High resolution spiral analyzer

CALSA

1. Introduction

In X-ray powder diffraction analysis, obtaining high resolution and high intensity diffraction data leads to the improved accuracy of analyses based on that data, such as qualitative and quantitative analysis of minor phases, crystallite size and strain analysis, lattice constants refinement and so on. Powder structure analysis has received a lot of attention in recent years. Powder structure analysis requires high-resolution, high-intensity data: accurate diffraction angles are needed for the determination of lattice parameters and accurate integrated intensity for each diffraction peak needs to be evaluated for the structure determination.

However, there is always a trade-off between resolution and intensity. Using older optical systems, it has generally taken quite a long time to collect high resolution, high intensity measurement data.

CALSA (Crystal Array on Logarithmic Spiral Analyzer), the high resolution spiral analyzer introduced here, is able to obtain very high resolution data using the Cu Ka_1 emission line, and gets an intensity ten times higher than conventional optics with the same angular resolution.

2. Advantages

2.1. Extremely high resolution data

The extremely high resolution is achieved using parallel optics: a parabolic graded multi-layer mirror and Ge(220) channel-cut monochromator on the incident side, and perfect Ge(111) crystals on the receiving side.

In order to compare resolution with legacy optics, measurement data for the 110 diffraction peak from LaB_6 (NIST SRM 660a) are shown in Fig. 1. The optics using CALSA obtained 0.017° in FWHM!

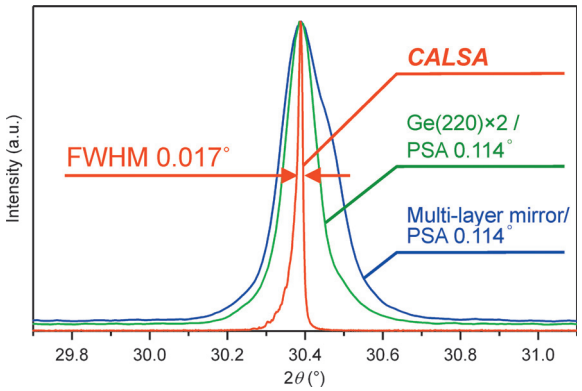


Fig. 1. Comparison of profiles for LaB_6 110 diffractions. The profiles are normalized with each peak height.

2.2. Ten crystals achieved ten times intensity

Ten perfect Ge(111) crystals are used to achieve CALSA's high intensity measurements. As a result, it is possible to obtain data with intensity ten times higher than that taken using conventional optics with the same angular resolution.

2.3. High P/B ratio data is obtained

Usually, measured data includes both diffraction peaks and background caused by fluorescent X-rays from the sample etc. If the obtained data has a high peak/background (P/B) ratio, it will enable qualitative

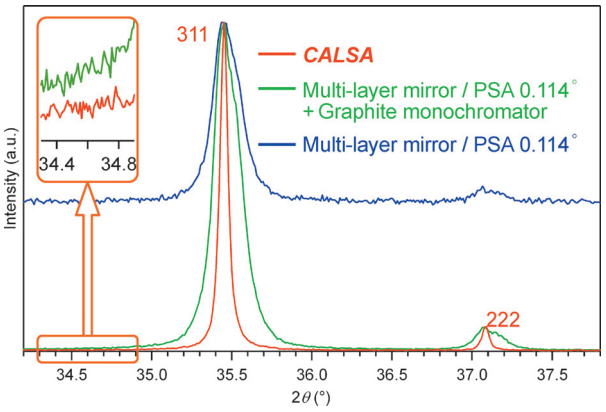


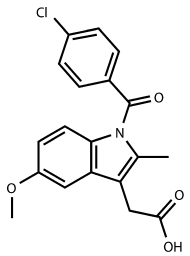
Fig. 2. Fe_3O_4 (magnetite) diffraction patterns of 311 and 222 diffraction peaks. The intensities are normalized by 311 diffraction peak heights.

and quantitative analysis of minor phases and improve the accuracy of lattice parameter determination.

Using the CALSA system with perfect Ge (111) crystals, it is possible to obtain high P/B ratio diffraction data without observing fluorescent X-rays (Fig. 2).

3. Application

γ-Indomethacin was assumed to be an unknown crystallographic structure here, its diffraction data was collected with the system using CALSA, and the crystal structure was analyzed. The analysis was performed using the PDXL Structure Analysis Package. Analyzing the crystal structure of γ-indomethacin using powder diffraction data is considered difficult because, due to the triclinic crystal system, many of the diffraction peaks overlap.



Triclinic *P*-1

a = 9.236(5) Å α = 69.897(5)°

b = 9.620(5) Å β = 87.328(5)°

c = 10.887(5) Å γ = 69.501(5)°

V = 847.865 Å^3 at 120 K

Fig. 3. The molecular structure of γ-indomethacin and its lattice parameters.[1]

As shown in Fig. 4, using data measured with CALSA, the analysis software was able to resolve the positions of very small peaks as well as overlapping peaks. From these, it was able to successfully determine the lattice parameters.

The initial structure was determined by the direct-space method using the molecular model optimized with the *ab-initio* molecular orbital method. The determined structure was refined using the Rietveld method (Fig. 5).

The refined structure is in good agreement with those from single-crystal structure analysis, as shown in Fig. 6.

CALSA is a powerful tool for structural analysis using powder diffraction data, particularly when a single-crystal cannot be obtained or measured.

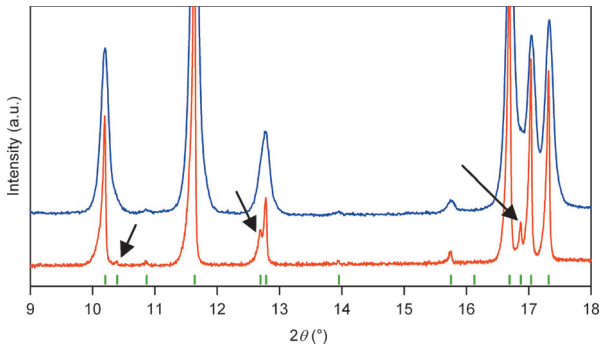


Fig. 4. Comparison of γ-indomethacin profiles.

—: CALSA system.

—: Ordinal-type parallel beam optics (Multi-layer mirror/ PSA 0.114°).

|: Peak positions calculated from lattice parameters.

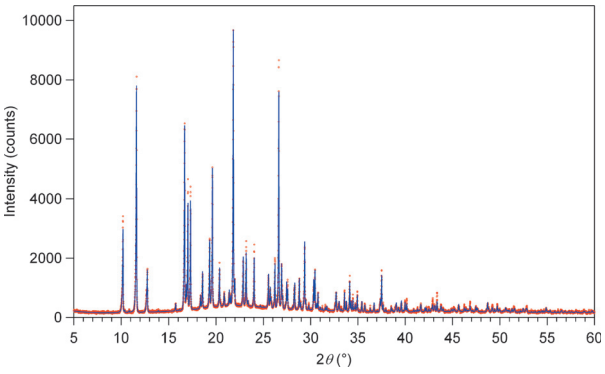


Fig. 5. The results of Rietveld refinement of γ-indomethacin.

·: Measured profile (measurement time is 98 hours).

—: Calculated profile.

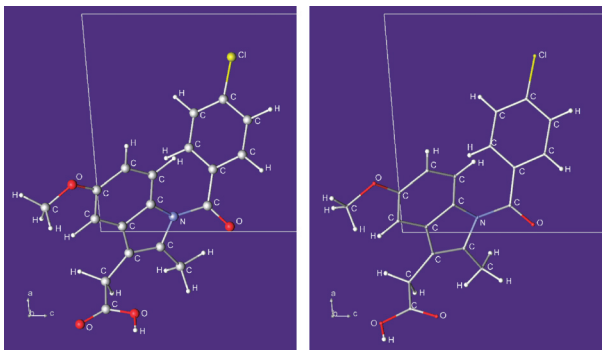


Fig. 6. Crystal structures of γ-indomethacin.

Left: Crystal structure analyzed with powder diffraction profile with CALSA.

Right: Crystal structure analyzed with single crystal diffraction data.[1]

References

(1) P. J. Cox and P. L. Manson: *Acta Cryst.*, **E59** (2003), o986–o988.