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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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Foreword

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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 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.

5.4 Coating-substrate parameters relevant to a test

The coating-substrate parameters relevant to a test include:

a) substrate hardness and roughness;

b) coating hardness and roughness;

c) coating thickness;

d) friction coefficient between coating and indenter;

e) internal stress in the coating.

Where a direct comparison is to be made between the test results for two or more samples of the same coating/substrate combination, all of the above parameters shall be the same for each sample.

6 Test procedure

6.1 General

Three modes of scratch testing are currently employed, depending on the apparatus available and the information sought. In the progressive-force scratch test (PFST) mode, the normal force applied by the indenter increases linearly as the indenter moves across the test surface at constant speed. In the constant-force scratch test (CFST) mode, the normal force is increased step by step between successive scratches carried out under constant normal force, at different locations on the specimen surface until failure occurs. In the multi-pass scratch test (MPST) mode, the specimen is subjected to repeated scratching, within the same scratch track, under a constant sub-critical normal force.

NOTE 1 In general, the PFST mode is used as a first-order assessment of critical forces corresponding to major coating damage and failure, while the CFST mode allows the statistical damage analysis of coatings along their surface. The MPST mode subjects the coated surface to a low-cycle fatigue-type contact, which is considered to better simulate real working conditions of most coated components.

NOTE 2 In most cases, the CFST mode allows better discrimination between better or poorer adhesion properties than does the standard PFST method. With the current state-of-the-art equipment, however, the CFST mode is very time- and effort-consuming. The MPST mode has been shown to better rank brittle coatings in terms of their adhesion properties. The current experimental effort required, however, is even higher than in the CFST mode (see Reference [2]).

NOTE 3 There is a trend towards the extension and automation of scratch-test operation modes to facilitate the use of more advanced test regimes (see Reference [3]).

6.2 Equipment preparation

The following actions shall be taken prior to testing.

1) The scratch tester shall be confirmed to be calibrated in accordance with normative Annex A.

2) The diamond stylus shall be confirmed to be free from surface contaminants (oil, grease, material picked up from the preceding test).

If necessary, the stylus can be cleaned by wiping with a soft tissue soaked in petroleum ether. If adhering debris is still observed under an optical microscope (recommended magnification: 200×), #1200 and #2400 SiC paper can be used, followed by wiping with a soft tissue soaked in petroleum ether. Ultrasonic cleaning of the stylus should not be used as cavitation damage can occur.

Following cleaning, the stylus shall be allowed to reach room temperature before testing.

6.3 Environmental conditions

Scratch testing requires frictional interaction between the indenter and the specimen surface, and the frictional properties may be sensitive to environmental conditions. Temperature and relative humidity of the test environment shall therefore be known and controlled, if possible, to ensure repeatability.

The recommended environmental conditions are

temperature: 22 °C ± 2 °C,

relative humidity: 50 % ± 10 %.

6.4 Scratching procedure

6.4.1 General

Select the test mode that will provide the information sought, see 6.1.

NOTE It may be necessary to use more than one test mode, depending upon the coating type, the coating substrate combination and the failure mode of interest.

6.4.2 Progressive-force scratch test

Hold the specimen rigidly in the sample holder and bring the stylus into contact with the coated surface. Apply the required start force to the stylus. Select the rate of increase of normal force and table traverse speed. Values of 100 N/min and 10 mm/min are recommended. Scratch the sample and determine the critical normal forces of the selected failure events, as described in 6.5.

Preliminary scratches should be used to define a minimum start force producing an indentation that can be observed by microscope observation (see 6.5.2), and the highest critical normal force of interest. The maximum normal force used in subsequent scratches can then be limited to prevent unnecessary wear of the scratch stylus.

If the critical normal force defining the failure event of interest is lower than 10 N, a rate of increase of normal force of 10 N/min and an indenter traverse speed of 10 mm/min are recommended.

6.4.3 Constant-force scratch test

Hold the specimen rigidly in the sample holder and bring the diamond stylus into contact with the coated surface. If the equipment is able to operate in the PFST mode, scratch the surface using the procedure in 6.4.2 to determine the normal-force range of interest. Move the sample so that a new, unscratched, region can be tested. Using one-fifth of the critical normal force determined by the PFST test, produce a series of scratches at increasing normal force using an indenter traverse speed of 10 mm/min and a scratch length of 10 mm. Following evaluation of the scratches produced, a new series of scratches using lower normal-force increments can be used to investigate any regions of interest more closely.

6.4.4 Multi-pass scratch test

Hold the specimen rigidly in the sample holder and bring the diamond stylus into contact with the coated surface. Using the PFST mode, scratch the surface to determine an approximate normal force at which the failure mode of interest occurs. Using a normal force of 50 % of that determined under the PFST mode, an indenter traverse speed of 10 mm/min and a scratch length of at least 3 mm, test the sample using the MPST mode until failure occurs.

NOTE Depending on the mechanical response of the specimen under investigation, it can be necessary to adjust the normal force, lowering it to obtain better discriminating capacity, or increasing it to obtain the results in an acceptable time-scale.

6.5 Scratch evaluation and critical normal-force determination

6.5.1 General

Several different methods are in use for evaluating scratches and for the determination of critical normal forces, but only microscope observation of the scratch is able to reliably differentiate between different failure modes and enable L_c values to be attributed to specific modes of failure.

NOTE To assist users of the scratch test in the standardized reporting of scratch test results, an atlas of scratch-test failure modes is included in Annex B. The major failure events have been classified in terms of plastic deformation, cracking (L_{c1}), spallation (where the coating flakes off, typically at the edges) (L_{c2}), and penetration of the coating to the substrate at the centre of the track (L_{c3}).

6.5.2 Microscope observation

Observe the scratch or scratches produced using a reflected-light microscope. Remove loosely adhering debris if it obscures the region of interest. Select the failure of interest and either make a sketch or take a micrograph for inclusion in the test report. Alternatively, reference may be made to a representative picture in Annex B. For scratches produced using the PLST mode, determine the critical normal force for the chosen failure event by measuring the distance along the axis of the scratch from the start (trailing edge) of the scratch to the point of failure extended perpendicular to the axis, see Figure 2, and multiplying the result by the rate of change of normal force, in newtons per millimetre, determined from the time rate of change of this force and the sample displacement velocity.

Care shall be taken when removing loosely adhered debris to cause no further damage, by the use of, for example, dry air or a clean, soft, paintbrush.

NOTE 1 It is normal to ignore isolated failures, and critical normal-force values generally refer to the normal force on the stylus at the start of clustered events, see Figure 2.

NOTE 2 The recommended magnification for optical observation is between 100× and 500×.

NOTE 3 More advanced observation tools, such as scanning electron microscopy (SEM) with energy dispersive analysis (EDX), SEM operating in the backscattered mode, scanning profilometry or a scanning acoustic microscope can be used to evaluate the coating damage more accurately. Scanning acoustic microscopy and scanning profilometry enable the detection of delamination events below the surface of the coating (blister formation).

6.5.3 Acoustic emission (AE) and frictional force (FF) recording

Failure events under the scratch stylus, during scratching, may result in perturbations of *in situ* monitored AE and FF signals (see Figure 4). If such monitoring is used, record the normal forces at which perturbations occur, so that these may be related to failure events observed during the microscope examination of the scratches.

Acoustic emission is generated by the elastic waves resulting from the energy released by the creation and propagation of cracks, but it can also be related to friction phenomena and instrumental noise (e.g. from the friction table). The operator may select a detection limit sensitivity to adjust the AE-recording to the agreed failure criterion. The AE-sensor should be of the resonant type, and the electronics should have a 30 kHz high pass filter (without energy integration) to avoid the mechanical vibration frequencies of the instruments (typically from 0 kHz to about 30 kHz).

NOTE *In situ* measurement of the specimen displacement is desirable, to enable the direct correlation between normal force, displacement, and other measuring signals.

7 Repeatability and limits

Because of the statistical nature of the failure probability, an exact critical normal-force value obtained in a single measurement is not significant, and at least five test operations shall be carried out. Consecutive test operations shall be performed in such a way that the critical normal forces cannot be influenced by the preceding scratch tracks.

Accepted limits for scratch testing are:

a) critical normal force L_c > 1 N;

b) resolution of normal force: 0,1 N;

c) coating thickness < 20 µm;

d) roughness parameter Ra < 0,5 µm.

NOTE The typical measurement uncertainty at the 95 % confidence level is 20 %, and different operators introduce errors in the range 5 to 10 %. Under optimum conditions, the reproducibility between instruments used for the scratch test is better then 15 % (see Reference [4]).

8 Test report

The test report shall include the following information:

a) the name of the testing establishment;

b) the date of the test, a unique identification of the report and of each page;

c) the name and address of the customer, and a signatory of the report;

d) a reference to this International Standard, i.e. “Determined in accordance with ISO 20502”;

e) the identification of the test material or product;

f) the procedure used for specimen preparation;

g) sample planarity;

h) rate of application of normal force;

i) traverse speed;

j) indenter tip radius;

k) environmental conditions;

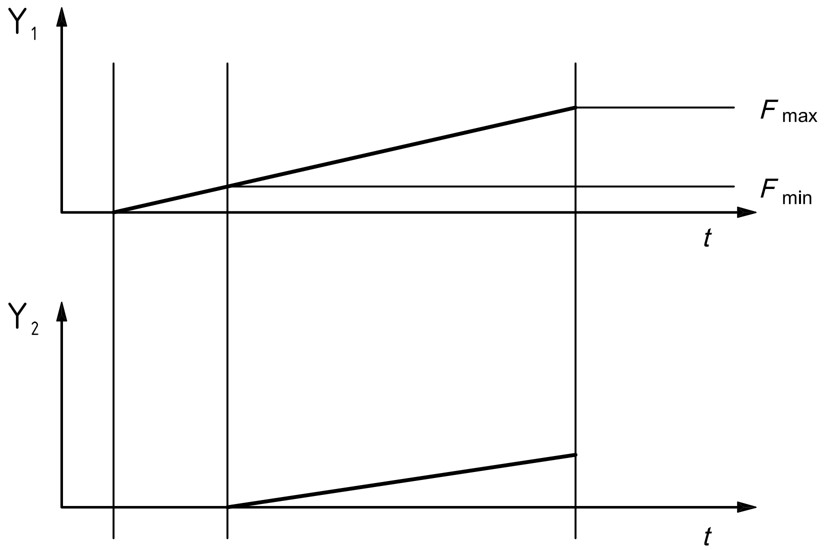
l) test results for L_{cn};

m) a description of the failure modes by reference to a micrograph, sketch or appropriate figure in Annex B;

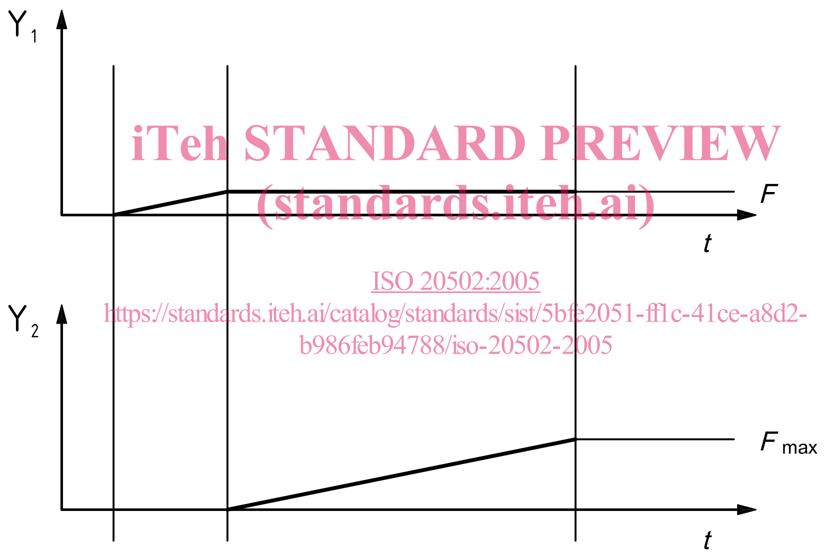
n) dates of last calibration of instrument and indenter;

o) comments on the test or test results, which shall be reported in accordance with ISO/IEC 17025;

p) cycles to failure if tested in the MPST mode.



a) **Progressive force operation mode**



b) **Constant-force operation mode**

Key

t time
Y_1 normal force
Y_2 indenter traverse distance
F_{max} maximum force
F_{min} minimum force

Figure 1 — Sketches illustrating the normal force and traverse distance versus time