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

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

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Introduction

Fine ceramics thin films are used in many applications such as anti-reflective coatings, infrared sensor cut filters, X-ray sensor cut filters, and band-pass filters for medical analysis equipment. Even though spectral transmittance of fine ceramics thin films are specified for each product, the refractive index and optical property change when water vapor or other vapor is adsorbed to the surface of fine ceramics thin films, because, generally, fine ceramics thin films have a columnar and voided structure. For this matter, an International Standard that could evaluate the reliability under a range of humidity was required. This International Standard provides test methods that will easily and accurately evaluate effects of humidity on optical properties of fine ceramic coatings. By establishing this International Standard, it aims for a rapid spread of this test method that will then lead to further growth of the industry.

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

1 Scope

This International Standard specifies a measurement method of spectral transmittance of fine ceramics thin films under humid condition. This International Standard provides test methods that will easily and accurately evaluate changes in optical properties of fine ceramic coatings in a humid atmosphere due to water adsorption.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9211-1, *Optics and photonics — Optical coatings — Definitions*

ISO 8980-3:2013, *Ophthalmic optics — Uncut finished spectacle lenses — Part 3: Transmittance specifications and test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8980-3 and ISO 9211-1, and the following apply.

3.1
half transmittance wavelength
$\lambda_{T1/2}$
center of a segment delimited by the wavelengths at which the transmittance is maximal and minimal in a given wavelength range

3.2
environmental mini-chamber
small chamber to keep the environmental condition of a test piece contained inside the chamber

Note 1 to entry: The chamber is equipped with optical feed-through ports with a ball valve for evacuation or air-circulation. The chamber shall be air-tight to keep the chamber in vacuum or with the atmosphere at a certain humidity by closing the ball valves after evacuation or air-circulating to expose the test piece inside to a certain atmosphere.

3.3
transmittance
fraction of intensity of incident optical beam transferring through the sample

3.4
monochromator
optical device that selects light beam with a certain wavelength with a range

3.5
spectrophotometer
optical instrument that measures spectral transmittance or reflectance

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

3.6
double beam spectrophotometer
type of spectrophotometer that is utilized to compare the light intensity between the reference sample and the test pieces

Note 1 to entry: Compared to single-beam spectrophotometer measures, a high stability of measurements is obtained. The instrument consists of optics that divide a source light beam into two beams and two detectors. The instability of a source beam mainly due to that of the source lamp is compensated by that of a reference beam.

3.7
integrating-sphere
optical component consisting of a hollow spherical cavity with its inner surface covered with a diffuse white reflective coating

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

4 Principle

Fine ceramics thin films sometimes possess a voided micro columnar structure, depending on film deposition conditions. If a fine ceramics thin film with a voided structure is exposed to a certain atmosphere with humidity, moisture in the atmosphere adsorbs on surfaces of the columns consisting the voided structures. The quantity of adsorbed water is affected by hydrostatic vapor pressure in the atmosphere. Depending on the quantity of water adsorbed, the refractive index of thin films changes, resulting in the change in transmittance and reflectance. To evaluate effects of water adsorption on to surface of voids of fine ceramics films on film optical properties, it is necessary to measure optical properties under a certain atmosphere, typically in an atmosphere with a high humidity. In addition to the measurements under a high humidity, for the sake of comparison, it is also necessary to measure the properties in vacuum, i.e. under the condition without water adsorption and low humidity. By comparing optical transmittance measured in vacuum, low humidity, and high humidity conditions, the stability of fine ceramics thin films shall be evaluated.

5 Ambient room environment

The test shall be carried out under an ambient laboratory room atmosphere where deviations in the temperature and humidity are small. In particular, to prevent the condensation of water in the environmental mini-chamber, after removing the mini-chamber from the humidity-temperature control chamber to the laboratory room, the ambient temperature should be controlled to a certain range. Recommended laboratory room conditions are the following:

a) temperature: 23 °C ± 2 °C;

b) relative humidity: below 70 %.

NOTE Attention is needed to dew condensation when environment temperature is low compared to that in the humidity-temperature control chamber.

6 Test piece

The test piece shall comprise a single layer or multilayer of thin films of fine ceramic deposited on to the surface of a plate or film substrate, such as glass, polymer material, with no restriction of material for the substrate if it is enough transparent in a certain range to measure transmittance of thin films. If it fits the environmental mini-chamber, in terms of size and shape, any test piece is acceptable.

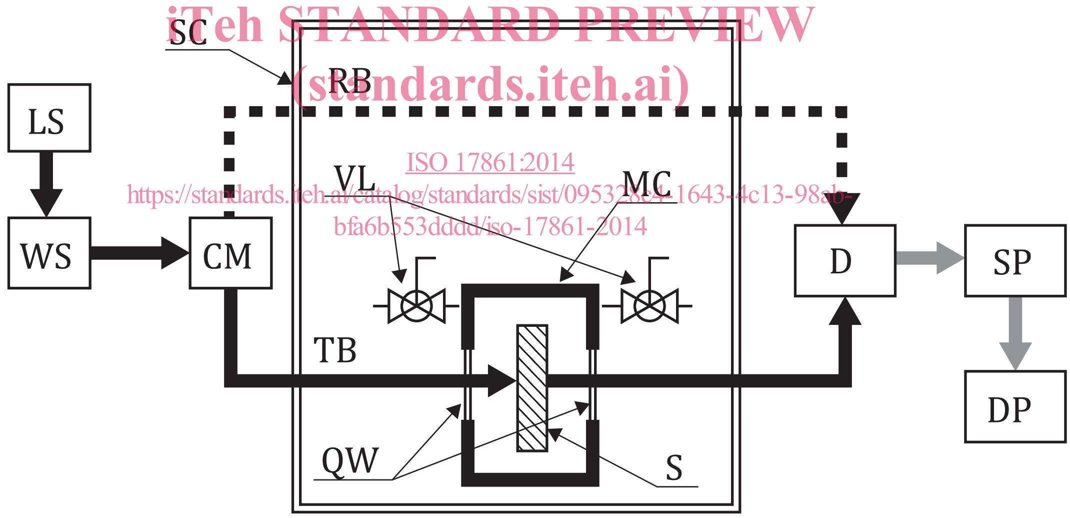
7 Measuring apparatus

7.1 Apparatus configuration

a) Spectrophotometer: Double beam method, wavelength accuracy ±0,2 nm or less at UV-visible light and ±1,0 nm or less at near-infrared range, repeatability of wavelength setting ±0,1 nm or less at UV-visible light and ±0,5 nm or less at near infrared range. For UV-visible and near-infrared range measurements, the range of 300 nm to approximately 2 500 nm is desirable. The use of the integrating sphere for photometer is recommended. In the case of measurements in a range in infrared and far-infrared, the wavelength range can be expanded by using an appropriate spectrometer.

b) Environmental mini-chamber: An airtight container which could maintain the test sample in vacuum and low- and high-humidity environments for the measurements of the spectral of each environment. Build two windows with transparent quartz glass with the sample fixed inside the chamber for the measurement of light transmittance. Also build an air inlet and exhaust ports, each with a valve. It is necessary to confirm that there is no vacuum leak from the chamber when all the valves are closed. In case of necessity, check the leak rate of the chamber by using a vacuum gauge.

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



Key

LS light source
CM chopper mirror
WS wavelength selector (monochrometer)
SC specimen compartment
D detector
SP signal processor
DP data processor
TB test beam
RB reference beam
MC environmental mini-chamber
VL valve
QW quartz window
S specimen

Figure 1 — device configuration for spectral transmittance measurement