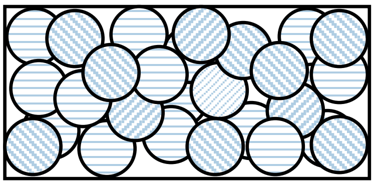
Texture / Preferred Orientation Analysis Group

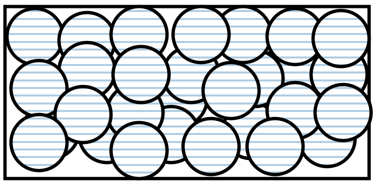
Contents

1. Overview

Polycrystalline materials are composed of numerous crystalline grains, which are generally found to be randomly oriented, as shown in Fig. 1.1 (a). But in certain cases, all crystalline grains are aligned in the same direction, as shown in Fig. 1.1 (b). When crystals are oriented in this way, a polycrystalline material is said to have *preferred orientation*.



(a) In random orientation



(b) In preferred orientation

Fig. 1.1 Orientations of polycrystalline materials (dotted lines indicate specific lattice planes)

If the lattice plane ($h\ k\ l$) of a certain crystalline grain with interplanar spacing d forms angle θ (Bragg angle) with respect to the incident x-ray beam to satisfy Bragg's formula, $2d\sin\theta = n\lambda$, the incident x-ray beam is diffracted by this lattice plane. The inclination of the diffracted ray is 2θ (the diffraction angle) with respect to the incident x-ray beam, equivalent to the sum of angle θ formed by the incident x-ray beam and the lattice plane and angle θ formed by the diffracted x-rays and the lattice plane (see Fig. 1.2).

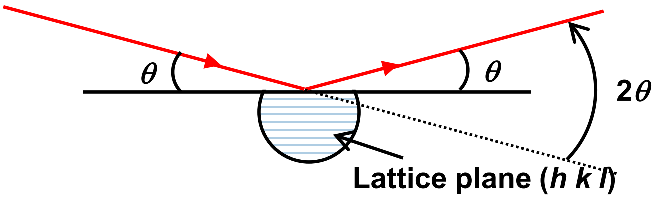
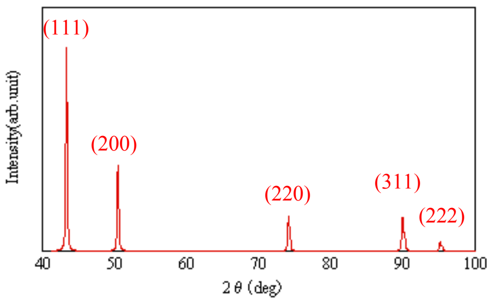


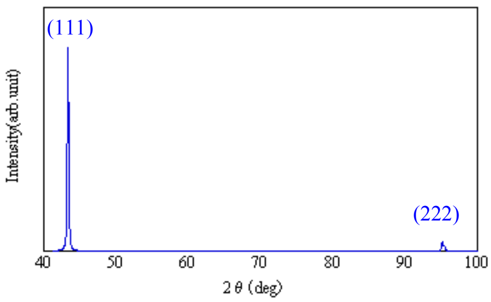
Fig. 1.2 X-ray diffracted by crystal

If the sample contains ample crystallites oriented randomly, for any given lattice plane a certain proportion of the crystallites will face whatever direction satisfies the diffraction condition. In ordinary diffractometry (theta/2-theta scan), many diffracted x-rays are observed.

Figure 1.3 shows the result of a theta/2-theta scan of a Cu sample. Graph (a) shows the diffraction pattern observed when the Cu crystalline grains are oriented randomly. Graph (b) shows the diffraction pattern observed when the Cu crystalline grains exhibit preferred orientation.



(a) In random orientation



(b) In preferred orientation

Fig. 1.3 Patterns of x-rays diffracted by polycrystalline materials

A theta/2-theta scan measures lattice planes parallel to the sample surface. If a large number of randomly-oriented crystallites are present, many diffracted rays of a certain relative intensity will be observed, due to the crystal structure of the substance, as shown in (a). In samples having preferred orientation, crystalline grains align in a certain direction. This means only specific diffracted x-rays can be observed. In the case of (b), only Cu [111] diffraction is observed, indicating that the sample surface has Cu [111] preferred orientation.

To a certain extent, it is also possible to determine the *presence/absence* of preferred orientation by performing a theta/2-theta scan (similar to the one shown in Fig. 1.3). “Pole figure measurement” and “preferred orientation/crystal quality measurement” (sometimes called “rocking curve measurement” in the field of polycrystalline materials) are representative methods for analyzing the preferred orientation type, directional relationships, and degree of orientation. Chapter 2 describes these measurement methods in detail.

2. Measurement principles

2.1 Pole figure measurements

In Fig. 2.1.1(a), x-ray diffraction by a crystal is indicated by red vectors. If we define the incident x-ray as $\boldsymbol{k}_0$ and the exiting x-ray as $\boldsymbol{k}_g$, the combined vector is $\boldsymbol{g}_{hkl}$. This is called the *reciprocal lattice vector*. The direction of $\boldsymbol{g}_{hkl}$ corresponds to the normal vector of the observed lattice plane *hkl*, and its length is $1/d$. For 2-theta/theta scans, which measures only lattice planes parallel to the sample surface, $\boldsymbol{g}_{hkl}$ has a constant orientation perpendicular to the sample surface, and we observe lattice planes with various d values (the length of $\boldsymbol{g}_{hkl}$ varies).

Pole figure measurements, on the other hand, are made using a constant diffraction angle, and the sample is rotated in all directions. Since the diffraction angle is fixed, the length of $\boldsymbol{g}_{hkl}$ remains constant. A semi-spherical pole figure is scanned by rotating the sample through (see Fig. 2.1.1 (b)) two preferred orientation axes: alpha (tilt) and beta (in-plane rotation). For a four-axis goniometer, alpha and beta correspond to the chi and phi axes, respectively.

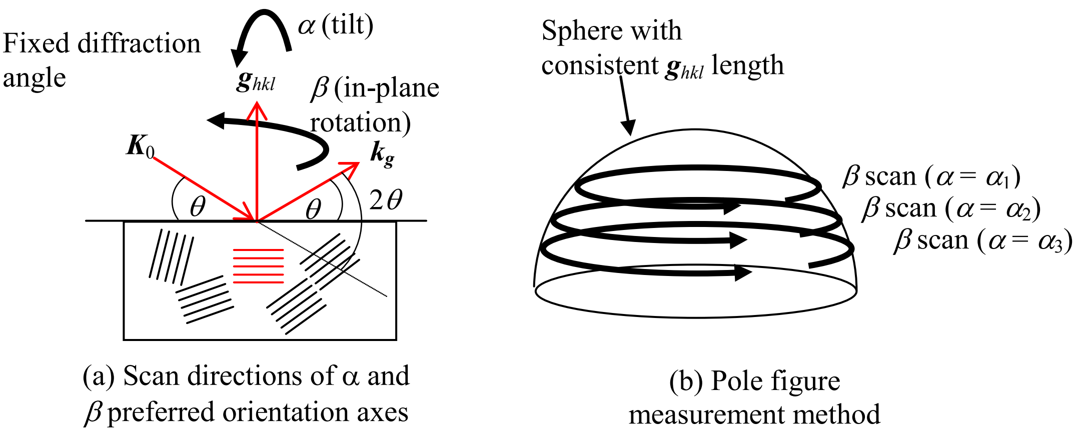


Fig. 2.1.1 Conceptual diagram of pole figure measurement

The measurement result is expressed by a pole figure, as shown in Fig. 2.1.2.

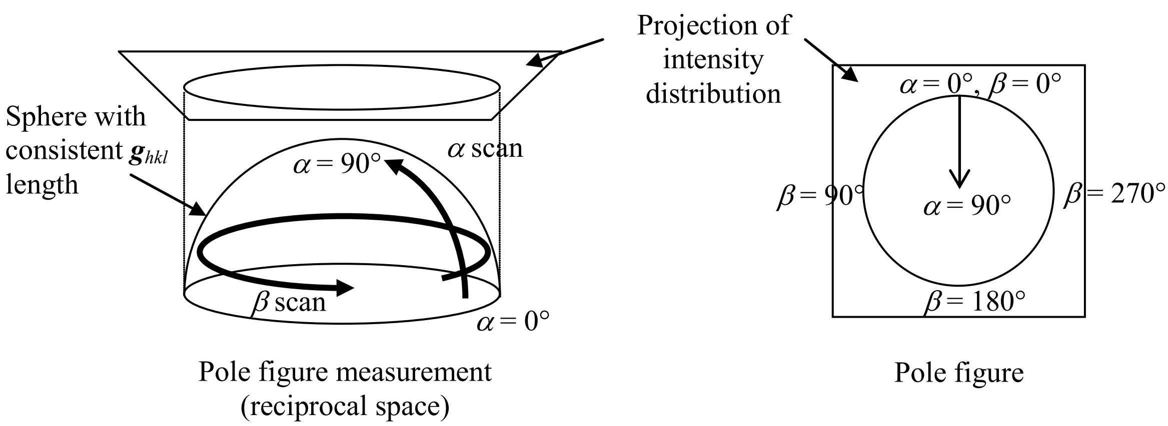


Fig. 2.1.2 Pole figure

As an example, let's examine three samples, each of which are composed of cubic crystals. The measurement of the (111) plane and (220) plane perpendicular to the (111) plane provides the results shown in Fig. 2.1.3:

Sample 1. Random orientation of crystals.
Diffraction intensity does not change with the sample orientation, and a flat intensity distribution is obtained.

Sample 2. (111) plane shows *fiber texture* aligned parallel to the sample surface.
High diffraction intensity is observed at the center (the point corresponding to the components parallel to the sample surface) in a pole figure of the (111) plane. This is because many crystallites satisfy the diffraction conditions in this orientation – i.e., their (111) planes are aligned parallel to the surface. In the case of cubic crystals, {111} ((-111), (11-1), (1-11) planes) are inclined 70.5° from the (111) plane, and all their diffraction angles are equal. Thus, we observe high diffraction intensity in the direction corresponding to alpha = 19.5°. However, since there is no preferred orientation in the plane of the sample (the in-plane), no distribution pattern is observed in the beta direction of the pole figure, result in a ring-shape intensity distribution. On the other hand, in the pole figure of the (220) plane, {220}, are inclined 35.3° and 90.0° from the (111) plane, provide diffraction intensity distribution rings at 90 minus those angles.

Sample 3. (111) plane consists of *single crystals* aligned parallel to the sample surface.
The (111) pole figure shows high diffraction intensity at the center and at = 19.5° (due to {111} planes' being inclined at 70.5°). In this case, the intensity distribution pattern in the β direction lacks a ring shape; the diffraction intensity is observed only for the specific direction of {111} for the single crystals (3. of Figure 2.1.3). Also, in the pole figure of the (220) plane, {220} planes inclined at 35.3° and 90.0° from the (111) plane provide the shown diffraction intensity distribution patterns.

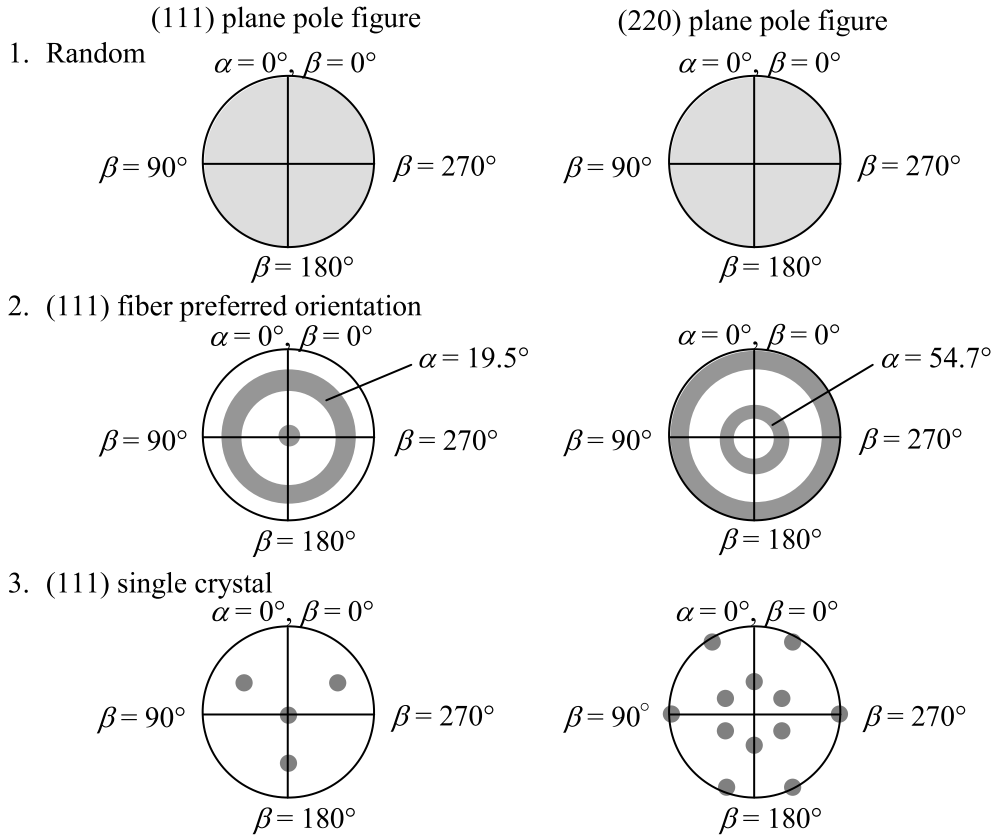


Fig. 2.1.3 Examples of pole figures

2.2 Method for measuring the pole figure

In pole figure measurements, the sample is rotated in various directions while the diffraction angle (2-theta axis, omega axis) remains fixed. Therefore, scanning is performed along the alpha and beta preferred orientation axes. There are two types of optical geometry to perform the pole figure measurement: the transmission method and the reflection method. In a transmission method, set the sample to the transmission geometry and use the omega axis and phi axis (in-plane rotation) of a four-axis goniometer. Also, in a reflection method, set the sample to the reflection geometry and use the chi axis (tilt) and phi axis (in-plane rotation) of a four-axis goniometer (however, an attachment for transmission method such as the $\alpha\beta$ attachment is required). As shown in Fig. 2.1.2, the outer area of the pole figure (alpha=0° to 30°) for the transmission method, or the center area of the pole figure (alpha=15° to 90°) for the reflection method can be measured. Fig. 2.2.1 shows the measurement geometry of transmission method and reflection method.

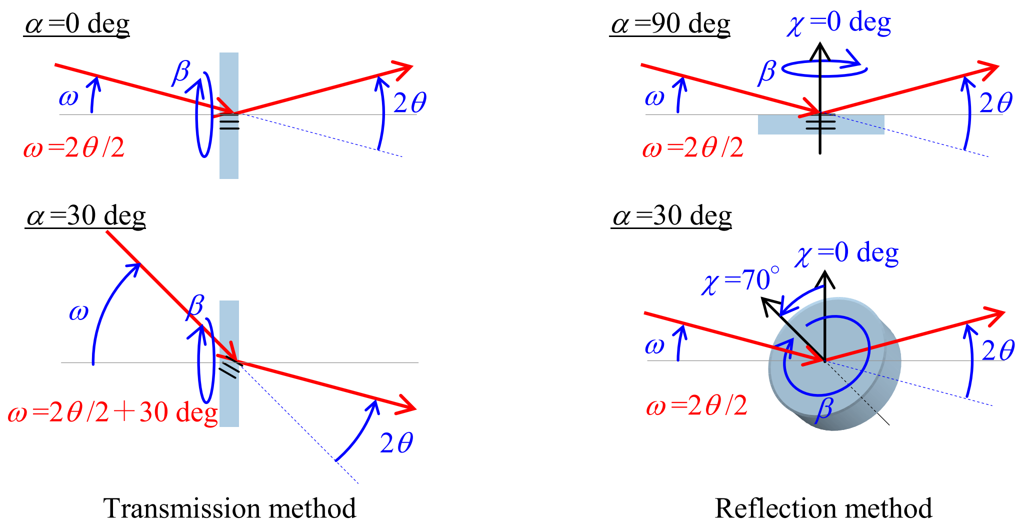


Fig. 2.2.1 Measurement geometry of pole figure measurement (transmission method and reflection method)

There are two methods for transmission method: the Decker method which uses the parallel beam, and the Schulz method which uses the divergent beam. Also, in reflection method, there is the Schulz method which uses the Schulz slit to minimize the longitudinal direction of X-ray divergence. Generally, use the Decker method for transmission method and Schulz method for reflection method to perform the pole figure measurement. (However, an attachment compatible with the Schulz slit such as $\alpha\beta$ or $\chi\phi$ attachment is required).

In the reflection method, since the diffraction angle in pole figure measurements remains constant (omega and 2-theta axes are fixed), the direction of the scattering vector (lattice plane vector) is constant. However, the direction of the surface normal vector changes as the chi axis is scanned (see Fig. 2.2.2). When the preferred orientation alpha axis approaches 0°, the x-ray beam strikes the side of the sample, and thus performing the measurement becomes difficult. In addition, the transmission method cannot be applied to thin film samples which the film is formed on the substrate or thick bulk samples, and thus it is virtually impossible to perform whole pole figure measurements in the range alpha = 0° to 90° by the reflection method only.

This limitation can be removed by performing in-plane pole figure measurements (see Fig. 2.2.3). In-plane measurements use the 2-theta-chi axis (in-plane axis) perpendicular to the 2-theta axis. The direction of the surface normal remains constant, while the 2-theta, omega, and 2-theta-chi axes are used to produce scattering vectors in differing directions. Note again that the scattering vector magnitudes remain constant.

In Fig. 2.2.2, when alpha = 90°, the scattering vector and surface normal vectors are pointing the same direction. As in the normal pole figure, the 2-theta-chi axis is at 0° and the omega axis and 2-theta axis are positioned at arbitrary angles. When alpha = 0°, the scattering vector is perpendicular to the normal vector, allowing lattice planes perpendicular to the sample surface to be measured. In the alpha = 0° case, the omega axis and 2-theta axis are at 0°, and the 2-theta-chi axis is positioned at an arbitrary angle. This in-plane geometry allows us to measure whole pole figures, including lattice planes perpendicular to the surface (using the 2-theta axis at the total reflection critical angle). In addition, in contrast to ordinary pole figure measurements, in-plane pole figure measurements do not require the sample to be tilted. This eliminates the risk of dropping the sample when using a horizontal sample mount goniometer like SmartLab.

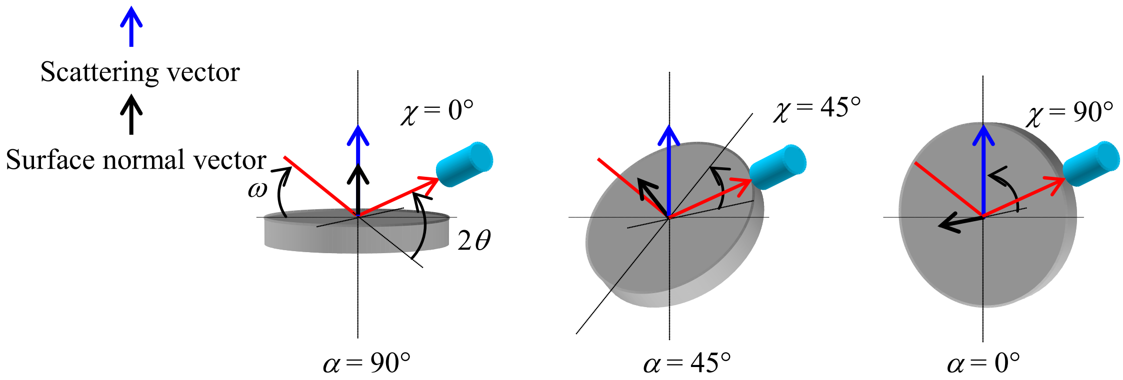


Fig. 2.2.2 Normal pole figure measurement

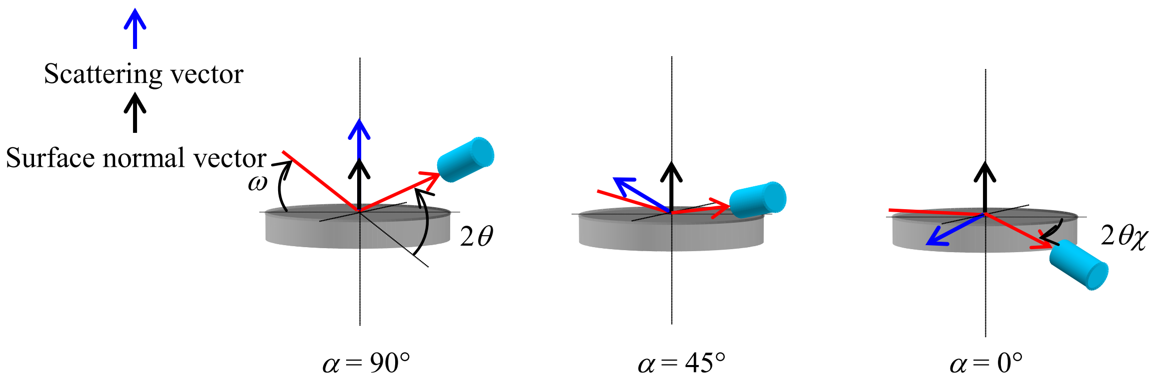


Fig. 2.2.3 In-plane pole figure measurement

2.3 Preferred orientation/crystal quality measurement (polycrystalline rocking curve)

Measuring the spread (width) of a cross-section of a pole figure intensity distribution pattern – that is, alpha scan (omega or chi scan) and beta scan (phi scan) – enables us to analyze of the degree of preferred orientation and the mosaic spread.

The degree of preferred orientation is a value indicating the degree to which crystallite prefer a given orientation, as compared to a randomly oriented sample. For example, when diffraction intensity is observed in the ranges of beta = 81° to 109° and beta = 261° to 279° in measurements of a sample having preferred orientation, the degree of preferred orientation can be obtained by the following formula.

(Spread of diffraction intensity)/360 = 36/360 = 10%

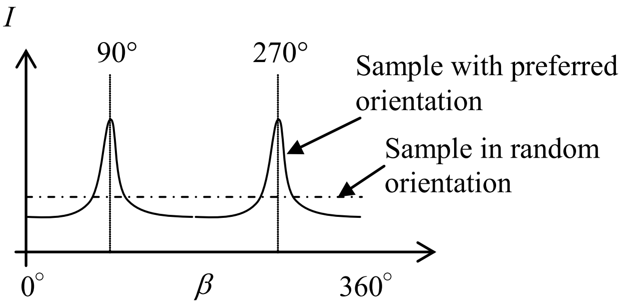


Fig. 2.3.1 Evaluation of degree of preferred orientation

On the other hand, the mosaic spread indicates the deviation of preferred orientation compared to perfect single crystals. No established quantitative definition exists for mosaic spread. If the diffraction intensity of a sample spreads ±0.5° from its center, the crystal orientation of the sample is deemed to have a deviation in the range of 1°, and the mosaic spread is said to be 1°.

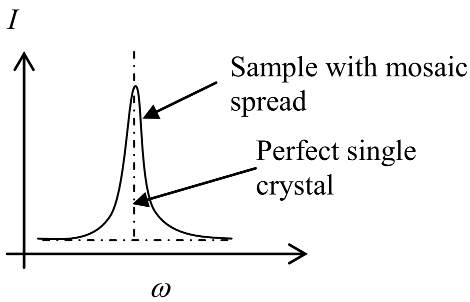


Fig. 2.3.2 Evaluation of mosaic spread

3. Selection of Package measurement

The Texture / Preferred Orientation Analysis Group includes three Package measurements: **Pole Figure (medium resolution PB)**, **In-Plane Pole Figure (medium resolution PB)** and **Transmission/Reflection Pole Figure (Bragg-Brentano focusing)**. Select a Package measurement based on the standards given in Table 3.1.

Table 3.1 Guidelines for selecting a Package measurement

Name of Package measurement	Guidelines for selecting a Package measurement
Pole Figure (medium resolution PB)	This Package measurement is used for pole figure measurements with the chi axis. Since it is capable of producing higher intensities than **In-Plane Pole Figure (medium resolution PB)** Package measurement, select this Package measurement when performing measurements that require higher intensity.
In-Plane Pole Figure (medium resolution PB)	This Package measurement is used for pole figure measurements using the 2-theta-chi axis. Since it is capable of performing whole pole figure measurements, generally, it is recommended to select this Package measurement when using a goniometer equipped with an in-plane axis.
Transmission/Reflection Pole Figure (Bragg-Brentano focusing)	This Package measurement is used for transmission pole figure measurements (option: $\alpha\beta$ attachment is required) with the sample transmission geometry using the omega axis, and also for reflection pole figure measurements with sample reflection geometry using the chi axis. Since it is compatible with reflection measurement by Schulz slit, select this Package measurement especially when obtaining the data of three dimensional orientation analysis (evaluation of orientation components, its dispersed states and its volume fraction).

Described below are the functions of the Parts included in each Package measurement.

3.1 Pole Figure (medium resolution PB) Package measurement

(1) Optics Alignment (medium resolution PB)
Performs direct beam alignment for slit collimation optics.

(2) Sample Alignment
Performs direct beam half cut alignment using a sample.

(3) Pole Figure Measurement
Performs pole figure measurement using the chi axis (tilt axis), as shown in Fig. 2.2.2.

3.2 In-Plane Pole Figure (medium resolution PB) Package measurement

(1) Optics Alignment (medium resolution PB)
Performs direct beam alignment for slit collimation optics. For in-plane pole figure measurements, performs 2-theta-chi axis alignment using the direct beam, since the 2-theta-chi axis (in-plane axis) is used for in-plane pole figure measurement.

(2) In-Plane Sample Alignment
Performs direct beam half cut alignment and in-plane total reflection alignment (surface normal alignment) using a sample.

(3) In-Plane Pole Figure Measurement
Performs pole figure measurement using the 2-theta-chi axis (in-plane axis), as shown in Fig. 2.2.3.

3.3 Transmission/Reflection Pole Figure (Bragg-Brentano focusing) Package measurement

(1) Optics Alignment (Bragg-Brentano focusing)
Performs direct beam alignment for para-focusing optics.

(2) Sample Alignment (Bragg-Brentano focusing)
Performs direct beam half cut alignment when using the wafer sample plate or the sample holder for $\alpha\beta$ attachment.

> Tip: No sample alignment is required if the sample is first placed in a glass sample holder or Al sample holder then inserted into the height reference sample plate.

(3) Transmission/Reflection Pole Figure Measurement
Performs the transmission pole figure ($\alpha\beta$ attachment is required) and the reflection pole figure measurement, as shown in Fig. 2.2.1.

～The optical system and hardware configuration to obtain the data suitable for three dimensional orientation analysis ~

The evaluation of samples with texture (preferred orientation) can be performed by the results indicated in the pole figure. Although the major crystal orientation component can be seen from the pole figure shown by two dimensional display, the quantitative information (how much crystal exist in which direction) cannot be obtained. The three dimensional orientation analysis is an analysis method which evaluates the orientation components of crystal, its dispersed states and its volume fraction by using a three angle variable function (Crystallite Orientation Distribution Function: ODF) based on the pole figure data obtained from the measurement. Methods such as the series expansion method and vector method are proposed.[1] [2] [3] To obtain a data suited for analysis, it is necessary to cover as much range of the pole figure as possible with an accurate intensity.

Especially to perform a measurement with an accurate intensity, keep in mind for the measurement range to not let the X-ray irradiate outside of the sample. However, for the reflection method which uses the chi axis as the step axis (tilt axis), the X-ray irradiation area dramatically increases in the area where alpha is around 15° ~30°, due to the width of longitudinal direction of the X-ray. Therefore, an optical system and device configuration to minimize the width of the longitudinal direction are required.

To obtain a data suited for three dimensional orientation analysis, the following optical system and device configuration are recommended.

- When the attachment base is standard Z stage
  - $\alpha\beta$ attachment (option: to use Schulz slit in reflection method)
  - $\chi\phi$ attachment (option: to use Schulz slit in reflection method)
  - ＊ Furthermore, the $\alpha\beta$ attachment is compatible with transmission measurement, and thus is able to perform measurement in broader range
- When the attachment base is standard χ cradle
  - MA optical system (option: to use length limiting slit 0.5 mm in reflection method)

[1] R. J. Roe: J. Appl. Phys., **36** (1965), 2024
[2] H. J. Bunge: Texture Analysis in Materials Science, Butterworths, (1982)
[3] D.Ruer and R. Baro: Adv. X-ray Anal., 20 (1977), 187