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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials — Measurement of water contact angle

Céramiques techniques — Méthode d'essai de la performance auto nettoyante des matériaux photocatalytiques semiconducteurs — Mesurage de l'angle de contact de l'eau

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 27448 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Introduction

Under the illumination of ultraviolet (UV) light, photocatalysts show diverse functions, such as the decomposition of air and water contaminants, as well as deodorization, self-cleaning, antifogging and antibacterial actions. These functions of photocatalysts are generally based on the action of active oxygen species such as hydroxyl (OH) radicals formed on the surface of photocatalysts. The energy- and labour-saving nature of photocatalysis has attracted keen interest when the photocatalyst is activated by sunlight (or artificial lighting).

Practical applications of photocatalysts, for both indoor and outdoor use, have rapidly expanded in recent years. Many kinds of photocatalytic materials have been proposed or are already commercialized, based on ceramics, glass, concrete, plastics, paper, etc. Such materials are produced by either coating or mixing of a photocatalyst, in most cases, titanium dioxide (TiO_2).

However, the effect of photocatalysis is not easily inspected visually, and no appropriate and official evaluation methods have been available to date. Some confusion has thus arisen as photocatalytic products have been introduced. Furthermore, the above-mentioned diverse functions of photocatalysts cannot be evaluated with a single method; thus, different evaluation methods are provided for self-cleaning, water decontamination, air purification and anti-bacterial actions, respectively.

As a result of continuing efforts to provide test methods for photocatalytic materials, this International Standard (covering the measurement of the water contact angle) for self-cleaning performance was prepared. For permeable, rough, or highly hydrophobic surfaces, etc., other test methods are required and are being developed.

It must be noted that self-cleaning performance could be evaluated with photo-induced hydrophylic property and photocatalytic decomposition ability, because many field test results concerning the self-cleaning properties of TiO_2-coated materials are in good agreement with the indexes obtained by measurement of the water contact angle and degradation of methylene blue.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for self-cleaning performance of semiconducting photocatalytic materials — Measurement of water contact angle

1 Scope

This International Standard deals with fine ceramics.

This International Standard specifies a test method for the determination of the self-cleaning performance of materials that contain a photocatalyst or have photocatalytic films on the surface, and which are usually made from semiconducting metal oxides such as titanium dioxide.

This method is used to measure the water contact angle under illumination with ultraviolet light, which is one of the indices influencing the self-cleaning performance of photocatalytic materials.

This International Standard does not include water-permeable substrates, rough surfaces which do not have exposed water droplets, highly hydrophobic, powder or granular materials, or visible light-sensitive photocatalysts.

2 Normative references

The following referenced documents are indispensable for the application 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 3534-1:2006, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 80000-1, *Quantities and units — Part 1: General*

3 Terms and definitions

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

3.1
photocatalyst
substance that carries out one or more functions based on oxidization and reduction reactions under photoirradiation, including decomposition and removal of air and water contaminants, deodorization, and antibacterial, self-cleaning and antifogging actions

3.2
photocatalytic materials
materials in which, or on which, the photocatalyst is added by coating, impregnation, mixing, etc.

NOTE Such photocatalytic materials are intended for use as building and road construction materials to obtain the functions mentioned in 3.1.

3.3
fluorescent UV lamp
lamp that provides UV-A irradiation within a wavelength range of 300 nm to 400 nm

NOTE A suitable lamp is the so-called black light blue (BLB) fluorescent lamp, with a maximum wavelength of 351 nm, as described in ISO 4892-3.

3.4
hydrophilicity
property in which the surface of the photocatalytic material shows a strong affinity for water

3.5
self-cleaning
function whereby a photocatalyst, attached to a surface like a building material, prevents fouling of the surface by utilizing at least one of the following: the phenomenon where fouling substances attached to the surface are broken down by oxidation and reduction when irradiated with light, and/or the phenomenon where fouling is washed away when the surface is exposed to rain or rinsed with water, due to the hydrophilicity which manifests itself at the same time

3.6
contact angle
when a tangent line is drawn from the contact point of the solid, liquid and vapour phases (normally air) along the curve of the liquid on the solid, the angle between the line and the solid surface is referred to as the contact angle

3.7
initial contact angle
contact angle immediately before starting UV irradiation

3.8
contact angle after n h of UV irradiation
contact angle after applying UV irradiation for n h

NOTE The unit of time may also be days, minutes or seconds, in addition to hours.

3.9
final contact angle
contact angle at the highest water affiliation after irradiation at a constant intensity on a photocatalytic material

3.10
pretreatment of test piece
preparation of a test piece by irradiating it with UV and then applying organic substances such as oleic acid

3.11
coefficient of variation
ratio of the standard deviation to the arithmetic mean

4 Symbols

θ_i initial contact angle, in degrees

θ_n contact angle after n h, in degrees

$\bar{x}$ average of the three consecutive points, in degrees

s standard deviation of the three consecutive points, in degrees

θ_f final contact angle, in degrees

5 Principle

This International Standard is for the development, comparison, quality assurance, characterization, reliability, and design data generation of photocatalytic materials.

This International Standard defines the test method, which provides a performance correlated with the contamination that occurred during the exposure test.

This test method is to evaluate the self-cleaning performance of a photocatalytic material by obtaining the final contact angle of a test piece on which organic substances are applied. The organic substance is first applied to a test piece (pretreatment), which is then irradiated by UV at a constant intensity. The water contact angle after n h can be measured during the process. This test simultaneously evaluates the decomposition of the organic substance and change of water affiliation. When the pretreatment of a certain test piece is impossible and the initial contact angle before UV irradiation is 20° or greater, the final contact angle can be obtained without pretreatment.

6 Apparatus

6.1 Black light fluorescent lamp

The fluorescent UV lamp shall be a black light blue (BLB) lamp with a peak wavelength of 351 nm and blue glass for absorbing visible light.

6.2 Ultraviolet irradiator

The equipment shall provide uniform irradiation from the lamp to the test piece and the irradiation intensity shall be adjustable by moving the position of the lamp or test piece.

6.3 Ultraviolet light radiometer

The irradiation intensity shall be measurable at the test sample position. The photoelectric sensor shall have good cosine characteristics. The irradiation-measuring instrument shall be calibrated with the light source to be used, or corrected to a certain sensitivity within the wavelength range to be absorbed by the photocatalytic test piece.

6.4 Contact angle meter

The instrument shall have a measurement range of 0° to 180°, a readout accuracy of 0,1° and a measurement accuracy of $\pm$ 1°. The instrument shall be capable of measuring the contact angle from the image of a liquid drop attached to a test sample by using the $\theta/2$ method after the dropping attachment has been used for a certain time.

7 Test chemicals

7.1 **Oleic acid**, of assay (cGC) 60,0 % or higher.

7.2 **n-Heptane**, of assay (cGC) 99,0 % or higher.

8 Temperature and humidity of test laboratory

The test laboratory should preferably be controlled at a temperature range of 23 °C $\pm$ 5 °C, and a relative humidity range of 40 % to 70 %.

9 Test piece

Cut out a 100 mm ± 2 mm square flat piece of a product as the standard-size test piece. Use caution not to contaminate the test piece with organic substances or other test pieces. It is recommended to obtain test pieces from the product but, if it is impossible to prepare test pieces due to the product's shape, then the test pieces may be created from a separate flat plate made from the same raw material and by using the same process. If it is impossible to cut a product into 100 mm ± 2 mm square pieces, then the test piece may have a different shape or size, as long as its shape and size allow the measurement of the contact angle at five different points. Test pieces shall be prepared in quantities of five.

10 Procedure

10.1 Pretreatment of test piece

10.1.1 Procedure of pretreatment

The following procedure shall be used for pretreatment of the test piece. When the test pieces are not used immediately after this pretreatment, store them in a sealed container. This pretreatment can be omitted if it is impossible to apply oleic acid and if the contact angle before pretreatment is not less than 20°.

In the case of a test in which the procedures in 10.1.2 and 10.1.3 were eliminated, in addition to the procedures in 10.2.3 and 10.2.4, the dark field test without any UV exposure shall be conducted.

The dark field test shall be performed in accordance with the same procedure as that in 10.2.3 and 10.2.4 by taking the number of test pieces as five, except for the procedure of performing UV irradiation. When the dark field test shall be performed simultaneously with the UV irradiation test, “the contact angle after n h without any UV exposure” in progress may not be measured.

10.1.2 Removal of organic substances

Use an ultraviolet irradiator adjusted so that the illuminance at the test piece surface is 2,0 mW/cm^2, recorded using an ultraviolet light radiometer, and perform ultraviolet irradiation on the test piece for at least 24 h. If necessary, the surface on the test piece may be washed before the illumination.

NOTE To prevent contamination of the test piece after pretreatment with hydrophobic substances, etc., wear gear such as polyethylene gloves during handling to prevent direct contact.

10.1.3 Application of oleic acid

Apply oleic acid to the photocatalyst-coated surface manually or by dipping.

a) Manual application

   Place the test samples with the photocatalyst-coated surface facing up and pour 200 µL of oleic acid around the centre of the coated surface; then spread the acid uniformly from the centre to the entire surface in all directions by using a piece of non-woven cloth. Wipe off excess acid and adjust the mass of acid to 2,0 mg ± 0,2 mg per 100 cm^2 by weighing the test piece.

b) Dipping

   Prepare a 0,5 % (by volume) oleic acid solution by diluting with *n*-heptane and dip the test piece in the solution. After lifting the test piece from the solution at a speed of 60 cm/min, dry it at 70 °C for 15 min.

10.2 Measurement of water contact angle

10.2.1 Measurement of UV intensity and preparation of UV irradiation equipment

Use the UV irradiation equipment with an adjusted irradiation intensity on the surface of the test pieces with an ultraviolet light radiometer at $(2{,}0 \pm 0{,}1)$ mW/cm^2 in the case of 10.1.3 a) Manual application, or at $(1{,}0 \pm 0{,}1)$ mW/cm^2 in the case of 10.1.3 b) Dipping.

10.2.2 Measurement of contact angle before the UV irradiation

Measure the initial contact angle at five points on each test piece and average the values. These averaged values are defined as the contact angle before UV irradiation of each test piece. The quantity of distilled water for dripping shall be determined by using the contact angle meter. Measurement of the contact angle shall be done within approximately 3 s to 5 s after dripping the distilled water and assume this measurement shall be used as 0 h data.

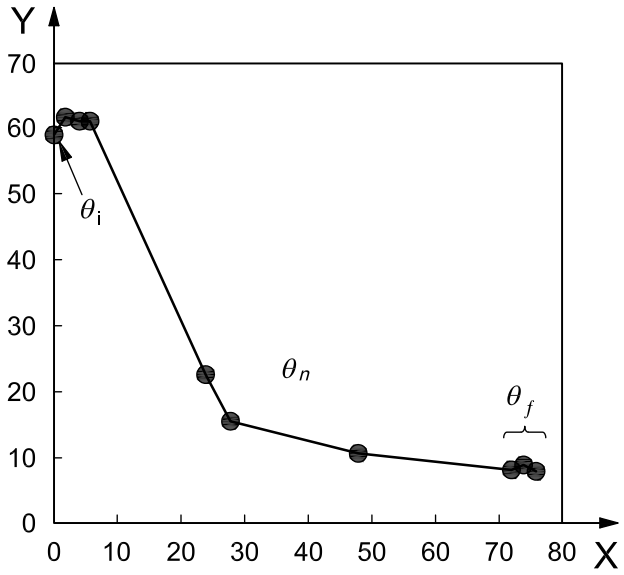
10.2.3 Measurement of contact angle after n h of UV irradiation

After starting UV irradiation, measure the contact angle at five places on each test piece at an appropriate irradiation time interval. The mathematical average value of the five measurements on each test piece is set as the "contact angle after n h of UV irradiation".

10.2.4 Measurement of final contact angle

Obtain the coefficient of variation, in accordance with ISO 3534-1, of the "contact angle after n h of UV irradiation" of the three consecutive measurements on each test piece. If the value is 10 % or smaller, then finish the measurement (see Note 1). The mathematical average of the three contact angle measurements is set as the "final contact angle".

NOTE 1 When the contact angle after n h of UV irradiation of each test piece is 5° or smaller, then the measurement is finished at that point and the measured contact angle of each test piece can be set as the "final contact angle".



Key

X time, h

Y water contact angle, degrees

Figure 1 — Typical data of contact angle during the test operation (see Note 2)

NOTE 2 Oleic acid was applied to the photocatalyst-coated surface by dipping, as a pretreatment of the test piece.

11 Calculation

11.1 Guide to the rounding of numbers

The test results shall be calculated as follows. The calculated values are usually rounded to the second decimal place in accordance with ISO 80000-1.

11.2 Condition for a valid test

The initial contact angle θ_i shall be greater than or equal to 20°.

NOTE When an initial contact angle θ_i is less than 20°, the test is invalid because it is difficult to judge whether contact angles decreased.

11.3 Determination of the final contact angle

The final contact angle is calculated by Equation (1):

$$\overline{x} = \frac{\left(\theta_{n1} + \theta_{n2} + \theta_{n3}\right)}{3} \tag{1}$$

$$\frac{s}{\overline{x}} \leqslant 10\ \% \tag{2}$$

$$\theta_f = \overline{x} \tag{3}$$

where

θ_{n1} is the contact angle after n1 h, in degrees;

θ_{n2} is the contact angle after n2 h, in degrees;

θ_{n3} is the contact angle after n3 h, in degrees;

$\overline{x}$ is the average of the three consecutive points, in degrees;

s is the standard deviation of the three consecutive points, in degrees;

θ_f is the final contact angle, in degrees.

12 Test report

The test report shall include the following information:

a) date of test, temperature, relative humidity, etc.;

b) description of the test piece (material, size, shape, etc.);

c) manufacturer's name of reagent used, and reagent grade type;

d) black-light fluorescent lamp: manufacturer's name, model, peak wavelength;

e) ultraviolet light radiometer: manufacturer's name, model;

f) contact angle meter: manufacturer's name, model;

g) test-piece pretreatment method and ultraviolet irradiation time;

h) method of oleic acid application (manually or dipping);

i) initial contact angle before irradiation for each test piece;

j) final contact angle and UV irradiation time for each test piece;

k) final contact angle without any UV exposure and the test time for each test piece in the case of the dark field test;

l) if necessary, contact angle after n h of UV irradiation for each test piece;

m) if necessary, contact angle after n h of the dark field test without any UV exposure for each test piece;

n) any other matters of special note, such as a change in the test piece noticed during the test.

Annex A
(informative)

Example of test results

An example of test results is given in Table A.1.

Table A.1 — Test result example

A1		**Contact angle: five measurements on test piece**, degrees					θ_n	s	$\overline{x}$	$\frac{s}{\overline{x}}$
		1	2	3	4	5	degrees	degrees	degrees	%
Ultraviolet radiation time, h	0	54,8	55,2	60,6	55,9	47,7	54,8			
	2	55,9	60,3	60,9	59,2	59,4	59,1			
	4	57,8	60,2	60,9	62,3	59,3	60,1	2,8	58,0	4,9
	6	57,4	55,7	58,7	54,9	61,3	57,6	1,3	58,9	2,1
	24	45,5	27,1	14,8	19,8	16,1	24,7	19,8	47,5	41,6
	28	48,5	34,2	19,7	23,6	35,0	32,2	17,2	38,2	45,2
	48	12,8	8,3	9,8	10,0	10,8	10,3	11,1	22,4	49,7
	72	8,3	7,4	8,2	8,8	7,6	8,1	13,3	16,9	79,0
	74	7,3	8,2	9,8	7,9	7,5	8,1	1,3	8,8	14,4
	76	9,8	9,7	9,5	8,6	9,3	9,4	0,75	8,5	8,8

NOTE Oleic acid was applied to the photocatalyst-coated surface by dipping, as a pretreatment of the test piece.

Determination of the final contact angle, in the case of test piece A1:

θ_{n1} is the contact angle after $n1$ h = 8,1 ($n1 = 72$);

θ_{n2} is the contact angle after $n2$ h = 8,1 ($n2 = 74$);

θ_{n3} is the contact angle after $n3$ h = 9,4 ($n3 = 76$);

For UV irradiation time = 76 h:

$\overline{x}$ is the average of the three consecutive points = 8,5;

s is the standard deviation of above three consecutive points = 0,75;

$s/\overline{x}$ is the coefficient of variation = 8,8 %;

θ_f is the final contact angle = 8,5°.

Bibliography

[1] ISO 4892-3:2006, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

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