

X-ray thin film measurement techniques

VII. Pole figure measurement

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1. Introduction

Since crystal orientation and the degree of preferred orientation in thin films have a great influence on the properties of thin film devices, it is crucial for thin film devices to control the preferred orientation texture of thin film materials for applications such as light-emitting devices, ferroelectric memory and transparent conductive film applications, etc.

The Electron Back Scatter Diffraction (EBSD) technique, combined with a Scanning Electron Microscope (SEM), has been a popular approach for determining crystallite orientation and distribution in the field of material science. Recently, this technique has become focused of great interest due to major improvements in the throughput speed of analysis, as well as its capability for inspections to the scale down to several tens to hundreds of nm.

On the other hand, the irradiation area on the sample surface in pole figure (PF) measurements with X-ray Diffraction (XRD) is ϕ 10 μ m to 50 mm. The advantages of PF measurements by XRD are as follows: firstly, it allows the analysis of textural information of samples averaged over a large area/volume and, secondly, this measurement can be performed under ambient conditions or at high/low temperature by changing the atmospheric conditions.

In the process of texture analysis of materials, it is necessary to express the distribution of crystallite lattices in the coordinates of the external field, such as applied stress, magnetic field, etc. For most thin film cases, it would be the lattice of a single crystalline substrate.

In earlier articles in this series about thin-film analysis techniques, out-of-plane measurement⁽¹⁾ and in-plane measurement⁽²⁾ techniques were explained in the 2nd and 4th articles. This article will explain the basics of the pole figure measurement/analysis technique using out-of-plane and in-plane measurement geometry, followed by their applications for thin films samples.

2. The basics for the pole figure measurement of XRD

Pole figure measurement is an XRD measurement technique where the diffraction angle (2θ) is fixed and the diffracted intensity is collected by varying two geometrical parameters, such as the α angle (tilt angle

from sample surface normal direction) and the β angle (φ rotation angle around sample surface normal direction). The obtained diffracted intensity data is plotted as a function of α and β .

The introduction of the concept of the scattering vector \mathbf{K} will greatly help the data analysis⁽³⁾. When the length of the scattering vector \mathbf{K} is fixed and a sample is rocked (this situation corresponds to the rocking curve measurement or PF measurement), the trajectory of the end of the scattering vector will trace out a sphere. It is more convenient to see the diffracted intensity variation on the plane of the stereographic projection (Fig. 1) with polar coordinates, α and β , rather than on the hemisphere surface.

In general, the center of pole figure is defined as $\alpha=0^\circ$ and the outer end is defined as $\alpha=90^\circ$. (Note: an alternative scheme for the definition of α is commonly employed for thin film samples, where, the center is $\alpha=90^\circ$ and the outer is $\alpha=0^\circ$.) $\alpha=0^\circ$ means that the lattice plane normal is parallel to the sample surface normal, and $\alpha=90^\circ$ means that the lattice plane normal is perpendicular to the sample surface normal.

β denotes a rotation angle around the surface normal relative to a certain reference position. It starts at the top of the figure and is circularly coordinated with a counter clockwise rotation, where 9 o'clock is defined as $\beta=90^\circ$ and 3 o'clock defined as $\beta=270^\circ$ (Fig. 2(a)).

The intensity variation along a varying α at a fixed β value corresponds to the variation due to the tilting motion⁽³⁾ of the sample. On the other hand, the intensity

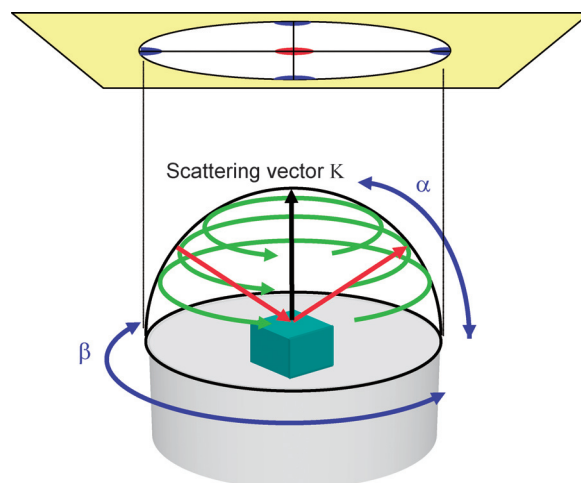


Fig. 1. Sphere of fixed-length scattering vector and stereographic projection.

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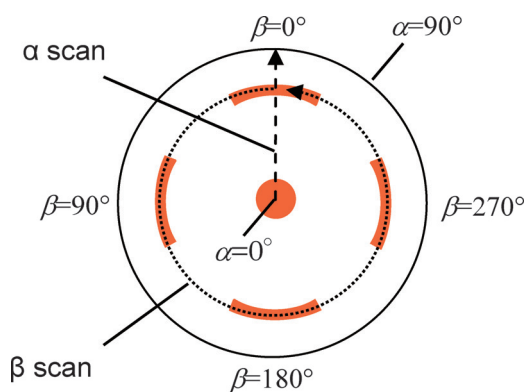
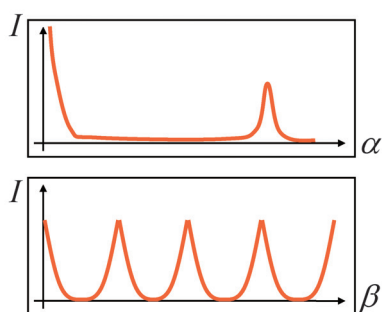
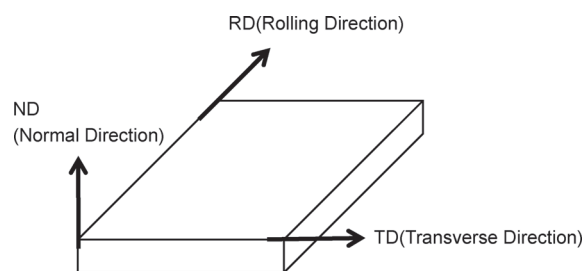

 (a) Definition of α and β .

 (b) α scan and β scan.

 Fig. 2. α and β of pole figure measurement.

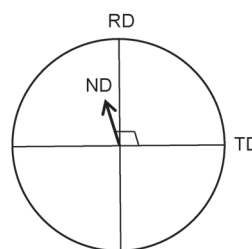
variation along a varying β with a fixed α corresponds to a twisting motion⁽²⁾ (Fig. 2(b)). The latter is sometimes used for the analysis of the azimuth distribution variation (inhomogeneity).

In the case of a precise quantitative analysis (i.e., the degree of preferred orientation etc.), normalization of the intensity is required, utilizing data obtained from a randomly oriented sample as a reference. (see Section 5) The relative intensity after normalization is known as pole density.

In this paper, coordinates orthogonal to the X, Y and Z axes are defined as RD, TD and ND based on the system used in metallurgy where texture analysis using pole figures has been performed (Fig. 3(a)). The normal direction to the sample surface is defined as ND (Normal Direction), the rolling direction of the sample is defined as RD (Rolling Direction). The term RD was originally used for a rolling process for samples such as steel materials. In thin film samples, RD is defined with reference to a specific direction from the shape of the substrate, or the crystallographic lattice of substrates. A direction perpendicular to RD within the surface plane is defined as TD (Transverse Direction). In the stereographic projection of pole figure data, the upward direction is defined as RD, the direction to the right is TD and the normal direction to the page is ND (Fig. 3(b)). Pole figures enable us to understand at a glance the angles between a lattice plane and RD, TD and ND.



(a) The definition of RD, TD and ND in a sample.



(b) The definition of RD, TD and ND in the pole figure.

Fig. 3. The definition of RD, TD and ND.

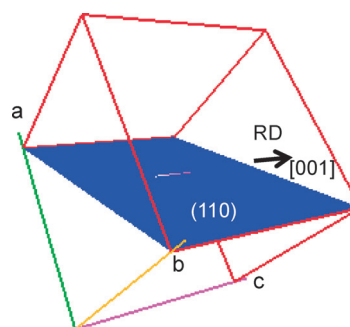


Fig. 4. Pattern diagram of cubic (110)[001] orientation.

3. The expression of crystal orientation

There are two ways to show crystal orientation distribution, such as pole figure and inverse pole figure. Coordinate axes for pole figures are RD, TD and ND, as shown in the previous section. This coordinate system is determined by the sample shape and is also called material coordinate system. On the other hand, the inverse pole figure coordinate system is a crystal coordinate system. For example, in an inverse pole figure of ND, it is possible to understand which lattice plane normal is parallel to the sample surface normal. When the (110) lattice plane of a cubic material is parallel to the sample surface, and its [001] axis is parallel to RD direction within the surface plane, the crystal orientation is described as (110)[001], as shown in Fig. 4. If all equivalent orientations are to be expressed, it is described as $\{110\}\langle 001\rangle$. Let us consider a crystal with cubic symmetry lying on the substrate where its lattice plane parallel to the sample surface is (hkl) and the [uvw] axis within the surface plane is aligned with RD. Since ND and RD intersect at a right angle, the relationship $hu + kv + lw = 0$ is satisfied. Pole

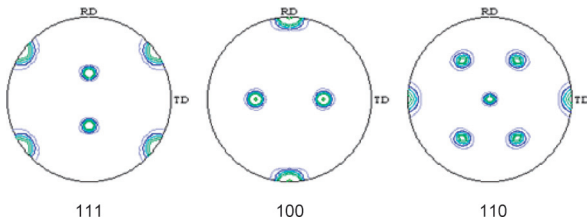


Fig. 5. Pole figure of $\{110\}\langle 001 \rangle$ orientation.

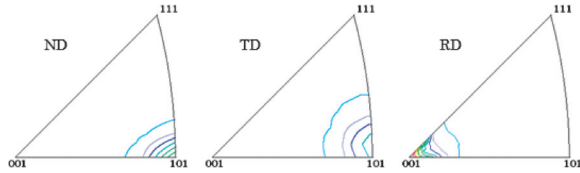


Fig. 6. Inverse pole figure of $\{110\}\langle 001 \rangle$ orientation.

figures of 111, 100 and 110 reflections for this sample with $\{110\}\langle 001 \rangle$ texture are shown in Fig. 5. It is found that the lattice plane parallel to the sample surface is $\{110\}$ because there is a pole maximum at the center location of the $\{110\}$ pole figure. Also, it is found that the $\langle 001 \rangle$ direction in the in-plane direction of lattice plane $\{110\}$ of the normal direction to the sample surface faces RD, because there is a pole at $\alpha=90^\circ$ on RD in the $\{100\}$ pole figure. The inverse pole figure for this sample with $\{110\}\langle 001 \rangle$ orientation texture is shown in Fig. 6. These figures indicate that ND faces $[110]$ and RD faces $[001]$.

4. Types of pole figure measurements and their geometries

4.1. Schulz reflection method and parallel beam reflection method

The Schulz reflection method is mostly used in pole figure measurement methods for bulk samples such as steel and light metal materials. In this method, sample tilting (α angle) is controlled using a four-circle goniometer equipped with a χ axis for thin film samples or the α -axis of a multipurpose attachment on a conventional powder XRD system. The detector is fixed at a particular 2θ position for the lattice plane of interest. The iterative motion of a χ -step (or α -step) and ϕ -rotation (or β -rotation) scan is performed to obtain pole figure data.

If Bragg–Brentano optics, which are generally used for powder samples, are utilized, the width of the diffraction peak becomes larger due to the defocusing effect, since the exposed area on the sample surface gets wider when tilting a sample. Therefore, a slit of about 1 mm in width is attached to minimize the defocusing effect. This slit, known as a Schulz slit, restricts the divergence of X-rays in the direction perpendicular to the incident beam. This geometry is called the Schulz reflection method (Fig. 7). The main advantage of the Schulz reflection method is the fact that the measurement can be conducted with a conventional powder XRD instrument capable of Bragg–Brentano

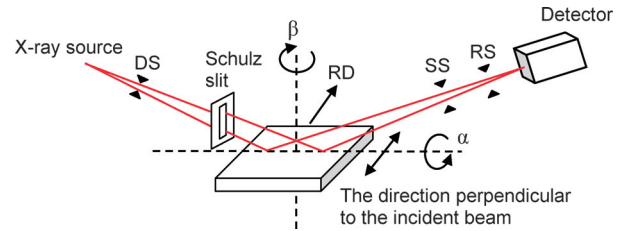


Fig. 7. Schulz reflection method.

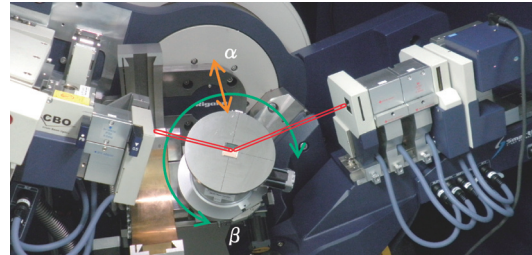


Fig. 8. Parallel beam reflection method.

geometry, so high intensity can be obtained. Its disadvantage is the fact that the background is high due to the scattering of continuous Bremsstrahlung radiation. With a thin film sample, the reflection of the single-crystal substrate is observed on the pole figure because continuous Bremsstrahlung radiation happens to satisfy the diffraction condition. An effective way to minimize this effect for thin film samples is to use a parallel beam that is monochromated to $K\alpha$ radiation using an optical device such as multilayer mirrors. This method is called the parallel beam reflection method. With this method, the divergence of the X-ray beam in the direction perpendicular to the incident beam can be restricted using a combination of a height-limiting slits and the incident Soller slit. The incident beam is turned into a quasi-point shape by setting the incident slit to about 1 mm (Fig. 8).

4.2. In-plane pole figure measurement method

In-plane pole figure measurement is a pole figure measurement performed using the in-plane arm. The details of in-plane measurement can be seen in the fourth article of this series⁽²⁾. It is possible to control the α , β and $2\theta_B$ angles by the combination of four axes: $2\theta_\chi$, ϕ , and 2θ , and ω , where χ is fixed to 0 all through the measurement. (Figure 9(a)–(c)) In particular, the control of the α axis (equivalent of the tilt angle of sample) can be performed with the combination of 2θ and $2\theta_\chi$. While the detector is fixed at a $2\theta_B$ position of interest, β (normally with a step of 2.5 to 5.0°) is scanned with the motion of ϕ at each α angle, recording the intensity as a function of α and β on a pole figure. Due to the geometrical constraint, this method is generally applied for the $2\theta_B$ below 90°.

This unique technique of in-plane pole figure measurement has two advantages comparison to the conventional Schulz reflection method. First, this method allows a complete pole figure to be recorded

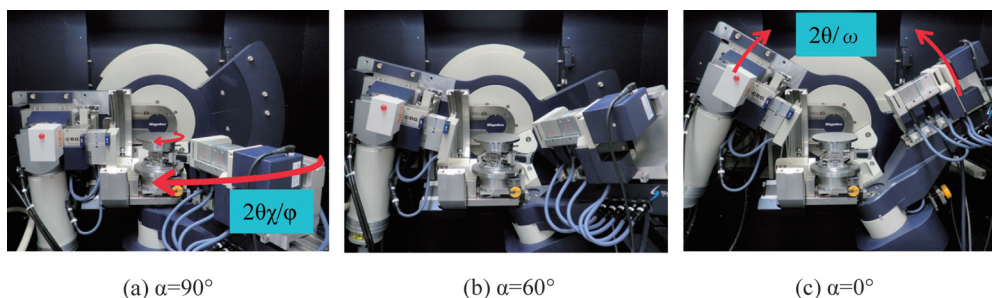


Fig. 9. The movement of the goniometer in in-plane pole figure measurement method.

from $\alpha=0^\circ$ to $\alpha=90^\circ$ because the goniometer used can detect diffraction from lattice planes perpendicular to the sample surface (this is the in-plane diffraction). Second, this method **does not** require the sample to be tilted—the sample is only rotated about the ϕ axis, maintaining a horizontal condition in the case of a horizontal sample goniometer, like SmartLab. This way, users will not have to worry about clipping or sticking samples to the stage to prevent them from falling off as when the in-plane pole figure measurement method is employed.

5. To realize better measurement and analysis

5.1. 2θ range covered during measurement (Schulz reflection method, parallel beam reflection method)

Though the 2θ position for the detector is fixed during pole figure measurement, the detector collects signals for the finite size of the capture angle for 2θ defined by the receiving optic slits/PSA (Parallel Slit Analyzer). When the capture angle for the 2θ range is controlled using the width of the receiving slit, the fact that the exposed area of the sample surface becomes large in accordance with sample tilting should be taken into consideration. If the 2θ range of the broadened peak due to sample tilting is $\Delta 2\theta$, the width of receiving slit to collect the entire signal can be calculated as follows:

$$RS = 2R \cdot \tan\left(\frac{\Delta 2\theta}{2}\right) \quad (1)$$

Where, R is the goniometer radius(mm). An absorption correction and a background correction (see. Section 5.2) are needed in addition to this defocusing correction (see. Section 5.3) when quantitative evaluation of orientation texture is carried out. When a PSA is employed on the receiving optics side, the width of the diffraction peak does not changed even if the sample is tilted because the PSA collects only the parallel component of the diffraction signal from the wide range on the sample surface. PSAs with aperture angles of 0.5° (for a standard set), 0.228° and 0.114° (for options) are available for the SmartLab system. It is important to select an appropriate PSA that can collect the 2θ range of the full width of the diffraction peak as possible. When using a 0.5° PSA, the measured 2θ range is equivalent to $\pm 0.5^\circ$ from the center of the peak position. If another diffraction peak position is close to the peak

of interest, it is useful to narrow the width of the receiving slit or to select a PSA with a smaller aperture angle to avoid collecting the other diffraction peak.

5.2. Absorption correction and background correction (Schulz reflection method, parallel beam reflection method)

If a sample is sufficiently thick, an absorption correction is not required because the sample thickness can be regarded as infinite. However, in the case of film samples, the length that X-rays pass through the thin layer changes according to the α angle because the incident X-ray reaches to a substrate by transmitting thin films. Therefore, the correction using Eq. (2) is required. The correction is performed by multiplying the inverse of the obtained K .

$$K = \frac{I_\alpha}{I_{\alpha=0^\circ}} = \frac{1 - \exp\left(\frac{-2\mu t}{\sin \theta \cos \alpha}\right)}{1 - \exp\left(\frac{-2\mu t}{\sin \theta}\right)} \quad (2)$$

K : intensity ratio to $\alpha=0^\circ$ (scale factor)

θ : Bragg angle of measured diffraction plane ($^\circ$)

μ : linear absorption factor of layer (cm^{-1})

t : thickness of layer (cm)

The signals collected include the diffraction signals from samples and background noise. Background measurement is carried out at a 2θ position where the diffraction peak is not detected anywhere the sample is tilted. Normally, the measured background position is approximately $\pm 2^\circ$ away from the peak position of interest. Background intensity is measured at each α angle, and the intensity is subtracted from the intensity of the peak position. These absorption and background corrections are performed before the intensity normalization process (defocusing correction) described in 5.3.

5.3. Defocusing correction (Schulz reflection method, parallel beam reflection method)

A powder sample in which the average particle size is sufficiently small and there is no preferred orientation for crystallites is generally treated as a sample with random orientation texture. The intensity of the pole figure for such a sample would be ideally constant with

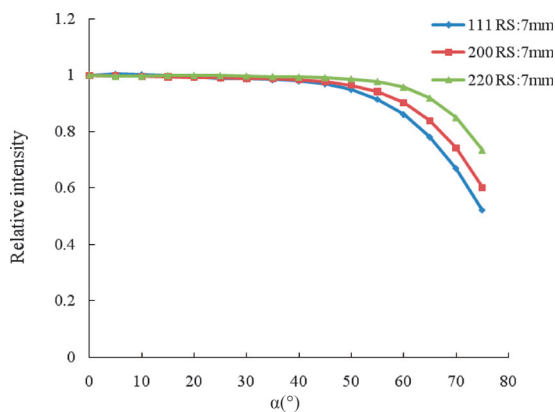


Fig. 10. The change in relative intensity of Al powder to α in the Schulz reflection method.

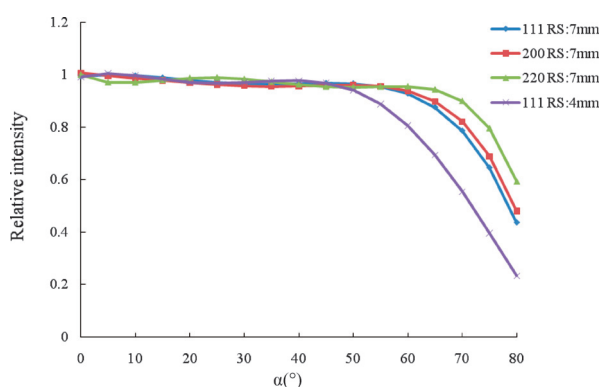


Fig. 11. The change in relative intensity of Al powder to α in the parallel beam reflection method.

varying α angle. However, the sample is tilted greatly on the high angle side of α , and the diffraction line is partly cut off at the receiving optics side. As a result, the relative intensity decreases at high values of the α angle, as shown in Fig. 10. Therefore, it is necessary to evaluate the degree of intensity loss at high values of α using a powder sample with random orientation texture. The correction factor at each α angle depends on the 2θ position and the width of the receiving slit⁽⁴⁾ as shown in Fig. 11. A sample with random orientation texture is measured using the same width of receiving slit as for the measurement sample and at as close a 2θ position as possible. It is preferable that substance used for the defocusing correction might be the same as the sample under analysis. Aluminum powder sample is frequently used instead because it is often the case that preparing a random orientation sample is difficult.

For large values of α , the correction factor is considerably high due to reduction in diffraction intensity, as shown in Fig. 10 and 11. It is estimated that the upper limit of the α angle measurable using the Schulz reflection method and parallel beam reflection methods is 75° considering the analysis precision.

5.4. 2θ range covered by measurement (In-plane pole figure measurement)

In in-plane pole figure measurement, it is common to

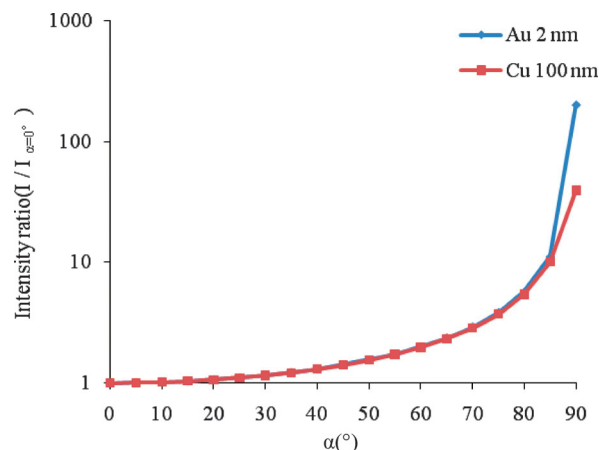


Fig. 12. The influence of absorption correction of thin film sample 111 pole figure to α (Schulz reflection method).

use an in-plane parallel slit collimator (in-plane PSC) on the incident optics side and an in-plane parallel slit analyzer (in-plane PSA) on the receiving optics side. At $\alpha=90^\circ$, the covering $2\theta_\chi$ range depends on the capture angle of the in-plane PSA on the receiving optics side. The configuration at $\alpha=0^\circ$ is the out-of-plane measurement geometry, as shown in Fig. 9(c). The covering 2θ range depends on the width of the receiving slit, as with the Schulz reflection and parallel beam reflection methods. Generally, the angular resolution of in-plane ($\alpha=90^\circ$) and out-of-plane ($\alpha=0^\circ$) geometry should be equal. When a 0.5° in-plane PSA is used, the appropriate width of the receiving slit is 2 to 3 mm. If another diffraction peak position is close to the peak of interest, it is useful to narrow the width of receiving slit or to select an in-plane PSA of smaller aperture angle to avoid collecting the other diffraction peak.

5.5. Absorption correction and background correction (In-plane pole figure measurement method)

Figure 12 shows the calculation of the influence of the absorption correction to the angle α in a 111 pole figure of 100 nm-thick Cu film and 2 nm-thick Au film. The formula used for the calculations in Fig. 12 is Eq. (2). The change in the relative intensity ratio is also applicable to in-plane pole figure measurement geometry. The incident angle between the X-ray beam and the sample surface is considerably smaller for α larger than 80° . Consequently, the intensity ratio relative to $\alpha=0^\circ$ expands exponentially because the irradiation area increases rapidly. Since the intensity of the pole figure may change greatly before and after an absorption correction in the range between $\alpha=80^\circ$ and 90° , quantitative data analysis in this range requires great attention. Data collection for the background measurement is carried out at approximately $\pm 2^\circ$ away from peak position of interest, as with the Schulz reflection method. Background intensity is measured at each α angle with the detector fixed, and this intensity is

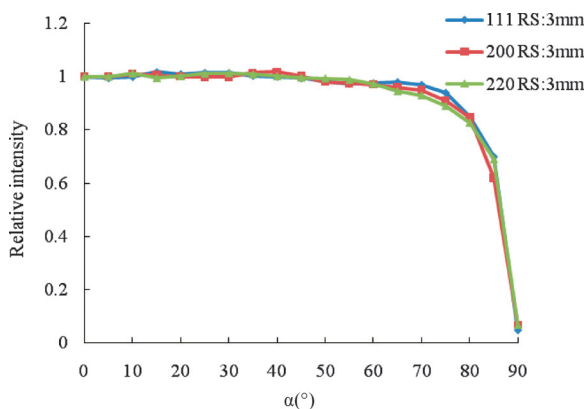


Fig. 13. The change in relative intensity of Al powder to α in in-plane pole figure measurement method.

subtracted from the intensity at the peak position of interest.

5.6. Intensity correction (In-plane pole figure measurement)

Figure 13 shows the change in relative intensity as a function of α in the case of an Al powder sample that is sufficiently thick for in-plane pole figure measurement. The width of the receiving slit in Fig. 13 is 3 mm. Since the relative intensity is almost constant from $\alpha = 0^\circ$ to 80° , it is obvious that an intensity correction is not required in this range for data obtained with the in-plane pole figure measurement technique. The relative intensity decreases greatly between $\alpha = 80^\circ$ and 90° . A sample with random orientation texture is measured with the same slit condition as the measurement sample when performing intensity correction. Even with in-plane pole figure measurement, the absorption and intensity corrections are large between $\alpha = 80^\circ$ and 90° , as described previously. Therefore, it is better to eliminate the range from $\alpha = 80^\circ$ to 90° when performing the quantitative evaluation of orientation texture.

6. Crystallite Orientation Distribution Function (ODF)

ODF⁽⁵⁾ is an analysis method where the three-dimensional crystalline orientation distribution function is deduced from pole figures of multiple lattice plane indices. The results are displayed as a Crystallite Orientation Distribution figure.

In ODF, the angle difference between $X_s(RD)$, $Y_s(TD)$, $Z_s(ND)$, the material coordinate system, and $X_c=[100]$, $Y_c=[010]$, $Z_c=[001]$ (suffix “c” denotes cubic symmetry), the crystal coordinate system, is expressed as the angle variables (ϕ_1, Φ, ϕ_2), as shown in Fig. 14 and 15. These angle variables (ϕ_1, Φ, ϕ_2) are known as Euler angles.

Figure 16 shows a Crystallite Orientation Distribution figure for the case of $\{110\}\langle 001\rangle$ orientation. A Crystallite Orientation Distribution figure is normally expressed using a cross-section diagram of ϕ_1 and Φ with fixed ϕ_2 and a step of 5° . In Fig. 16, one pole is located at $\phi_1 = 0.0^\circ$, $\Phi = 45.0^\circ$, $\phi_2 = 0.0^\circ$. Presently, the

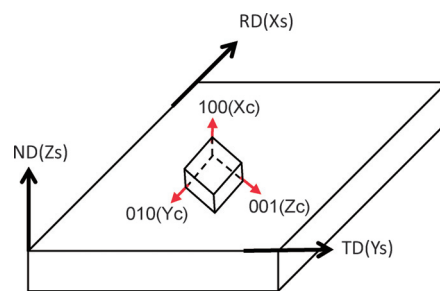


Fig. 14. Material coordinate system (RD, TD and ND) and crystal coordinate system ([100], [010] and [001]).

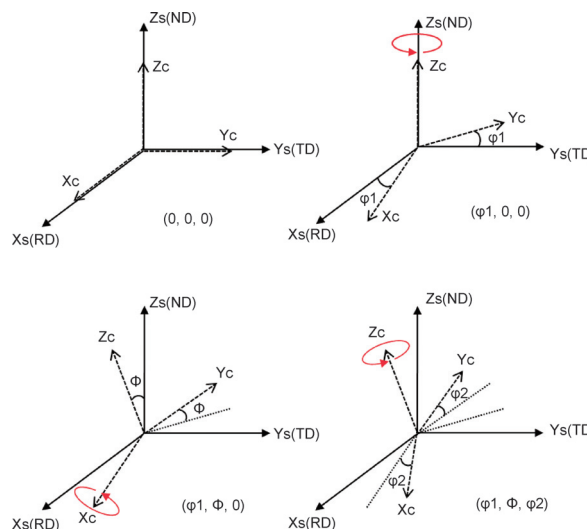


Fig. 15. Crystal orientation and Euler angle.

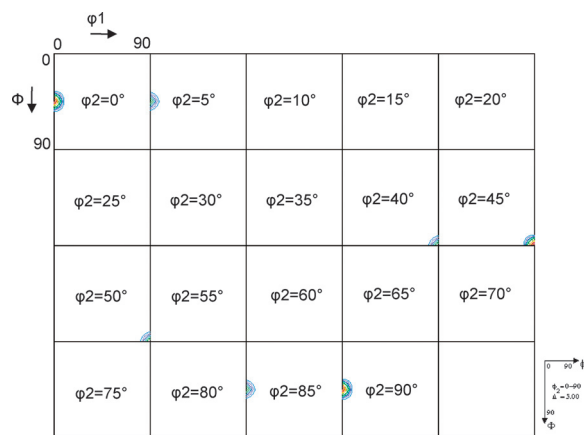


Fig. 16. Crystal orientation distribution figure of $\{110\}\langle 001\rangle$ orientation.

operation of commercially available ODF software is simplified and easy to use, enabling us to analyze the crystal orientation texture automatically from a Crystallite Orientation Distribution figure.

The features of the ODF are as followed;

- A pole figure obtained by the Schulz reflection method is incomplete because it cannot measure to $\alpha = 90^\circ$. However, the ODF allows us to create a complete pole figure by recalculation using a

Crystallite Orientation Distribution figure, which expresses a positional relation between the material coordinate system and the crystal coordinate system.

- It is possible to create pole figures of arbitrary lattice planes that have not been measured, using a Crystallite Orientation Distribution figure.
- Quantitative analysis of orientation texture is possible, and the volumetric ratio between the different orientation textures in a sample can be calculated.⁽⁶⁾

7. In-plane pole figure measurement and orientation texture analysis of a copper electrode

Figure 17(a) shows pole figures of a copper electrode (film thickness: 100 nm) using in-plane pole figure measurement. The poles can be observed in the shape of ring, such as $\alpha=70^\circ$ of 111 reflection, $\alpha=55^\circ$ of 200 reflection and $\alpha=35^\circ$ of 220 reflection. This indicates that this sample exhibits a fiber texture where a specific crystallographic axis of Cu stands perpendicular to the sample surface and the distribution around the axis is

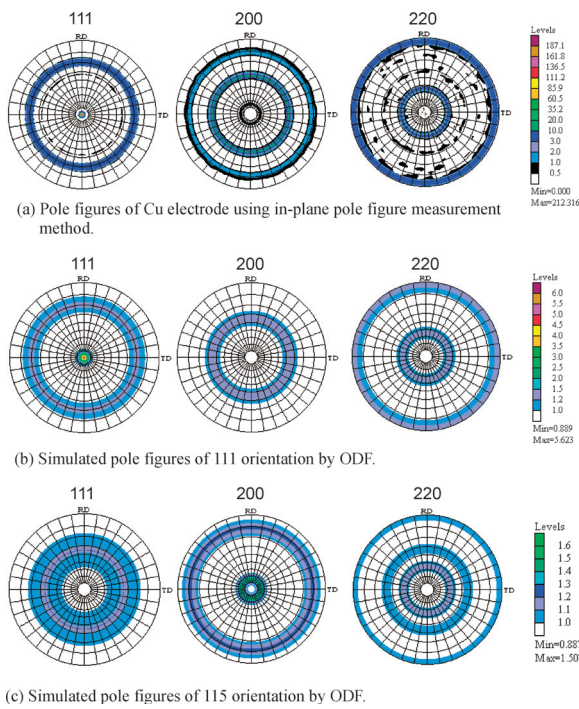


Fig. 17. Comparison of pole figures of Cu electrode using in-plane pole figure measurement method and simulated pole figures by ODF.

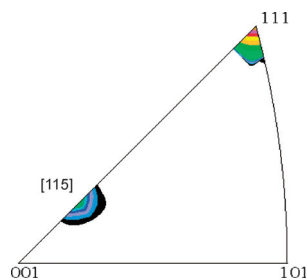


Fig. 18. Inverse pole figure of Cu electrode (ND).

random. It indicates that the plane normal to (111) is in the normal direction to the sample surface because there is a pole maximum at the center position of the 111 pole figure. This orientation texture is called uniaxial orientation or fiber texture. The α angle of the ring in a sample with fiber texture can be analyzed with the aid of a standard stereo projection figure, reciprocal simulation⁽⁷⁾ or ODF, etc. ODF software enables us to simulate the Crystallite Orientation Distribution figure for a sample with an orientation texture of (hkl)[uvw] and to create pole figures of an arbitrary lattice plane by recalculation. Figure 17(b) shows the pole figures simulated for a case of [111] fiber texture. It is readily apparent that some of the rings in Fig. 17(a) are reproduced in Fig. 17(b), but other rings, such as, those at $\alpha=57^\circ$ of the 111 pole figure, $\alpha=15^\circ, 78^\circ$ of the 200 pole figure and $\alpha=58^\circ$ of the 220 pole figure, are not observed in Fig. 17(b). The inverse pole figure of ND of the identical Cu film (Fig. 18) shows that an additional pole of 115 reflection co-exists with a pole of 111 reflection. The α angle of the ring in the simulated pole figure of [115] fiber texture (Fig. 17(c)) corresponds with the measured pole figures (Fig. 17(a)).

8. In-plane pole figure measurement and texture analysis of ferroelectric thin film

Figure 19 shows pole figures of a PLT ((Pb, La)TiO₃) epitaxial layer (film thickness: 380 nm) on a Pt buffer layer (film thickness: 150 nm) epitaxially grown on MgO substrate, measured with the in-plane pole figure method. The 110 Bragg reflections of Pt and PLT are measured. In this measurement, the incident X-rays were monochromated to CuK α_1 using a Ge220 2-bounce channel-cut monochromator in the incident optics together with a parabolic multilayer mirror. Using only a parabolic multilayer mirror, the CuK α_1 and CuK α_2 lines are included and the intensity of K β line remains to the extent of 0.5% of the K α line. Furthermore, there are characteristic X-rays, such as W-L lines caused by contamination of W from filaments in the X-ray generator chamber. These characteristic X-

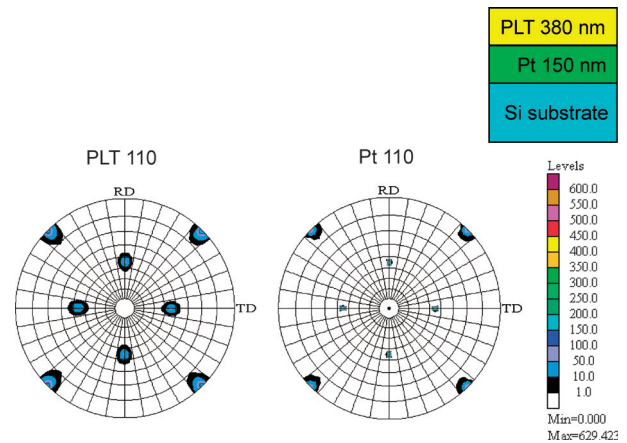


Fig. 19. 110 pole figure of PLT and Pt epitaxial film measured with in-plane pole figure measurement method.

rays other than the $K\alpha$ line are ambiguous because the intensity of diffraction from a single crystal substrate is very high. After introducing a Ge monochromator into the incident optics side, the incident X-rays are monochromatized to only the $CuK\alpha_1$ line. Therefore, diffraction by the $W-L$ line and $K\beta$ line are not observed. Employing a $K\beta$ filter will also work in the parallel beam optics with a parabolic multilayer mirror.

To analyze epitaxial films, the introduction of Ge monochromators in the incident optics is recommended to eliminate the signals caused by non- $CuK\alpha_1$ lines and to lower the background noises, though the peak intensity will be lowered by one order of magnitude. In contrast, when measuring polycrystalline film, the use of parallel beam optics only with a parabolic multilayer mirror is generally preferable, since it is necessary to collect weak signals.

PLT was treated as of cubic symmetry in this analysis. The positions of poles in the 110 pole figure shows that the crystal orientation texture is analyzed as $\{001\}\langle 100\rangle$ both for the Pt layer and the PLT layer with the aid of a standard stereo projection figure. It is supposed that PLT was epitaxially grown in Cube-on-Cube fashion on Pt/MgO because of the good lattice-matching of PLT to Pt and MgO. It is reported that a tetragonal phase is stable for PLT in La-poor composition. Reciprocal mapping is also used in order to evaluate domain and orientation relationship between substrate and single crystalline films⁽⁷⁾.

9. The employment of 2D detector for Pole Figure measurements

The previously mentioned pole figure measurement techniques, such as Schulz reflection, parallel beam reflection and in-plane pole figure measurement, are normally performed by changing the α and β angles using a zero-dimensional detector. The employment of two-dimensional detector allows us to collect signals in a certain range of α and β in a single frame of the picture. In other words, the pole figure measurement method using a two-dimensional detector enables us to obtain a pole figure by just measuring the intensity in the β direction at one χ angle (tilt angle). It is necessary to turn the X-ray beam into a point-like shape in order to suppress the umbrella effect for the effective use of a two-dimensional detector.

Advantages and disadvantages of pole figure measurement using two-dimensional detector are as follows;

Advantages:

- It is possible to expand the 2θ range measured at the same time by shortening the camera length, the distance from the sample to the face of the two-dimensional detector.
- It is possible to collect Debye rings from multiple diffraction planes simultaneously, and to create multiple pole figures from one measurement.
- The background can be measured simultaneously.

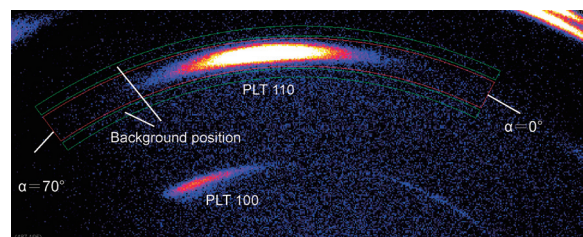


Fig. 20. Two-dimensional image of PLT/Pt thin film sample on Si substrate ($\chi=30^\circ$).

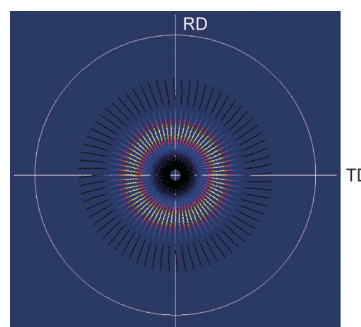


Fig. 21. The creation of PLT 110 pole figure using 2D image.

Thus, it is not required to move to the 2θ position of the background for the Schulz reflection method and in-plane pole figure method.

The greatest feature is the remarkable reduction in measurement time compared to the Schulz reflection method and the in-plane pole figure method performed with point detector, since it is not necessary to change the α angle.

Disadvantages:

- The α range measured in a pole figure depends on the 2θ position of lattice planes and χ angle.
- The α range measured in a pole figure is narrower than with the Schulz reflection method and parallel beam reflection method.

Figure 20 shows the two-dimensional image measured at $\chi=30^\circ$ for a film with a layer structure: PLT layer (film thickness: 400 nm) on a Pt buffer layer (film thickness: 150 nm) on Si substrate. The PLT film is not an epitaxial film but instead has a fiber texture. When creating a pole figure using a two-dimensional image, the range to cut out from the Debye ring (2θ , α and background position) should be determined. Then, the clipped Debye rings are arranged on the pole figure as shown in Fig. 21. Subsequently, the intensities on the pole figure are interpolated with 2.5 to 5° steps so that data points are placed equally spaced in the α and β directions.

10. Conclusion

This paper has explained data plotting and orientation analysis of pole figure data, and the features of several

pole figure measurement methods, data correction processing of intensity, absorption and background correction, and the use of two-dimensional detectors. The orientation texture can be analyzed from the pole figure data obtained by pole figure measurement. It is very useful and convenient to obtain a pole figure with a complete hemisphere by the in-plane pole figure measurement method where a sample is kept on the horizontal plane without tilting it.

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