INTERNATIONAL STANDARD

ISO 22278

First edition
2020-08

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

Reference number
ISO 22278:2020(E)

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
2θ
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

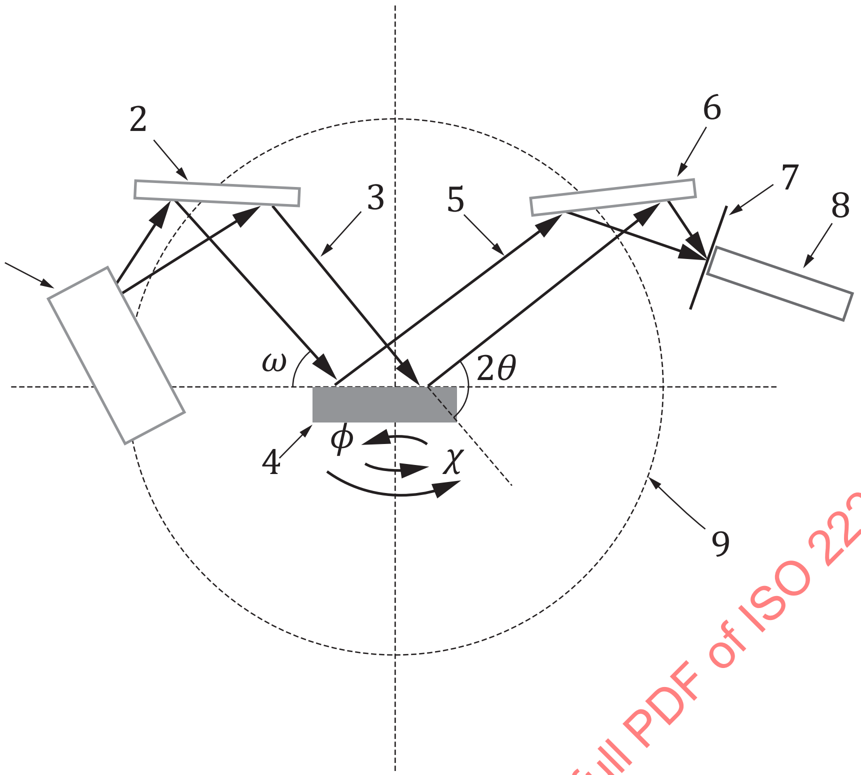
4 Fundamentals

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 $K_{\alpha 1}$, $K_{\alpha 2}$ and K_{β}. For instance, if the target is Cu, Cu $K_{\alpha 1}$ (λ = 0,154 056 nm), Cu $K_{\alpha 2}$ (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 ($K_{\alpha 1,}$ $K_{\alpha 2}$), K_{β} ray can be easily filtered using a thin Ni filter. However, as the wavelengths of $K_{\alpha 1}$ and $K_{\alpha 2}$ 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 $K_{\alpha 1}$ 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:

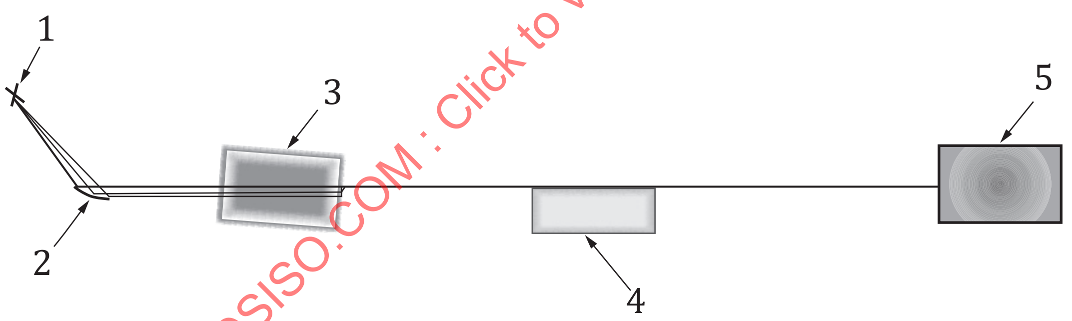
a) Make sure that nothing unwanted obstructs the beam between the source and detector. The sample attachment shall be out of the beam.

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 routines, and can make use of other methods of alignment, such as range finders or position monitors. The following basic requirements shall be met:

a) The instrument shall be aligned correctly, with appropriate slit widths, with the incident beam accurately over the centre of rotation. Set the X-ray source slit width to minimize spill off (typically 1 mm in a laboratory system) and set the detector slits significantly wider than the source slits (many times wider).

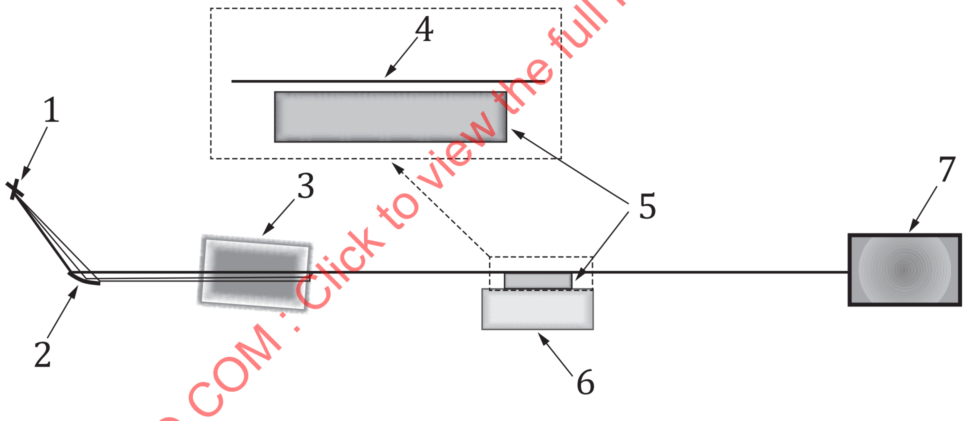
b) The angle, ω, between sample surface and incident beam shall be calibrated so that zero sets the sample surface approximately parallel to the incident beam.

c) Where applicable, set the X-ray generator power to the manufacturer's recommended operating level. Ensure stable operation.

d) Make sure that no obstructions (such as magnet or tape to attach the sample firmly on the sample plate) can interfere with the incident or diffracted beams. In the case of a wafer sample, make sure that the direction of the flat zone faces the bottom when it is placed above the sample plate.

   If there is no high-quality single-crystal thin film or wafer available for XRD machine checking and calibration, the certified internationally standard single-crystal wafer [e.g. silicon (Si) single-crystal wafer for crystalline orientation] should be used.

e) If required, insert an attenuator in the beam so that the detected intensity is well within the linear or linearized regime of the detector.



Key

1 X-ray generator
2 X-ray mirror
3 monochromator
4 incident beam
5 sample
6 sample attachment
7 detector

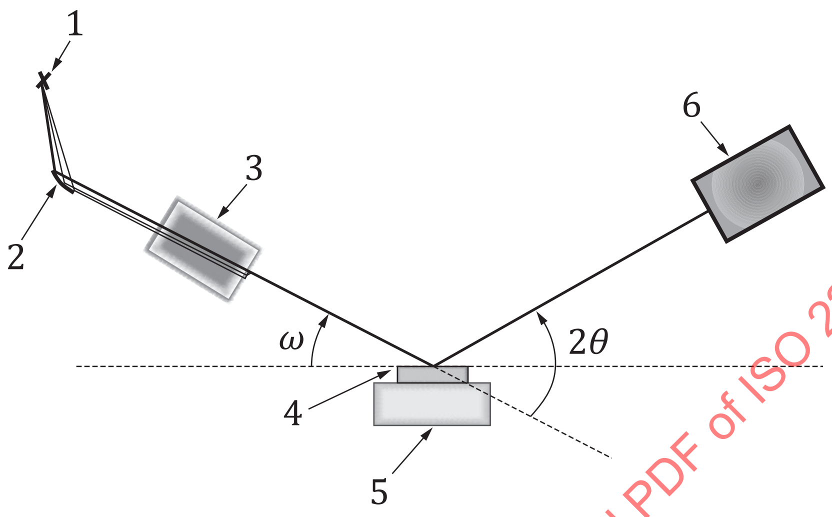
Figure 3 — Schematic diagram of sample alignment

7.3 Adjusting the initial position of goniometer

7.3.1 Symmetric diffraction

Single-crystal thin film (wafer) has several specific crystal planes depending on the growing method. Examples for d, 2θ, ω and relative ideal intensity values of symmetric diffraction of the grown crystal plane are shown in Annex A, Table A.1, using the method given in Annex B. Once 7.2 is completed, adjust the X-ray generator (mirror), detector (analyser) and the position of the sample plate by as much as an angle equivalent to ω (an angle equivalent to half of 2θ) and 2θ value on the corresponding crystal plane (see Figure 4).

When cutting the single crystal at a specific angle (off-cut angle), the cutting angle shall be added to or subtracted from the 2θ and ω values. For instance, for a 4 off-cut on a 6H-SiC single-crystal thin film (wafer) that has the crystal growth face of (006), ω = 29,548 7° −4° = 25,548 7°, 2θ = 59,097 5° and −8° = 51,097 5° (see Annex A, Table A.1, and Figure 5)



Key

1 X-ray generator

2 X-ray mirror

3 monochromator

4 sample

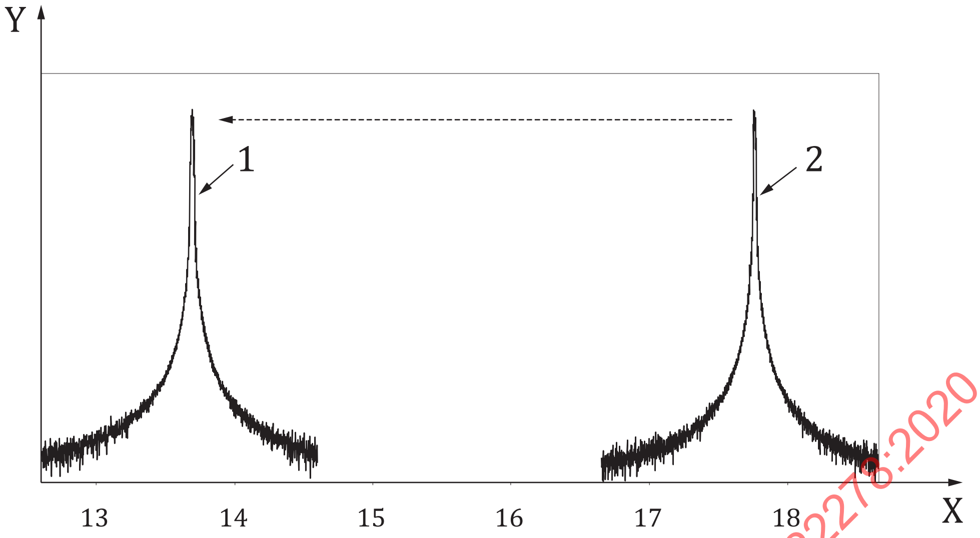
5 sample attachment

6 detector

ω angle between the sample surface and the incident X-ray beam 2θ angle between the detected beam and the extension of the incident X-ray beam

2θ angle between the detected beam and the extension of the incident X-ray beam

Figure 4 — Schematic diagram of the initial position adjustment of goniometer (symmetric diffraction observation)



Key

X omega angle (°)

Y arbitrary unit (au)

1 RC with off-cut angle 4°

2 RC with off-cut angle 0°

Figure 5 — Change of ω value depending on off-cut angle

7.3.2 Asymmetric diffraction

Examples for d, 2θ, ω and χ values of asymmetric diffraction of the grown crystal plane are shown in Annex A, Table A.2, using the method given in Annex B. Once 7.2 is completed, adjust the X-ray generator (mirror), detector (analyser) and the position of the sample plate by as much as an angle equivalent to ω, 2θ and χ values on the corresponding growing faces.

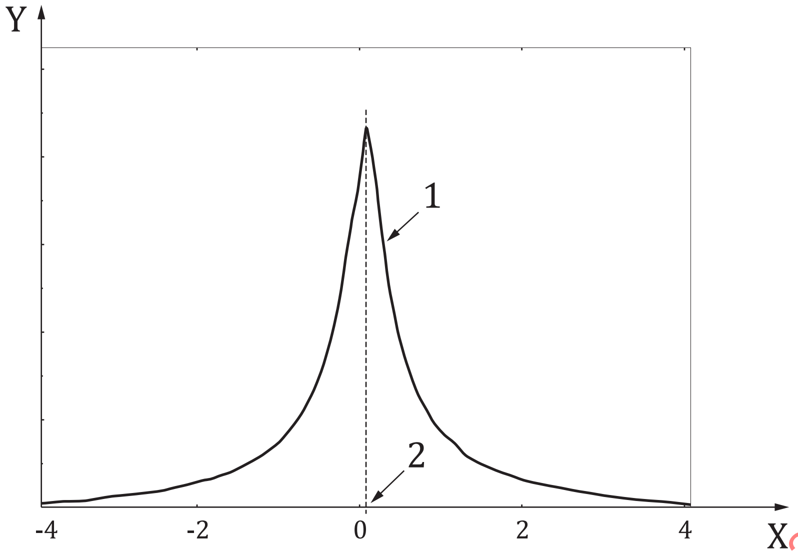
Moving fundamentals and structure of the χ-axis (see Figure 10) can vary depending on the equipment manufacturer. The χ-value shall also refer to the reference saved in the corresponding software.

7.4 Microscopic position adjustment of goniometer (Φ and χ axes) and ω scan

7.4.1 Symmetric diffraction

If the crystal plane of a grown single-crystal thin film (wafer) is symmetric diffraction, the optimum χ value shall be calculated from the initial ω position. There are two methods of calculation. Method A is to set the initial ω position, and then, from 0°, to scan the χ values within a range of −4° to +4°, as shown in Figure 6, to obtain a certain shape of the peak and a value of χ corresponding to the maximum intensity (key reference 2 in Figure 6). If an optimum χ value (deg) is found, perform the ω scan again within the range of –1° ~ +1° immediately after moving to that position (angle) to derive the FWHM value of RC (see Figure 7).

NOTE 1 The range of chi and omega scan can be set at random according to the shape of peak.



Key

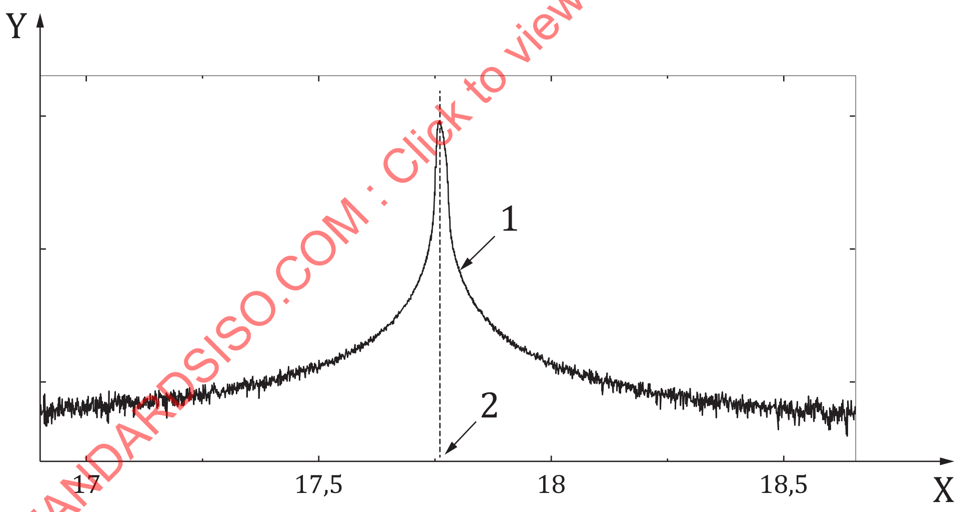
X chi angle (°)

Y arbitrary unit (au)

1 chi curve at initial omega position

2 chi angle at maximum peak intensity

Figure 6 — Optimum χ value at the position of initial ω for the crystal plane



Key

X omega angle (°)

Y arbitrary unit (au)

1 RC at initial omega position after moving to the optimum χ value

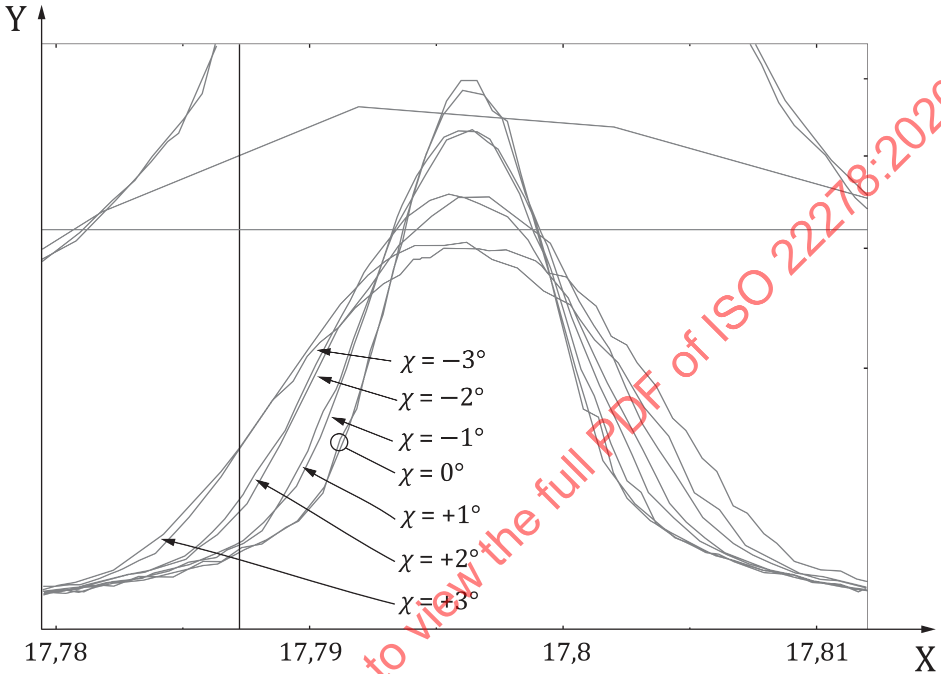
2 omega angle at maximum peak intensity

Figure 7 — FWHM value of RC under optimum χ value for the crystal plane

Method B is to set the range of the χ value to −1° to +1° (from 0°) and then divide it by intervals of 0,1° or 0,2° to get RC for each χ value. After deriving a value of χ with the maximum intensity, move the sample to the corresponding χ value and then perform the ω scan in the range of −1° to +1° to get the FWHM value of RC.

NOTE 2 The range of chi and omega scan can be set at random according to the shape of peak.

Find an optimum χ value since the FWHM value of RC changes considerably even with a microscopic change of χ-axis as shown in Figure 8 and Figure 9. The optimum χ value varies depending on the quality of single crystal (defect, curvature or dislocation) and the χ value of a single crystal with better symmetry becomes closer to 0.

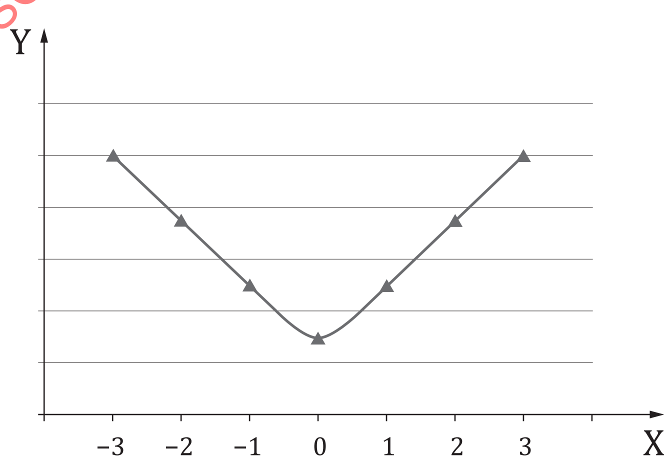


Key

X omega angle (°)

Y arbitrary unit (au)

Figure 8 — Change of RC according to the change of χ-axis for the crystal plane



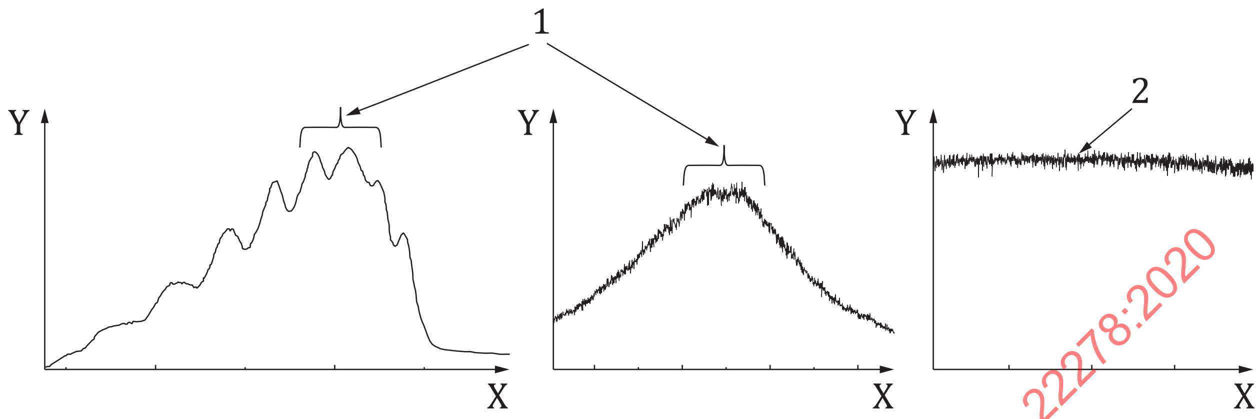
Key

X chi angle (°)

Y FWHM values (au)

Figure 9 — Change of FWHM according to the change of χ-axis

Method B can be more useful in cases where it is difficult to obtain the maximum intensity only from the shape of the χ peak. For instance, when an anomalous peak appears as in Figure 10, it is not easy to derive a value of χ representing the maximum intensity. By adopting method B, the optimal χ value can be derived (if necessary, the scan interval of χ value can be set smaller than 0,1°).



Key

X chi angle (°)

1 multi-peak shape of chi scan

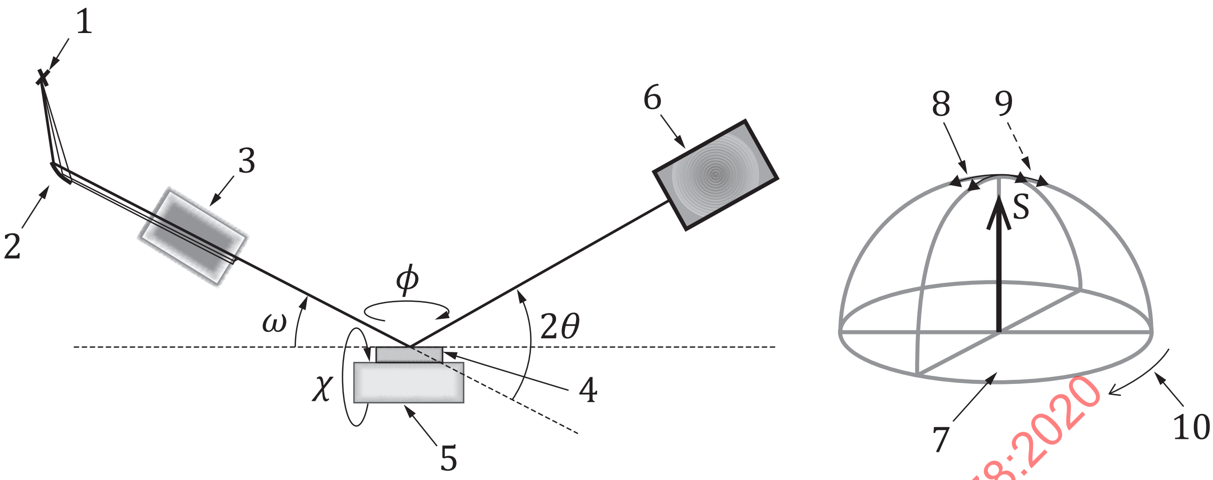
2 flat-peak shape of chi scan

Figure 10 — Irregular curves of χ-axis

7.4.2 Asymmetric diffraction

If the crystal plane of a grown single-crystal thin film (wafer) is asymmetric diffraction, the optimum Φ value shall be calculated from the initial ω position. Here, Φ value stands for the angle of peak indicating the maximum peak intensity among the number of peaks gained by rotating the sample 0° ~ 360° clockwise (or counterclockwise) (see Figure 11). Figure 12 shows optimum Φ value at the position of initial ω for the crystal plane with hexagonal structure. The periodicity of the diffraction peaks (every 60°) and the number of six peaks signifies a highly oriented hexagonal structure; sixfold azimuthal symmetry.

NOTE 1 The number of phi(Φ) peaks gained by rotating the sample which used to confirm the single crystalline nature by measuring in-plane structure order.



Key

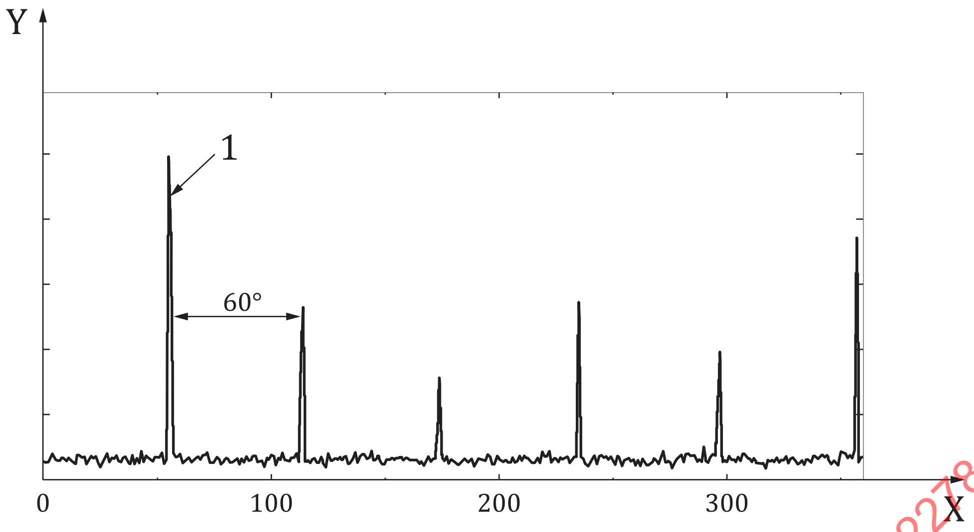
1 X-ray generator
2 X-ray mirror
3 monochromator
4 sample
5 sample attachment
6 detector
7 sample surface
8 omega direction
9 chi direction
10 phi direction
ω angle between the sample surface and the incident X-ray beam
2θ angle between the detected beam and the extension of the incident X-ray beam
Φ 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
S vector perpendicular to sample surface

Figure 11 — Schematic diagram of the microscopic position adjustment of goniometer (when crystal plane is asymmetric diffraction)

After finding an optimum Φ value, move to that position and calculate the optimum χ value immediately. In asymmetric diffraction, the optimal χ value can be obtained in two methods. Method A is to set the initial ω and χ position of the crystal plane to be measured according to Annex B, then scan the χ values within a range of −2° to +2° as shown in Figure 13 to obtain a certain shape of the peak and a value of χ corresponding to the maximum intensity (e.g. +70,6° in Figure 13). If an optimum χ value (deg) is found, perform the ω scan again within the range of −1° ~ +1° immediately after moving to that position (angle) to derive the FWHM value of RC (see Figure 14).

NOTE 2 The range of chi and omega scan can be set at random according to the shape of peak.

Since there is risk of dropping the sample placed on the sample attachment while measuring the asymmetric diffraction, be sure to attach the sample firmly to the sample plate using magnet or tape.



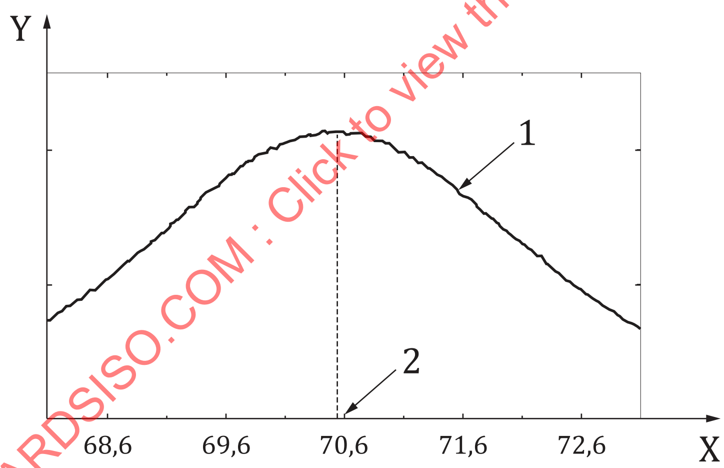
Key

X phi angle (°)

Y arbitrary unit (au)

1 peak with maximum intensity

Figure 12 — Optimum *Φ* value at the position of initial *ω* for the crystal plane with hexagonal structure



Key

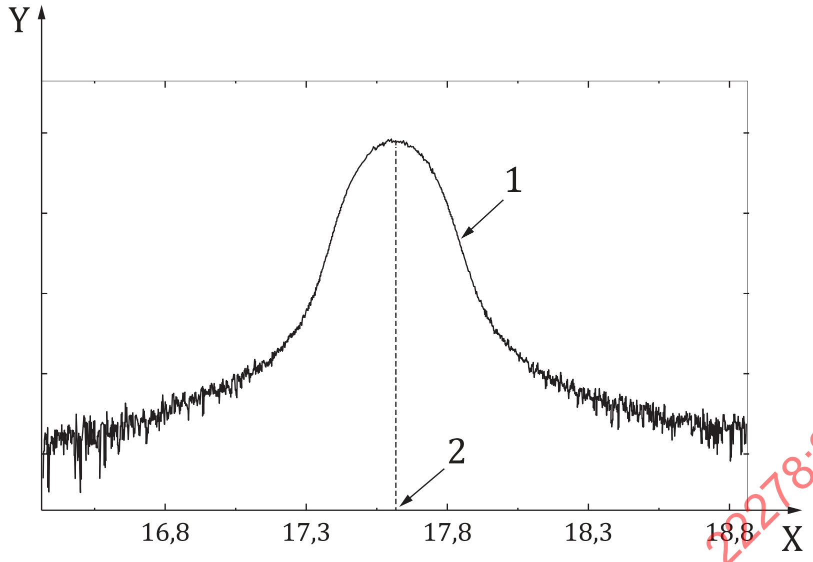
X chi angle for the crystal plane (°)

Y arbitrary unit (au)

1 chi curve at initial omega position after move to the optimum *Φ* value

2 chi angle at maximum peak intensity

Figure 13 — Optimum *χ* value at the position of initial *ω* for the crystal plane



Key

X omega angle (°)

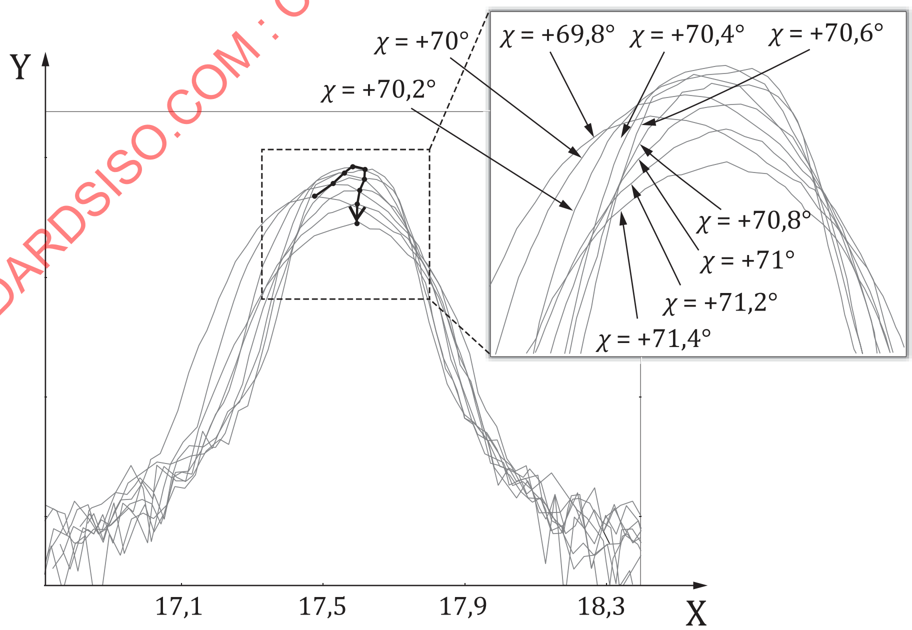
Y arbitrary unit (au)

1 RC at initial omega position after moving to the optimum χ value

2 omega angle at maximum peak intensity

Figure 14 — RC under optimum χ value for the crystal plane

Method B is to set the range of the calculated χ value to –1° to +1° (e.g. from +70,6°) as shown in Figure 15, and then divide it by intervals of 0,1° or 0,2° to obtain RC for each χ value. After deriving a value of χ with the maximum intensity, move the sample to the corresponding χ value and then perform the ω scan in the range of –1° to +1° to get the FWHM value of RC (see Figure 16).



Key

X omega angle (°)

Y arbitrary unit (au)

Figure 15 — Change of RC according to the change of χ-axis for the crystal plane

NOTE 3 The range of chi and omega scan can be set at random according to the shape of peak

If the RC cannot be identified even after performing the ω scan with the optimal χ value, the ω angle might have been affected by off-cut angle and therefore is currently larger or smaller than the first ω angle. In such cases, conduct the measurement again by setting the ω angle to a wider range (e.g. −8 to +8°). For this reason, it is recommended that the tester check the information on off-cut angle before conducting a sample analysis.

NOTE 4 Even in the asymmetric diffraction (like the symmetric diffraction), method B can be useful when it is not easy to determine the maximum intensity from only the shape of the χ peak (see Figure 10).

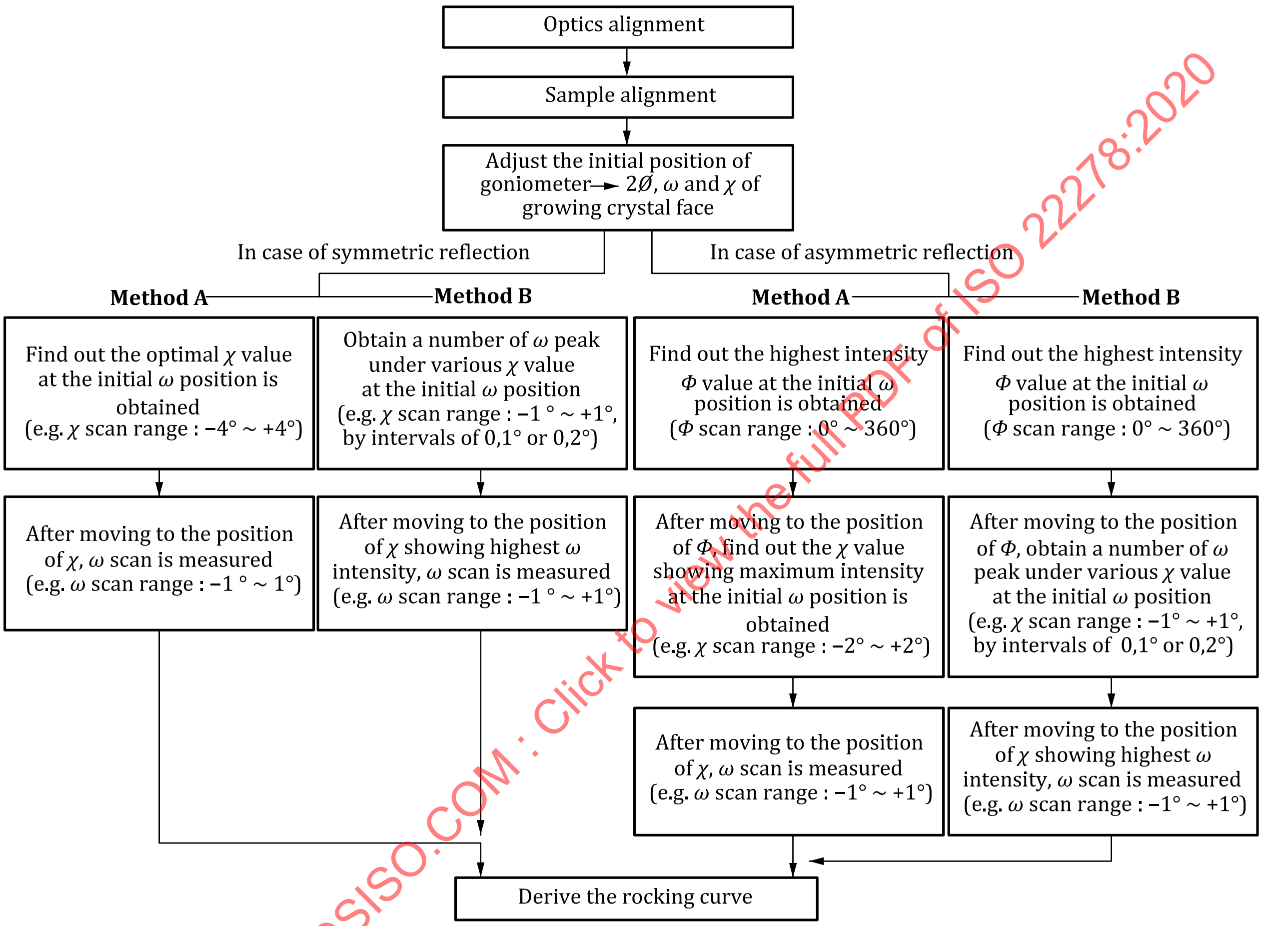


Figure 16 — Schematic diagram showing the order of deriving the RC

7.5 Crystalline quality measurement method of single-crystal wafer

7.5.1 General

For wafers of specific size (e.g. 2 inches, 4 inches, 6 inches, 8 inches), the method of calculating the FWHM value of the RC is the same as the procedures in 7.1 to 7.4. However, since an FWHM value of RC representing the quality of crystalline on the entire area is necessary in the case of wafers, evaluation shall be performed using the following method.

7.5.2 Selecting the flat zone position of wafer

Place above the plate after making the position of flat zone made to check the structure of grown single-crystal wafer to face down. At this time, be sure to attach firmly using magnet or tape. The centre of wafer shall coincide with the centre of sample plate.

7.5.3 Arranging the fixed size of the square

As shown in Figure 17, arrange the fixed size of the square regularly on the wafer area of a specific size to set the distance from the centre of the square to the centre of another square as *x* mm. At this time, the size of all the squares filling up the area of the wafer shall be consistent.

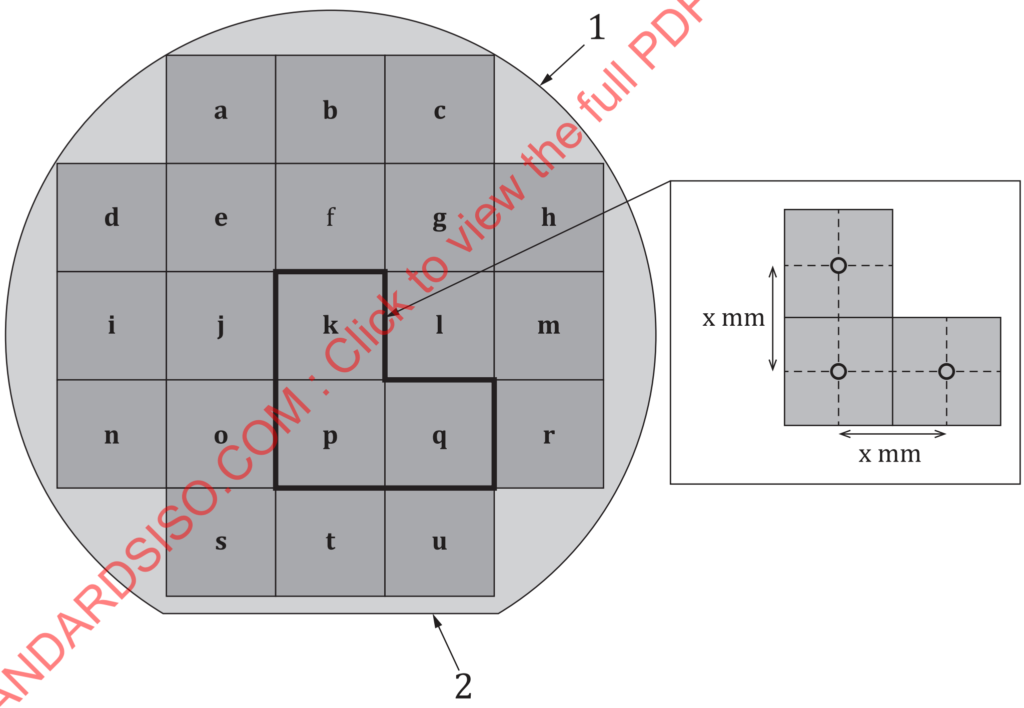
NOTE The size of the square can be determined by the user (customer).

7.5.4 Measuring the FWHM value of RC

Measure the FWHM value of RC by going through the procedures from 7.1 to 7.4 at the k point which is the centre point of wafer. Once the measurement is complete, mean values and standard deviation values shall be calculated on all points (a ~ u) after measuring the RC sequentially from a specific point desired by the user to the last point. The unit shall be expressed in arc seconds.

7.5.5 Interference effect by the wafer's curvature

If the curvature of a wafer is high, the FWHM of the RC can increase significantly due to the curvature effect. For this reason, in order to reduce the effect of the curvature and thereby prevent an increase in the width of X-ray beam, the width of X-ray incident beam needs to be properly narrowed or a diffraction crystal face of high index with a relatively bigger Bragg angle shall be applied.



Key

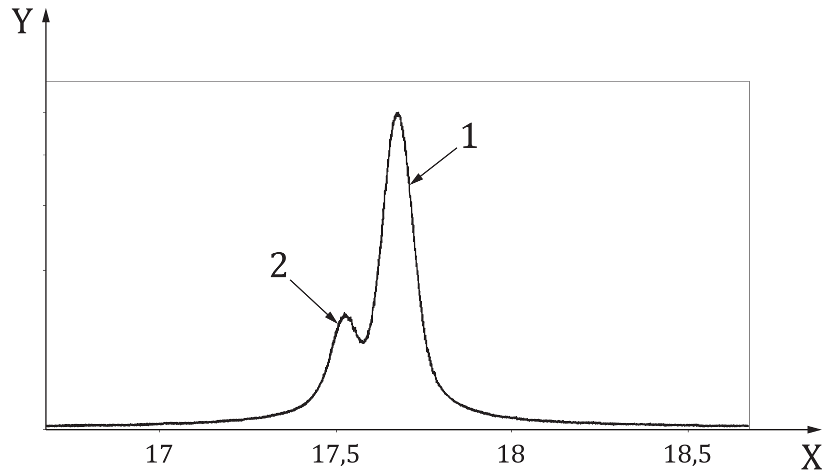
1 wafer sample

2 flat zone

Figure 17 — Crystalline quality mapping method of single-crystal wafer

7.5.6 Doped epitaxy film on a single-crystal thin film substrate

In general, in accordance with specific properties, epitaxial thin film is grown on a single-crystal thin film substrate. In such cases, the two peaks that are overlapped shown in Figure 18 can be obtained (peaks can shift depending on the amount of the doping element). Therefore, in order to gain accurate RC of doped epitaxial thin film, it is necessary to separate it from the peak of single-crystal thin film substrate. The overlapped peaks can be separated using dedicated software linked to the corresponding XRD.



Key

X omega angle (°)

Y arbitrary unit (au)

1 peak of single-crystal thin film substrate

2 peak of doped epitaxial film

Figure 18 — Example of RC for the doped epitaxial film on single-crystal thin film

8 Data analysis

The FWHM of RC obtained means is the width of a spectrum curve measured between those points on the y-axis which are half the maximum diffraction intensity. In general, the FWHM value can be gained by processing a dedicated software program installed in XRD; diverse fitting algorithms can be chosen. The degree of FWHM alteration can be checked and referred to according to each algorithm. A special caution shall be given in the course of RC processing as smoothing RC too much can distort its shape.

9 Test report

In the test report, the following information shall be included as a minimum.

a) detailed explanation of the method used:

- X-ray source type;
- X-ray beam size and shape;
- attenuator factors for the radiation used (if applicable);
- incident full beam intensity and detector background;
- details of incident and diffracted beam conditioning (monochromator, slits, collimator and other optics);
- detector type;
- scan parameters: step size, scan speed, start/stop range;
- FWHM calculation method (the report should briefly specify the calculation approach used and what type of software was employed; if a commercially available or custom-made software was employed, it should be indicated in the text);

b) a reference to this document (i.e. ISO 22278:2020);

c) analysed date and name/model of the devices used;

d) a description of the wafers (material manufacturer, growth conditions);

e) name, laboratory name and address of the analyser;

f) identification of reports;

g) mean and standard deviation on the RC values of multiple points (in the case of wafers);

h) the date of the test.

Annex A
(informative)

Example of d-spacing, 2θ, χ value (tilt angle) and relative ideal intensity of the symmetric and asymmetric diffraction on the SiC single-crystal thin film (wafer)

Table A.1 and Table A.2 indicate d, 2θ, ω and relative ideal intensity values of an example of silicon carbide (SiC) where the grown crystal plane is symmetric and asymmetric diffraction, respectively, using the method given in Annex B.

Table A.1 — Example of the calculated d, 2θ, ω angles and relative ideal intensities of symmetric diffractions of 4H-SiC and 6H-SiC

Diffraction/ growth face indexes	4H-SiC				6H-SiC			
	d Å	2θ °	ω °	Relative ideal intensity %	d Å	2θ °	ω °	Relative ideal intensity %
002/(002)	5,030 7	17,615 5	8,807 7	0	7,562 4	11,692 4	5,846 2	0
004/(004)	2,515 4	35,665 4	17,832 7	63	3,781 2	23,508 9	11,754 4	0
006/(006)	1,676 9	54,691 3	27,345 7	0	2,520 8	59,097 5	29,548 7	57
008/(008)	1,257 7	75,537 6	37,768 8	2	1,890 6	48,087 6	24,043 8	0
0012/(0012)	1,008 7	99,920 0	49,960 0	1	1,512 5	61,367 5	30,683 8	3

Table A.2 — Example of the calculated d, 2θ, ω, χ angles and relative ideal intensities of asymmetric diffractions of 4H- SiC and 6H-SiC

Diffraction/ growth face indexes	4H-SiC					6H-SiC				
	d Å	2θ °	ω °	χ °	Relative ideal intensity %	d Å	2θ °	ω °	χ °	Relative ideal intensity %
101/(101)	2,572 7	34,845 0	17,422 5	75,172 4	100	2,620 8	34,184 4	17,092 2	79,991 6	40
102/(102)	2,352 0	38,235 6	19,117 8	62,100 9	98	2,509 6	35,749 6	17,874 8	70,559 2	100
103/(103)	2,084 0	43,384 5	21,692 2	51,544 1	17	2,352 0	38,234 2	19,117 1	62,101 7	20
110/(110)	1,536 5	60,176 6	30,088 3	90,000 0	49	1,536 5	60,174 9	30,087 4	90,000 0	35
116/(116)	–	–	–	–	0	1,310 9	71,971 1	35,985 5	58,560 9	40
106/(106)	1,417 9	65,813 1	32,906 5	32,193 7	35	–	–	–	–	0
114/(114)	1,310 9	71,974 0	35,987 0	58,560 1	33	–	–	–	–	0

NOTE 1 Original crystallographic data for this calculation are shown in Table A.3 and the wavelength using this calculation is 1,540 56 Å (Cu K $_{a1}$). Since there are no standard materials for 4H-SiC or 6H-SiC, no accuracy can be expected even with the calculated results from the structure previously reported or experiments.

NOTE 2 In both 4H-SiC and 6H-SiC, the 00l (l = odd) diffractions cannot be observed because the diffractions are systematically absence ordered by their crystal symmetry (space group $P6_3mc$).

NOTE 3 Relative ideal intensity values for the 4H-SiC and 6H-SiC are normalized to the 4H-SiC (101) and 6H-SiC (102) diffraction (100 %), respectively (see Table A.2).

Table A.3 — Original crystallographic information of 4H-SiC and 6H-SiC for the calculation

Compound	Space group	a Å	c Å	Reference
4H-SiC	$P6_3mc$	3,081 5	10,061 4	[8]
6H-SiC	$P6_3mc$	3,081 0(2)	15,124 8(10)	[9]

Annex B
(informative)

Determination of *d*-spacing, 2*θ*, *χ* value (tilt angle) and relative ideal intensity for the symmetric and asymmetric diffraction on the single-crystal thin film (wafer)

B.1 General

This annex provides details of determination of *d*-spacing, 2*θ*, *χ* value (tilt angle) and relative ideal intensity for the symmetric and asymmetric diffraction on the single-crystal thin film (wafer).

B.2 Method for determining *d*-spacing and 2*θ* calculation

Calculate the value of d_{hkl} using *d*-spacing as shown in Formulae (B.1) to (B.7) for different crystal systems.