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Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral reflectance of fine ceramic thin films under humid conditions

Céramiques techniques — Méthode de mesurage de la transmittance spectrale des films minces de céramiques fines en conditions humides

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Foreword

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

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

Fine ceramic thin films are used in many optical applications, such as anti-reflective coatings, infrared sensor cut filters, X-ray sensor cut filters and band-pass filters. Even though the spectral reflectance of fine ceramic thin films is specified for each product, the refractive index and the optical properties change if these thin films are exposed to a humid environment. The reason for such changes in the optical properties is that water is adsorbed onto the surfaces inside the voids of fine ceramic thin films. A standard for evaluating the reliability of these films under a range of humidity conditions is therefore necessary. A standard for determining the spectral transmittance under humid and dry conditions was developed in ISO 17861. This document provides test methods that enable changes in the spectral reflectance of fine ceramic coatings in a humid environment due to water adsorption to be evaluated easily and accurately. The aim of this document is to promote the rapid adoption of this test method to facilitate further growth in this industry.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral reflectance of fine ceramic thin films under humid conditions

1 Scope

This document specifies the procedure for measuring the spectral reflectance of fine ceramic thin films in an environment with variable relative humidity by using a general-purpose spectrophotometer.

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 8980-3, *Ophthalmic optics — Uncut finished spectacle lenses — Part 3: Transmittance specifications and test methods*

ISO 9211, *Optics and photonics — Optical coatings*

ISO 17861, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method of spectral transmittance of fine ceramics thin films under humid condition*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8980-3, ISO 9211 and ISO 17861 and the following 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
half of maximum/minimum reflectance wavelength
$\lambda_{R1/2}$
wavelength at which the reflectance is equal to half of the maximum and minimum values observed in a spectral reflectance curve obtained for a certain range of wavelength

3.2
environmental mini-chamber
small chamber that is used to control humidity

Note 1 to entry: This chamber is equipped with a quartz window for the optical beam inlet and outlet, an evacuation feedthrough to evacuate the mini-chamber and an inlet to feed air with a controlled humidity to the mini-chamber.

3.3
spectral reflectance
regularly reflected or spectral component of the illumination (i.e. light that is reflected in a mirror-like way off a surface at the same angle and in the same plane as the incident ray) that is measured by using a spectral reflectometer

3.4
monochromator
optical device that transmits a specific range of wavelengths from a polychromatic light

3.5
spectrophotometer
optical instrument that measures spectral transmittance or reflectance

Note 1 to entry: This instrument consists of an optical source, a monochromator, a sample chamber, an optical detector, a signal processor, a data processor, and an interface

3.6
double-beam spectrophotometer
type of spectrophotometer that is utilized to compare the light intensity of a reference sample with that of a test piece

Note 1 to entry: Compared with a single-beam spectrophotometer, this instrument provides a higher level of measurement stability. In a double-beam spectrophotometer, the energy of the source light beam is divided into two beams: a reference beam and a sample beam. The energy of the sample beam is detected by referring to the energy of the reference beam.

3.7
integrating sphere
optical component consisting of a hollow spherical cavity with an inner surface that is covered with a diffuse white reflective coating

Note 1 to entry: The cavity is equipped with small holes for the mounting of a test piece and for the inlet and outlet of the light beam.

4 Principle

Fine ceramic thin films sometimes possess a voided micro columnar structure. Due to this structure, moisture in the air is adsorbed onto the surfaces of columns with voids between them. The quantity of adsorbed water is affected by the hydrostatic vapour pressure and temperature in the environment. Depending on the quantity of water that is adsorbed, the refractive index of a thin film changes, resulting in a change in its spectral reflectance. To evaluate what effects the adsorption of water on the surface of voids in fine ceramic thin films has on the spectral reflectance of thin films, it is necessary to measure the optical properties under a vacuum, a high-humidity environment and a low-humidity environment. Comparing the results measured under a vacuum, a high-humidity environment and a low-humidity environment facilitates an evaluation of changes in the spectral reflectance due to the environmental humidity.

5 Measurement room ambient environment

The tests shall be carried out in a measurement laboratory under a room atmosphere in which the temperature and humidity changes are negligible. In particular, to prevent the condensation of water in the environmental mini-chamber, the ambient temperature shall be controlled to restrict it to a certain range.

a) Measurement room temperature: (23 ± 2) °C.

b) Measurement room relative humidity: below 70 %.

6 Test pieces

The test pieces shall comprise single-layer or multilayer thin films of fine ceramics deposited on substrates such as glass or polymer materials. There are no restrictions on the types of materials that can be used for the substrates, provided the measurements will not be disturbed by any of the

properties of the substrates. Any test piece is acceptable as long as it fits in the environmental mini-chamber in terms of its size and shape.

7 Measuring apparatus

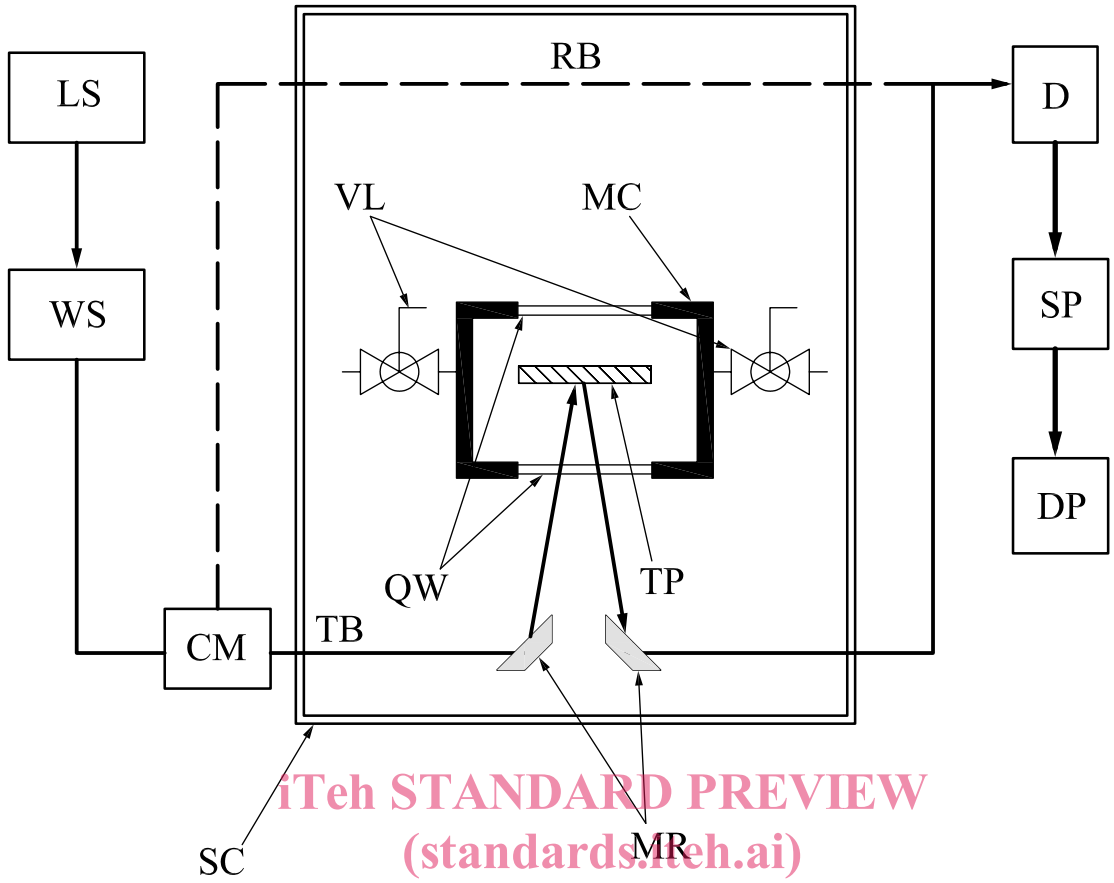
7.1 Apparatus configuration

a) Spectrophotometer: an instrument that utilizes a double-beam method with a wavelength accuracy of ±0,2 nm or less in UV-visible light and ±1,0 nm or less in the near-infrared domain. The repeatability of the wavelength setting is ±0,1 nm or less in UV-visible light and ±0,5 nm or less in the near-infrared domain, with a desirable measurement range of 300 nm to 2 500 nm.

   The use of a double-beam spectrophotometer is strongly recommended. However, the use of an integrating sphere is not mandatory.

b) Environmental mini-chamber: an airtight container that maintains test pieces under a vacuum, a high-humidity environment and a low-humidity environment for the measurement of the spectral reflectance under each condition. The chamber consists of an optical feedthrough that comprises a transparent quartz glass plate, a backside window, and two valves for the inlet of environmental air and evacuation. A test piece is fixed inside the chamber for the measurement of light reflectance. It is necessary to confirm that there is no vacuum leak from the chamber when the valves are closed.

   The spectrometry device configuration is shown in Figure 1. An example of the blueprint is shown in Annex A.



Key

LS	light source	VL	valves
WS	wavelength selector (monochromator)	QW	quartz window
CM	chopper mirror	MR	mirrors
SC	specimen compartment	D	detector
MC	environmental mini-chamber	SP	signal processor
TB	test beam	DP	data processor
RB	reference beam	TP	test piece

Figure 1 — Device configuration for spectral reflectance measurements

c) Vacuum evacuation: an exhaust equipment rotary pump or a dry pump shall be used. Any type can be used, provided the ultimate pressure is less than 40 Pa.

d) Temperature/humidity-controlled chamber: a type of chamber that allows the temperature and relative humidity to be set to 23 °C and from 40 % to 80 %, respectively. It is desirable to measure the humidity around the test pieces by using a hygrometer.

7.2 Spectrophotometer calibration

Perform a wavelength and reflectance calibration for the spectrophotometer apparatus as follows:

a) Wavelength calibration: apply a test method that uses an optical glass filter.

b) Baseline correction: set the spectral reflectance to 100 % within the range of the measuring wavelength by using the baseline adjustment function of the apparatus without setting an environmental mini-chamber.

8 Environmental conditions and control procedures

Expose the test piece to the required environmental conditions before measuring the spectral reflectance. The test piece should not be removed from the environmental mini-chamber during the series of measurements conducted under a vacuum, a high-humidity environment and a low-humidity environment. The recommended measurement conditions for a vacuum, a high-humidity environment and a low-humidity environment are as follows.

a) Vacuum: evacuate the environmental mini-chamber containing the test piece by connecting one of its valves to a vacuum pump. Evacuate for more than 30 min.

b) High-humidity environment: place the environmental mini-chamber containing a test piece in the temperature/humidity-controlled chamber. Open both of the valves and expose the test piece to the required temperature and humidity conditions. The temperature and relative humidity of the temperature/humidity-controlled chamber are (23 ± 2) °C and (80 ± 2) %, respectively. The recommended retention time is 20 min.

c) Low-humidity environment: place the environmental mini-chamber containing a test piece in the temperature/humidity-controlled chamber. Open both of the valves and expose the test piece to the required temperature and humidity conditions. The temperature and relative humidity of the temperature/humidity-controlled chamber are (23 ± 2) °C and (40 ± 2) %, respectively. The recommended retention time is 20 min.

9 Reflectance measurement procedure

Measure the spectral reflectance within the required wavelength range. For a wavelength range that is shorter than 850 nm, use the following conditions: a wavelength interval of 0,2 nm, a scanning speed below 120 nm/min, a fast response and a light incident angle of 5° to the test piece. For a wavelength range that is longer than 850 nm, use the following conditions: a wavelength interval of 0,2 nm, a scanning speed below 150 nm/min, a fast response and a light incident angle of 5° to the test piece.

a) Measurement position: measure the spectral reflectance at the centre of the test piece. An optical diaphragm is not required if the dimensions of the test piece are larger than the window diameter of the environmental mini-chamber.

b) Measurement of reflectance under a vacuum: measure the reflectance under a vacuum after finishing the evacuation procedure.

c) Measurement of reflectance under a high-humidity environment: measure the reflectance under the required humidity conditions after exposing the test piece to the required conditions in a temperature/humidity-controlled chamber. The recommended temperature and relative humidity are (23 ± 2) °C and (80 ± 2) %, respectively.

d) Measurement of reflectance under a low-humidity environment: measure the reflectance under the required humidity conditions after exposing the test piece to the required conditions in a temperature/humidity-controlled chamber. The recommended temperature and relative humidity are (23 ± 2) °C and (40 ± 2) %, respectively.

10 Evaluations of spectral shifts between dry and humid conditions

10.1 General

Evaluate changes in the spectral reflectance of fine ceramic thin films. The evaluation methods used for several typical types of changes observed in the spectra are shown in this clause. The evaluation method should be optimized to obtain appropriate results for evaluating the film optical properties based on the applications.