 26423 [1].

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

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

2 Normative references

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

ISO 3290-1, *Rolling bearings — Balls — Part 1: Steel balls*

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

3 Terms and definitions

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

3.1

abrasive wear rate

abrasive wear coefficient

K

volume of material removed in unit sliding distance under a normal contact load of 1 N

4 Significance and use

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

The purpose of this International Standard is to provide a method for measuring the abrasion resistance of both thin and thick coatings and of bulk materials. The test can be carried out on flat surfaces or surfaces with a known radius of curvature and requires test pieces measuring only a few square millimetres. However, the calculations described in Clause 9 apply only to flat test pieces and are applicable only to homogeneous single-layer coatings. Errors may occur if the test is used on inhomogeneous coatings. References [4] and [5] give details of analytical treatments for determining the wear rate of coatings on curved surfaces.

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

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

5 Principle

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

6 Apparatus and materials

6.1 Test system

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

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

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

6.2 Test balls

The balls used are typically 25-mm-diameter hardened steel, e.g. UNS G52986 (SAE 52100) and shall, prior to any conditioning, conform to the requirements of ISO 3290-1.

NOTE 1 Balls can be used in a polished condition, but it has been found ^[7] that the test behaviour is erratic and poor results are obtained if balls are used without conditioning.

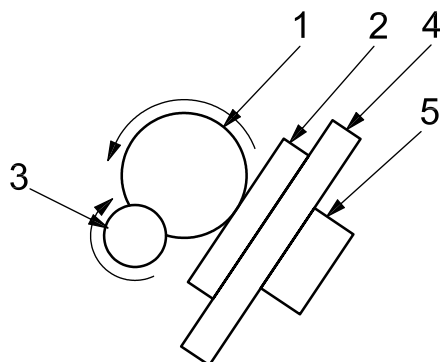
The recommended conditioning treatment consists of running the new test ball for at least 300 revolutions on a non-critical part of the test piece, or another suitable surface, under normal test conditions and repeating this for at least five different orientations of the ball before starting the test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

NOTE 2 A flat, ground steel coupon with a hardness of between 200 HV30 and 800 HV30 has been found to be suitable for conditioning the ball.

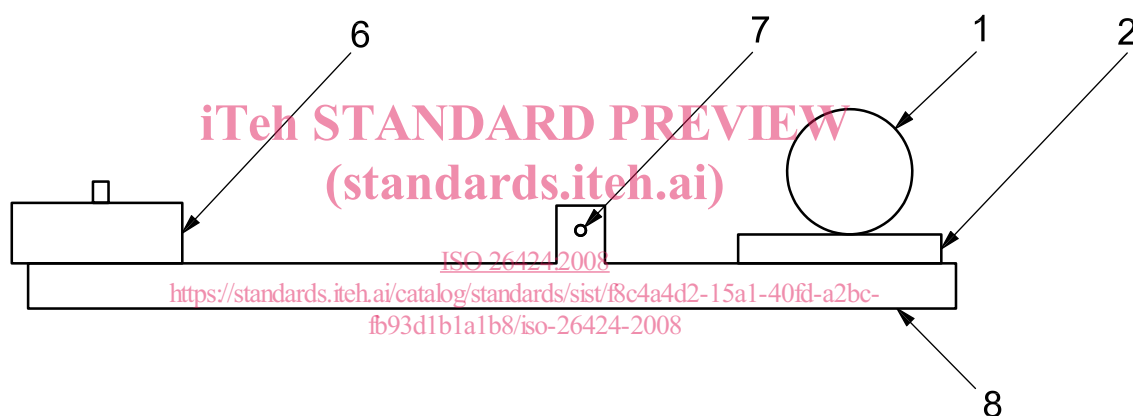
NOTE 3 Following conditioning, balls have been found to be usable for around 50 individual craters, depending on the precise conditions used.

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

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



a) Free-ball system



b) Fixed-ball system

Key

- | | |
|----------------------|---------------|
| 1 ball | 5 load cell |
| 2 test piece | 6 weight |
| 3 drive shaft | 7 pivot point |
| 4 test piece support | 8 lever |

Figure 1 — Two different types of ball cratering system

6.3 Abrasive slurry

In all cases, a slurry of silicon carbide (SiC) or another suitable abrasive in a suitable liquid, normally water, shall be used.

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

The use of different abrasive media will produce different wear rates, and results shall not be compared unless they are obtained from craters produced under identical conditions.

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

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

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

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

a) Dilute (promotes grooving wear)

Concentration 2 % by volume.

For SiC, for example, with a density of $3,2 \text{ g}\cdot\text{cm}^{-3}$, this is achieved by mixing 6,4 g of SiC into 98 cm^3 of distilled or deionized water.

b) Concentrated (promotes rolling wear)

Concentration 20 % by volume.

For SiC, for example, with a density of $3,2 \text{ g}\cdot\text{cm}^{-3}$, this is achieved by mixing 80 g of SiC into 100 cm^3 of distilled or deionized water.

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

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

It is recommended that preliminary testing be undertaken to ensure that the slurry concentration chosen produces the wear mode(s) of interest during the test.

6.4 Measurement of crater dimensions

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

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

Profilometry may lead to results which are different from those obtained by optical-microscopy evaluation of the crater size, due to rounded crater edges. Results of tests evaluated by different measurement methods shall not be compared to each other.

7 Preparation of test pieces

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

NOTE Test pieces with non-flat surfaces can also be tested, but the analysis required to determine the wear rate of coating and substrate will be different to that given in this International Standard (see References [4] and [5]).

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

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

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

- a) ultrasonically clean in a suitable solvent;
- b) rinse;
- c) dry in an oven at $110\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$ for 10 min.

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8 Test procedure

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8.1 Different types of test

8.1.1 Type A: no perforation of coating

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

8.1.2 Type B: perforation of coating

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

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

8.2 Type A test: no perforation of coating

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

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

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

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

In free-ball systems, the friction due to the ball rotation causes the normal force acting on the test piece to be different from that when the ball is stationary (see Reference [8]). In such test systems, a load cell should preferably be employed to measure the true normal force.

8.2.3 Start the slurry feed and ball rotation and ensure that the ball is completely coated in the contact zone during its first complete revolution. The feed rate of the slurry shall be sufficient to ensure that the area of contact between the ball and test piece is always well wetted by the slurry. The slurry shall not be recirculated. Report the ball rotation speed used.

8.2.4 Record the ambient temperature during the test series. Also record the humidity if this is likely to affect the viscosity of the slurry, e.g. where a hygroscopic liquid is being used.

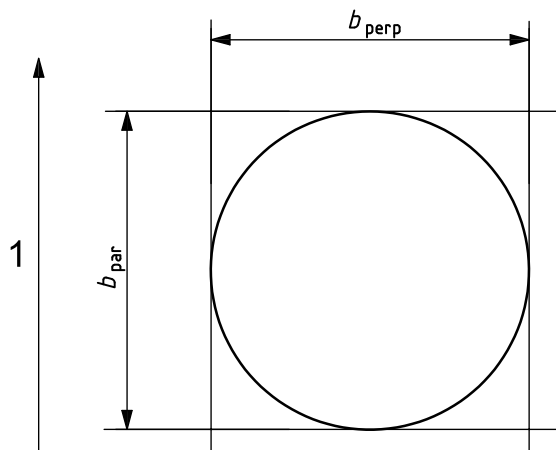
8.2.5 Record the normal load and any variation during the test.

8.2.6 Stop the test (motor and slurry feed) after the predetermined test duration.

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

8.2.7 When the test has been completed, remove the test piece and clean it using the same procedure as that used prior to testing (see 7.3).

8.2.8 Measure the diameter, b , of the crater both parallel and perpendicular to the direction of ball rotation (see Figure 2). If b_{par} and b_{perp} differ by less than 10 %, then take the average of these measurements as the diameter of the crater. Craters that do not meet this condition shall not be used for the calculation of wear rates.



Key

- 1 direction of ball rotation
- b_{perp} crater diameter perpendicular to the direction of ball rotation
- b_{par} crater diameter parallel to the direction of ball rotation

Figure 2 — Measurement of crater with no perforation of coating

8.2.9 Calculate the abrasive wear rate of the coating using the method described in 9.1.

8.2.10 Perform the test at least three times on each test piece.

8.3 Type B test: perforation of coating

8.3.1 For apparatus that allows exact relocation of the ball in the crater after each measurement of crater diameter, a single crater, which is measured after each test, may be used. Otherwise, use a series of craters produced using increasing test durations.

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

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

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

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

In free-ball systems, the friction due to the ball rotation causes the normal force acting on the test piece to be different from that when the ball is stationary (see Reference [8]). In such test systems, a load cell should preferably be employed to measure the true normal force.

8.3.4 Start the slurry feed and ball rotation and ensure that the ball is completely coated in the contact zone during its first complete revolution. The feed rate of the slurry shall be sufficient that the area of contact between the ball and test piece is always well wetted by the slurry. The slurry shall not be recirculated. Report the ball rotation speed used.

8.3.5 Record the ambient temperature during the test series. Also record the humidity if this is likely to affect the viscosity of the slurry, e.g. where a hygroscopic liquid is being used.

8.3.6 Record the normal load and any variation during the test.

8.3.7 Stop the test (motor and slurry feed) at predetermined intervals. Check if perforation of the coating has occurred. If perforation has occurred and if *in situ* measurement of the crater diameter and precise relocation of the ball is possible, remove the ball and, after appropriate cleaning to enable the crater in the substrate to be seen clearly, measure the diameter, a , of the crater in the substrate, both parallel and perpendicular to the direction of ball rotation (see Figure 3). When the diameter has been measured, replace the ball and restart the test (motor and slurry feed). Alternatively, move the test piece so that a new position may be worn during the next test in the series.

For small degrees of penetration into the substrate, the nature of the the abrasion process leads to an uneven edge to the crater in the substrate. This introduces a measurement uncertainty in the crater size that can be greater than the measurement error. It has been found^[10] that the measurement error typically dominates when the depth of penetration into the substrate exceeds the abrasive-particle size, d . This condition is satisfied^[10] if the diameter of the crater in the substrate is greater than the product $8Rd$, where R is the ball radius and d is the mean abrasive-particle size. If the measured diameter of the crater in the substrate is less than $8Rd$, then this measurement shall not be used in the analysis of results in Clause 9.

NOTE The number of revolutions that is required will depend on the material being tested and the test conditions employed. A series of craters produced in 400, 600, 900, 1 200, 1 500 and 2 000 revolutions has been found to be appropriate for a test on a titanium nitride coating of about $3 \mu\text{m}$ thickness deposited on a hardened and tempered high-speed steel substrate, using a 20 % by volume SiC abrasive slurry, a load of 0,2 N, a ball diameter of 25 mm and a speed of $0,1 \text{ m}\cdot\text{s}^{-1}$.