

Phase ID and Structural Analysis from Powders Group

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1. Overview

Most solid substances exist in a crystalline state. Many are comprised of aggregates of extremely fine crystal particles. These materials are called *polycrystalline substances*. The study of polycrystalline substances using x-rays is known as *x-ray powder diffractometry*.

Used primarily to measure crystals, x-ray powder diffraction can also be used to measure vapors, liquids, and non-crystalline solids in which molecules are arranged at sufficiently regular intervals. Since measured angle and diffraction intensity are unique to each crystal, x-ray analysis provides the following information about the sample structure.

- (1) The diffraction pattern varies with the crystal structure and composition of the sample. This makes it possible to **identify** (i.e., **perform qualitative analysis** of) a substance by comparing its measurements against known data. The x-ray powder diffraction method allows one to differentiate between polymorphic compounds of the same chemical composition, such as quartz, tridymite, cristobalite, and quartz glass (all of which have the same chemical formula: SiO_2).
- (2) We can measure crystal interplanar spacing. If the structure is known, we can obtain the **lattice constant** to an accuracy of about $\pm 0.1\%$ in some cases.
- (3) Measuring the intensity of diffraction permits **quantitative analysis** of individual components. However, obtaining accurate quantitative values becomes difficult if the components are present in minute quantities or if the sample has a preferred orientation.
- (4) Measuring the spread of diffracted rays lets us assess **crystallite size and lattice distortion**.
- (5) **Degree of crystallinity**. The diffraction pattern of an amorphous sample shows broad peaks (halos). Based on this diffraction pattern, we can obtain a **radial distribution pattern**. Based on the ratio of integrated intensity values of crystalline components and non-crystalline components, we can obtain the degree of crystallinity.
- (6) Rietveld analysis permits **precise analysis of crystal structures**.

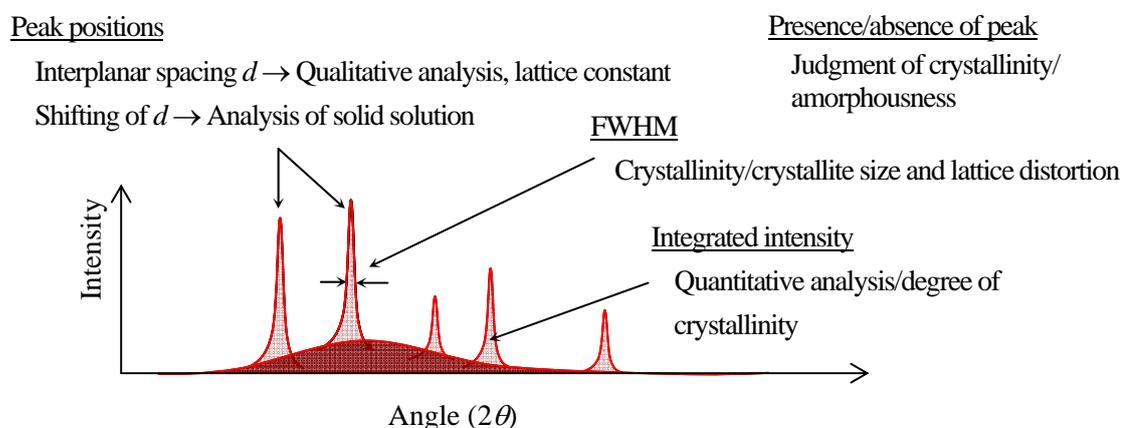
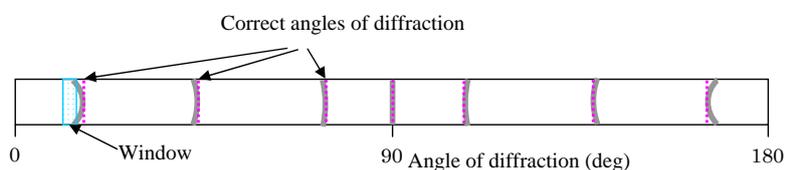


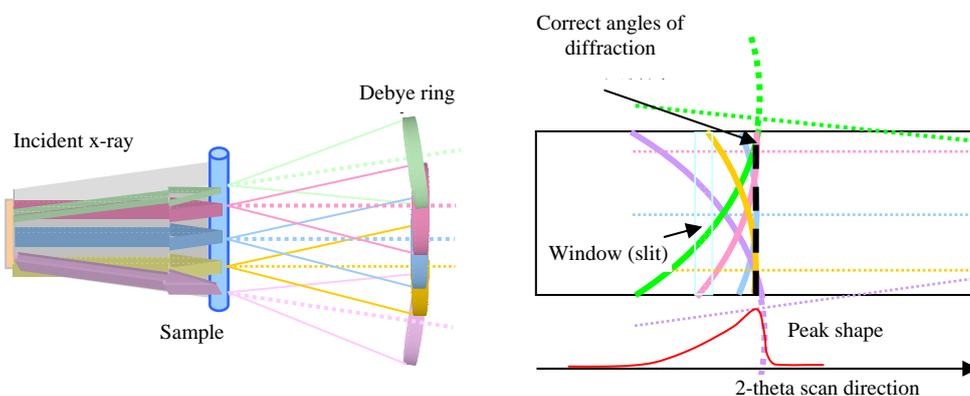
Fig. 1.1 Information provided by x-ray powder diffraction pattern and analysis

Umbrella effect

When a monochromatized x-ray beam irradiates a powder sample, circles of diffracted rays concentric around the axis of the incident x-ray can be observed. These circles are called Debye rings. Since a detector such as a scintillation counter is generally used for x-ray powder diffraction measurements, we can obtain diffraction patterns by scanning parts of the Debye rings through a “window” of a certain size. This means x-rays are observed even at locations where the angle of diffraction is not correct and does not satisfy Bragg’s diffraction condition, as shown in the diagram below. When the angle of diffraction is less than 90° , x-rays are observed on the low-angle side of the correct angle of diffraction. When the angle of diffraction is greater than 90° , x-rays are observed on the high-angle side.



Since diffraction devices such as SmartLab use a line-focus beam, the rays diffracted by a given plane index spread in the longitudinal direction from the line-focus beam, as shown in the following schematic diagram. This results in significant asymmetry of the peak displays. This is called the umbrella effect.



A Soller slit reduces the longitudinal divergence of incident x-rays and diffracted x-rays from the sample and suppresses the umbrella effect. Although smaller aperture angles are more effective, they increase the number of parallel plates and reduce x-ray intensity.

2. Measurement principles

Let's examine a case in which a monochromatic and parallel x-ray beam irradiates a powder sample. Consider a particle in the sample with lattice plane ($h k l$) and interplanar spacing d , which is tilted at angle θ (Bragg's angle) from the incident x-ray beam. This satisfies Bragg's formula, $2d\sin\theta = n\lambda$, and the incident x-ray beam is reflected by this lattice plane. The direction of the diffracted ray is inclined at an angle of 2θ (diffraction angle) from the incident beam, which is the sum of angle θ between the incident x-ray beam and the lattice plane and angle θ between the diffracted x-rays and the lattice plane (see Fig. 2.1 (a)).

If there are ample crystallites in the sample and they are randomly oriented, we will always encounter crystallites on any lattice plane oriented in the direction satisfying the diffraction condition. As shown in Fig. 2.1 (b), x-rays diffracted by the lattice plane ($h k l$) travel along the generator of a cone having a half-apex angle of 2θ when $2\theta < 90^\circ$ or a half-apex angle of $(180^\circ - 2\theta)$ when $2\theta > 90^\circ$. Similarly, x-rays diffracted by the lattice plane ($h' k' l'$) having a different interplanar spacing travel along the generator of a cone having a half-apex angle of $2\theta'$. In other words, x-rays diffracted by a powder sample form numerous cones with different center angles.

When such cones are captured on a flat film (see Fig. 2.2 (a)) or cylindrical film (see Fig. 2.2 (b)), the diffraction pattern shows concentric circles, with the incident x-ray beam at the center. These concentric circles are called Debye rings.

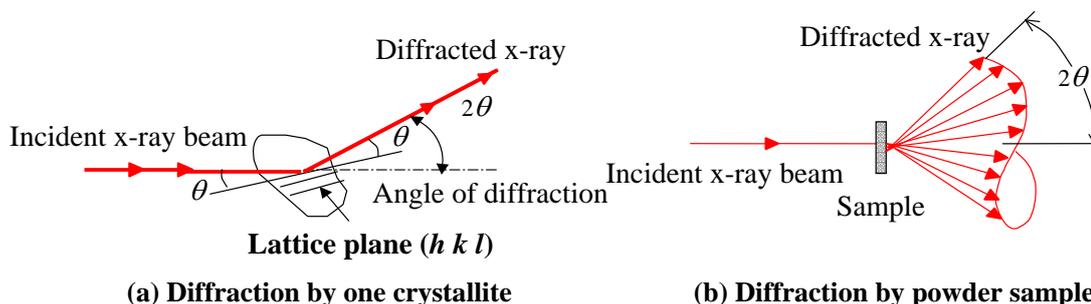


Fig. 2.1 X-ray diffraction by crystal

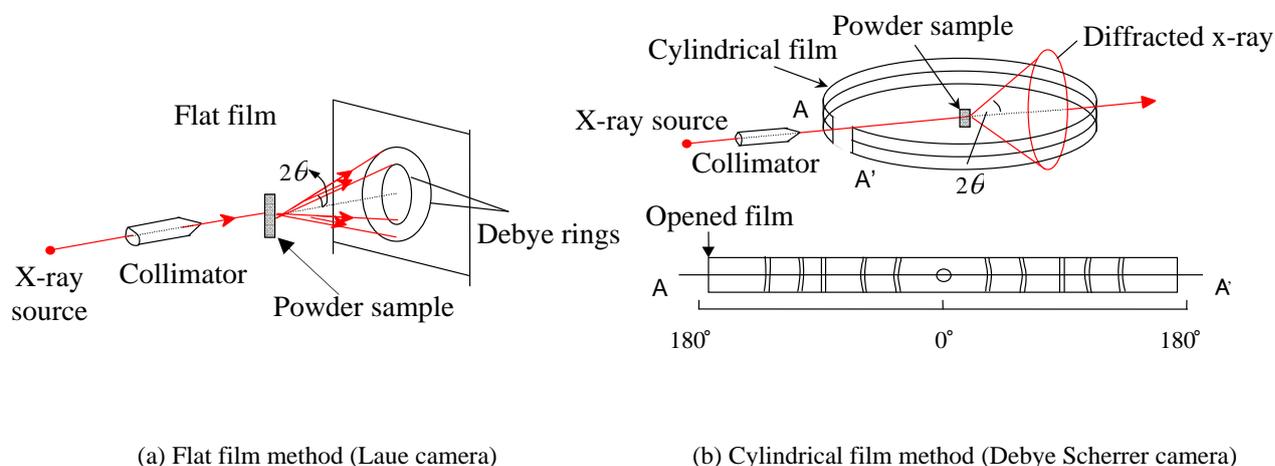


Fig. 2.2 X-ray powder diffraction method

2. Measurement principles

As shown in Fig. 2.3, an “x-ray diffractometer” is a device that measures and records the x-ray intensity of each Debye ring. It does this by scanning the detector along the circumference of a circle with the sample at the center. The output is x-ray intensity as a function of angle 2θ . We can obtain interplanar spacing d using Bragg’s formula, $2d\sin\theta = n\lambda$.

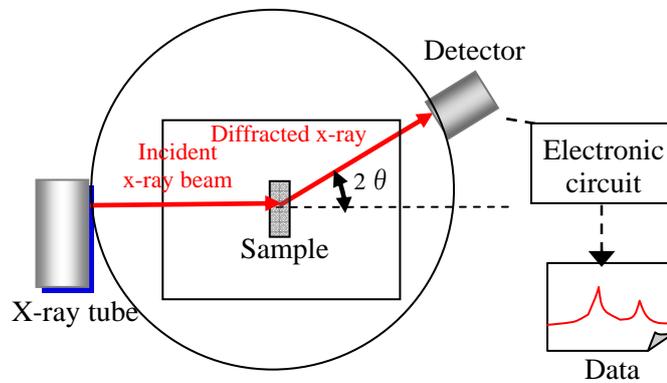


Fig. 2.3 Basic diffractometer principles

The optics used in the x-ray powder diffraction method are applied by the “para-focusing method” or the “parallel beam method.” The parallel beam method uses slits to generate parallel x-rays. The para-focusing method is more commonly used, because it achieves higher resolution and intensity than the parallel beam method.

2.1 Para-focusing optics

The para-focusing method assumes a “focusing circle” as shown in Fig. 2.1.1. The focusing circle is a virtual circle, which contacts the surface of the sample, and incorporates the following three points: the x-ray source, the center of goniometer rotation, and the receiving slit. The radius of the focusing circle varies depending on diffraction angle 2θ , as shown in Fig. 2.1.1.

For this method, the sample is pulverized into a fine powder, packed onto a sample plate, then placed so that the sample surface contacts the focusing circle. When x-rays emerge from the x-ray source (located on the focusing circle) and strike the sample, they are diffracted by the sample and converge at another point on the circle (the focusing point: where the receiving slit is positioned). To achieve this, the diffractometer must satisfy two conditions:

- (1) The distance between the x-ray source and the center (sample surface) of goniometer rotation must be equal to the distance between the center of goniometer rotation and the receiving slit (this length is called the **goniometer radius**).
- (2) The ratio of the angle (θ) formed by the incident x-ray beam and the sample surface to the angle (2θ) formed by the incident x-ray beam and the diffracted x-ray remains 1:2.

Optical systems meeting conditions (1) and (2) above are known as “Bragg-Brentano para-focusing optics.”

To satisfy condition (2) precisely, the sample must have the same surface curvature as the focusing circle. However, even when the sample has a flat surface, this condition also applies approximately. For this reason, flat samples can be used with the diffractometer.

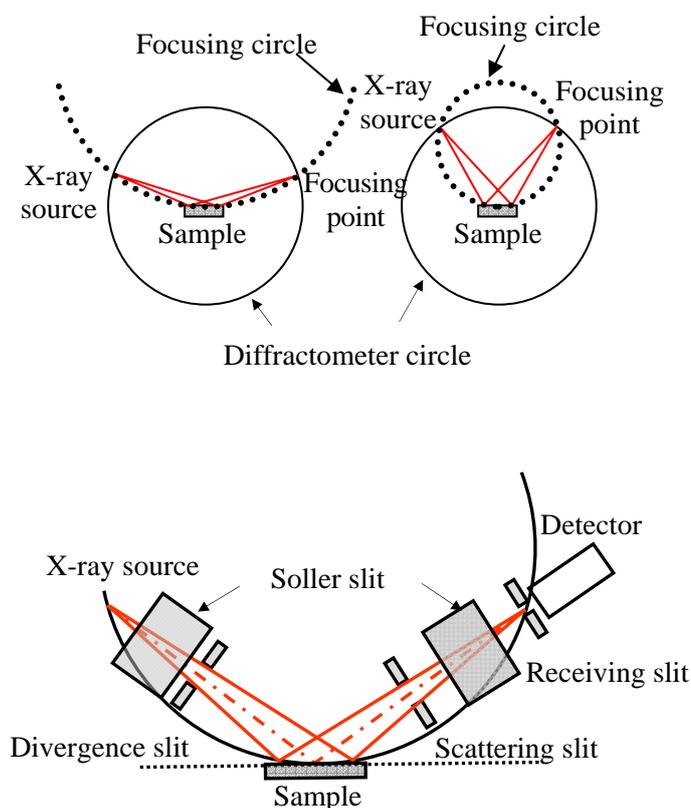


Fig. 2.1.1 Para-focusing optics

The Bragg-Brentano para-focusing optics involve a Soller slit and divergence slit (DS) on the incident side and a scattering slit (SS), Soller slit, and receiving slit (RS) on the receiving side.

The incident x-ray beam diverges not only along the scattering plane direction, but in a direction perpendicular to the scattering plane (this is hereafter referred to as “axial divergence”). The diffracted x-rays spread at an angle, a phenomenon known as the umbrella effect. At $2\theta = 90^\circ$, the Debye rings are perpendicular to the RS, so the umbrella effect does not cause deviations of the diffracted x-rays. When 2θ moves further along the low-angle side (or high-angle side), the diffracted x-rays spread toward the low-angle side (or high-angle side). Soller slits restrict the axial divergence of incident x-rays and diffracted x-rays. They consist of many thin metal plates configured in parallel with very narrow gaps. Fig. 2.1.2 shows that, when x-rays from the oblong focal point enter the Soller slit, the slit suppresses the axial divergence of the incident x-rays. The Soller slit on the receiving side extracts sections of the Debye rings.

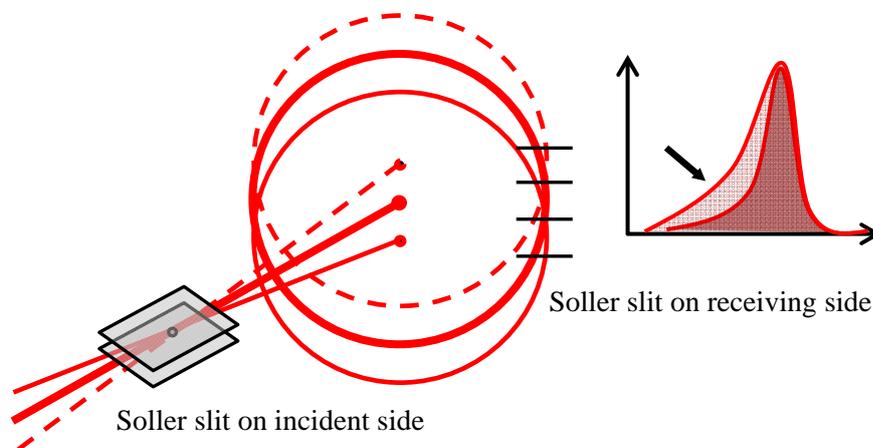


Fig. 2.1.2 Axial divergence of Debye rings

2.2 Parallel beam optics

In the parallel beam method, incident x-rays are parallelized using monochromator crystals, extra-fine slits, parallel slits or the like. Similarly, the x-rays diffracted from the sample are parallelized using a parallel slit analyzer (PSA) or crystal analyzer (see Fig. 2.2.1). Since this method is geometrically unconstrained by sample shape or optics, it is often used to measure thin-film samples or when performing measurements with a collimator. But since the x-ray source is a divergent light source, parallelizing the beam reduces intensity.

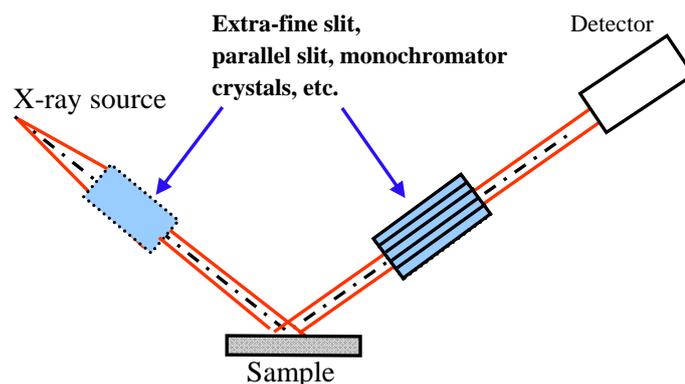


Fig. 2.2.1 Parallel beam optics

3. Selection of Package measurement

The Phase ID and Structural Analysis from Powders Group includes the following eight Package measurements: **Quick Theta/2-Theta Scan (Bragg-Brentano focusing)**, **Quick Theta/2-Theta Scan (Bragg-Brentano focusing) D/teX**, **Precise Theta/2-Theta Scan (Bragg-Brentano focusing)**, **General (Bragg-Brentano focusing)**, **General (Bragg-Brentano focusing) D/teX**, **Quick Theta/2-Theta Scan (medium resolution PB/PSA)**, **Precise Theta/2-Theta Scan (medium resolution PB/PSA)**, and **General (medium resolution PB/PSA)**. The application of each Package measurement is as follows:

Table 3.1 Guidelines for selecting a Package measurement

Package measurement	Guideline for selecting a Package measurement	Remarks
Quick Theta/2-Theta Scan (Bragg-Brentano focusing)	These Package measurements are used to determine crystalline or amorphous states, to detect trace components, to perform phase ID analysis, and to make pre-measurements for other analyses.	The incident slit and receiving slit widths determine the optical resolution.
Quick Theta/2-Theta Scan (Bragg-Brentano focusing) D/teX	Select Quick Theta/2-Theta Scan (Bragg-Brentano focusing) D/teX if D/teX Ultra is used as a detector.	
Precise Theta/2-Theta Scan (Bragg-Brentano focusing)	This Package measurement is used to analyze the intensity or width of diffracted x-rays quantitatively – for example, for quantitative analysis or precision measurement of lattice constants, crystallite size, percent crystallinity, or crystal structure.	

**General
(Bragg-Brentano
focusing)**

These Package measurements are used to measure a divided profile using para-focusing optics.

**General
(Bragg-Brentano
focusing) D/teX**

Select **General (Bragg-Brentano focusing) D/teX** if D/teX Ultra is used as a detector.

**Quick
Theta/2-Theta
Scan (medium
resolution
PB/PSA)**

This Package measurement is used to determine crystalline or amorphous states, phase ID analysis, and pre-measurements for other analyses. This Package measurement is effective for measurements of samples for which the para-focusing method can generate errors (samples that are not easily crushed or molded, samples with irregular surfaces or curvature, samples with strong preferred orientation, etc.)

The aperture angle of the parallel slit analyzer (PSA) determines the optical resolution. These Package measurements can also be used for symmetric transmission measurement and asymmetric measurement (2-theta scan) with a fixed incidence angle.

**Precise Theta/
2-Theta Scan
(medium
resolution
PB/PSA)**

This Package measurement is used to quantitatively analyze the intensity or width of diffracted x-rays – for example, for quantitative analysis and precision measurement of lattice constants, crystallite size, percent crystallinity, or

**General
(medium
resolution
PB/PSA)**

crystal structure.

This Package measurement is used to measure a divided profile using parallel beam optics.

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

3.1 Quick Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement

- (1) Optics Alignment (BB)
Performs direct beam alignment for para-focusing optics.
- (2) Sample Alignment (BB)
Performs direct beam half-cut alignment when using a wafer sample plate.



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) Quick Theta/2-Theta Measurement (BB)
Determines the scan range and slit based on the sample type, then performs a theta/2-theta scan.

3.2 Quick Theta/2-Theta Scan (Bragg-Brentano focusing) D/teX Package measurement

- (1) Optics Alignment (BB)
Performs direct beam alignment for para-focusing optics.
- (2) Si Peak Measurement
Measures the diffraction peaks from Si using the Si powder reference sample.



Tip: Replace the scintillation counter with D/teX Ultra after the Si peak measurement.

- (3) D/teX Detector Center Correction
Measures the diffraction peaks from Si using D/teX Ultra in the same way as step (2). After the measurement, the center of the detector plane of D/teX Ultra will be determined from the difference between the Si diffraction angles obtained in steps (2) and (3).
- (4) Quick Theta/2-Theta Measurement (BB)
Determines the scan range and slit based on the sample type, then performs a theta/2-theta scan.

3.3 Precise Theta/2-Theta Scan (Bragg-Brentano focusing) Package measurement

- (1) Optics Alignment (BB)
Performs direct beam alignment for para-focusing optics.
- (2) Sample Alignment (BB)
Performs direct beam half-cut alignment when using a wafer sample plate.



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) Precise Theta/2-Theta Measurement (BB)
The important slit for quantitative analysis is determined based on the scan range, sample width, and sample height. The FWHM and intensity of the sample determine the optimum scan step width and duration, and a theta/2-theta scan is performed.

3.4 General (Bragg-Brentano focusing) Package measurement

- (1) Optics Alignment (BB)
Performs direct beam alignment for para-focusing optics.
- (2) Sample Alignment (BB)
Performs direct beam half cut alignment when using a wafer sample plate.



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) General Measurement (BB)
Performs the scan using para-focusing optics and based on conditions (including tube voltage and tube current settings) set by the user.

3.5 General (Bragg-Brentano focusing) D/teX Package measurement

- (1) Optics Alignment (BB)
Performs direct beam alignment for para-focusing optics.
- (2) Si Peak Measurement
Measures the diffraction peaks from Si using the Si powder reference sample.



Tip: Replace the scintillation counter with D/teX Ultra after the Si peak measurement.

- (3) D/teX Detector Center Correction
Measures the diffraction peaks from Si using D/teX Ultra in the same way as step (2). After the measurement, the center of the detector plane of D/teX Ultra will be determined from the difference between the Si diffraction angles obtained in steps (2) and (3).
- (4) General Measurement (BB)
Performs the scan using para-focusing optics and based on conditions (including tube voltage and tube current settings) set by the user.

3.6 Quick Theta/2-Theta Scan (medium resolution PB/PSA) Package measurement

- (1) Optics Alignment (PB/PSA)
Performs direct beam alignment for parallel beam optics using a PSA.
- (2) Sample Alignment (PB/PSA)
Performs direct beam half cut alignment when using a wafer sample plate.



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) Quick Theta/2-Theta Measurement (PB/PSA)
Determines the scan range and slit based on the sample type and shape, and performs a theta/2-theta scan.

3.7 Precise Theta/2-Theta Scan (medium resolution PB/PSA) Package measurement

- (1) Optics Alignment (PB/PSA)
Performs direct beam alignment for parallel beam optics using a PSA.
- (2) Sample Alignment (PB/PSA)
Performs direct beam half cut alignment when using a wafer sample plate.



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) Precise Theta/2-Theta Measurement (PB/PSA)
The important slit for quantitative analysis is determined based on the scan range, sample width, and sample height. The FWHM and intensity of the sample determine the optimum scan step width and duration, and a theta/2-theta scan is performed.

3.8 General (medium resolution PB/PSA) Package measurement

- (1) Optics Alignment (PB/PSA)
Performs direct beam alignment for parallel beam optics using a PSA.
- (2) Sample Alignment (PB/PSA)
Performs direct beam half cut alignment when using a wafer sample plate.



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) General Measurement
Performs the scan using parallel beam optics and based on conditions set by the user.

4. Supplementary information

4.1 Preparation of samples for x-ray powder diffractometry

X-ray diffractometry is used for a wide range of samples, both crystalline and amorphous. Among these sample types, polycrystals can be classified as powder samples or bulk samples based on their physical form.

Samples appropriate for x-ray powder diffractometry are ultrafine crystals, so this analysis is generally undertaken on the assumption that the orientation of crystals in the sample is free of any particular bias. However, certain powder samples have cleavability, while many bulk samples have textures. This means the above assumption is not met in certain cases.

Described below are the methods for preparing ordinary samples.

(1) Powder samples

The size of the particles in powder samples significantly affects diffraction intensity. Generally, a particle size of about 10 μm is considered ideal in terms of diffraction intensity reproducibility. If the crystal grains are coarse, the Debye rings appear patchy and diffraction intensity reproducibility degrades. If the particle diameter is 30 μm or larger, the extinction effect reduces diffraction intensity. If the materials are crushable, we recommend pulverizing samples with a mortar or automatic crusher. Table 4.1.1 gives $\alpha\text{-SiO}_2$ particle diameters and diffraction intensity reproducibility. As this table indicates, the larger the particle diameter, the greater the relative standard deviation (diffraction intensity dispersion).



CAUTION: Note that in certain samples pulverization can change crystal structures, generate distortion, or cause changes in the amorphousness.

Table 4.1.1 Change in diffraction intensity reproducibility due to particle size (unit: counts)

Measu remen	Particle diameter
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
Avera	
Standa	
Relati	

The above table is based on data appearing on page p. 336 of *X-ray Diffraction Procedures*, 2nd Edition by H.P. Klug and L.E. Alexander, John Wiley & Sons, New York (1974).

(2) Bulk samples

Bulk samples include metals, sintered materials, polymers, and coagulated materials. Materials that can be pulverized should be ground into powder form. The flatness of the sample surface is a crucial factor in measurements based on the x-ray powder diffraction method, and it has a major effect, particularly when using the para-focusing method. Irregular or curved sample surfaces will generate phenomena such as deviations in diffraction angles and deformation of diffracted rays (separation or spreading). For this reason, it is important to ensure that the sample surface flatness is at most 0.02 mm. If a sample that does not meet this condition, it is important to consider the effects on the diffraction pattern when analyzing the resulting data.

4.2 Setting measurement conditions

Diffraction patterns can vary greatly, depending on measurement conditions, even when measuring the same sample. It is important to select the most appropriate measurement conditions based on the purpose of the measurement and the sample type. To obtain good x-ray powder diffraction data, set the measurement conditions based on the ratio (P/B ratio) of peak intensity to background intensity, integrated intensity, FWHM, etc. Shown below is a method for setting conditions shared by all measurement applications, including measurements for qualitative and quantitative analyses.

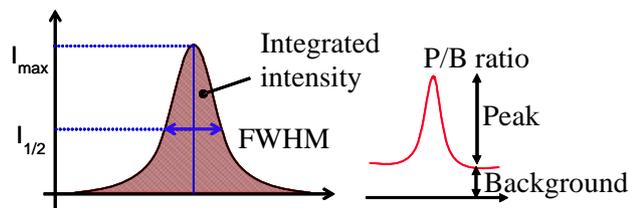


Fig. 4.2.1 Integrated intensity, FWHM, and P/B ratio

- (1) Set the conditions to elevate the P/B ratio.
If the background intensity is high relative to peak intensity, weak peaks will be difficult to detect.

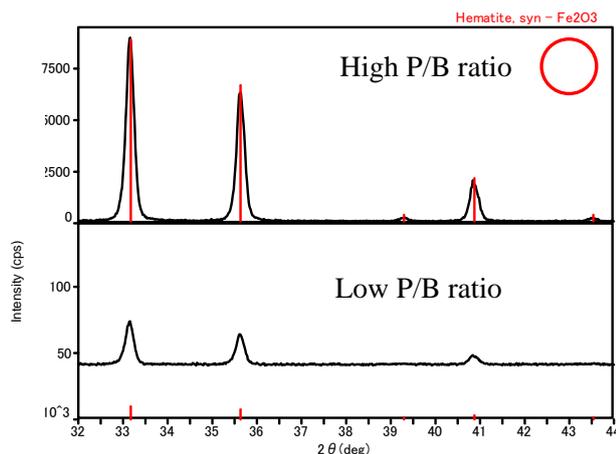


Fig. 4.2.2 P/B (peak/background) ratio

- (2) Set the conditions so that statistical counting errors will be negligible (high x-ray diffraction intensity).
If the data suffers from significant statistical counting errors, it will be difficult to obtain accurate diffraction angles, integrated intensities, and peak widths.

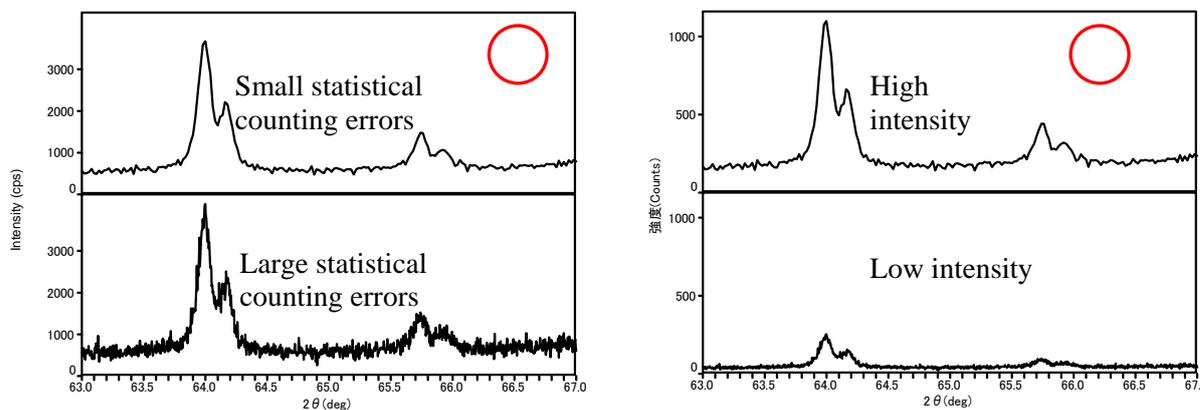


Fig. 4.2.3 Statistical errors for counting and x-ray diffraction intensity

- (3) Set conditions to increase resolution.

If the resolution of the data is low, it will be difficult to obtain accurate diffraction angles, integrated intensities, and peak widths.

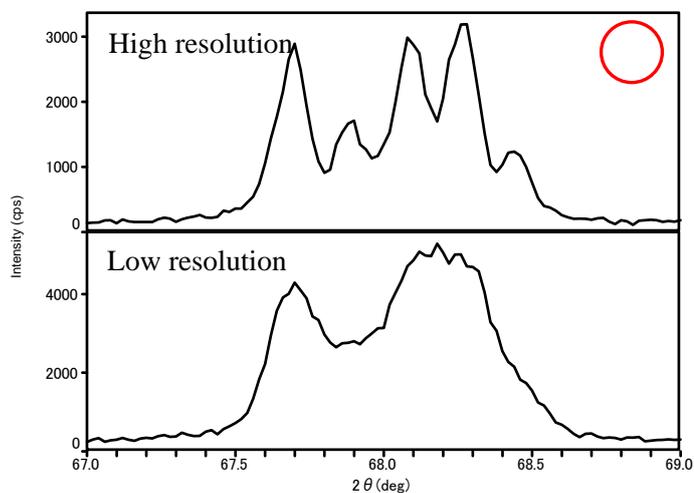
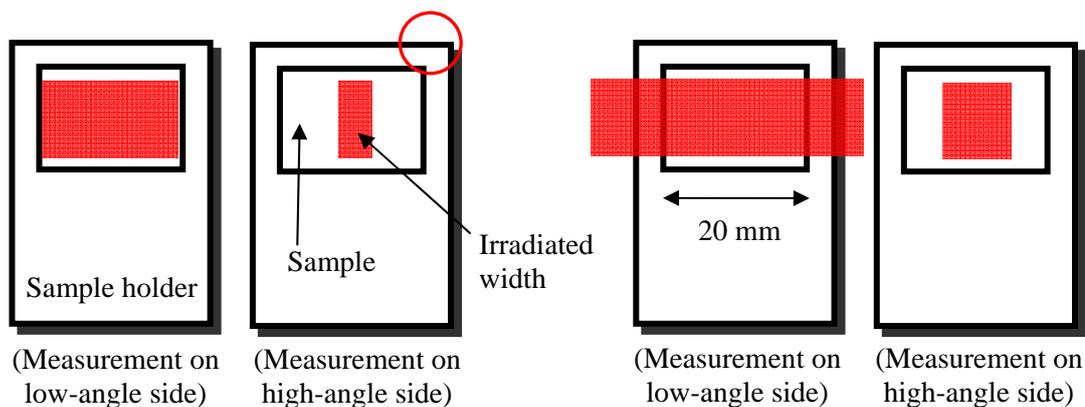


Fig. 4.2.4 Profile differences resulting from different resolutions

- (4) Ensure that the incident x-ray beam does not irradiate areas beyond the sample.

This is especially critical in analyses (quantitative analysis, percent crystallinity analysis, Rietveld analysis, etc.) for which the intensity ratio of diffracted x-rays is important. For symmetric reflection measurements (x-ray incidence angle is the same as the exit angle), the irradiated width of the incident x-ray beam varies with 2-theta.



(a) X-ray beam does not extend from edges on either low-angle or high-angle side.

(b) X-ray beam extends from edges on low-angle side.

Fig. 4.2.5 Diffraction angle 2-theta and irradiated area

Optics and irradiated width of incident x-ray beam

Both the para-focusing method and parallel beam method normally perform symmetric reflection measurements with theta/2-theta scans. However, the parallel beam method also enables symmetric transmission and asymmetric measurements with the fixed-incidence-angle 2-theta scan (omega fixed).



CAUTION: When determining the irradiated width resulting from the aperture angle of the incident slit, note that the goniometer radius (standard value: 300 mm) of SmartLab exceeds those in the previous Rigaku powder diffractometer models.

In the para-focusing method, the irradiated width W is expressed by the following formula, based on the aperture angle of the incident slit.

[Irradiated width of x-rays resulting from aperture angle of incident slit in para-focusing optics]

$$W(\text{mm}) = \left[\frac{1}{\sin\left(\theta + \frac{IS}{2}\right)} + \frac{1}{\sin\left(\theta - \frac{IS}{2}\right)} \right] R \sin\left(\frac{IS}{2}\right)$$

Where:

θ : = Diffraction angle $2\theta/2$ (deg.)

IS : Aperture angle of incident slit (deg.)

R : Goniometer radius (mm)

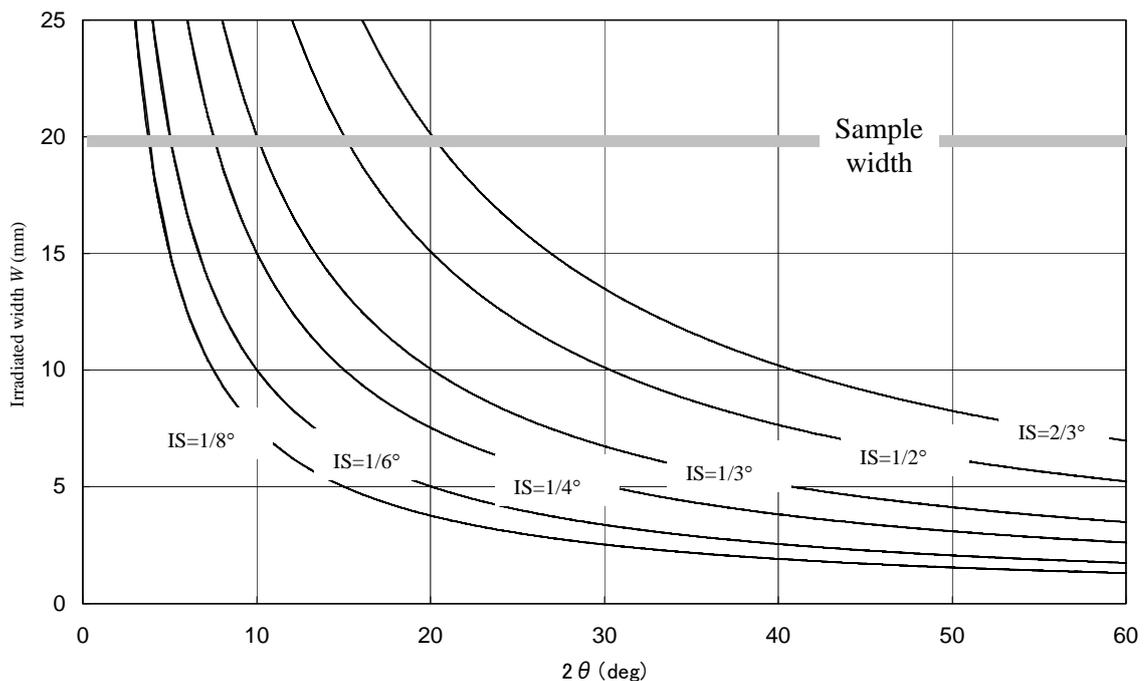


Fig. 4.2.6 Irradiated width of incident x-ray beam in para-focusing optics for goniometer radius of 300 mm

Since the parallel beam method uses a parallel x-ray beam approximately 1 mm wide, that exits from a multilayer mirror, it uses $IS = 1$ mm to remove scattered rays. RS1 and RS2 are opened to prevent blocking of the diffracted x-rays. Irradiated width W on the sample surface is expressed by the following equation:

[Irradiated width of x-rays resulting from aperture angle of incident slit in parallel beam optics]

$$W(mm) = \frac{IS}{\sin \theta}$$

θ : = Angle of diffraction $2\theta/2$ (deg.)

IS : Width of incident slit (mm). In general, "1 mm" is used for calculations.

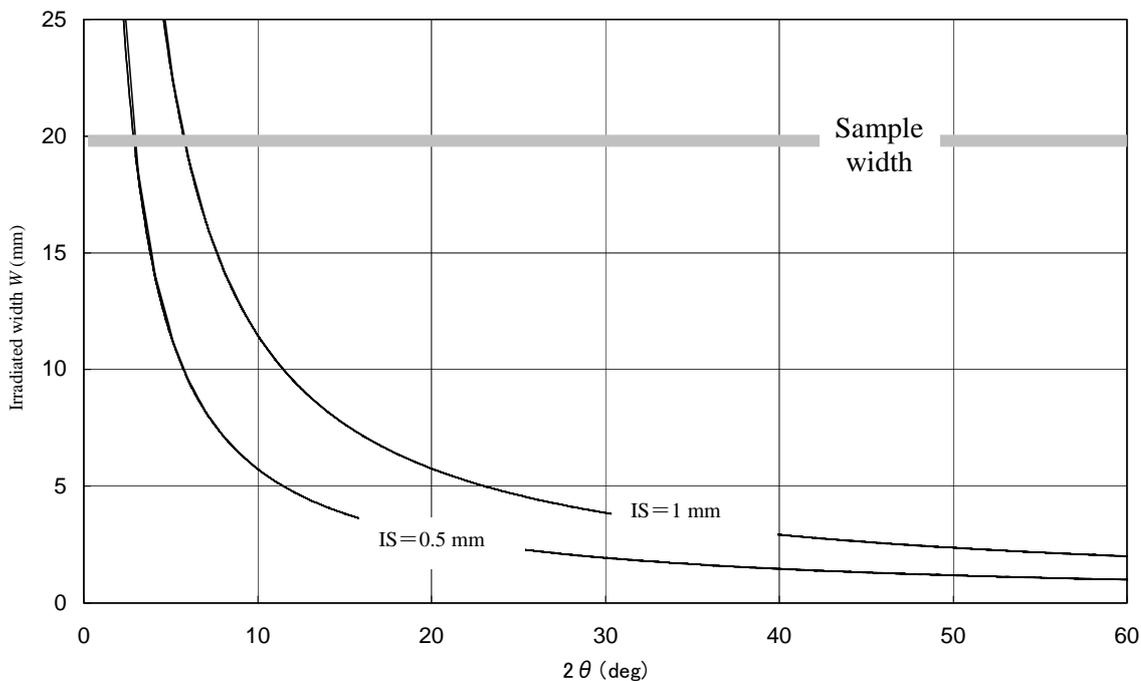


Fig. 4.2.7 Irradiated width of incident x-rays in parallel beam optics for goniometer radius of 300 mm

4.3 Measurements using parallel beam optics

Optical configuration is one of the most important elements in x-ray diffractometry. Various optical systems are available for different types of measurement. The para-focusing method is the most commonly used, since it provides high resolution and high intensity and is easy to use. It currently represents the standard optics for x-ray diffractometry.⁽¹⁾ While the para-focusing method is easy to use, it has the following drawbacks.⁽²⁾

- (1) The incidence angle cannot be set flexibly.
- (2) It is susceptible to optical aberrations, since flat samples are used and x-ray diffraction occurs inside samples.
- (3) The diffraction pattern may vary depending on the irregularity of the sample surface.
- (4) Information can be obtained only from crystallites whose diffraction surfaces parallel the sample surface.
- (5) The diffraction peaks tend to be asymmetrical.
- (6) Improving resolution requires reducing the width of the x-ray focal point and the divergence angle as much as possible while increasing the goniometer radius, which presents difficulties when accounting for intensity degradation and averaging sample crystals.

For the above reasons, we recommend using parallel beam optics for the following: thin-film samples requiring low-angle incidence, samples with strong preferred orientation, samples that cannot be pulverized or molded, profile analyses such as the Rietveld method⁽³⁾ and Pawley method,⁽⁴⁾ precision measurements of diffraction peak positions and widths, and measurements in special environments (temperature/atmosphere adjustment, etc.).

The para-focusing method is based on the inscribed angle theorem. However, for measurements, samples are formed into flat plates, and diffraction occurs inside samples. If the reference position on the sample surface is displaced vertically or the sample surface is irregular, an eccentric error is generated. Additionally, since x-rays emitted from the source and the receiving slit have finite widths, even an ideally adjusted optics has the six error factors shown in Fig. 4.3.1.

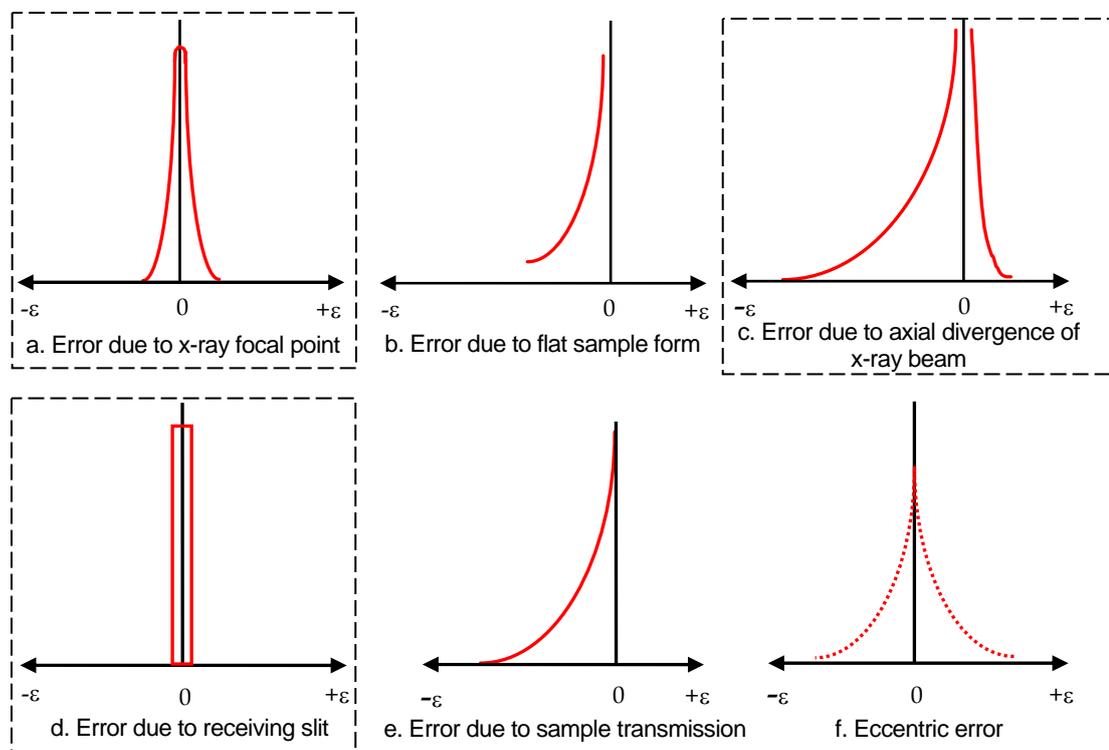


Fig. 4.3.1 Error factors in para-focusing optics
(those in dotted-line boxes also occur in parallel beam optics)

In contrast, the parallel beam method has virtually no error factors associated with sample shapes. As shown in Fig. 4.3.1, the associated error factors are limited to three categories: (a) errors due to x-ray focal point; (c) errors due to axial divergence of x-ray beams; and (d) errors due to the receiving slit. And while the para-focusing method requires an infinitely small focal point width to suppress errors due to the x-ray focal point, the parallel beam method can suppress this error by reducing the divergence angle with an incident monochromator or other parallelizing optics.

Profile analysis methods such as the Rietveld method and Pawley method have become increasingly important in the recent practice of powder diffractometry. Most profile analysis methods perform approximations of measured diffraction patterns using an analytical profile function – an asymmetric function based on the Pseudo-Voigt or Person VII function – but the degree of coincidence is low for highly asymmetric profiles.⁽⁵⁾ However, in the parallel beam method, the asymmetrical factors are caused only by the umbrella effect (which is a result of axial divergence). Suppressing this effect provides high-resolution, symmetrical profiles. In addition, by suppressing the symmetrical error factors of the x-ray focal point and receiving slit, we also improve the precision of absolute-value diffraction angles, potentially facilitating the determination of the lattice constant and index.

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