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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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Foreword

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

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Introduction

Single-crystals are important in many applications ranging from synthetic gemstones for jewelry to hosts for solid-state lasers. For some applications, ceramic materials must be prepared as single-crystals. When used as substrates for thin film growth (e.g. 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, e.g. the use of ruby and yttrium–aluminum–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, e.g. quartz, the optimum properties are obtained in single-domain single-crystals. Some of the applications that utilize the desirable 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 wide area (2 inch, 4 inch and 6 inch, etc.) is 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 sample, etc.

Therefore, a standard on universal measurement methods and conditions is absolutely necessary.

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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

1 Scope

This standard is the one for measuring the crystalline quality of single-crystal thin film (wafer) XRD method with parallel X-ray beam. All of the single-crystal thin film (wafer) as bulk or epitaxial layer structure is included in the scope of this standard.

2 Normative references

There are no normative references in this document.

3 Terms, definitions, symbols and abbreviated terms

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 http://www.iso.org/obp

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

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3.1 Terms and definitions

3.1.1
Single-crystal
While all atoms that exist in one crystal form a fixed regular arrangement in a three dimensional space, a single-crystal at this time stands for a crystalline material having identical atomic arrangement on all areas of the material

3.1.2
Off-cut angle
Angle that a specific crystallographic orientation forms with surface in a single-crystal thin film (wafer). Off-cut angle is a key condition determining the growth behaviour of thin film during epitaxial growth on a single-crystal thin film (wafer)

3.1.3
CMP (chemical mechanical polishing)
A process to planarize the thin film surface using the combination of chemical action by a slurry composed of chemical liquid / abrasive particles and mechanical action of a grinder

3.1.4
Bragg diffraction
Shows the 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. The formula is $2d \cdot \sin\theta = n \cdot \lambda$.

Here, d is the width of periodic structure, θ is the angle between crystal plane and incident light, λ is the wavelength of light and n is the constant

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3.1.5
Parallel X-ray beam
Refers to X-ray beam obtained by collimating incident and/or diffracted X-ray beam by using solar slit, analyser crystal and/or x-ray mirror. As comparison with the focused beam, parallel X-rays, it does not suffer the sample condition (e.g. surface roughness) and geometrical limitations of the optical system (e.g. mechanical focal-circle deviation)

3.1.6
Slit
Device for controlling the size and photon flux amount of X-ray beam

3.1.7
Symmetric diffraction
State where the surface of sample and the Bragg diffraction are parallel

3.1.8
Asymmetric diffraction
State where the surface of sample and the Bragg diffraction are not parallel

3.1.9
2-theta
2θ
Angle of the detected X-ray beam with respect to the incident X-ray beam direction

3.1.10
omega
ω
Angle between the incident X-ray beam and the sample surface

3.1.11
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 is also may define as psi (ψ) depending on equipment companies

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3.1.12
phi
Φ
Angle of rotation about the normal to the nominal surface of the sample

3.1.13
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 Φ = 0; Y is the direction in the plane of the sample, perpendicular to the incident beam when Φ = 0; and Z is the direction normal to the plane of the sample

3.1.14
Flat zone wafer
Since crystal structure in the thin film (wafer) cannot be visually identified with human eye, the location is classified by making one section flat in order to distinguish the structure of thin film (wafer)

3.1.15
Rocking curve
ω rocking
ω scan
A term which indicates the intensity of peak and the 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. It is normally an indicator showing crystalline quality of the sample.**NOTE** Rocking curve is normally called an RC by abbreviation.

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3.1.16
Crystalline quality
The "crystalline quality" used in this standard shall mean the FWHM value of a rocking curve based on various degrees of defects (dislocation density, mosaic spread, curvature, misorientation, and inhomogeneity etc.) in the sample.

3.1.17
Arc seconds
FWHM unit of rocking curve as 1/3600 of a degree of an angle

3.2 Symbols and abbreviated terms

2θ 2Theta, the angle of the detected X-ray beam with respect to the incident X-ray beam

ω Omega, the angle between the incident X-ray beam and the sample surface

ϕ Phi, the angle of rotation about the normal to the nominal surface of the sample

χ Chi, the 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 (may define as psi (ψ))

λ Wavelength of the incident X-ray beam

4 Fundamentals

The purpose of this standard is to provide an 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 (dislocation density, mosaic spread, curvature, misorientation and inhomogeneity, etc.) 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 crystal plane are very consistent in an almost perfect crystal, most diffraction conditions are gained from one angle (2 theta) in all crystal planes so that a very sharp peak can be gained. On the contrary, the single-crystals with more defects show a broader peak.

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5 Devices and instruments

5.1 Schematic diagrams

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

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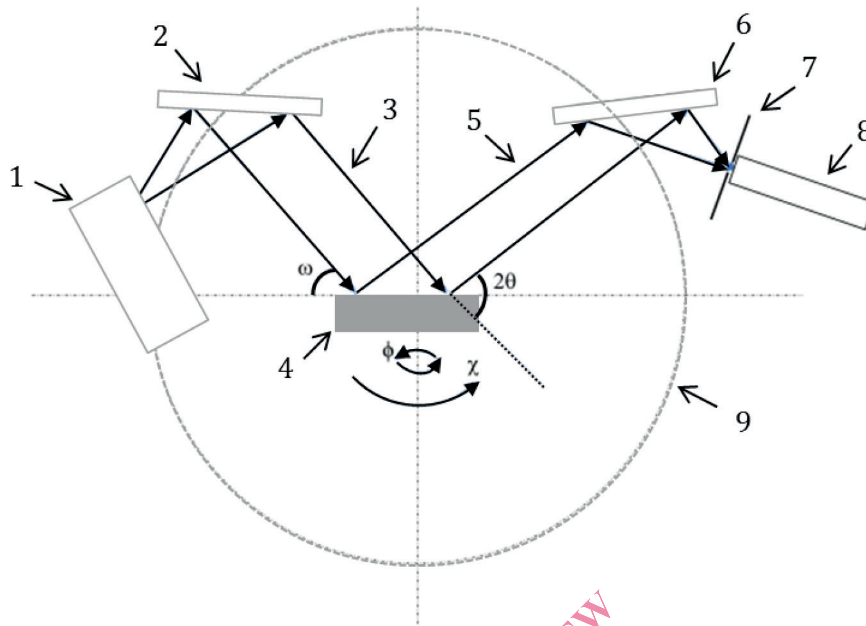

Key

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
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

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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 the one that can control the amount of X-ray beam that reaches 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 may not be used as the case may be.

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5.4 Monochromator

Device which monochromatizes the parallel beam generated from X-ray mirror to have a specific resolution. Selecting an appropriate monochromator is critical in the XRD analysis. In the case where the crystalline quality (FWHM of rocking curve) of the sample is similar to the resolution of a monochromator, the monochromator needs to 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 rocking curve) 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 = 0,154056 nm), Cu K_{a2} (λ = 0,154439 nm), and Cu K_{β} (λ = 0,139221 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. Therefore, it causes a problem in measuring the accurate angle of diffraction (2θ). For this reason, using a monochromator that takes only K_{a1} ray from incident X-rays to make a single wavelength is required 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 x-axis, y-axis, z-axis, 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 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 may have different moving fundamentals and structure depending on the equipment company.

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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 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 Analyser crystal works only for zero-dimensional detector (Scintillation detector). Multidimensional X-ray detector can be drastically reduced total measurement time and can be observed detail information of the sample very quick. Moreover, 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 above 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 rocking curve, 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 specific size by cutting with a multi-wire saw, etc. Polish using a chemical mechanical polishing (CMP) for the planarized surface of processed single-crystal thin film (wafer).

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