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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for optical properties of ceramic phosphors for white light-emitting diodes using a gonio-spectrofluorometer

PROOF/ÉPREUVE

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

White light-emitting diode (LED)-based solid-state lighting (SSL) has been widely used for a variety of applications as an alternative for incandescent and fluorescent lamps. In the beginning, white LEDs (comprising blue LEDs and yellow phosphors) became popular as backlight sources for small-size liquid-crystal displays (LCDs) used in mobile phones and digital cameras. These were followed by white LEDs (consisting of blue LEDs combined with green and red phosphors) applied to backlight sources for large-area LCDs. Subsequently, LED lamps have been commercialized for general lighting, replacing conventional luminaires and capitalising on their advantages, such as compactness, high luminous efficiency, high brightness below 0°C or higher ambient temperatures, long life and controllability of light intensity and colour temperature.

Optical performance of a phosphor material for use in a white LED is one of the most important factors influencing the performance of the white LED. Accordingly, it is of great importance not only for researchers and manufacturers of phosphors for use in white LEDs but also for researchers and manufacturers of white LED devices to evaluate optical properties of the phosphors in a well-established manner. Photoluminescence quantum efficiency is one of the key optical parameters of phosphors for use in white LEDs and has been measured extensively by using an integrating sphere-based absolute method.

ISO 20351 was developed in accordance with the demand for standardizing the test method of internal quantum efficiency of phosphors using an integrating sphere. This standard test method has the advantage of short measurement time and being available to those with no expertise in precise optical measurement. Despite their importance in terms of the performance of ceramic phosphor products, however, external quantum efficiency and absorptance are out of the scope of ISO 20351 due to insufficient understanding of the source of variation in these measurement values.

This document provides the absolute measurement methods of external quantum efficiency and absorptance as well as internal quantum efficiency and related optical properties for ceramic phosphors for use in white LEDs using a gonio-spectrofluorometer. This equipment is regarded as one of the variations of a gonio-reflectometer commonly used to evaluate optical properties of material surfaces.

In this document, measurement conditions and procedures, which can affect the measurement values, are described in detail, helping those who address the high-performance phosphors for competitive SSL products to obtain the proper information on their competitiveness.

This document can also be adopted to phosphors used in non-white LEDs, for example green, orange, pink and purple.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for optical properties of ceramic phosphors for white light-emitting diodes using a gonio-spectrofluorometer

1 Scope

This document specifies a method for use of a gonio-spectrofluorometer to measure internal quantum efficiency, external quantum efficiency, absorptance, luminescent radiance factor and relative fluorescence spectrum of ceramic phosphor powders which are used in white light-emitting diodes (LEDs) and emit visible light when excited by UV or blue light.

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 20351, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Absolute measurement of internal quantum efficiency of phosphors for white light emitting diodes using an integrating sphere*

CIE S 017/E, *International Lighting Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20351 and CIE S 017/E 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
gonio-spectrofluorometer
apparatus measuring the observation angle dependence of the spectral distribution of fluorescent light or scattered light emitted by a sample irradiated on its surface by a monochromatic light

3.2
in-plane
<optical radiation> emitted or reflected, with a propagation vector located in a plane of incidence

3.3
out-of-plane
<optical radiation> emitted or reflected, with a propagation vector not located in a plane of incidence

4 Spherical coordinate system

The coordinate system used in gonio-spectrofluorometry shall be a spherical coordinate system (r, θ, ϕ). In a gonio-spectrofluorometer, the plane including the sample surface shall be taken as the horizontal plane and the centre of the surface of the sample shall be taken as the origin. The radial distance r

of the observation point shall be held constant during the measurement. The geometrical parameters of measurement are defined by the angle of incidence θ_i, the zenith angle of observation θ_r and the azimuth angle of observation ϕ_r. The vertical axis is defined as the direction where $\theta_i = \theta_r = 0°$, and the plane of incidence is defined as $\phi_r = 0°$.

5 Measurement apparatus

5.1 Apparatus configuration

The apparatus comprises elements including a light source unit, a sample unit, a detection unit, a rotational positioning unit, an enclosure and a signal/data processing unit. Figure 1 illustrates the typical measurement apparatus configuration.

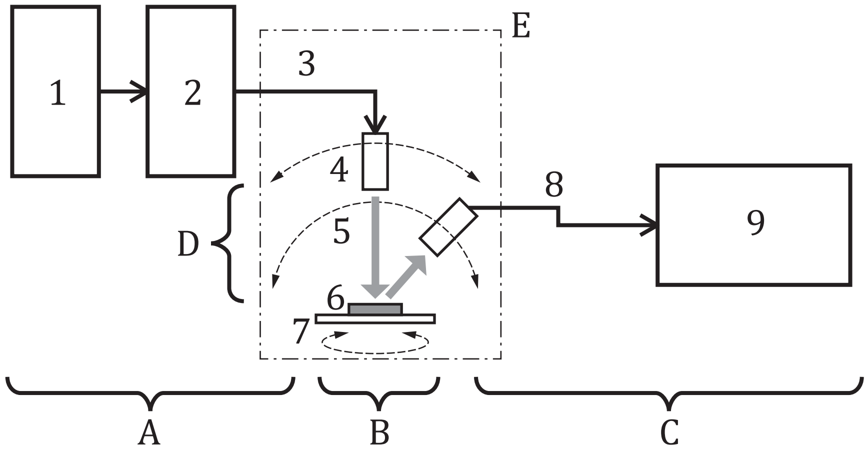
The light source unit generates monochromatic excitation light and comprises a white light source, a power supply for the white light source, a focusing optical system, a wavelength selection unit (monochromator for the white light source) and an optical system for irradiation. A collimated laser beam can also be used as the monochromatic light source.

The sample unit comprises a cell, a diffuse reflectance standard and a sample stage.

The detection unit comprises a directing optical system for collecting light, a spectrometer, a detector and an amplifier.

Example measurement configuration is illustrated in Figure 2, where the geometrical parameters are defined in Clause 4. The rotational positioning unit for measuring in-plane spatial distribution comprises a mechanism for setting the angle of incidence and a mechanism for setting the zenith angle of observation. When out-of-plane spatial distribution is measured, the rotational positioning unit also includes mechanism for setting the azimuth angle of observation. When gonio-spectrofluorometric measurement is performed with a certain fixed angle of incidence, a non-adjustable optical system for irradiating incident beam onto the centre of a sample surface may be used. For measuring in-plane spatial distribution, the incident optical axis is located in the same plane as that where the observation optical axis is rotating by the mechanism for setting the zenith angle of observation. To prevent the mechanism for setting the zenith angle-of-observation from interfering with the mechanism for setting the angle-of-incidence, each can be given radial distance that differs substantially from the other. Alternatively, one or both of these mechanisms can be provided with a supplemental positioning mechanism for preventing collision.

Each component incorporated inside the enclosure should have a matte black surface to reduce stray light.

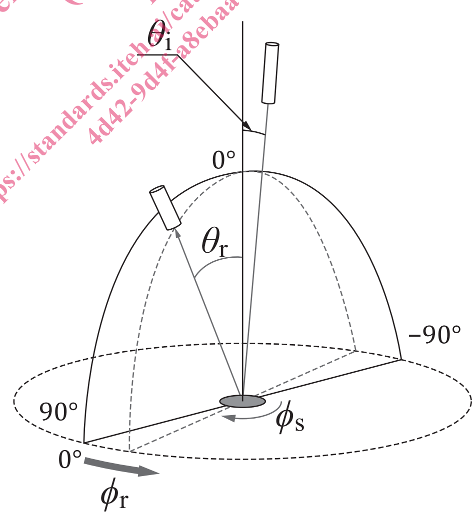


Key

A light source unit
B sample unit
C detection unit
D rotational positioning unit
E enclosure

1 light source
2 spectrometer
3 optical system for irradiation (optical fibre probe)
4 mechanism for setting angle of incidence
5 mechanism for setting zenith angle of observation
6 sample (cell)
7 sample stage
8 directing optical system (optical fibre probe)
9 array spectrometer

Figure 1 — Typical measurement apparatus configuration



Key

θ_r zenith angle of observation
θ_i angle of incidence
ϕ_r azimuth angle of observation
ϕ_s sample rotation angle

Figure 2 — Example configuration of each geometrical parameter

5.2 Light source unit

The spectral width of the excitation light is limited by the spectrometer. The half-width of the excitation light spectrum is preferably 15 nm or less.

The excitation light passes through an optical system for irradiation and irradiates a sample or a diffuse reflectance standard. One example of an optical system for irradiation is an optical fibre probe. One end of the fibre probe is attached to the exit slit of the monochromator and the monochromated light is emitted from the other end. A focusing optics attached to the end of the fibre probe provides a circular, nearly circular or oval-shaped beam of light to illuminate the sample surface.

The optical system for irradiation should be designed to optimize the size of illuminating area on the sample and the diffuse reflectance standard for detecting scattered light and fluorescence efficiently.

5.3 Sample unit

5.3.1 Cell

The area of a sample shall be substantially larger than the area irradiated by the excitation light, and the thickness of a sample in the normal direction shall be at least 2 mm.

A cell shall be made of chemically and physically stable material which does not contaminate the sample inside and can be used in conjunction with a cell adapter provided for a sample stage.

When a less absorptive sample is measured, a cell with a transparent window at its bottom can be used to examine fluorescence or scattered light emitting from the bottom of the cell (see Annex A).

The top surface of the cell shall have a cover glass or a lid to prevent a sample powder from dispersing and contaminating its surroundings during transport or preparation for installation. However, the cover glass or a lid shall be removed during the gonio-spectrofluorometric measurement to expose the sample surface.

5.3.2 Diffuse reflectance standard

A diffuse reflectance standard is used as a reference standard in gonio-spectrofluorometric measurement for calibrating a spectral radiance factor or bi-directional reflectance distribution function (BRDF) in the wavelength range including the excitation light wavelength and the fluorescence light wavelength. The diffuse reflectance standard used shall be an item with a diffuse reflectance of 90 % or greater and a spatial distribution of diffused light close to Lambertian at an incident angle from 0° to 30°, examples of which include a sintered polytetrafluoroethylene material. A secondary diffuse reflectance standard (working standard) can also be used, where the working standard provides calibration for a spectral radiance factor or BRDF using a gonio-spectrofluorometer or another spatial light distribution measurement apparatus, based on a diffuse reflectance standard used to calibrate a spectral radiance factor or BRDF. The working standard used shall be an item with a diffuse reflectance of 90 % or greater and a spatial distribution of diffused light close to Lambertian at an incident angle from 0° to 30°, an example of which is a cell filled with pressed barium sulfate powder.

5.3.3 Sample stage

A sample stage allows a cell to be placed with a cell adapter, if any, at the centre portion of the stage such that the sample surface is always kept horizontal. The sample stage is preferably provided with an automatic or manual mechanism rotating about the vertical axis for setting a sample rotational angle ϕ_s when a surface of a ceramic phosphor sample is not confirmed in advance to be substantially uniform with respect to the sample rotation.

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