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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for crystalline quality of single-crystal thin film (wafer) using XRD method with parallel X-ray beam

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Contents

Page

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

Single crystals are important in many applications ranging from synthetic gemstones for jewellery to hosts for solid-state lasers. For some applications, ceramic materials are prepared as single crystals. When used as substrates for thin film growth (such as gallium-on-sapphire technology or the growth of superconductor thin films) it is the crystalline perfection of a single crystal that is important. Wide bandgap semiconductors such as silicon carbide (SiC) and gallium nitride (GaN) have drawn a lot of attention in power applications due to their superior material properties such as high critical electric field resulting in a minimum of 10 times higher breakdown voltage or a 100 times smaller on-resistance than Si. These unique properties of SiC and GaN materials have made them promising candidates for future high-power, high-frequency semiconductor devices. In optical applications, such as the use of ruby and yttrium–aluminium–garnet (YAG) for laser hosts and quartz and sapphire for optical windows, single crystals are used to minimize scattering or absorption of energy. In piezoelectric materials, such as quartz, the optimum properties are obtained in single-domain single crystals. In addition, there are many other applications that require the optical, electrical, magnetic or mechanical properties of ceramic single crystals.

Substrate diameters for the single crystal have been steadily increasing since the commercial introduction of substrates in 1990 and crystal defects have been greatly reduced in the past 15 years. Commercial devices are available, but their widespread use will depend on the ability of growers to make large, inexpensive, defect-free materials available.

While various methods for measuring the defect of single-crystal thin films have been presented until now, the most typical method for measuring the crystalline quality (degree of average defect) of single-crystal thin films that have a wide area (e.g. 2 inches, 4 inches, 6 inches) is the X-ray diffraction (XRD) method with parallel X-ray beam. However, this method can easily create a great error margin as the result value is analysed to be very different depending on the measuring process and conditions of the user or the pre-treatment of samples, for example. A standard on universal measurement methods and conditions, therefore, is absolutely necessary.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for crystalline quality of single-crystal thin film (wafer) using XRD method with parallel X-ray beam

1 Scope

This document specifies the test method for measuring the crystalline quality of single-crystal thin film (wafer) using the XRD method with parallel X-ray beam. This document is applicable to all of the single-crystal thin film (wafer) as bulk or epitaxial layer structure.

2 Normative references

There are no normative references in this document.

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
single crystal
crystalline material having identical atomic arrangement on all areas of the material

3.2
off-cut angle
angle that a specific crystallographic orientation forms with surface in a single-crystal thin film (wafer)

Note 1 to entry: Off-cut angle is a key condition determining the growth behaviour of thin film during epitaxial growth on a single-crystal thin film (wafer).

3.3
chemical mechanical polishing
CMP
process to planarize the thin film surface using a combination of chemical action by a slurry composed of chemical liquid or abrasive particles and the mechanical action of a grinder

3.4
Bragg diffraction
width between the wavelength of light and the width of crystal structure, or relationship between the reflecting surface and the angle formed by the ray

Note 1 to entry: The formula is $2d \cdot \sin\theta = n \cdot \lambda$

where

d is the width of periodic structure;

θ is the angle between the crystal plane and incident light;

λ is the wavelength of light;

n is the constant.

3.5
parallel X-ray beam
X-ray beam obtained by collimating an incident X-ray beam or diffracted X-ray beam by using a solar slit, an analyser crystal or an x-ray mirror

Note 1 to entry: In comparison with the focused beam, the parallel X-ray beam does not suffer the sample condition (such as surface roughness) and geometrical limitations of the optical system (such as mechanical focal-circle deviation).

3.6
slit
device for controlling the size and photon flux amount of X-ray beam

3.7
symmetric diffraction
state where the surface of sample and the Bragg diffraction are parallel

3.8
asymmetric diffraction
state where the surface of sample and the Bragg diffraction are not parallel

3.9
2 theta
2*θ*
angle of the detected X-ray beam with respect to the incident X-ray beam direction

3.10
omega
ω
angle between the incident X-ray beam and the sample surface

3.11
chi
χ
angle of tilt of sample about an axis in the plane of the sample and in the plane of the incident X-ray beam, X-ray source and detector

Note 1 to entry: It can also be defined as psi (*ψ*) depending on the equipment manufacturer.

3.12
phi
Φ
angle of rotation about the normal to the nominal surface of the sample

3.13
X, Y, Z coordinate system
orthogonal coordinate system in which X is the direction in the plane of the sample, parallel to the incident beam when $\Phi = 0$; Y is the direction in the plane of the sample, perpendicular to the incident beam when $\Phi = 0$; and Z is the direction normal to the plane of the sample

3.14
flat zone
flat section in order to distinguish the structure of thin film (wafer)

Note 1 to entry: Since crystal structure in the thin film (wafer) cannot be visually identified with the human eye, the location is classified by making one section flat.

3.15
rocking curve
RC
ω rocking
ω scan
intensity of peak and change of FWHM (full width at half maximum) on the incident angle ω as the optimum Bragg diffraction condition on a specific crystal plane of a single crystal

Note 1 to entry: It is normally an indicator showing crystalline quality of the sample.

3.16
crystalline quality
FWHM value of a rocking curve based on various degrees of defects (such as dislocation density, mosaic spread, curvature, misorientation and inhomogeneity) in the sample

3.17
arc second
FWHM unit of rocking curve as 1/3 600 of a degree of an angle

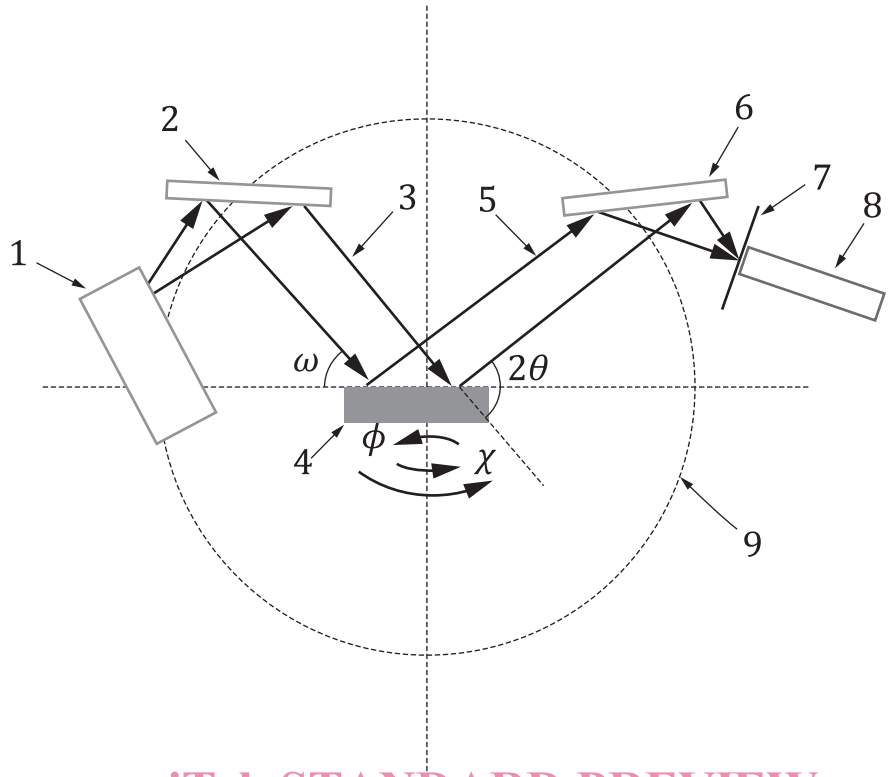
4 Fundamentals

The purpose of this document is to provide information to minimize the measuring error on the crystalline quality evaluation of a single crystal. An X-ray beam shall be investigated in order to satisfy the Bragg diffraction conditions on a single-crystal thin film (wafer) that has grown into a specific crystal plane. Information on the internal defects of single crystal (such as dislocation density, mosaic spread, curvature, misorientation and inhomogeneity) can be gained using the microscopic angle change of X-ray beam (or microscopic change in the position of sample) investigated at this time. Since the crystal plane interval and the arrangement of the crystal plane are very consistent in an almost perfect crystal, most diffraction conditions are gained from one angle (2θ) in all crystal planes so that a very sharp peak can be gained. In contrast, the single crystals with more defects show a broader peak.

5 Devices and instruments

5.1 Schematic diagrams

Figure 1 shows an example configuration for XRD with parallel X-ray beam system.



Key

1 X-ray source
2 X-ray mirror
3 incident X-ray beam
4 sample
5 diffracted X-ray beam
6 X-ray mirror
7 slit
8 detector
9 roland circle
2θ angle between the detected beam and the extension of the incident X-ray beam
ω angle between the specimen surface and the incident X-ray beam 2θ angle between the detected beam and the extension of the incident X-ray beam
ϕ angle of rotation about the normal to the nominal surface of the sample
χ angle of tilt of sample about an axis in the plane of the sample and in the plane of the incident X-ray beam, X-ray source and detector

Figure 1 — Example schematic layout of an XRD experimental configuration with parallel X-ray beam system, projected into the plane of the source, detector, incident and diffracted X-ray beams

5.2 X-ray generator

Device which generates an X-ray beam of fixed intensity.

5.3 X-ray mirror

Device which makes the dispersed beam generated from an X-ray generator parallel or one that can control the amount of X-ray beam that reaches the monochromator by passing through the slit.

NOTE Since the X-ray mirror is related only to the intensity of the peak (regardless of the beam's resolution), it might not be used.

5.4 Monochromator

Device which monochromatizes the parallel beam generated from the X-ray mirror to have a specific resolution. Selecting an appropriate monochromator is critical in the XRD analysis. In cases where the crystalline quality [FWHM of rocking curve (RC)] of the sample is similar to the resolution of a monochromator, the monochromator shall be replaced with a new one having higher resolution for measurement purposes. This is because the resolution of the monochromator is likely to be lower than the crystalline quality (FWHM of RC) of the single crystal.

NOTE X-rays (coming from the X-ray tube) have various wavelengths such as K_{a1}, K_{a2} and K_{β}. For instance, if the target is Cu, Cu K_{a1} (λ = 0,154 056 nm), Cu K_{a2} (λ = 0,154 439 nm) and Cu K_{β} (λ = 0,139 221 nm) are emitted in the ratio of 10:5:2. Because of a large gap between the wavelength of K_{β} ray and the rest ($K_{a1,}$ K_{a2}), K_{β} ray can be easily filtered using a thin Ni filter. However, as the wavelengths of K_{a1} and K_{a2} are quite similar, they cannot be filtered through a general Ni filter. Likewise, with the incident X-rays on a specimen having diverse wavelengths, a diffraction peak broadening occurs, disrupting interpretation of the diffraction peak. This causes a problem in measuring the accurate angle of diffraction (2θ). For this reason, a monochromator that takes only K_{a1} ray from incident X-rays to make a single wavelength must be used for accurate diffraction tests with high resolution.

5.5 Sample attachment

Plate where the measured sample gets placed to become parallel.

5.6 Goniometer

Device designed for the sample to move in the x-axis, y-axis and z-axis (X, Y, Z coordinate system), in Φ and χ directions. A mechanically well-aligned and stable X-ray goniometer is required. The sample height (z-axis) shall be capable of being set accurately on the centre of rotation of the ω and 2θ axes, and the sample stage angle of tilt (χ) shall enable setting the sample parallel to the incident beam slits.

NOTE A goniometer can have different moving fundamentals and structure depending on the equipment manufacturer.

5.7 Detector

Device which changes into a form of peak on the software by receiving the X-ray beam that has passed through the slit of the analyser crystal. The detector response shall be stable within the time frame of the experiment. It is usual and recommended that the acceptance slits at the detector be set to match the incident beam width and divergence.

NOTE An analyser crystal works only for zero-dimensional detector (scintillation detector). A multidimensional X-ray detector can drastically reduce total measurement time and can observe detailed information of the sample very quickly. Moreover, the X-ray sensitivity and angular resolution of modern multidimensional X-ray detector devices are comparable to scintillation systems.

5.8 Instrument calibration

The aligned state of devices mentioned in these subclauses is critical in the single-crystal thin film (wafer) analysis. As even a minor misalignment makes it different to get a precise FWHM of the RC, calibration on a regular basis shall be conducted in accordance with the procedures and methods in the manual provided by the XRD manufacturer.

6 Preparation of sample

Process the single crystal under a state of ingot into a form of wafer with a specific size by cutting with a multi-wire saw. Polish using a chemical mechanical polishing (CMP) for the planarized surface of processed single-crystal thin film (wafer).

7 Test method and procedure

7.1 Optics alignment

The X-ray generated from the X-ray generator shall be made to reach the detector by passing through the monochromator after going through the X-ray mirror as shown in Figure 2. Alignment checks might be part of automated (or manual mode) routines available on particular equipment. The following basic requirements shall be met:

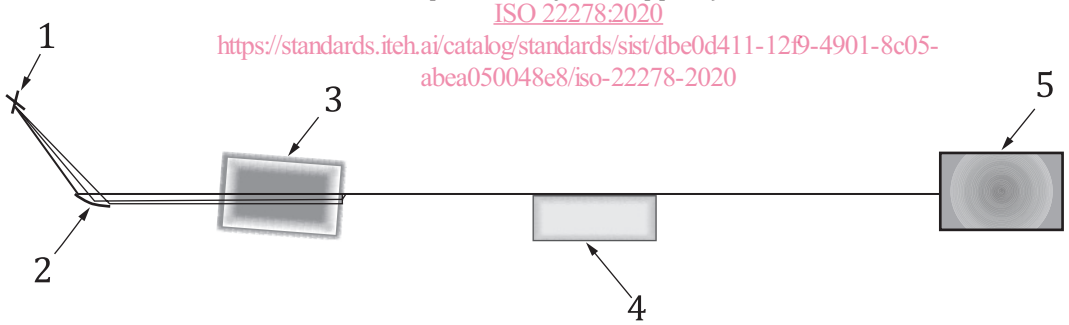
a) Make sure that nothing unwanted obstructs the beam between the source and detector. The sample attachment shall be out of the beam.

b) The incident X-ray beam shall be accurately centred on the centre of the sample attachment and detector axes.

c) The incident X-ray beam shall be made to become parallel with the surface of the sample attachment.

NOTE 1 It is possible with modern control software that corrections to axes motions can take into account a non-ideal instrument alignment.

NOTE 2 To perform accurate evaluation on a high-quality single-crystal thin film (wafer), the resolution of the monochromator must not be lower than the crystalline quality of the single crystal sample (refer to the resolution data of the monochromator provided by the supplier).



Key

1 X-ray generator
2 X-ray mirror
3 monochromator
4 sample attachment
5 detector

Figure 2 — Schematic diagram of optics alignment

7.2 Sample alignment

After placing the single-crystal thin film (wafer) to be measured on the sample attachment above the sample plate, make the incident X-ray beam become parallel with the sample surface as shown in Figure 3. The sample surface also coincides with the centre of rotation of the goniometer axes. Equipment and its controls can include automatic sample alignment, data collection and analysis

