SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17629:2021

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Nanotehnologije - Nano in mikro preskus praskanja

Nanotechnologies - Nano- and micro- scale scratch testing

Nanotechnologien - Nano- und Mikro-Ritzprüfung

Nanotechnologies - Tests de résistance à l'échelle nanométrique et microscopique

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Nanotechnologies - Nano- and micro- scale scratch testing

Nanotechnologies - Tests de résistance à l'échelle nanométrique et microscopique

Nanotechnologien - Nano- und Mikro-Ritzprüfung

This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 352.

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Contents

Page

European foreword

This document (FprCEN/TS 17629:2021) has been prepared by Technical Committee CEN/TC 352 “Nanotechnologies”, the secretariat of which is held by AFNOR.

This document is currently submitted to the Vote on TS.

Introduction

The test procedure is intended to complement other standards which are concerned with the scratch resistance of materials. This procedure extends the use of the nano- and micro- single pass scratch test to bulk and coated materials, additionally covering the use of multiple pass nano- and micro- scratch tests.

The method described is not intended to be used to define how particles are released from a surface under this type of damage.

Several measurement techniques are described, according to the following procedures:

— Constant force scratch test

Single movement of a normally loaded probe (constant force) onto a test piece; friction force and displacement of the probe (relative to the test piece) are measured along the scratch path.

— Ramped force scratch test

Single movement of a progressively normally loaded probe (ramped force) onto a test piece; friction force and displacement of the probe (relative to the test piece) are measured along the scratch path.

— Multi-pass unidirectional constant force scratch test

Repeated movement of a normally loaded probe (constant force) onto a test piece, following the same track; the variation in friction force and displacement of the probe (relative to the piece test) are measured along the scratch path. First introduced by Bull and Rickerby [1], this test is also called “nanowear” when used in the nano scratch range and provides information regarding the fatigue behaviour of the test piece as an effective low cycle fatigue test.

— Progressive force “3-scan” scratch test

Three repetitive unidirectional movement of a normally loaded probe onto a test piece, along the same track. The first movement of the probe is carried out at constant force (low force) and performed as a topography scan of a non-scratched test piece surface. The second movement of the probe is achieved with a progressively increased normal force onto the test piece (from low to high forces). The third movement of the probe is similar to the first movement, at low force, to acquire a topography of the scratch carried out in the test piece. This test is also called “scratch topography multi-pass test” and was first reported by Wu and co-workers [2,3], which enables identification of failure mechanisms and provides more details regarding the impact of stress such as the critical force for onset of non-elastic deformation and the yield pressure (estimated from mean pressure at critical force).

1 Scope

This document specifies a method for measuring the scratch resistance and failure behaviour for advanced materials and coatings by means of nano- and micro- scale scratch experiments. The method provides data on both the physical damage to test-pieces and the friction generated between the probe and the test-piece under single pass and multiple pass conditions. The force range in these tests is from 1 μN up to 2 N.

The test method is not applicable to coatings as defined in EN ISO 4618 [18].

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
nanoscale
size range between approximately 1 nm and 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size are predominately exhibited in this size range.

Note 2 to entry: The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as nano-objects or elements of nanostructures, which might be implied by the absence of a lower limit.

Note 3 to entry: EN ISO 14577-1 defines nano range for indentation depth as less than 200 nm and has a force criterion for tests in the micro range.

[SOURCE: CEN ISO/TS 80004-1:2015, 2.1 [17], modified]

3.2
microscale
size range between 100 nm and 100 μm

3.3
topographical profiling
scans carried out for topographical profiling sequence (e.g. 3-pass scratch test: pre-scanning and post-scanning under minimal force), the purpose of which is to measure the topographical profile of the surface before and after the scratch test

Note 1 to entry: The load of the scan should be kept to a minimum to avoid plastic deformation.

Note 2 to entry: Scans have to move in the same direction to avoid uncertainties in displacement recording and scanning movements have to be longer than scratching ones to cover the starting- and ending part of the scratch and providing undeformed areas for checking instrument drift. The force during the scanning movements shall be low enough to ensure that any deformation is elastic.

Note 3 to entry: The probe radius needs to be small enough to give sufficient resolution for the analysis of the profile of the surface

3.4
critical points on the scratch track
points on the scratch track, where any new damage process starts as a function of the track length, normal-force or other measured signal (e.g. tangential force, acoustic emission, etc.)

Note 1 to entry: These processes can be identified as characteristics of the scratching track itself or as characteristic of the recorded tracks (scanning – scratching – post-scanning).

3.4.1
onset of plastic deformation
point on the scratch track where the post- scanning track becomes significantly deeper (4 × instrument displacement noise floor) than the pre-scanning track, if necessary, after thermal drift correction

Note 1 to entry: The normal force at this point is the threshold force for onset of plastic deformation (L_y).

Note 2 to entry: When the objective is to identify coating properties one has to confirm that the yield event takes place in the coating.

3.4.2
onset of cracking
critical point in the scratch experiment where initial cracking is experienced as evidenced by subsequent imaging of the scratch path, or through analysis of the friction force, acoustic emission [4], or displacement data (L_{c1})

3.4.3
onset of partial coating failure
critical point in the scratch experiment where the beginning of material removal inside or outside the scratch track can be identified

Note 1 to entry: Typically, the removed material is smaller than the film thickness. This critical point is called L_{c2}. The failure mode correlated to L_{C2} is not always occurring. In this case L_{C2} should not be given.

3.4.4
onset of severe coating failure
critical point in the scratch experiment where a significant removal of material can be identified by subsequent imaging of the scratch path or by the difference of post- and pre-scan depth

Note 1 to entry: If this is greater or equal to the coating thickness, then delamination may have been occurred. This critical point is called L_{c3}. If the difference of post- and pre-scan depth is less than the coating thickness the failure mode may be cohesive.

3.5
probe area function
geometrical relationship between projected normal area of probe and distance from the end of the probe

Note 1 to entry: The probe area function should be verified periodically to determine the shape of the tip (refer to Annex A).

3.6
spherical probe effective radius
radius of an ideal spherical probe that gives same depth as an imperfect probe

Note 1 to entry: For other probes with other nominal shapes such as pyramidal probes, there is always some imperfection in the shape of the tip at the point such that the tip has an effective radius.

3.7
scratch depth
h_t is the scratch depth under force and h_r is the residual depth determined from a subsequent topographical profile as the depth determined by subtracting the pre-scan profile from the post-scan profile

3.8
friction force
resisting force tangential to the interface between two bodies when, under the action of an external force, one body moves or tends to move relative to the other

Note 1 to entry: See also coefficient of friction.

[SOURCE: ASTM G40-17:2017]

4 Symbols and abbreviations

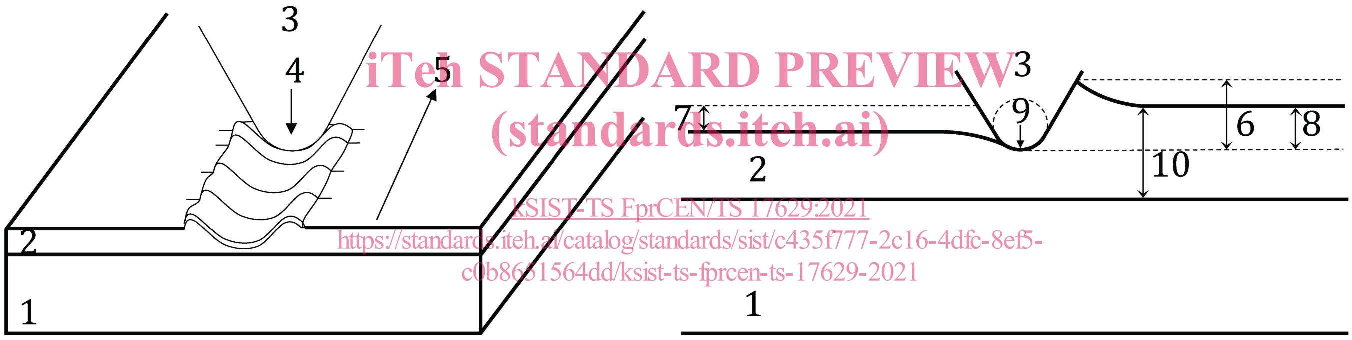
For the purposes of this document, the following symbols and abbreviations apply.

Table 1 — Symbols and abbreviations

Symbol	**Definition**	**Unit**
h_t	On-force scratch depth	mm
h_c	Contact depth	mm
h_0	Initial depth (pre-scan)	mm
h_r	Residual depth (after scratch)	mm
t_f	Coating thickness	mm
R_a	arithmetic average of the roughness	mm
S	Effective adhesion strength	GPa
R	Tip radius	mm
A_c	Tip contact area	mm²
x	Scratch distance	mm
t	Scratch time	s
F_N	Normal force	N
F_S	Scan force	N
F_T	Tangential force	N
P_m	Contact pressure	GPa
L_y	Initial yield force	N
L_{c1}	Force at which first cracking occurs	N
L_{c2}	Force at which partial failure occurs	N
L_{c3}	Force at which complete failure occurs	N
L_c	Critical force	N
μ_{tot}	Friction coefficient	
$\mu_{interfacial}$	Interfacial friction component	
$\mu_{ploughing}$	Ploughing friction component	

Symbol	Definition	Unit
C	Contact compliance	nm/mN
C_f	Frame compliance	nm/mN
E	Young’s modulus	GPa
E_i	Young’s modulus of the probe	GPa
E_s	Young’s modulus of test specimen	GPa
E_r	Reduced Young’s modulus	GPa
H	Hardness	GPa
ν_i	Poisson’s ratio of the probe	
ν_s	Poisson’s ratio of the test specimen	
a	Probe contact radius	mm
ε	Epsilon factor	

Some of the abbreviations listed above are schematically described in Figure 1.



Key

1	substrate	6	h_c contact depth
2	coating	7	h_r residual depth (after scratch)
3	probe	8	h_t on-force scratch depth
4	F_N normal force	9	R tip radius
5	x scratch distance	10	t_f coating thickness

Figure 1 — Schematic representation of a scratch test and its cross-section

5 Principle

5.1 General

In the test, a probe of known geometry is loaded against a test-piece and moved across the test-piece. The depth of penetration of the probe into the test-piece is measured to provide a real-time measure of damage to the test-piece as the test is being carried out, and the force generated by the resistance of the motion is measured to provide information on friction. To determine the response of the material to repeated contacts, multiple pass experiments can be carried out. Both constant loading and ramping loading can also be carried out.

A key parameter in these measurements is the measurement and control of the geometry of the probe that is used to carry out the experiments.

5.2 Friction

The measured friction force in the nano- or micro-scratch test typically varies with the applied force. When a hard surface is slid over a softer surface part of the frictional resistance is due to the force required to plough asperities of the harder surface through the softer. The friction coefficient (frictional force/normal force) can therefore be separated into its interfacial and ploughing components so that the interfacial friction coefficient can be reported:

$$\mu \text{ total} = \mu_{\text{interfacial}} + \mu_{\text{ploughing}} \quad [5] \qquad (1)$$

Interfacial friction comes from the adhesion force that normally occurs between two surfaces in contact. No change to the surface occurs from this effect. Interfacial friction can be determined by different approaches:

1) performing constant force friction test at very low force where contact is completely elastic and the ploughing contribution is zero;

2) performing repetitive scratches to eliminate the ploughing contribution;

3) performing progressive force scratch and extrapolating the low force friction data to zero force.

Typically, the friction coefficient at yield is of the order of 0,05, rising to about 0,2 to 0,5 at failure. The impact of ploughing on friction has a complex mechanism and depends strongly on mechanical properties of the test specimen.

Although frictional measurements are often reported to differ at different length scales it appears that when the extent of deformation is taken into account there is much better agreement [6-8].

5.3 Factors influencing the critical forces

5.3.1 General

In addition to the strength of adhesion between any coating that is present and substrate, the critical force can be influenced by a range of extrinsic and intrinsic factors [9] as well as the mechanical properties of the test-piece (E and H) and the lateral stiffness of the instrument [10,11]. The intrinsic factors include scratching speed, loading rate, tip radius and extrinsic factors can include the mechanical properties of both the coating and the substrate, and roughness, thickness and friction force generated in the contact of the probe with the surface of the test-piece. In addition, the environment conditions, namely, the temperature, pressure, relative humidity and gas composition may have a signification impact on the test results.

5.3.2 Probe radius

For coated samples, the ability of the test to investigate features of the structure like the interface between coating and substrate is controlled by the magnitude of the applied force and the ratio of the probe radius to coating thickness, R/t_f. This ratio is much less than the ratio used in macro-scale scratch tests. For coatings, the probe radius can be chosen to generate a maximum stress above, at, or below an interface, depending on what information is most critical in the application. The different R/t_f ratio can associated with different stress field, enabling to focus on cohesive or adhesive failure of coatings.

Higher critical forces are observed when larger probe radii are used (low stresses). For the condition where plastic deformation starts in the substrate a rough estimation of the critical force may follow a power law dependence on the probe radius [7] of the type shown in Formula (2) where S is a parameter which can be correlated with the effective adhesion strength in the scratch test. The exponent m is usually in the range 1 to 2 when spherical probes are used.

$$L_c = SR^m \quad [10] \qquad (2)$$

If coatings are present, their thickness can influence the critical force since

a) thicker films that are harder than the underlying substrate provide more load support and so delay the onset of substrate deformation that can occur before film failure (higher critical force);

b) thicker films can be more highly stressed and more easily through-thickness crack and delaminate when deformed (lower critical force) since the driving force for spallation to reduce stored elastic energy is greater.

In practice when the film is not highly stressed the ratio L_c/t_f may be approximately constant.

Materials, such as metals, that show an indentation size effect will also show a size effect in the nano-scratch test. For materials such as these, the yield stress determined from the onset of plastic deformation in the nano-scratch test will be higher than when determined at greater length scale in bulk testing. This means that the yield stress can increase for smaller radii probes.

5.3.3 Scan speed and loading rate

The critical force may vary with choice of scan speed and loading rate, but practice has shown that within the typical range of these most materials show relatively low sensitivity [12]. When tests are performed at a constant dF_N/dx ratio (the increase in normal force per unit scratch distance), the critical force can be approximately constant over a large range of scratching speeds. While nano-scratch tests carried out under varying loading rate (<1 N/mm) can be compared to one another (low sensitivity), micro-scratch tests with loading rates exceeding 1 N/mm have an effect on the critical force; the critical force decreases as the dF_N/dx ratio decreases as a result of higher probability to encounter a defective adhesion region [10].

5.3.4 Roughness

The critical force can be influenced by test-piece roughness and by the direction of scratching relative to polishing marks on the surface made prior to coating deposition [13]. The critical force may be lower when scratching perpendicular to the grinding marks than parallel to them. In addition, high roughness tends to decrease the critical force but load carrying capability of thick coating has a higher effect; the critical force increases with coating thickness when coating is harder than substrate, which delays substrate deformation [5,14].

5.4 Multiple pass testing

Multiple pass constant force scratch tests can be carried out with the nano- and micro-scratch technique. The test effectively becomes a low cycle [15] nano- or micro-wear test where the test parameters are less severe than in a progressive force scratch test. Constant force wear tests are often used to determine rates of wear and investigate the role of surface fatigue. The low-cycle wear experiments can often be much more informative regarding the influence of, e.g. coating stress leading to poor adhesion than single pass scratch tests. When compared to progressive force scratch testing, wear testing has the advantage that the force can be varied to tune the maximum stress to be close to the coating-substrate interface.

The applied force used in the multiple pass scratch test is usually lower than the critical force (Lc_1) in a progressive force scratch test. The number of scratch passes to failure is a convenient parameter to compare the durability of different trial coatings. Either the variation in friction or the on-force depth with number of scratches can be used to determine the number of cycles to film failure.

Typically, a test is composed of 10 to 20 scratch cycles; an example of the multi-pass test is presented in Figure 2. Optionally, it can be combined with alternate low-force passes. For example, a test involving 20 scratches under constant force could be set as a total of 41 passes over the same scratch track - an initial low force scan would be followed by 20 times (scratch-topography) pairs.

In contrast to progressive force scratch tests, failure may initially occur at isolated regions of the film surface requiring several subsequent passes until the film is completely removed from the scratch track. The mean depth over the constant force region is a convenient parameter to follow the evolution of the damage process.