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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*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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. Another related term is “lotus effect” which arises for droplets in the 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 identifies 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/

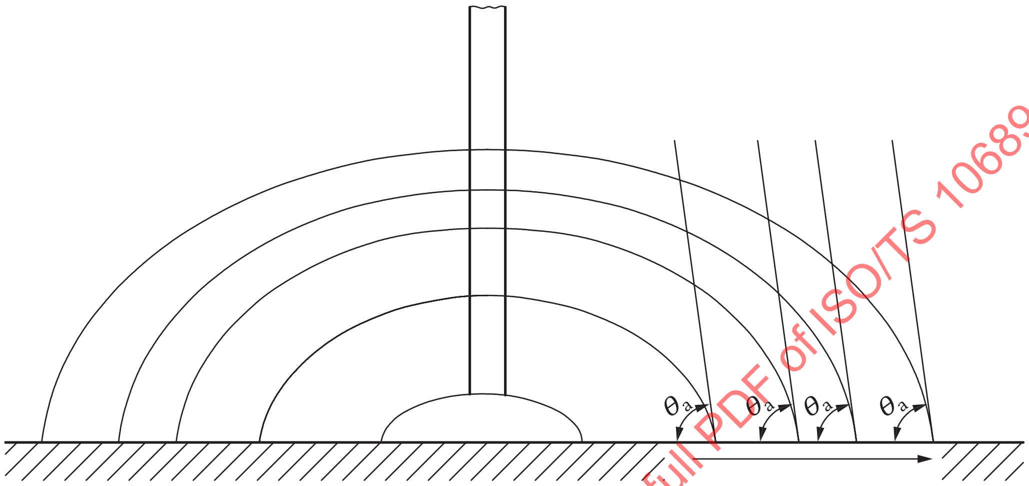
3.1
abrasion
wear which is caused by removal of coating materials on a surface

[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

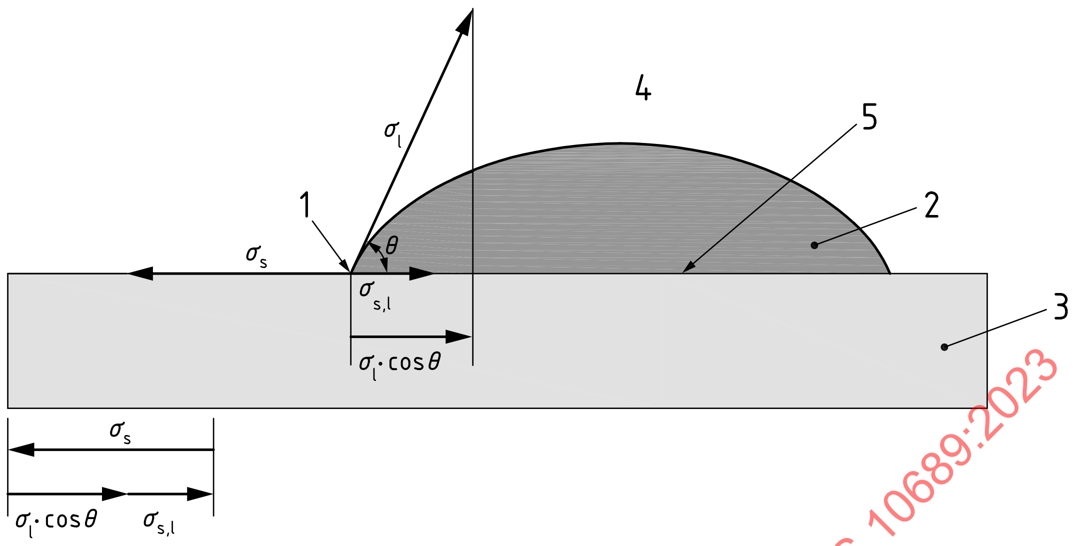
θ_a advancing angle

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

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.

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

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]

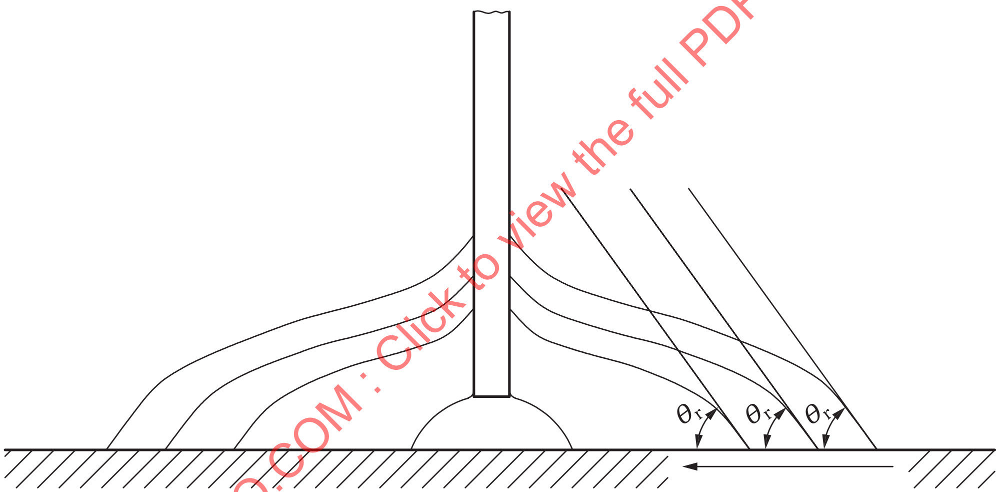
3.7
dynamic contact angle
contact angle (3.3), which is measured during advancing or receding of the three-phase point

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 the 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) in accordance with 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.

4.3 Pre-treatment of the test piece

Measurements and determination of contact angle is extremely surface sensitive, especially to any contamination. The risk of measuring useless results is therefore immense. Store the test pieces when they will not be used immediately. Storage depends on the superhydrophobic material and substrate; storage specifications shall be agreed upon by the interested parties.

4.4 Contact angle measurement — Dynamic method

4.4.1 Advancing angle

The advancing angle shall be measured in accordance with ISO 19403-6. In this method, by adding the test liquids (i.e. water) to a drop on a surface, advancing angles are measured. The measurement should be carried out close to the equilibrium. The standard deviation for the advancing angle should not be more than 5°. In case the standard deviation is more than 5° for the dynamic method, the surface chemical and topological homogeneities shall be checked. In order to improve the reliability, the mean value can be calculated for smaller periods.

4.4.2 Receding angle

The receding angle shall be measured in accordance with ISO 19403-6. In this method, by subtracting the test liquid (i.e. water) from a drop on a surface, receding angles are measured. The measurement should be carried out close to the equilibrium. The standard deviation for the receding angle should not be more than 5°. In case the standard deviation is more than 5° for the dynamic method, the individual measuring values shall be checked. In order to improve the reliability, the mean value can be calculated for smaller periods.

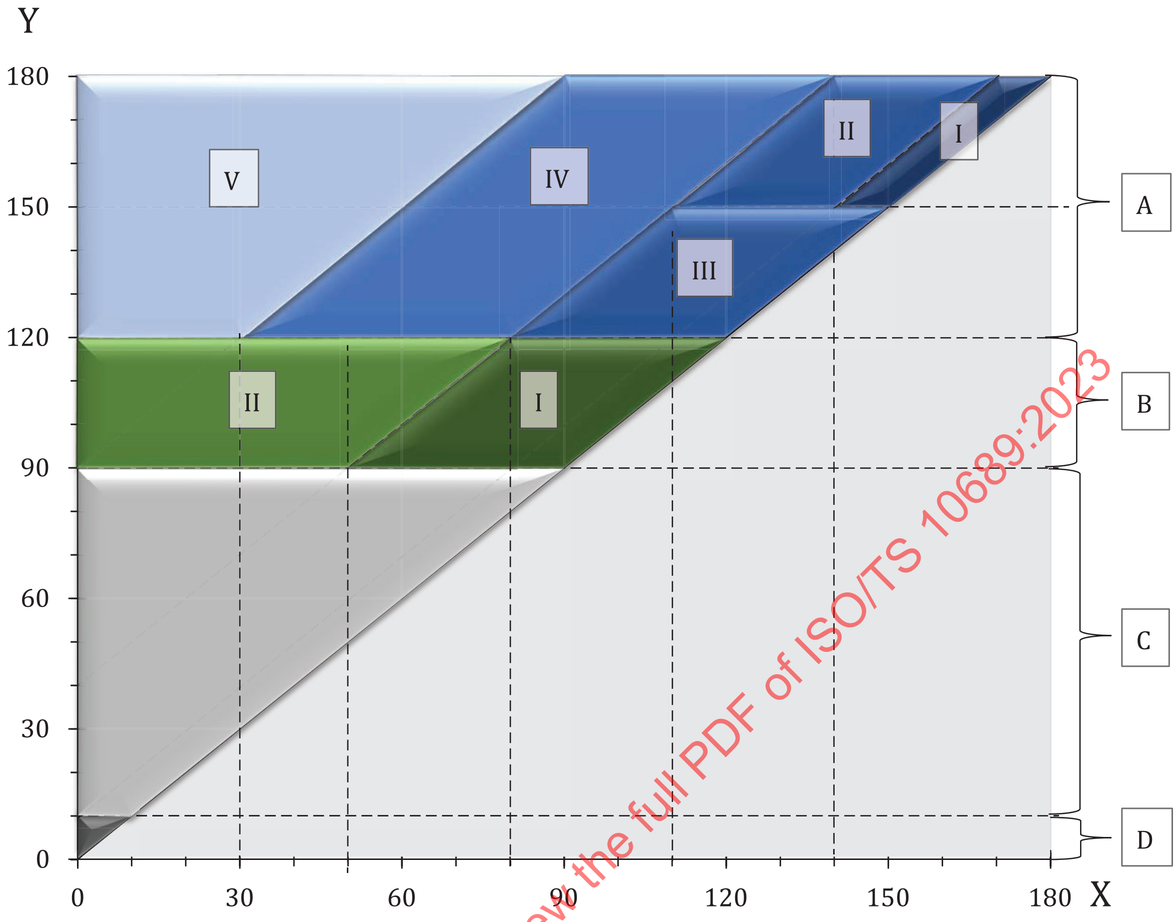
4.4.3 Contact angle hysteresis

The difference between advancing angle and receding angle is the contact angle hysteresis.

4.5 Wettability regions

The maximum water contact angle that can be attained on smooth surfaces (with lowering the surface free energy) is 120° for a surface covered with CF_3 groups, e.g. on Teflon. For heterogeneous surfaces as shown by Cassie-Baxter relation, the apparent contact angle is the weighted average of the contact angles of patches. As the contact angle of water on air is 180°, by roughening the surface, contact angles can be larger than 120°. On rough or heterogeneous surfaces, the apparent contact angle is not unique. The apparent contact angle changes between a minimum (receding angle) and a maximum (advancing angle). From the wettability perspective, surfaces are categorized into: superhydrophobic, hydrophobic, hydrophilic and superhydrophilic.

Adhesion of drops to substrates is linearly correlated to the contact angle hysteresis. The lower the contact angle hysteresis, the lower the force required to shed (detach) a drop from its substrate. The shedding force is usually gravity and/or stream. Comparing two drops with equal volumes, the one with larger contact angle has larger area exposed to air. So, when the drops are exposed to free/forced streams, to facilitate the shedding of drops, along with the low contact angle hysteresis, large contact angle is important. As such, drops easily detach from superhydrophobic surfaces. In this document, the following borders are suggested to better distinguish the superhydrophobics, hydrophobics, hydrophilics and superhydrophilics. See Figure 4 and Table 1.



Key

X receding angle (degree)

Y advancing angle (degree)

A superhydrophobic

B hydrophobic

C hydrophilic

D superhydrophilic

Figure 4 — Superhydrophobic, hydrophobic, hydrophilic and superhydrophilic wettability regions

Table 1 — Grades of superhydrophobic (×5), hydrophobic (×2), hydrophilic and superhydrophilic wettability regions

Wettability	Advancing angle	Receding angle	Contact angle hysteresis
Superhydrophobic I	$\theta_a > 150°$	$\theta_r > 140°$	$\theta_{ar} < 10°$
Superhydrophobic II	$\theta_a > 150°$	$\theta_r > 110°$	$10° < \theta_{ar} < 40°$
Superhydrophobic III	$150° > \theta_a > 120°$	$\theta_r > 80°$	$\theta_{ar} < 40^o$
Superhydrophobic IV	$\theta_a > 120°$	$\theta_r > 30°$	$40° < \theta_{ar} < 90°$
Superhydrophobic V	$\theta_a > 120°$	—	$90° < \theta_{ar}$
Hydrophobic I	$120° > \theta_a > 90°$	$\theta_r > 50°$	$\theta_{ar} < 40°$
Hydrophobic II	$120° > \theta_a > 90°$	—	$40° < \theta_{ar}$
Hydrophilic	$90° > \theta_a > 10°$	—	—
Superhydrophilic	$10° > \theta_a$	—	—

Based on the above-mentioned discussion in this subclause, superhydrophobic grades IV and V (or pseudo-superhydrophobics) are not always preferred to hydrophobic grade I. For example, for the applications that the shedding force is not a free/forced stream, smaller contact angle hysteresis is preferred to larger contact angle (i.e. hydrophobic grade I is preferred to superhydrophobic grades IV and V). For the application that the surface is exposed to air, a higher contact angle is more important, so superhydrophobic grades IV and V are preferred to hydrophobic grade I.

5 Procedure

5.1 General

Considering the fact that superhydrophobic surfaces and coatings are in practice subjected to different environmental and working conditions, the performance assessment of superhydrophobic surfaces and coatings shall be carried out and reported based on measuring the dynamic angles and calculating the contact angle hysteresis before and after applying such working conditions. The working conditions include mechanical stress (water impacting, and wear resistance), solar radiation and weathering, liquid attacks, and thermal cycling (see Annex B). Depending on the application(s), the subject of working condition(s) to be applied shall be the matter of agreement between the interested parties.

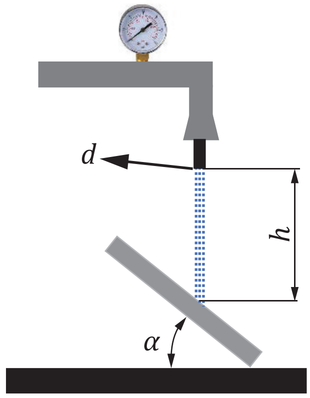
5.2 Mechanical stress methods

In practical applications, superhydrophobic surfaces and coatings are unavoidably rubbed or scratched by washing pads, sand or rain drops, which can lead to deterioration of the hierarchical structures. Therefore, assessing the mechanical durability and adhesion is essential to applicability of the superhydrophobic surfaces and coatings.

5.2.1 Water impacting test

5.2.1.1 General

The purpose of this test is to evaluate the resistance of superhydrophobic surfaces and coatings to rain and thunderstorm. During heavy rain in a thunderstorm, the maximum diameter and velocity of rain drops are 4 mm to 5 mm and 7 m/s to 9 m/s, respectively[4]. Considering the precipitation of heavy rain during thunderstorm is 2,5 cm/h, the following setup can be used to simulate the rainfall and Table 2 can be used to relate the duration of water impacting test to the duration of constant heavy rainfall in real-life. For such drops, the Weber number (We), a dimensionless number which compares inertia to surface tension, is in the order of 10^3. So, the effect of surface tension is negligible, and inertia is the main driving force on micro/nano-textures.



Key

h spacing from pipe to the specimen

d pipe diameter

α sample inclination angle

Figure 5 — Water impacting test setup

Table 2 — Duration of water impacting test related to the real-life rainfall duration.

Velocity of water exiting the pipe m/s	Experiment duration equivalent to 1-year constant rain s
7	32
9	24,7

5.2.1.2 Preparation

Calculate the flowrate by weighing the water exiting the pipe and measuring the time. Calculate the water exiting velocity by dividing the volume flowrate to the faucet surface area. Make sure the water velocity is at least 7 m/s and preferably less than 9 m/s. The spacing from pipe to the specimen should not exceed 50 cm.

5.2.1.3 Procedure

Place the sample as shown in Figure 5 preferably put the sample at 45° (i.e. $\alpha = 45^o$) and examine the falling water stream hits the center of the specimen. Subsequent to the water impacting test, and before measuring the contact angle, the specimen shall be dried.

Before and after each test, measure the advancing angle and the receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4.

5.2.1.4 Test report

The test report shall contain at least the following information:

a) specimen description;

b) velocity of water exiting the pipe, duration of the test and sample inclination angle;

c) a reference to this document (i.e. ISO/TS 10689:2023);

d) agreed sample preparation and conditioning requirements between interested parties;

e) the results of the test, as identified in 5.2.1.3;

f) the method used;

g) any deviation from the procedure;

h) any unusual features observed;

i) the date of the test.

5.2.2 Wear resistance tests

5.2.2.1 Abrasion resistance test

5.2.2.1.1 General

The purpose of this test is to evaluate to what extent the superhydrophobic surfaces and coatings are affected when subjected to specific abrading condition at ambient atmospheric condition. In this test by applying the test load, a rigid abrasive paper is pressed on the superhydrophobic surfaces and coatings. Then the abrasive paper is moved on the surface. The abrasion resistance test shall be performed in accordance with ISO 7784-3. Then the possible alteration of advancing and receding angles and contact angle hysteresis of the as-received sample and those subjected to abrasion resistance test shall be reported.

5.2.2.1.2 Preparation

It is recommended to use a new fresh portion of the abrasive paper before each double stroke (see ISO 7784-3).

5.2.2.1.3 Procedure

Subsequent to the abrasion, the specimen shall be cleaned as per agreement between interested parties. To see if the cleaning procedure was effective, the chemical and topological homogeneities can be checked.

Abrasive paper can release chemicals, abrasive and dust on the surface. The surface shall not show evidence of any residue. If the surface is suspected of having foreign material embedded in it, clean the surface per agreement between interested parties, after the abrasion resistance test and before the contact angle measurement.

Before and after each double stroke abrasion, measure the advancing angle and receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4.

The number of iterations of the test should be agreed between the interested parties.

5.2.2.1.4 Test report

The test report shall contain at least the following information:

a) all items identified in ISO 7784-3:2022, Clause 10;

b) a reference to this document (i.e. ISO/TS 10689:2023);

c) agreed sample preparation and conditioning requirements between interested parties;

d) the results of the test, as identified in 5.2.2.1.3;

e) the method used;

f) any deviation from the procedure;

g) any unusual features observed;

h) the date of the test.

5.2.2.2 Falling-sand test

5.2.2.2.1 General

This test method covers the determination of the resistance of superhydrophobic surfaces and coatings to abrasion produced by abrasive falling and dunes onto the surface. The test shall be performed in accordance with ISO/TR 21555:2019, 5.5.

5.2.2.2.2 Preparation

Align the apparatus until the inner concentrated core of the sand stream falls in the center of the flow when viewed at two positions at 90° to each other.

5.2.2.2.3 Procedure

Pour a quantity of standard sand specified in ISO/TR 21555:2019, 5.5, into the funnel and examine the sand stream falling from the lower end of the guide tube.

Before and after each test, measure the advancing angle and the receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4.

The superhydrophobic surface/coating shall not show evidence of residue. To see if the cleaning procedure was effective, the chemical and topological homogeneities can be checked. If the surface is suspected of having foreign material embedded in it, clean the surface per agreement between interested parties.

The number of iterations of the test should be agreed between the interested parties.

5.2.2.2.4 Test report

The test report shall contain at least the following information:

a) all necessary details identified in ISO/TR 21555:2019, 5.4.1;

b) a reference to this document (i.e. ISO/TS 10689:2023);

c) agreed sample preparation and conditioning requirements between interested parties;

d) the results of the test, as identified in 5.2.2.2.3;

e) the method used;

f) any deviation from the procedure;

g) any unusual features observed;

h) the date of the test.

5.2.2.3 Scrub test with brush

5.2.2.3.1 General

The ability of superhydrophobic surfaces and coatings to withstand wear caused by repeated cleaning operations is an important consideration. This test is designed to evaluate the washability of painted and coated surfaces. The test shall be performed in accordance with ISO/TR 21555:2019, 5.4.1. This test method covers the determination of the resistance of superhydrophobic surfaces and coatings to abrasion produced by wet abrasion onto the surface.

5.2.2.3.2 Preparation

Adjust the stroke length of the wet-scrub tester apparatus to (300 ± 10) mm and operate at approximately 30 scrub cycles per minute. A counter for recording the number of scrub cycles shall be provided. Hold the sample with the scrub pad holder and replace the abrasive pad for every test.

5.2.2.3.3 Procedure

The superhydrophobic surface/coating is subjected to a number of wet-scrub cycles in a scrub testing machine. The number of iterations of the test should be agreed between the interested parties.

Before and after each test, measure the advancing angle and the receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4.

The superhydrophobic surface/coating shall not show evidence of residual. To see if the cleaning procedure was effective, the chemical and topological homogeneities can be checked. If the surface is suspected of having foreign material embedded in it, clean the surface per agreement between interested parties.

5.2.2.3.4 Test report

The test report shall contain at least the following information:

a) all necessary details identified in ISO/TR 21555:2019, 5.4.1;

b) a reference to this document (i.e. ISO/TS 10689:2023);

c) agreed sample preparation and conditioning requirements between interested parties;

d) the results of the test, as identified in 5.2.2.3.3;

e) the method used;

f) any deviation from the procedure;

g) any unusual features observed;

h) the date of the test.

5.3 Determination of the resistance to solar radiation and weathering

5.3.1 General

Radiation, temperature and humidity contribute to the aging process of surfaces and coatings. The aim of this test is to compare and assess the performance of superhydrophobic coatings/surfaces after the exposure to solar radiation and weathering. The test can be performed according to ISO 16474-2, where methods are described to expose coatings to xenon-arc radiation, to simulate the aging process, which occurs when materials are exposed in their actual end-use environments to solar radiation or to solar radiation filtered through window glass. For outdoor conditions optical systems in accordance with ISO 16474-2, method A shall be used to simulate solar radiation. For indoor conditions optical systems in accordance with ISO 16474-2, method B shall be used to simulate solar radiation filtered by window glass. Also, for the simulation of outdoor conditions, specimens may be exposed to moisture in the form of water spray, condensation, or by immersion.

5.3.2 Specimen preparation and conditioning

The method used for test panel preparation shall be agreed upon by the interested parties. It should preferably be closely related to the method normally used to process the material in typical applications.

Attach the specimens to the specimen holders in the test chamber in such a manner that the specimens are fixed, but not compressed.

Before testing, condition the test specimens for at least 16 h under standard conditions as specified in ISO 3270, i.e. (23 ± 2) °C and (50 ± 5) % relative humidity.

5.3.3 Procedure

Test cycle, test duration, iteration, specimen conditioning and intermediate inspections shall be agreed upon by the interested parties.

Operate the equipment to simulate outdoor conditions, for example, according to ISO 16474-2 Cycle 1 (black standard temperature control) or Cycle 4 (black panel temperature control) and for indoor conditions, for example, according to ISO 16474-2 Cycle 2 (black standard temperature control) or Cycle 5 (black panel temperature control).

Expose the specimen to the agreed test cycle. If a test cycle with specimen wetting was used, the test should be interrupted for inspection at the end of a dry phase. Before examination, the specimen shall be conditioned in accordance with the agreed procedure and duration.

When removing the specimen for contact angle measurement, take care not to touch the exposed surface or alter it in any way. The advancing angle, receding angle and contact angle hysteresis shall be reported. The examination shall be performed as specified in 4.4.

The superhydrophobic surface shall not show evidence of residual. If the surface is suspected of having foreign material embedded in it, dry and clean the surface as agreed between the interested parties.

If after the examination the test needs to be continued, return the specimen to its holder with its exposed surface oriented in the same direction as before and continue the exposure.

5.3.4 Test report

The test report shall contain at least the following information:

a) specimen description;

b) description of exposure test conducted in accordance with ISO 16474-2;

c) the date of the exposure test;

d) a reference to this document (i.e. ISO/TS 10689:2023);

e) the agreed sample preparation and conditioning requirements between interested parties;

f) the results of the test, as identified in 5.3.3;

g) the method used;

h) any deviation from the procedure;

i) any unusual features observed.

5.4 Determination of resistance to liquids

5.4.1 General

Superhydrophobic surfaces and coatings are exposed to rain which is sometimes acidic in industrial areas, and detergents which are usually basic. Acidic, basic and ionic solutions may contribute to the deterioration of the superhydrophobic surfaces and coatings. This test is devised to assess the resistance of superhydrophobic surfaces and coatings to different liquids. The test shall be performed in accordance with ISO 2812-1. ISO 2812-1 specifies general methods for determining the resistance of an individual-layer or multi-layer system of coating materials to the effect of liquids, other than water, or paste-like products.

5.4.2 Preparation

Both sides of the sample should be superhydrophobic and the edges protected, otherwise, the reverse side and edges should be protected with a sufficiently resistant coating agreed by interested parties. The suggested liquids used for assessing the resistance of superhydrophobic surfaces are: acidic water (3 mol/l of sulfuric acid), alkaline water (6 mol/l of sodium hydroxide) and ionic solution (mass fraction of 3,5 % of sodium chloride) or any other liquid as agreed between interested parties.

5.4.3 Procedure

Superhydrophobic surface or surface with superhydrophobic coating is exposed to a test liquid by immersion in the test liquid according to ISO 2812-1.

After the test period has expired, unless specified and agreed between interested parties, wipe the test piece with a cloth. Then clean off any dried residue of test liquids under running water or with a solvent that does not attack the surface and coating. To see if the cleaning procedure was effective, the chemical and topological homogeneities can be checked.

The effects of the exposures are assessed by measuring the contact angles. Before and after each immersion followed by cleaning and drying, measure the advancing angle, receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4. Evaluate only the areas which have been in direct contact with the test liquid.

The number of iterations and duration of the test should be agreed between the interested parties.

5.4.4 Test report

a) all necessary details identified in ISO 2812-1;

b) a reference to this document (i.e. ISO/TS 10689:2023);

c) agreed sample preparation and conditioning requirements between interested parties;

d) the results of the test, as identified in 5.4.3;

e) the method used;

f) any deviation from the procedure;

g) any unusual features observed;

h) the date of the test.

5.5 Thermal cycling test

5.5.1 General

The thermal cycling test covers the determination of the resistance of superhydrophobic surfaces and coatings during prolonged exposure in harsh climates. The suggested thermal cycling is described in ISO 11997-3 daily cycle C (−15 °C to 50 °C, where each cycle is 24 h).

5.5.2 Procedure

Before and after each cycle, measure the advancing angle and the receding angle, and calculate the contact angle hysteresis. The examination shall be performed as specified in 4.4.

The number cycles should be agreed between the interested parties.