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Fine ceramics (advanced ceramics, advanced technical ceramics) — Ultraviolet photoluminescence image test method for analysing polytypes of boron- and nitrogen-doped SiC crystals

Céramiques techniques — Méthode d'imagerie de photoluminescence ultraviolette pour l'analyse des polytypes dans les cristaux de SiC dopés à l'azote et au bore

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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

Silicon carbide (SiC), which has a close-packed crystal structure, is a promising wide-bandgap (WBG) material applicable to laser diodes (LDs), light-emitting diodes (LEDs) and electronic power devices.

Polytype inclusion generated during SiC growth is a common problem. During crystal growth, many types of SiC-stacking can occur within the bulk of a single sample. These different stacking-order types are called “polytypes.” Polytypes have identical close-packed planes but differ in the stacking sequence on the axis that is perpendicular to these planes.

SiC has more than 200 known polytypes, but most polytypes are rare, except types 2H, 4H, 6H, 15R and 3C. For example, 4H-SiC is the material used for power production in devices because of its excellent physical properties. These SiC polytypes have the same density and Gibbs-free energy but different electronic band structures. The different band structures cause different wavelengths of luminescence induced by incident ultraviolet (UV) light.

SiC can be grown using several crystal-growth techniques, such as physical vapor transport (PVT), chemical vapor deposition (CVD) and top-seeded solution growth (TSSG). Polytype inclusion in bulk SiC is one of the drawbacks during production.

Therefore, a rapid test method to discriminate between polytypes would be useful for the development and mass production of SiC crystals.

This document specifies a test method to evaluate the polytypes and their SiC ratios by UV-induced photoluminescence using non-contact and full-field measurement techniques.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Ultraviolet photoluminescence image test method for analysing polytypes of boron- and nitrogen-doped SiC crystals

1 Scope

This document specifies a test method for determining the polytypes and their ratios in silicon carbide (SiC) wafers or bulk crystals using ultraviolet photoluminescence (UVPL) imaging. The range of SiC is limited to semiconductor SiC doped with nitrogen and boron to have a deep acceptor level and a shallow donor level, respectively. The SiC wafers or bulk crystals discussed in this document typically show electrical resistivities ranging from 10^{-3} ohm · cm to 10^{-2} ohm · cm, applicable to power electronic devices.

This method is applicable to the SiC-crystal 4H, 6H and 15R polytypes that contain boron and nitrogen as acceptor and donor, respectively, at concentrations that produce donor-acceptor pairs (DAPs) to generate UVPL. In 4H-SiC the boron and nitrogen concentrations typically range from 10^{16} cm^{-3} to 10^{18} cm^{-3}. Semi-insulating SiC is not of concern because it usually contains minimal boron and nitrogen; therefore deep level cannot be achieved.

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/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
ultraviolet photoluminescence
UVPL
wavelength shifting to a longer wavelength by the interaction of photons with matter

3.2
donor-acceptor pair
DAP
state of a solid in which an electron-hole is created when a photon or other energy is absorbed

3.3
DAP recombination energy
photon energy emitted by the recombination of the *donor-acceptor pair* (3.2)

3.4
ultraviolet photoluminescence image test method
method to identify the polytypes and their ratios using the *donor-acceptor pair recombination energy* (3.3) of materials as the *ultraviolet photoluminescence* (3.1) colour

4 Symbols

Symbol	Designation	Unit
C	velocity of light	299 792 458 m/s
E	bandgap energy (or DAP recombination energy)	eV
H	Planck's constant	$6{,}626\ 070\ 04 \times 10^{-34}$ J·s
I	intensity of the light source	W/cm^2
T	temperature	K
λ	wavelength	nm
ρ	resistivity	Ω.cm
ω	angular velocity	radian/s

5 Principle

When UV light illuminates a material having DAP recombination energy in the visible-light range, the material will emit a specific colour depending on the DAP recombination energy. Thus, the polytypes of SiC can be identified by their luminescence colour because different SiC polytypes have their own DAP recombination energy. The polytype area in CIE 1931 colour space is mapped and transformed from the UVPL image; subsequently, the polytype ratios are calculated.

6 Apparatus

6.1 Stage. The experiment should be performed in a non-vibrational state or on an anti-vibration table to prevent noise caused by vibration.

6.2 Digital camera. This should be a charge-coupled device (CCD) camera with an ISO rating of (500 ± 100), (f/#) of f/1,8–f/3,2 and zero exposure compensation. The shutter speed should be 1/30 to 1/50 and the white balance should be 6 500 K. These are the standard conditions for the UVPL image test, but they can be varied.

The purpose and function of a CCD camera is to produce an image similar to the object recognized by the human eye. This is achieved by synthesizing the image consisting of the artificial fine shapes and colours. It should be noted that the CCD camera is neither a spectra analyser nor a spectra recorder.

Before evaluating the polytype, the CCD camera should be calibrated. The UVPL image captured by the CCD camera should be significantly expanded and carefully checked. If any artificial pattern is observed, the CCD camera should be replaced with another.

6.3 Lens. This is used to focus the incident UV beam onto the specimens. The position and focal length of the lens should be selected for the beam to spread over every sample with uniform intensity.

6.4 Light source. The wavelength (λ) of the UV light source (UVLS) should be 180 nm to 365 nm in the nonvisible range and monochromatic range. The intensity of the light source should be 600 mW/cm^2 or above. The working distance between the sample and the light source should be approximately (10~30) cm. In reflectance mode, adjust the angle (0~360)° between the direction of the light source and the direction normal to the sample to prevent the UV source from being incident directly on the CCD camera and measure only the luminescence beam. In transmission mode, the luminescence beam produced by the sample should be measured using the CCD camera after having been transmitted through the sample.

6.5 Filter. UVLSs can emit a visible beam as well as a UV beam. If the visible beam is incident on the CCD camera, it can disturb the data measurements. Therefore, a bandpass filter that passes only UV beams should be positioned in front of the UVLS.

7 Sampling

7.1 The UVPL technique can be applied to SiC samples grown by various techniques. The necessary condition is to have boron and nitrogen dopant at concentrations that are sufficient to emit the luminescence of the DA pair at an effectively high intensity.

7.2 The flat surface of a sample should be polished finely to improve the measurement accuracy by preventing the UV beam from being scattered for the beam to remain incident directly on the digital camera.

7.3 Should there be any treatment (e.g. polishing) before the test, it shall be recorded in the test report.

8 Procedure

8.1 Cleaning of SiC wafer surface

The samples should be handled in a clean environment, such as a clean-booth or clean-room. The clean surface of a wafer should be maintained. Wiping with a soft tissue soaked with a volatile solvent, such as acetone or ethanol, can render the sample surface free of foreign contaminations, such as dust, other particles, metals, organic compounds, inorganic compounds or moisture. However, if the surface contamination remains and thereby influences the measurements, the sample surface should be wet cleaned in a clean environment, using ultra-high-purity water and chemicals, such as H_2SO_4: H_2O_2: H_2O, NH_4OH: H_2O_2: H_2O, HCl: H_2O_2: H_2O or other suitable chemicals.

Figure 1 shows examples of polytype shapes. Typically, they have clear and sharp lines revealing the grain boundaries.

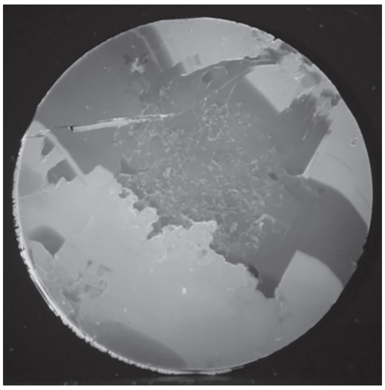


Figure 1 — Crystal grains in UVPL image (clear lines of grain boundary expressing crystal shapes)

8.2 Optical setup

8.2.1 The optical setup should be constructed as shown in Figure 2 a).

8.2.2 The experiment should be performed in a darkroom or with a blackout curtain to prevent extraneous light, which can disturb the UVPL beam.

8.2.3 The experiment should be performed in a non-vibrational state or on an anti-vibration table to prevent noise caused by vibration.

8.2.4 Beam expanders (BEs) should be positioned adjacent to the UVLS to expand the beam.

8.2.5 Optical bandpass filters (BF_1) are positioned after the BE to filter light except in the UV range. The transmittance distribution along the wavelength of BF_1 when it passes only the intended UV light is shown in Figure 2 b).

8.2.6 Support for the sample (SiC) should be positioned such that the UV beam is not incident directly on the CCD camera.

8.2.7 The normal axes of the CCD camera and the SiC sample should coincide to maximize the UVPL intensity.

8.2.8 The photoluminescent beam emitted from the SiC sample incident on the CCD camera should be filtered by a bandpass filter (BF_2) to filter the reflected or scattered UV beam from the SiC sample for only PL light to be incident on the CCD camera. The transmittance distribution along the wavelength when it filters UV-range light is shown in Figure 2 c).

8.2.9 The image taken by the CCD camera should be transformed into colour space.

8.3 Measurement

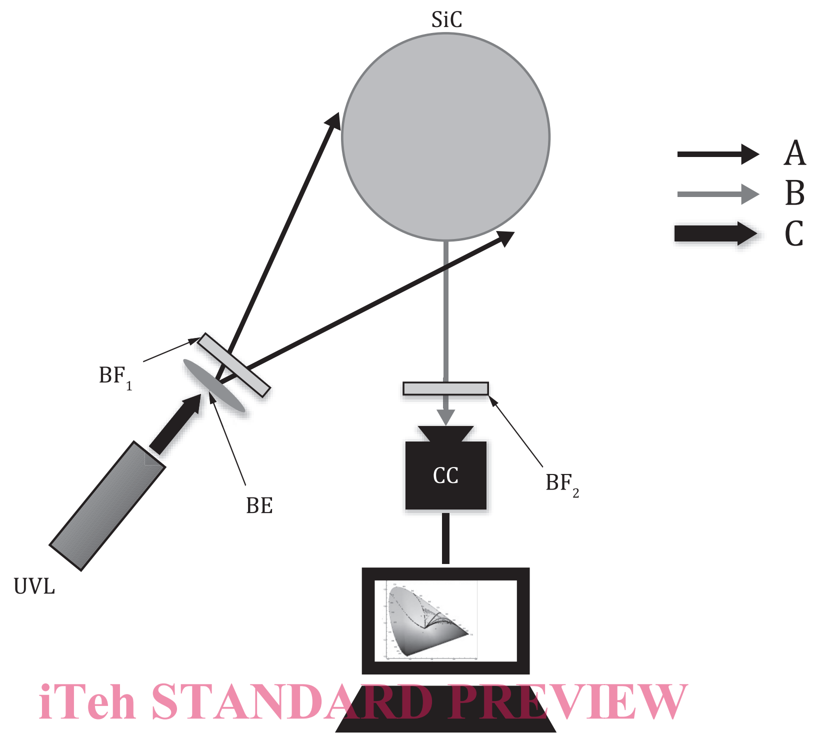
8.3.1 Calibration is necessary before measurement. See Annex D.

8.3.2 The UV beam emitted by the UVLS passing through BE and BF_1 then irradiates the SiC sample.

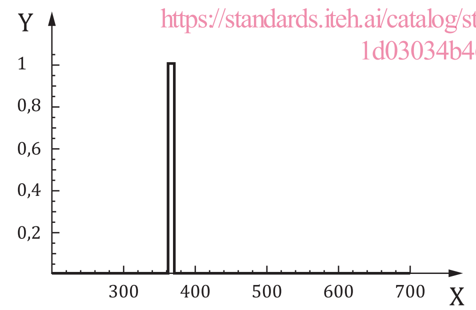
8.3.3 Take pictures of the UVPL image using the CCD camera and save the images in RGB format.

8.3.4 Transform the RGB images to the CIE 1931 colour space.

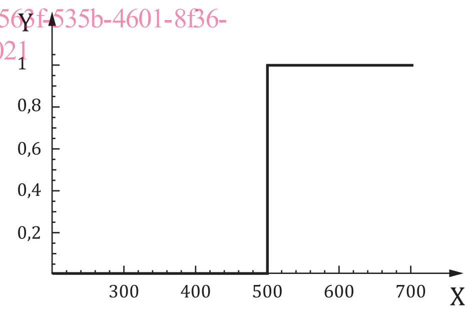
8.3.5 The polytype can be determined by the position of the colour point when it is plotted on the colour space.



a) Schematic of a UVPL imaging system



b) Transmittance distribution of BF_1



c) Transmittance distribution of BF_2

Key

A	expanded UV beam
B	UV induced PL beam
C	UV LED
UVL	ultraviolet light source
BF	bandpass filter
BE	beam expander
SiC	silicon carbide
CC	CCD camera
DAQ	data acquisition
X	wavelength, nm
Y	transmittance

Figure 2 — Optical setup for UVPL measurement

9 Expression of results

9.1 Expression of boundaries of polytypes in CIE 1931 colour space

The polytype boundaries are defined in the CIE 1931 colour space as shown in Figure 3. The dotted lines are the polytype lines. When the temperature of a SiC sample is near 0 K, the transformed points from the UVPL image are positioned near the edge of the colour space; as the temperature increases, the colour points approach the centre of the colour space (1/3,1/3,1/3). The black lines in Figure 3 represent the boundary lines, which are between polytype lines and help to determine the major polytypes.

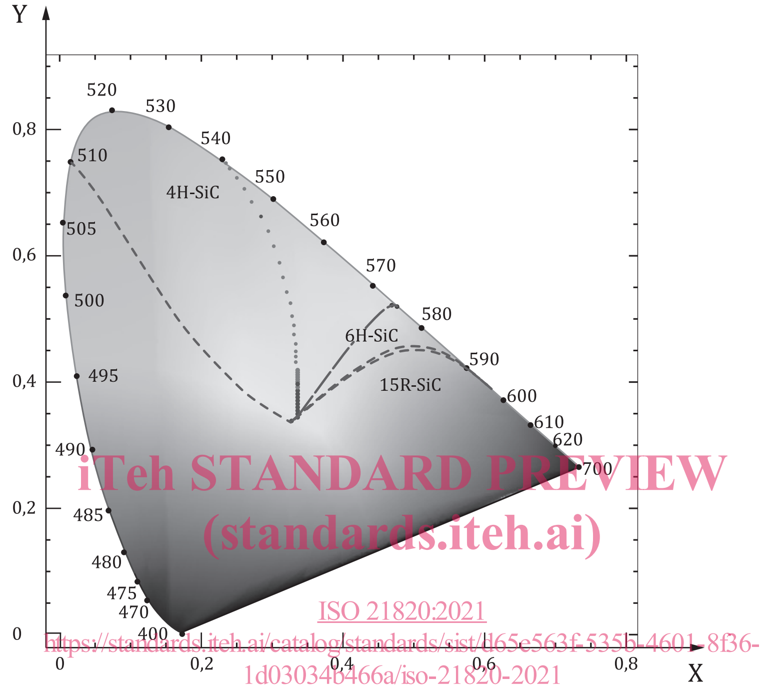


Figure 3 — Polytype lines (dotted lines) and boundary lines (dashed lines) of polytypes in CIE 1913 colour space

The detailed procedure for producing the polytype lines and boundaries is described in Annex A.

Annex B shows the boundary points table of each polytype in the CIE 1931 colour space. The specific polytype regions are defined as the inner regions of polygons made by connecting these boundary points, as shown in Figures B.1 to B.3.

9.2 Transformation of the UVPL image to CIE 1931 colour space with polytype boundaries

The UVPL colour data should be transformed to the CIE 1931 colour space using the polytype boundaries to identify the polytype.

Each polytype has a different DAP recombination energy, which indicates that each polytype has a different colour. Therefore, when a UVPL image is transformed into the CIE 1931 colour space, each polytype colour has a different position in the colour space.