SLOVENSKI STANDARD
SIST ENV 1071-3:2000

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 3: Determination of adhesion by a scratch test

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Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 3: Bestimmung der Haftung mit dem Ritztest

Céramiques techniques avancées - Méthodes d'essai pour revetements céramiques - Partie 3: Détermination de l'adhérence par essai de rayure

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CEN

European Committee for Standardization
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Foreword

This European prestandard has been prepared by Technical Committee CEN/TC 184 'Advanced technical ceramics', of which the secretariat is held by BSI.

ENV 1071 has six Parts:

- Part 1 : Determination of coating thickness by contact probe profilometer
- Part 2 : Determination of coating thickness by cap grinding method
- Part 3 : Determination of adhesion by a scratch test
- Part 4 : Determination of chemical composition
- Part 5 : Determination of the porosity
- Part 6 : Determination of the topography

CEN/TC 184 approved this European prestandard by resolution 2 during its sixth meeting held in Alkmaar, Netherlands, on 30 September 1992.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to announce the existence of this European prestandard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This Part of ENV 1071 describes a method of testing the adhesive strength of ceramic coatings by scratch adhesion testing assessed by microscopic examination, acoustic emission analysis and tangential force recording.

2 References

This European prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ENV 1071-1 Advanced technical ceramics - Methods of test for ceramic coatings - Part 1 : Determination of thickness by contact probe profilometer

EN 45001 General criteria for the operation of testing laboratories

ISO 4287-1 Surface roughness - Terminology - Part 1 : Surface and its parameters

3 Principle

The scratch test is designed for the assessment of coating-substrate mechanical strength. The test method consists in generating scratches with a diamond stylus which is drawn across the coating/substance system to be tested, either under constant or progressive load (see figure 1).

In general, spring displacement controlled normal load instruments are used, in which a spring displacement is practised to achieve the chosen loading programme. Magnetically driven assemblies are equally appropriate.

The driving forces for loss of adhesion of coatings in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses and the residual internal stress present in the coating.

During the scratch test, a number of coating failure modes may be observed, only some of which depend on the adhesion. Failure by cracking through the thickness (through-thickness cracking) normally occurs at lower loads than detachment of the coating.

The onset of through-thickness cracking, which can be either conformal or tensile (see figure 2) defines the critical normal load L_{C1}.

The onset of coating detachment, which can be by spalling, buckling or chipping (see figure 3) defines the critical normal load L_{C2}.

NOTE: In the case of spalling failure mode, the thus obtained step can be used to determine the thickness of the coating with the step height measurement method in accordance with ENV 1071-1.

The critical loads at which these phenomena appear depend on the coating adhesion strength but also on the parameters given in clause 4, some of which are directly related to the test itself, while others are related to the coating/substrate system.

4 Test parameters

4.1 The test specific parameters include:

a) loading rate;
b) indenter traverse speed;
c) indenter tip radius;
d) diamond tip wear.

4.2 The coating/substrate specific parameters 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.

4.3 All of the above parameters are to be kept constant if a direct comparison is made between two or more samples.

5 Instrumentation

5.1 Diamond stylus

The diamond stylus has a Rockwell C profile (tip radius R = 200 μm; 120° cone). The indenter surface should be freed from surface contaminants (oil, grease, material picked up from the preceding test) by cleaning it prior to each test.

NOTE 1: Such reconditioning can be achieved by rubbing size 1000 emery paper lightly over the tip and then wiping the tip with an acetone soaked clean tissue paper, leaving no residue.

Periodically (at least every 10 scratches) the tip surface shall be inspected by means of a reflected light microscope (magnification > 100:1) for crater wear (see figure 4). If this type of damage is observed the diamond stylus shall be exchanged.

NOTE 2: To check the repeatability of the diamond stylus, a reference sample (e.g. TiN-coated steel) presenting a repeatable critical load can be tested regularly.

5.2 Critical load determination

5.2.1 Microscope observation. Reflected light microscopy is the most reliable method for detecting coating damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating/substrate system. For the increasing load operation mode, the distance between the start (or the end) of the scratch and the damage site can easily be measured and directly related to the load.

NOTE 1: The recommended magnification for optical observation is 100:1. However, in some cases (very thin films < 1 μm) higher magnifications are needed.

NOTE 2: More advanced observation tools, such as a scanning electron microscope, electron microprobe (chemical mapping) or a scanning acoustic microscope may also be used to establish the position of the coating damage more accurately, as described in annex A, reference A.4.

5.2.2 Acoustic emission (AE) detection. The elastic waves generated as a result of the formation and propagation of microcracks are detected. The operator can select a detection limit sensitivity to adjust the AE-recording to the agreed failure criterion. The AE-sensor should be insensitive to the mechanical vibration frequencies of the instruments (typically from 0 kHz to about 30 kHz). See figure 5.

NOTE 1: The tangential force (and/or friction coefficient) seems to be useful as a complement to the AE-information and microscopic observations, especially for very thin coatings (< 1 μm). See annex A, reference A.5.

NOTE 2: Scratch depth measurement: highly valuable information can be acquired if the scratch tester can be used as a profilometer: by overlapping the pre-scan profile (before scratching) with the post-scan profile (after scratching), the separation between the two curves is then the plastic depth along the scratch track. From the scratch depth profile, one can clearly distinguish between the initial wearing and thinning of the coating, the initiation of microcracks, and the final sudden increase in depth due to delamination of the coating. See annex A, reference A.6.

6 Sampling and surface preparation

6.1 General requirements

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

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

6.2 Surface roughness

The surface roughness shall be known and kept constant when comparing data assessed for different samples. The surface roughness value R_a (see ISO 4287-1) should not exceed 0,5 μm in order to ensure reliable critical load assessment.

6.3 Surface preparation

The specimen surface shall be freed from surface contaminants such as oil and grease by cleaning it prior to testing. Fingerprints on the test area shall be avoided.

NOTE: In general, ultrasonic cleaning for 5 min in clean acetone followed by drying in ambient air is sufficient.

6.4 Surface levelling

For spring displacement controlled normal load instruments, the specimen shall be levelled with respect to the stylus/specimen traverse displacement direction (see clause 7). In practice, this is easily attained for flat specimens held on the sample holder. For curved specimens such as cylinders, such instruments require delicate additional alignment facilities.

NOTE: For spring displacement controlled normal load, instruments with a spring constant of 50 μm/N and a load range of 100 N, a surface roughness value R_a = 0,5 μm corresponds to load oscillations of 0,1 N. Load variations of less than 1 N (1 % of the load range) require a waviness and a levelling defect smaller than 50 μm.