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

Céramiques fines (céramiques avancées, céramiques techniques avancées) — Mesurage absolu du rendement quantique interne des luminophores des diodes électroluminescentes blanches en utilisant une sphère intégrante

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Contents

Page

Foreword

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

Introduction

White light-emitting diode (LED) based solid-state lighting (SSL) has been widely used for a variety of applications as alternatives 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 capitalizing 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.

The optical performance of a phosphor 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 the optical properties of the phosphors in a well-established manner. However, standard measurement methods of studying the optical properties of luminescent powder materials commercially used for white LEDs have never been developed.

Photoluminescence quantum efficiency is one of the key parameters of phosphors for use in white LEDs and has been measured extensively by using an integrating sphere-based absolute method. This method was originally developed to determine the photoluminescence quantum efficiency for fluorophore-doped organic thin films and solutions, and has also been applied to phosphor powders. However, those who measure the quantum efficiency of phosphor materials have frequently noted that the measured quantum efficiency may deviate beyond their tolerance level, depending on the measurement equipment, the geometrical configuration of the integrating sphere and the arrangement of the sample cell, even if the measurement procedure is common in principle. This document provides the absolute measurement method of internal quantum efficiency of phosphors for use in white LEDs with reduced deviation of measured values. In this document, measurement equipment and procedures, which can be the sources of the deviation, are described in detail, helping those who address the high performance phosphors for competitive SSL products to obtain the proper information on their competitiveness.

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

1 Scope

This document specifies a method of absolute measurement (using an integrating sphere) of internal quantum efficiency of phosphor powders which are excited by UV or blue light and emit visible light, and which are used for white light-emitting diodes (LEDs).

This document can be adopted for the measurement of phosphors used in non-white LEDs, for example, green, orange, pink or purple LEDs.

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.

CIE S 017/E:2011, *International Lighting Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CIE S 017/E:2011 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
internal quantum efficiency
ratio of the number of photons emitted in free space from the phosphor to the number of excitation light photons absorbed by the phosphor

3.2
cell
container filled with a sample or a white material such as barium sulfate

Note 1 to entry: A cell is typically a flat plate sample holder with a cylindrical hollow, a Petri dish or a rectangular cell used in a spectrophotometer.

3.3
reference cell
cell (3.2) filled with a white powder which has a high spectral diffuse reflectance over the whole visible spectrum (such as barium sulfate or alumina), used when measuring the excitation light spectrum

3.4
white diffuser
white plate which has a high spectral diffuse reflectance over the whole visible spectrum [such as barium sulfate or polytetrafluoroethylene (PTFE)], used when measuring the excitation light spectrum

3.5
secondary absorption
absorption of indirect incident light from every direction of the sphere wall by the phosphor sample

Note 1 to entry: The excitation light illuminating the sample is not entirely absorbed by the sample but is partially scattered or reflected and then repeatedly reflected on the sphere wall. Some of the scattered/reflected light can illuminate the sample again and be absorbed.

3.6
self absorption
absorption of photoluminescent photons emitted by the sample itself

4 Measuring equipment

4.1 Equipment configuration

The equipment comprises a light source unit, a sample unit, a detecting unit, and a signal and data processing unit.

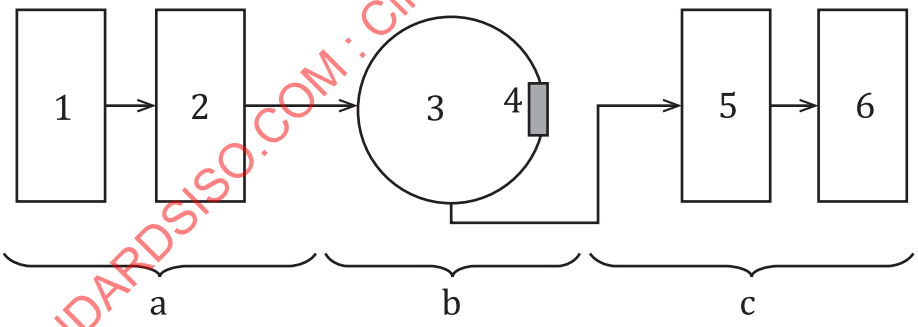
The light source unit generates excitation light and comprises a white light source, a power supply for the light source, a focusing optics and a wavelength selection unit (monochromator for the light source). A laser can also be used as the light source.

The sample unit comprises a cell and an integrating sphere.

The detecting unit comprises a directing optics, a spectrometer, a detector and an amplifier.

The fluorescence spectrophotometer equipped with a sample unit (including an integrating sphere), and the equipment combining a light source unit and an array spectrometer together with the sample unit, are typical examples.

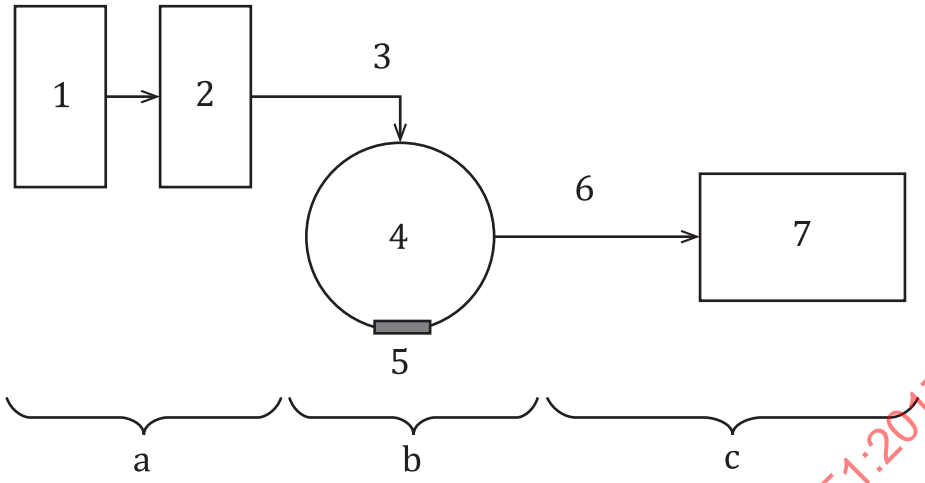
Typical configurations for measuring equipment are shown in Figure 1 and Figure 2.



Key

1	light source	6	detector
2	excitation monochromator	a	light source unit
3	integrating sphere	b	sample unit
4	cell (sample)	c	detecting unit
5	emission monochromator		

Figure 1 — Example configuration of measuring equipment (fluorescence spectrophotometer type)



Key

1	light source	6	optical fibre
2	monochromator	7	array spectrometer
3	optical fibre	a	light source unit
4	integrating sphere	b	sample unit
5	cell (sample)	c	detecting unit

Figure 2 — Example configuration of measuring equipment (array spectrometer type)

4.2 Light source unit

The spectral width of the excitation light is limited by the monochromator. It is desirable that the half-width of the excitation light spectrum is less than 15 nm.

The generated excitation light is introduced into the integrating sphere via the excitation light port to illuminate a cell or a white diffuser. It is important to ensure that the beam diameter of the excitation light illuminating the sample is sufficiently smaller than the diameter of the sample facing the interior of the integrating sphere.

4.3 Sample unit

4.3.1 Cell

Ensure that the area of the sample facing the interior of the integrating sphere is sufficiently larger than that illuminated by the excitation light, and that the thickness of the sample is at least 2 mm.

When using a flat plate sample holder, place a cover glass on the sample. Cover with the lid when using a Petri dish.

Ensure that the cover glass, at least one side of the rectangular cell, or the Petri dish and its lid have sufficient optical transmittance over the entire measured wavelength range. Quartz is generally used for these items. Ensure that the thickness of the cover glass or the transparent side of the rectangular cell is no more than 1,25 mm.

Use a material with high spectral diffuse reflectance (e.g. white alumina) in the flat plate sample holder. When the reference cell is used, the sample cell should be the same type as the cell used for the reference cell.

4.3.2 White diffuser or reference cell

Place a white diffuser or reference cell in the integrating sphere sample mount when measuring the excitation light spectrum.

4.3.3 Integrating sphere

The inner wall is spherical in shape and covered with white diffused reflective material. The integrating sphere incorporates the excitation light port, the measurement port, and the sample port where the cell can be mounted. For the integrating sphere with the sample port aperture, ensure that the total aperture area is less than 10 % of the total area of the integrating sphere wall. For the integrating sphere without the sample port aperture, ensure that the total aperture area in addition to the surface area of the sample is less than 10 % of the total area of the sphere wall. Use a material with sufficiently high spectral diffuse reflectance (e.g. barium sulfate, PTFE) over the entire range of measured wavelengths for the inner wall. The diameter of the integrating sphere shall be between 60 mm and 150 mm.

Note that the presence of light-absorbing material inside the sphere or on the vicinity of the sphere aperture results in measurement errors.

Cells are placed on the inner surface of the integrating sphere or pressed against or inserted into the sample port of the integrating sphere from the outside. The integrating sphere should be designed so that the detector is blind for the direct incidence of reflection, scattering and photoluminescence from the surface of the sample.

It is desirable that the design is such so as to prevent leakage of Fresnel-reflected light in the surface of the cover glass via the aperture to the outside of the integrating sphere when the cell is set in place and illuminated by the excitation light.

4.4 Detecting unit

4.4.1 Directing optics

This optical component guides light collected from the measurement port of the integrating sphere into the spectrometer. The guides need to be sufficiently transparent over the entire measured spectral range. For example, a focusing lens system or an optical fibre bundle can be used.

4.4.2 Spectrometer and detector

This equipment converts light collected from the measurement port of the integrating sphere via directing optics to an electrical signal in proportion to its intensity spectrum. For example, a photomultiplier tube or a CCD detector, with its ample spectral response over the entire measured spectral range, can be used as a detector.

4.4.3 Amplifier

This amplifies the electrical signal from the detector for data processing.

4.5 Signal and data processing unit

This unit separates and processes signals required for measurement, outputs light intensity for each measured wavelength as photon number or energy and saves the data.

5 Calibration, checking and maintenance of measuring equipment

5.1 General

Measuring equipment should be calibrated in a proper manner for accurate optical measurement. In addition, the equipment as well as its accessories should be maintained to keep its condition optimal. The quality control manager should make sure to undertake a regular checking procedure according to the manufacturer's suggestions. Routine factory checking by the manufacturer is also desirable.

5.2 Wavelength calibration of light source unit

When using a monochromated light source, use a monochromator whose wavelength is calibrated with the line source (e.g. a low-pressure mercury lamp) of known wavelength.

When using a laser light source, separately verify its wavelength using a spectrometer or a wavemeter calibrated for wavelength.

5.3 Cells and cover glasses

Handle cells and cover glasses carefully to avoid damage. Replace damaged cells and cover glasses with new items.

5.4 Integrating sphere walls and white diffusers

Verify that there is no contamination, damage or peeling of the walls regularly. If damage or contamination which may interfere with the high diffuse reflection factor in the range of measured wavelengths is found, clean (if possible), recycle or repaint, or replace with a new item.

5.5 Wavelength calibration of detecting unit

Use a spectrometer whose wavelength is calibrated with the line source (e.g. a low-pressure mercury lamp) of known wavelength.

5.6 Spectral responsivity correction

The relative spectral responsivity of the entire measurement system from the integrating sphere to the detector should be calibrated using a spectral irradiance standard light source. All measurement spectra should be corrected based on the relative spectral responsivity calibration results.

6 Samples

6.1 Storage and pre-processing

Phosphor samples shall be stored appropriately according to their properties and pre-processed as necessary. Samples are normally stored at room temperature in a desiccator. However, samples which react with moisture in the air, or which may be degraded with UV or visible light, shall be stored in a sealed container (filled with an inert gas using a glove box) or a coloured bottle.

Samples which readily absorb moisture shall be dried before measurement in a vacuum dry oven at a temperature at which they do not deteriorate.

6.2 Filling cells with samples

When using a flat plate sample holder, overfill its hollow with an excessive amount of sample, press it down with the flat plate, scrape off the excess and place the cover glass over the top. When using a rectangular cell, place the powder sample in the cell and tap it to ensure that it is densely packed, and

cover with the lid if necessary. When using a Petri dish, place the powder in the dish and smooth its surface by tapping it, for example, and cover with the lid.

When the cell is mounted outside the integrating sphere, ensure that the thickness of the sample layer is sufficient to prevent leakage of excitation light. When using a Petri dish, ensure that the amount of the sample is sufficient for the results obtained to be invariant with respect to its amount.

7 Measurement methods

7.1 Measurement environment

Locate the measuring equipment in a room temperature environment, out of direct sunlight and not subject to sudden temperature changes. Samples are handled and measured in stable ambient conditions with a room temperature of 10 °C to 30 °C and a relative humidity of 20 % to 80 %. For samples that are hygroscopic or that have poor durability, an appropriate measurement environment should be prepared according to their properties, and measurement shall be conducted in as short a time as possible. Turn on the measuring equipment at least 30 min prior to the measurement.

7.2 Light spectrum without phosphor sample

Place the white diffuser or the reference cell on the sample port of the integrating sphere. When using the white diffuser, place the same cover glass as used for the sample on the diffuser. When using a Petri dish as a cell, place the dish with its lid closed on the cell mount of the integrating sphere. The cell type of the reference cell, filled with white powder as described in 6.2, should be the same as that of the phosphor sample.

Introduce the monochromated excitation light through the aperture at the excitation light port of the sphere to illuminate the white diffuser or the reference cell, and measure the intensity of the light for each wavelength at the measurement port to find the excitation light spectrum (see Figure 3). The intensity of the excitation light should be controlled so as not to saturate the detector signal and deteriorate the sample, as noted in 7.3.

7.3 Light spectrum with phosphor sample

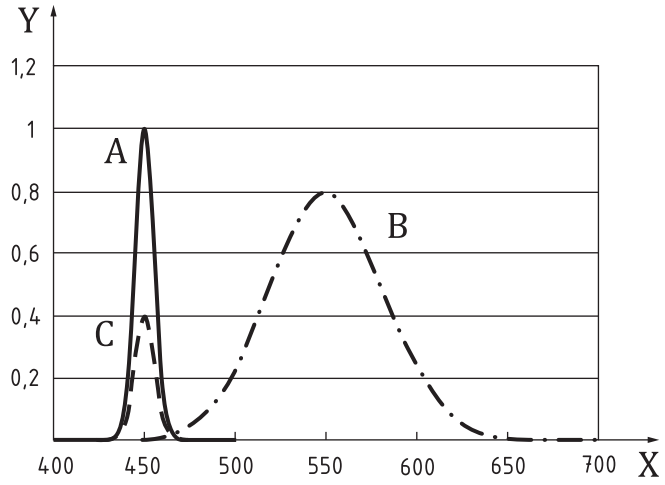
Attach the cell filled with the sample onto the sample port of the integrating sphere, or mount the cell on the cell mount inside the integrating sphere.

As described in 7.2, illuminate the sample with excitation light then measure the light intensity with the detector to obtain the spectrum with excitation light and photoluminescence components.

8 Calculations

8.1 Conversion to photon-number-based spectra

Convert the spectra measured as described in 7.2 and 7.3 to photon-number-based spectra if they are energy-based spectra.



Key

X wavelength (nm)
Y photon flux (arbitrary units)
A excitation light spectrum obtained in 7.2
B photoluminescence spectrum obtained in 8.2
C excitation light spectrum obtained in 8.3.1

Figure 3 — Excitation light and photoluminescence spectra obtained in Clause 7 and Clause 8

8.2 Photoluminescence spectrum

8.2.1 General

To obtain the photoluminescence spectrum of the phosphor sample separately as shown in Figure 3, use the excitation light spectrum obtained in 7.2 to remove the excitation light spectrum component from the spectrum measured in 7.3. The excitation light component can be removed, for example, as follows.

8.2.2 Method 1

Apply a suitable scale factor to the excitation light spectrum obtained in 7.2, and subtract the resulting spectrum from that obtained in 7.3 so as to obtain a smooth subtracted spectrum at the vicinity of the excitation light. Irregularities in the vicinity of the excitation wavelength can be removed, for example, by linear fitting of data points adjacently outside of the spectral area of irregularities.

8.2.3 Method 2

Approximate the baseline in the vicinity of the excitation light as a linear plot.

8.3 Internal quantum efficiency

8.3.1 Relative number of absorbed photons

Extract the excitation light component (see Figure 3) of the measured spectrum in 7.3 by subtracting the photoluminescence component extracted in 8.2 from the measured spectrum. Then, obtain the difference spectrum by subtracting the extracted excitation light spectrum from the measured excitation light spectrum in 7.2 divided by the diffuse reflectance in the excitation wavelength region of the white diffuser or reference cell, and integrate it at the spectral region of excitation light to obtain the relative number of absorbed photons for the phosphor sample.

8.3.2 Relative number of photoluminescent photons

Find the relative number of the photoluminescent photons emitted from the sample from the photoluminescence spectral component obtained in 8.2.

8.3.3 Internal quantum efficiency

Calculate internal quantum efficiency using Formula (1):

$$\eta = F \times 100/A \quad (1)$$

where

η is the internal quantum efficiency (%);

F is the number of photoluminescent photons;

A is the number of absorbed photons.

Corrections such as secondary absorption correction[1] and self-absorption correction[2] may be adopted, if required, in accordance with agreements between involved personnel.

9 Report

Report the following measurement results:

a) a reference to this document, i.e. ISO 20351;

b) date of measurement and measurement personnel;

c) sample name;

d) measurement equipment;

e) wavelength of excitation light and its spectral full width at half maximum;

f) measurement spectral range;

g) internal quantum efficiency and spectral range used in calculation;

h) corrections (e.g. secondary absorption correction, self-absorption correction) used (Y/N);

i) ambient conditions;

Report the following as necessary:

j) type of light source;

k) type of detector;

l) dimensions and material of cell, thickness of sample, angle of incidence of excitation light;

m) internal diameter and material of integrating sphere;

n) material of white diffuser or powder for reference cell;

o) spectral diffuse reflectance of white diffuser or reference cell;

p) excitation light spectrum (indicate clearly whether energy-based or photon number-based;

q) measured spectrum of phosphor sample (see 7.3) (as above);