SLOVENSKI STANDARD
SIST EN ISO 20502:2016

01-junij-2016

Nadomešča:
SIST EN 1071-3:2005

Fina keramika (sodobna keramika, sodobna tehnična keramika) - Ugotavljanje adhezije keramičnih prevlek s preskusom praskanja (ISO 20502:2005, vključno s popravkom Cor 1:2009)

Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of adhesion of ceramic coatings by scratch testing (ISO 20502:2005 including Cor 1:2009)

Hochleistungskeramik - Bestimmung der Haftung von keramischen Schichten mit dem Ritztest (ISO 20502:2005 einschließlich Cor 1:2009)

Céramiques techniques - Détermination de l'adhérence des revêtements céramiques par essai de rayure (ISO 20502:2005 y compris Cor 1:2009)

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EN ISO 20502

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English Version

Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of adhesion of ceramic coatings by scratch testing (ISO 20502:2005 including Cor 1:2009)

Céramiques techniques - Détermination de l'adhérence des revêtements céramiques par essai de rayure (ISO 20502:2005, y compris Cor 1:2009)

Hochleistungskeramik - Bestimmung der Haftung von keramischen Schichten mit dem Ritztest (ISO 20502:2005 einschließlich Cor 1:2009)

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

The text of ISO 20502:2005 including Cor 1:2009, has been prepared by Technical Committee ISO/TC 206 “Fine ceramics” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 20502:2016 by Technical Committee CEN/TC 184 “Advanced technical ceramics” the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by October 2016, and conflicting national standards shall be withdrawn at the latest by October 2016.

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Endorsement notice

The text of ISO 20502:2005 including Cor 1:2009, has been approved by CEN as EN ISO 20502:2016 without any modification.

INTERNATIONAL STANDARD

ISO 20502

First edition
2005-08-15

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of adhesion of ceramic coatings by scratch testing

Céramiques techniques — Détermination de l'adhérence des revêtements céramiques par essai de rayure

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Case postale 56 • CH-1211 Geneva 20
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Fax + 41 22 749 09 47
E-mail copyright@iso.org
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 20502 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of adhesion of ceramic coatings by scratch testing

1 Scope

This International Standard describes a method of testing ceramic coatings by scratching with a diamond stylus. During a test, either a constant or increasing force normal to the surface under test is applied to the stylus so as to promote adhesive and/or cohesive failure of the coating-substrate system. The test method is suitable for evaluating ceramic coatings up to a thickness of 20 μm and might also be suitable for evaluating other coating types and thicknesses.

The International Standard is intended for use in the macro (1 to 100 N) force range. The procedures may also be applicable to other force ranges. However, appropriate calibration is essential if the normal forces at which failure occurs are to be quantified.

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 4288, *Geometric Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

ISO 6508-2, *Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T)*

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

3 Principle

The scratch test is designed for the assessment of the mechanical integrity of coated surfaces. The test method consists of generating scratches with a stylus of defined shape (usually a diamond with a Rockwell C geometry) by drawing it across the surface of the coating-substrate system to be tested, either under a constant or progressive normal force (see Figure 1). Failure events are detected by direct microscopic observation of the scratch and sometimes by using acoustic emission and/or friction force measurement.

The driving forces for the failure of the coating-substrate system in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses and the residual internal stress present in the coating. The normal force at which failure occurs is called the critical normal force L_C.

NOTE 1 The term “critical load” is frequently used in place of “critical normal force”. The use of the term “critical load” is deprecated because the failure is typically initiated by the application of a force rather than a load.

NOTE 2 In a scratch, a number of consecutive coating-failure events may be observed at increasing critical normal-force values. Failure by cracking through the coating thickness (through-thickness cracking) usually occurs at lower normal forces than detachment of the coating. Therefore, it is quite common to characterize the onset of cracking by the critical normal force L_{c1}, while the onset of coating detachment defines the critical normal force L_{c2}. In general, a series of failure modes are observed and used to study the mechanical behaviour of the coated surface, where the onset of the nth failure mode defines the critical normal force L_{cn} (see Figure 2).

NOTE 3 The critical normal forces at which the failure events appear depend not only on the coating adhesion strength but also on other parameters, such as rate of increase of normal force, traverse speed, diamond-tip wear, substrate and coating roughness, some of which are directly related to the test itself, while others are related to the coating-substrate system.

4 Apparatus and materials

4.1 Scratch tester

A scratch tester is an instrument used to rigidly hold the stylus and to apply both the normal force and the driving force to produce scratches. A schematic of a typical arrangement is shown in Figure 3.

NOTE 1 In general, spring-deformation-controlled normal-force instruments are used in which the deformation of a spring is used to achieve the chosen force programme. Magnetically driven assemblies are also available.

Where required, the scratch tester can be equipped with acoustic emission (AE) and/or friction force (FF) transducers.

NOTE 2 Although it is attractive to use such methods for the on-line automatic quality control of coated parts, these techniques cannot discriminate between cohesive and adhesive failures, nor do they always detect the first occurrence of failure. Hence, AE and FF signals cannot be used as a reliable means for determining scratch-test critical normal forces. These techniques can at best be used as a warning system in the quality control of coated components, and then only after a large series of experiments on the same coating type to establish the statistics of correlation with a certain failure mode. Inspection of the scratch track by microscopic observation remains the only reliable means of associating a failure event with a measured critical normal force.

To meet the requirements of this International Standard, scratch testers shall comply with the calibration requirements of Annex A.

4.2 Diamond stylus

This consists of a rigidly mounted diamond normally having a Rockwell C geometry in accordance with the requirements of ISO 6508-2.

The stylus shall be inspected regularly to check for contamination and changes in geometry. If damage is observed at 200× or lower magnification then the stylus shall be changed (see Reference [1]), and if either damage or contamination is observed, the test results since the last inspection shall be disregarded. If the friction force increases at a constant normal force during operation, this is a presumption of contamination of the stylus.

NOTE 1 Uncertainties in the Rockwell C stylus tip shape and manufacturing defects are a major source of error for the scratch test method. The use of an imperfect stylus may result in different values of critical normal force when the stylus is rotated in its holder. Control of the stylus shape is imperative, in the as-received condition as well as during usage, to detect wear at the tip. Wear usually occurs in the form of ring cracks or crater wear, which are easily visible under a reflected-light microscope (magnification > 100×).

NOTE 2 A certified reference material (BCR-692) has been developed and is available from the Institute of Reference Materials and Measurements, European Commission Joint Research Centre, Retieseweg, B-2440 Geel, Belgium (www.irmm.jrc.be)[1)]. This material, a diamond-like carbon coated substrate, presents three repeatable failure events at known critical normal-force intervals, and is available for verification purposes. This can provide a good indication of overall performance, including stylus condition and calibration.

5 Preparation of test piece

5.1 General requirements

A representative specimen of the product to be tested shall be used.

Substrate, interface and coating shall be as homogeneous as possible with respect to composition, microstructure, density, residual stress and thickness along the entire scratch length (test zone).

5.2 Surface roughness, waviness and levelling

The surface of the specimen shall have a uniform statistical roughness. The surface roughness Ra, measured according to the procedures specified in ISO 4288, shall not exceed 0,5 µm.

NOTE 1 For spring-deformation-controlled normal-force instruments (typical spring constant: 0.02 N/µm), the normal force depends on the roughness and waviness of the surface. A surface roughness value Ra of 0,5 µm may lead to normal-force oscillations of 0,1 N. Normal-force variations of less than 1 N (1 % of the typical force range) require a waviness and/or levelling error smaller than 50 µm.

NOTE 2 In general, the critical force is reduced with increasing surface roughness by the concentration of stresses at roughness peaks, as well as by the poorer cleanliness properties of rough substrates prior to coating.

The test surface shall be levelled with respect to the stylus/specimen traverse-displacement direction, see Annex A. In practice, this is easily attained for flat specimens held on the sample holder. Cylindrical specimens require additional alignment facilities.

The specimen-levelling mechanism should be stiff to preclude the variation of rate of change of normal force due to the compliance of the specimen support. It has been shown that the rate of change of normal force may vary considerably with the rotational position of the spring, and the compliance of the test specimen. Ideally, mechanisms with *in situ* control of the normal force should be used.

5.3 Specimen cleaning

The specimen surface shall be freed from surface contaminants, such as oil, grease and moisture by cleaning it prior to testing.

The following cleaning procedure is adequate if no anomalous contamination has occurred: place in an ultrasonic bath for 5 min in clean analytical-grade petroleum ether. Allow to reach room temperature before testing. If drying stains are observed, wipe with a soft tissue soaked in petroleum ether. Allow at least 3 min equilibration time before testing.

During testing, the specimen surface and stylus tip shall be kept free of fingerprints.

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.