SLOVENSKI STANDARD

SIST EN 1071-3:2005

01-december-2005

Nadomešča:

SIST ENV 1071-3:2000

Advanced technical ceramics - Methods of test for ceramic coatings - Part 3: Determination of adhesion and other mechanical failure modes by a scratch test

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Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 3: Bestimmung der Haftung und Formen des mechanischen Versagens mit dem Ritztest

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

Ta slovenski standard je istoveten z: EN 1071-3:2005

ICS:

25.220.99	Druge obdelave in prevleke	Other treatments and coatings
81.060.30	Sodobna keramika	Advanced ceramics

SIST EN 1071-3:2005 en

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 1071-3

August 2005

ICS 81.060.30

Supersedes ENV 1071-3:1994

English Version

Advanced technical ceramics - Methods of test for ceramic coatings - Part 3: Determination of adhesion and other mechanical failure modes by a scratch test

Céramiques techniques avancées - Méthodes d'essai pour revêtements céramiques - Partie 3 : Détermination de l'adhérence et autres modes de défaillance mécanique par essai de rayure

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 3: Bestimmung der Haftung und Formen des mechanischen Versagens mit dem Ritztest

This European Standard was approved by CEN on 27 June 2005.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Ref. No. EN 1071-3:2005: E

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Foreword

This European Standard (EN 1071-3:2005) 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of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

This European Standard supersedes ENV 1071-3:1994.

EN 1071 – *Advanced technical ceramics – Methods of test for ceramic coatings* has eleven Parts:

Part 1: *Determination of coating thickness by contact probe profilometer*

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*

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 testing*

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: *Measurement of internal stress with the Stoney formula*

Parts 7 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, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

The test method in this Part of EN 1071 consists of drawing a loaded stylus across a coated surface. The load on the stylus is increased until failure of the coating/substrate system occurs. Alternatively repetitive scratching at a fixed load can be used to promote failure. The normal load on the stylus at which a particular failure mode initiates is recorded and is referred to as the critical load, L_c, for that mode of failure. Failure events are detected by the use of microscopic examination, acoustic emission and/or friction force measurement.

Knowledge of the failure mode is essential to assess the mechanical behaviour of coated surfaces. Only some of the observed failure events in scratch testing are related to detachment at the coating-substrate interface and are thus relevant as a measure of adhesion. Other failures, such as cracks and cohesive damage within the coating or substrate may be equally important to determine the behaviour of a coated component in a particular application.

1 Scope

This Part of EN 1071 describes a method of testing ceramic coatings by scratching with a loaded diamond stylus so as to promote adhesive and/or cohesive failure of the coating-substrate system. The test is suitable for evaluating ceramic coatings up to a thickness of 20 µm and can also be suitable for evaluating other coating types and thicknesses. The test is intended for use with specimens of limited surface roughness.

This European Standard is intended for use in the macro (1 N – 100 N) load range. The procedures can also be applicable to other load ranges. However, appropriate calibration is essential if the normal loads at which failure events occur are to be quantified.

2 Normative references

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

EN ISO 4287, *Geometrical product specifications (GPS) - Surface texture: Profile method - Terms, definitions and surface texture parameters (ISO 4287:1997)*

EN 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 6508-2:1999)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

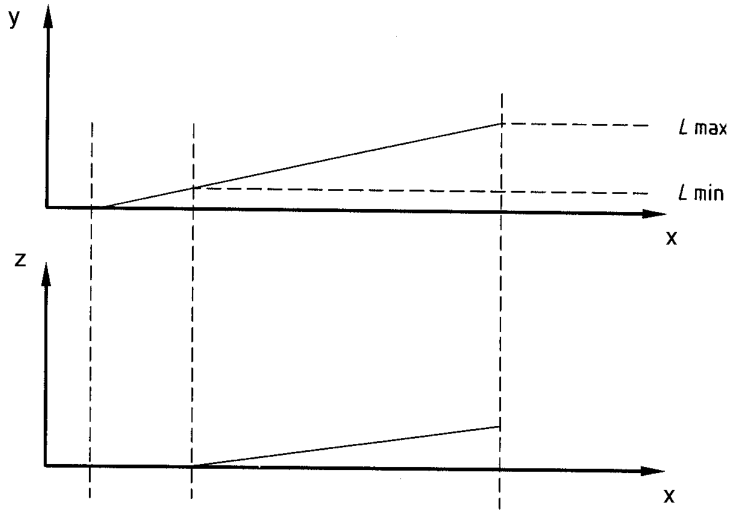
3 Principle

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 diamond with a Rockwell C geometry) by drawing it across the surface of the coating-substrate system to be tested, either under constant or progressive load (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.

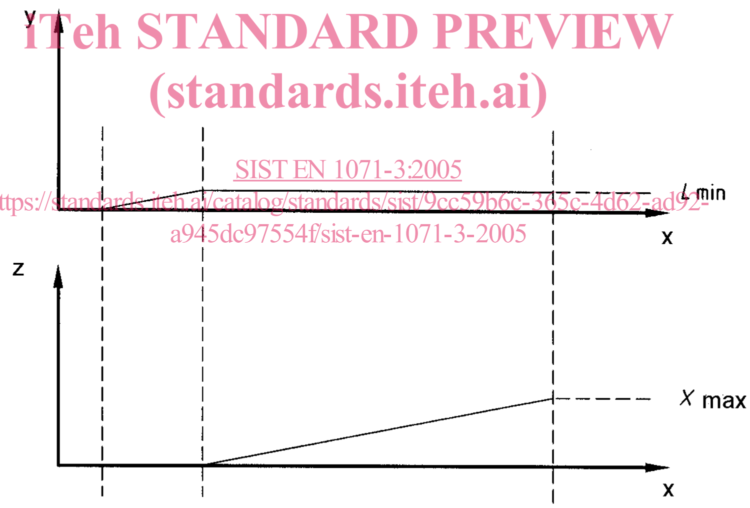
The driving forces for the failure of the coating-substrate system in the scratch test are a combination of elastic-plastic indentation stresses, frictional stresses and the residual internal stress present in the coating. The normal load at which failure occurs is called the critical load L_c.

NOTE 1 In a scratch test, a number of consecutive coating failure events may be observed at increasing critical load values. Failure by cracking through the coating thickness (through-thickness cracking) usually occurs at lower loads than detachment of the coating. Therefore, it is quite common to characterise the onset of cracking by the critical normal load L_{c1}, while the onset of coating detachment defines the critical normal load 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 n^{th} failure mode defines the critical load L_{cn} (see Annex B).

NOTE 2 The critical loads at which the failure events appear depend not only on the coating adhesion strength but also on other parameters, such as loading rate, 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.



(a)



(b)

Key

X Time
Y Normal load
Z Indenter traverse distance

Figure 1 — Sketches illustrating the normal load and traverse distance versus time in (a) the progressive load, and (b) the constant load operation modes

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 load and the driving force to produce scratches. A schematic of a typical arrangement is shown in Figure 2.

NOTE 1 In general, spring deformation controlled normal load instruments are used, in which the deformation of a spring is used to achieve the chosen loading 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 loads. 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 load.

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

4.2 Diamond stylus

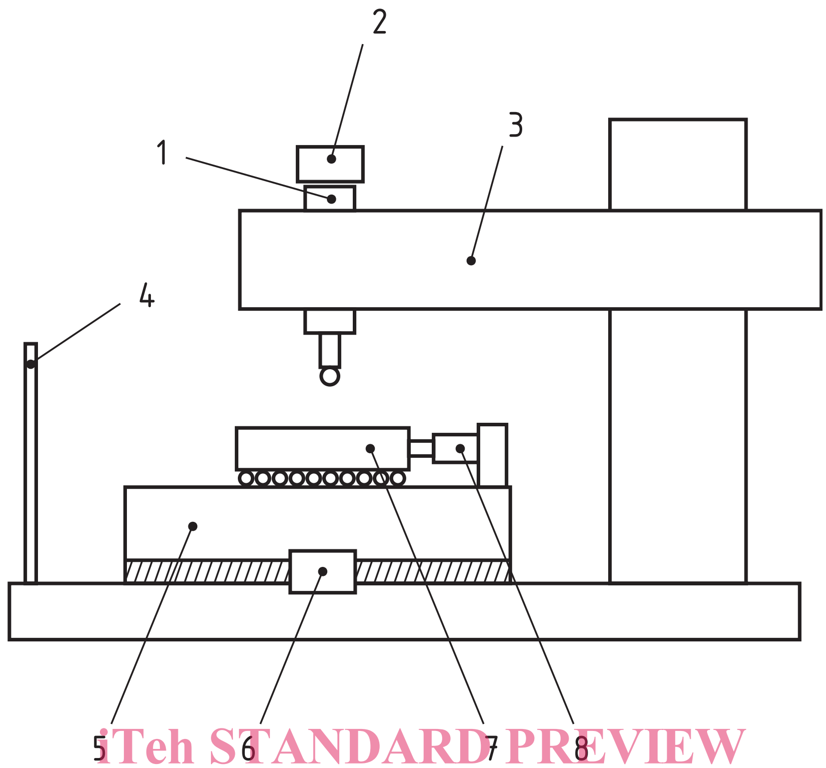
This consists of a rigidly mounted diamond normally having a Rockwell C geometry compliant with the requirements of EN ISO 6508-2.

The stylus shall be inspected regularly to check for contamination and changes in geometry. If damage is observed at 200x or lower magnification then the stylus shall be changed (see [1]) and if either damage or contamination is observed, the test results since the last inspection shall be disregarded. If during constant load operation the friction force increases, it should be presumed that the stylus has been contaminated.

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 load 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 is easily visible under a reflected light microscope (magnification > 100:1).

NOTE 2 A certified reference material, BCR-692[1)], which presents three repeatable failure events at known critical load intervals is available for verification purposes. This can provide a good indication of overall performance, including stylus condition and calibration.

1) European Commission – Directorate-General Joint Research Centre, Institute for Reference Materials and Measurement (IRMM), Retieseweg 111, B-2440 Geel, Belgium.



Key

1 Stylus shaft
2 Vertical load transducer
3 Upper support assembly
4 Base reference
5 XY stage to manoeuvre
6 XY stage drive arrangement
7 Low friction sample table
8 Horizontal force transducer

Figure 2 — Schematic diagram of a typical scratch tester

5 Preparation of the test piece

5.1 General requirements

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

The 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 value Ra, established in accordance with EN ISO 4287, shall not exceed 0,5 µm.

NOTE 1 For spring deformation controlled normal load instruments (typical spring constant: 0,02 N/µm) the normal load depends on the roughness and waviness of the surface. A surface roughness value Ra of 0'5 µm may lead to load oscillations of 0,1 N. Load variations of less than 1 N (1 % of the typical load range) require a waviness and/or levelling error smaller than 50 µm.

NOTE 2 In general, the critical load 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.

NOTE 3 The specimen levelling mechanism should be stiff to preclude the variation of load rate due to the compliance of the specimen support. It has been shown that the load rate 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 load 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.

NOTE The following cleaning procedure is adequate if no anomalous contamination has occurred: place the specimen in an ultrasonic bath for 5 min, using clean analytical grade petroleum ether. Allow it 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.

5.4 Coating-substrate specific parameters

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.

If a direct comparison is to be made between two or more samples of the same coating/substrate combination, it is essential that the above parameters are the same for all the samples compared.

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 loading scratch test (PLST) mode the load on the indenter increases linearly as the indenter moves across the test surface at constant speed. In the constant load scratch test (CLST) mode the normal load is increased stepwise between successive scratches carried out under constant load 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 load.

NOTE 1 In general, the PLST mode is used as a first order assessment of critical loads corresponding to major coating damage and failure, while the CLST 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 CLST mode allows better discrimination between better or poorer adhesion properties than does the standard PLST method. With the current state-of-the-art equipment, however, the CLST 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 CLST mode (see [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 [3]).

6.2 Equipment preparation

Before starting a test the following actions shall be taken:

1) Scratch tester shall be confirmed as having been calibrated in accordance with Annex A.

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

NOTE 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 X), #1 200 and #2 400 SiC paper can be used, followed by the 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.

NOTE The recommended environmental conditions are:

i) temperature: (22 ± 2) °C

ii) relative humidity: (50 ± 10) %

6.4 Scratching procedure

6.4.1 General

Select the test mode that will provide the information sought.

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 loading scratch test

Hold the specimen rigidly in the sample holder and bring the stylus into contact with the coated surface. Load the stylus to the required starting load. Select the loading rate and table traverse speed. Values of 100 N/min and 10 mm/min are recommended. Scratch the sample and determine the critical loads of the selected failure events, as described in 6.5.

NOTE 1 Preliminary scratches should be used to define a minimum start load producing an indentation that can be observed using microscope observation (see 6.6) and the highest critical load of interest. The maximum load used in subsequent scratches can then be limited to prevent unnecessary wear of the scratch stylus.

NOTE 2 If the critical load defining the failure event of interest is lower than 10 N, a loading rate of 10 N/min and an indenter traverse speed of 10 mm/min are recommended.

6.4.3 Constant load 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 PLST mode, scratch the surface using the procedure in 6.4.2 to determine the load range of interest. Move the sample so that a new, unscratched, region can be tested. Using one fifth of the critical load determined by the PLST test, produce a series of scratches at increasing load using an indenter traverse speed of 10 mm/min and a scratch length of 10 mm.

NOTE Following evaluation of the scratches produced, a new series of scratches using lower load increments can be used to investigate any regions of interest more closely.

6.4.4 Multipass scratch test

Hold the specimen rigidly in the sample holder and bring the diamond stylus into contact with the coated surface. Using the PLST mode, scratch the surface to determine an approximate load at which the failure mode of interest occurs. Using a load of 50 % of that determined under the PLST 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 may be necessary to adjust the normal load, to obtain better discriminating capacity by lowering the load, or to obtain the results in an acceptable time-scale by using higher loads.

6.5 Scratch evaluation and critical load determination

Several different methods are in use for evaluating scratches and for the determination of critical loads but of these 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 standardised 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}).