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Nanotechnologies — Superhydrophobic surfaces and coatings: Characteristics and performance assessment

Nanotechnologies — Surfaces et revêtements superhydrophobiques : Caractéristiques et évaluation de la performance

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Foreword

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This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

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Introduction

Surfaces or coatings which are extremely difficult to wet with water can be considered as superhydrophobic. Based on the scientific literature, superhydrophobic surfaces and coatings show contact angles of above 150° as well as contact angle hysteresis less than 10°. Superhydrophobicity phenomena is seen in some natural species, e.g. lotus leaves. Other related terms are “lotus effect” which arises for droplets being in “Cassie-Baxter” wetting state.

Various methods have been utilized for the production of superhydrophobic surfaces and coatings, e.g. chemical vapour deposition, spin coating, sputtering, plasma deposition, chemical etching, sol-gel, photolithography, anodizing and plasma electrolyte oxidation. The superhydrophobic surfaces and coatings have numerous applications in different industries due to their properties, which can include self-cleaning, anti-corrosion, anti-icing, anti-fog and antibacterial effects. Such coatings and surfaces are gradually entering automotive, building and construction, healthcare, optical and electrical industries. The market for superhydrophobic surfaces and coatings for 2020 was about $1,8 billion[1].

A common characteristic of superhydrophobic surfaces and coatings is their proper two-level topography (i.e. micro- and nano-sized asperities) combined with low surface energy. This multiscale (hierarchical) roughness would result in large water contact angle, low contact angle hysteresis, and high wetting stability against the Cassie–Baxter to Wenzel transition. In other words, a large contact angle is already achievable with a microscale surface roughness but for having a large contact angle combined with small contact angle hysteresis, nanoscale roughness is needed[3]. In other words, water cannot penetrate into nano-scale surface asperities which results in small contact angle hysteresis. In the absence of nano roughness, penetration of water into the micro-scale surface asperities results in high contact angle hysteresis (see Annex A). Such surfaces (surfaces with contact angles above 150° and contact angle hysteresis more than 10°) are called “pseudo-superhydrophobic” surfaces[3]; another related term for pseudo-superhydrophobic is: “sticky superhydrophobic” that arises due to the rose petal effect for droplets being in the Wenzel state.

Water droplets easily bead up and roll-off on superhydrophobic surfaces and coatings and this easy roll-off is the root cause of all the interesting properties of superhydrophobic surfaces and coatings. Advancing and receding angles are the parameters used to quantify the droplet mobility on surfaces. As such, measuring the advancing and receding angles identify if a coating/surface has superhydrophobic properties. Also, measuring the advancing and receding angles before and after exposing the surface to different working/environmental conditions can be used to assess the performance of superhydrophobic surfaces and coatings.

The superhydrophobic surfaces and coatings are normally subjected to different working/environmental conditions, for example, mechanical stress, ultra-violet (UV), visible and infrared (IR) exposure, exposure to different liquids and thermal cycling. These conditions may lead to possible alteration of the performance of superhydrophobic surfaces and coatings. Unfortunately, despite the huge market, there is currently no standard to assess the durability of superhydrophobic surfaces and coatings. This document aims to specify performance assessment methods of superhydrophobic surfaces and coatings under different working/environmental conditions, where applicable based on the agreement between interested parties. The assessment criteria are comparison of advancing angle, receding angle and contact angle hysteresis of the samples before and after being subjected to the above-mentioned working/environmental conditions. Further, this document facilitates the communication between the interested parties. Also, this document supports UN sustainable development goals (SDGs) 8 and 12 which are “decent work and economic growth” and “responsible consumption and production”.

Nanotechnologies — Superhydrophobic surfaces and coatings: Characteristics and performance assessment

1 Scope

This document specifies requirements and recommendations for performance assessment methods for superhydrophobic surfaces and coatings subjected to mechanical stress, solar radiation and weathering, liquids, and thermal cycling, where applicable based on the agreement between interested parties. The performance assessment is carried out based on comparative measurements of the advancing and receding angles and the calculation of the contact angle hysteresis before and after the above-mentioned working/environmental conditions. This document does not address safety and environmental related issues of such coatings.

This document is applicable to any superhydrophobic surfaces and coatings (i.e. nanostructured) on which the measurement of the advancing and receding angles is possible.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2812-1, *Paints and varnishes — Determination of resistance to liquids — Part 1: Immersion in liquids other than water*

ISO 7784-3, *Paints and varnishes — Determination of resistance to abrasion — Part 3: Method with abrasive-paper covered wheel and linearly reciprocating test panel*

ISO 11997-3, *Paints and varnishes — Determination of resistance to cyclic corrosion conditions — Part 3: Testing of coating systems on materials and components in automotive construction*

ISO 16474-2, *Paints and varnishes — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

ISO 19403-6:2017, *Paints and varnishes — Wettability — Part 6: Measurement of dynamic contact angle*

ISO/TR 21555:2019, *Paints and varnishes — Overview of test methods on hardness and wear resistance of coatings*

3 Terms and definitions

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

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

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

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

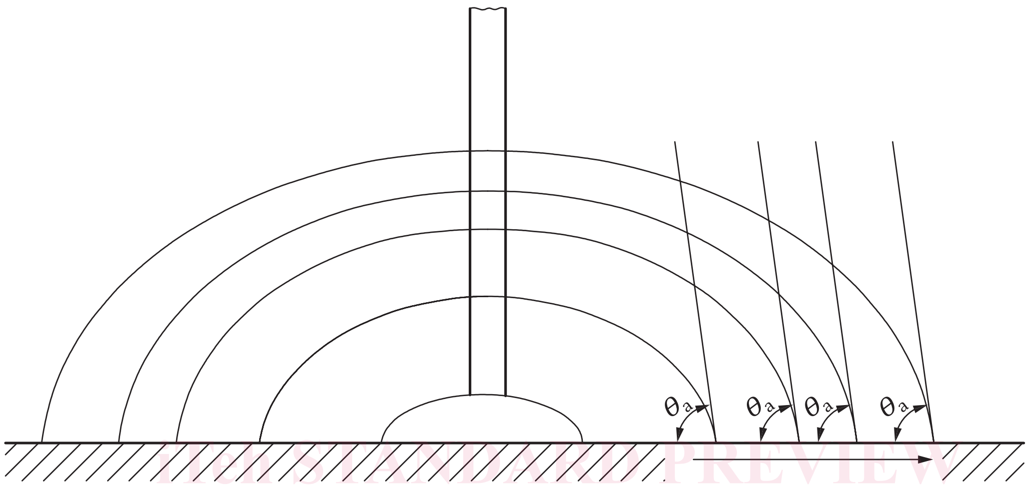
3.1
abrasion
wear which is caused by removal of coating materials on a surface

[SOURCE: ISO/TR 21555:2019, 3.6]

3.2
advancing angle
θ_a
contact angle (3.3), which is measured during advancing of the three-phase point

Note 1 to entry: Generally, the advancing angle is used for the determination of the interface energy, in which case, the measurement should be carried out close to the thermodynamic equilibrium. This is approximately reached if there is no influence of, for example, the dosing speed on the contact angle.

Note 2 to entry: See Figure 1.



Key

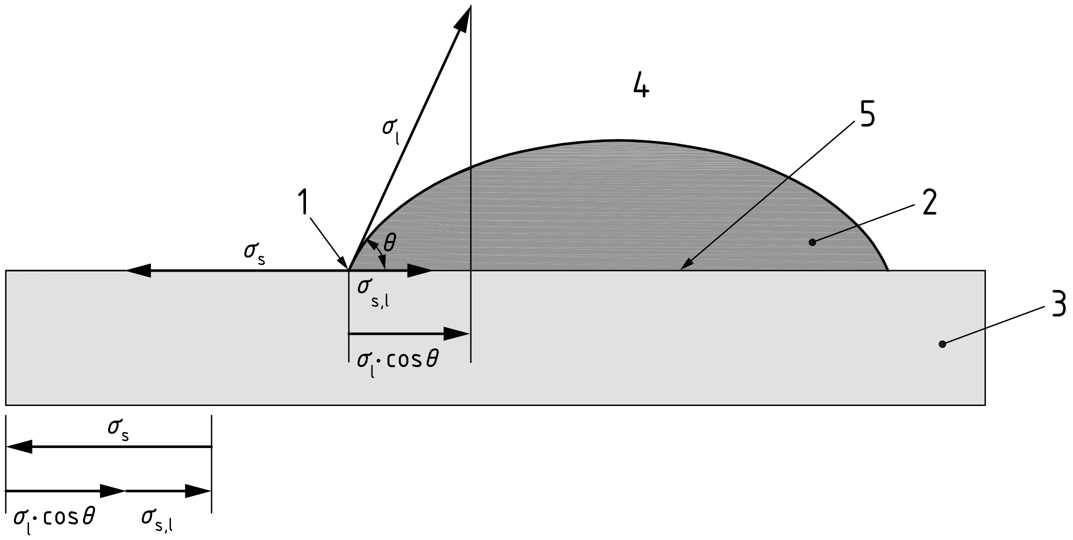
θ_a advancing angle

Figure 1 — Illustration of an advancing angle by needle application of a drop

[SOURCE: ISO 19403-6:2017, 3.2, modified — Note 2 to entry has been added.]

3.3
contact angle
θ
angle to the base line within the drop, formed by means of a tangent on the drop contour through one of the three-phase points

Note 1 to entry: see Figure 2.



Key

1 three-phase point
2 liquid phase
3 solid phase
4 gas phase
5 base line
σ_l surface tension of the liquid surface
σ_s surface free energy of the solid surface
σ_{sl} interfacial energy between solid surface and liquid surface
θ contact angle

Figure 2 — Illustration of a contact angle in wetting equilibrium

[SOURCE: ISO 19403-1:2022, 3.1.9, modified — the title of Figure 2 has been slightly modified and Note 2 to entry has been deleted.]

Note 2 to entry: The contact angle is preferably indicated in degrees (°). $1^o = \pi / 180$. If the system is in thermodynamic equilibrium, this contact angle is also referred to as thermodynamic equilibrium contact angle.

3.4
contact angle hysteresis
θ_{ar}
difference between *advancing angle* (3.2) and receding angle

[SOURCE: ISO 19403-6:2017, 3.4]

3.5
chemical homogeneity
chemically homogeneous composition of a surface to be examined

Note 1 to entry: The definition regards a purely qualitative assessment of the surface. Regarding the measurement of the *contact angle* (3.3), a surface is considered chemically and topologically sufficiently homogeneous if no significant differences of the contact angles can be determined when measuring on several areas on the surface. The significance limits can be specified by the user in accordance with standard laboratory methods.

[SOURCE: ISO 19403-1:2022, 3.1.1, modified — "locations" has been replaced with "areas" in Note 2 to entry.]

3.6
double stroke
ds
complete reciprocal movement made by the abrasive wheel

[SOURCE: ISO 7784-3:2022, 3.2]

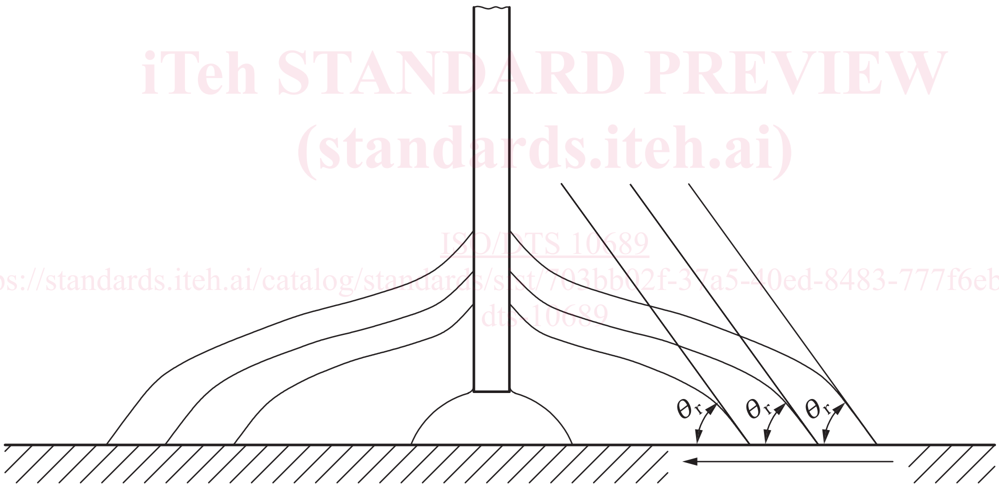
3.7
dynamic contact angle
contact angle (3.3), which is measured during advancing or receding of the three-phase point

Note 1 to entry: The advancing or receding of the three-phase point can be achieved by changing the volume of the liquid drop to be measured, by relative movement (immersing and pulling out) of a solid body to an interface, or by moving the drop over the interface (e.g. rolling off).

[SOURCE: ISO 19403-6:2017, 3.1]

3.8
receding contact angle
θ_r
contact angle (3.3), which is measured during receding of the three-phase point

Note 1 to entry: See Figure 3.



Key

θ_r receding angle

Figure 3 — Illustration of receding angle by needle extraction of a drop

[SOURCE: ISO 19403-6:2017, 3.3, modified — Note 1 to entry has been added.]

3.9
static contact angle
angle between a plane solid surface and the tangent drawn in the vertical plane at the interface between the plane solid surface and the surface of a droplet of liquid resting on the surface

[SOURCE: ISO 15989:2004, 3.4, modified — the symbol "θ" has been deleted.]

3.10
superhydrophobic coating
coated surface for which the *contact angle* (3.3) with a water droplet exceeds 150° and *contact angle hysteresis* (3.4) is less than 10°

3.11
superhydrophobic surface
surface made from hydrophobic material having nano-scale textures for which the *contact angle* (3.3) with a water droplet exceeds 150° and the *contact angle hysteresis* (3.4) is less than 10°

3.12
topological homogeneity
uniformity of the macroscopic surface, including evenness and smoothness

Note 1 to entry: The definition regards a purely qualitative assessment of the surface. Regarding the measurement of the *contact angle* (3.3), a surface is considered chemically and topologically sufficiently homogeneous if no significant differences of the contact angles can be determined when measuring on several areas on the surface. The significance limits can be specified by the user in accordance with standard laboratory methods.

[SOURCE: ISO 19403-1:2017, 3.1.2]

3.13
wear
irreversible change of a coating which is caused by mechanical impact of moved objects

[SOURCE: ISO/TR 21555: 2019, 3.2]

3.14
wettability
degree of wetting

Note 1 to entry: *Contact angle* (3.3) $\theta = 0$ indicates fully wetted and $\theta = 180^o$ indicates not wetted.

[SOURCE: ISO 19403-1:2017, 3.3.2, modified — Note 1 to entry has been added.]

4 Characteristics and measurement methods

4.1 General

The contact angle of water on superhydrophobic surfaces and coatings is larger than 150° and contact angle hysteresis is less than 10°. Measuring the static contact angle on a superhydrophobic surface/coating according to ISO 19403-2:2017, 7.2.2 is not possible (or at least it is challenging) as the drop adheres to the needle and detaches from the surface during the procedure. As such, only dynamic method (advancing and receding angles) shall be used. In other words, the following characteristics shall be measured/calculated and reported after each test: advancing angle, receding angle and contact angle hysteresis. The superhydrophobic surfaces and coatings to be tested for this document shall be rigid, planar, macroscopically homogeneous and macroscopically smooth, on which measuring the advancing and receding angles (dynamic contact angles)according to ISO 19403-6 is possible.

A commercially available contact-angle meter, including a light source, optical system, specimen stage, automatic liquid delivery system, and image processing algorithm is used according to ISO 19403-6.

4.2 Test piece

Cut out flat pieces of the substrate coated by superhydrophobic coating or substrate with superhydrophobic surface. The cut pieces shall be proper representatives of the whole material used in the real-world application. Caution shall be made not to contaminate the test piece with contaminants. The shape and size of the test piece should allow the measurement of the advancing/receding angle at minimum five different points, also allow performing the required tests mentioned in Clause 5 and agreed by interested parties.

