PRAMANA
— journal of
physics

Vol. 63, No. 1
July 2004
pp. 175–181

A possibility of parallel and anti-parallel diffraction measurements on neutron diffractometer employing bent perfect crystal monochromator at the monochromatic focusing condition

YONG NAM CHOI[1], SHIN AE KIM[1], SUNG KYU KIM[1], SUNG BAEK KIM[1], CHANG-HEE LEE[1] and PAVEL MIKULA[2]
[1]HANARO Center, Korea Atomic Energy Research Institute, Yuseong, Daejeon 305-600, Korea
[2]Nuclear Physics Institute, 25068 Rez, Czech Republic
E-mail: dragon@kaeri.re.kr

Abstract. In a conventional diffractometer having single monochromator, only one position, parallel position, is used for the diffraction experiment (i.e. detection) because the resolution property of the other one, anti-parallel position, is very poor. However, a bent perfect crystal (BPC) monochromator at monochromatic focusing condition can provide a quite flat and equal resolution property at both parallel and anti-parallel positions and thus one can have a chance to use both sides for the diffraction experiment. From the data of the FWHM and the $\Delta d/d$ measured on three diffraction geometries (symmetric, asymmetric compression and asymmetric expansion), we can conclude that the simultaneous diffraction measurement in both parallel and anti-parallel positions can be achieved.

Keywords. Neutron diffractometer; bent crystal monochromator; monochromatic focusing.

PACS No. 28.41.Rc

1. Introduction

Neutron diffraction has two measurement positions, the parallel and the anti-parallel positions, according to the relative direction of neutron beam between the source and the monochromator line and the sample and the detector line. Conventional neutron diffractometers employing a monochromator of single reflection are using only one measurement position, the parallel position, because the resolution property of the anti-parallel position is worse than the other one [1].

There are many efforts to increase measurement efficiency, the higher intensity within a shorter time, such as modernization of the neutron source, collimators of high transmission, monochromator crystals of high reflectivity, detectors of high efficiency and new electronics. If we can add the possibility of the simultaneous

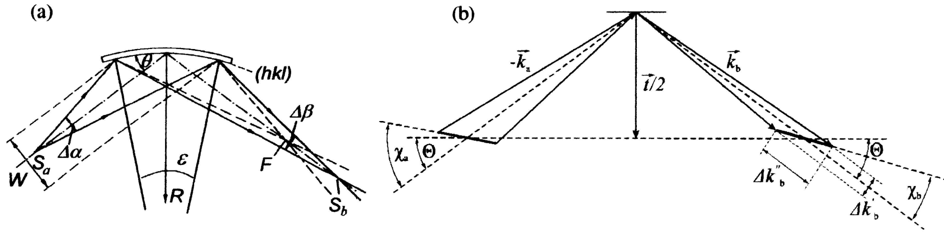

Figure 1. (**a**) RSR (real space representation) and (**b**) MSR (momentum space representation) of the diffraction by BPC.

measurement in the anti-parallel as well as the parallel positions, the measurement efficiency would be twice that of the traditional measurement mode.

A mosaic crystal monochromator has determinent characteristics of the neutron reflectivity and the beam divergence. The resolution or FWHM of the diffraction peaks from a sample depends on the take-off angle (θ_M), the diffraction angle (θ_S) and collimation angles (α_i). The following is an expression of the Caglioti's formula for the simple case, $\alpha_1 = \alpha_2 = \alpha_3 = \beta$ (mosaicity) $\equiv \alpha$ [1].

$$\mathrm{FWHM} = \alpha[(11 - 12a + 12a^2)/6]^{1/2}, \tag{1}$$

where $a = (\mathrm{d}\lambda/\mathrm{d}\theta)_{\mathrm{monochr}}/(\mathrm{d}\lambda/\mathrm{d}\theta)_{\mathrm{sample}} = \tan\theta_S/\tan\theta_M$ and the sign of a, plus and minus, means the parallel and the anti-parallel positions, respectively. As shown in eq. (1), the square of the FWHM is a parabola in variable a and whence it diverges monotonically with a or the scattering angle θ_S in the anti-parallel position.

Bent perfect crystal (BPC) monochromator has tunable properties based on the Bragg diffraction optics [2–6]. Characteristics of diffracted beams by BPC strongly depend on the geometric parameters such as bending radius, diffraction geometry (symmetric or asymmetric), crystal thickness etc. One of the prominent characteristics of a BPC monochromator compared to a conventional mosaic crystal monochromator is that BPC can produce focusing beams in the momentum space as well as in the real space. Another one is that the beam diffracted by BPC has strong correlation between wavelength and angular dispersion.

Diffraction by the BPC monochromator can be represented in momentum space as well as real space as shown in figure 1 [7]. Parameters shown in figure 1 are strongly correlated with each other [8]. The resolution property or the FWHM of the powder diffraction by a BPC monochromator depends on the bending radius, R, for a given configuration. The optimum R for the smallest $\Delta\theta_S$ in one position can be reduced to eq. (3) by minimizing $\Delta\theta_S$ in eq. (2) [8].

$$\Delta(2\theta_S) = \Delta\beta[2(\tan\theta_S/\tan\theta_M)(1 - L_{\mathrm{MS}}/R\sin\beta) - 1] \tag{2}$$

$$R_{\mathrm{opt}} = (L_{\mathrm{MS}}/\sin\beta)(1 - 2\tan\theta_S/\tan\theta_M)^{-1}. \tag{3}$$

When there are some grains satisfying the following condition (eq. (4)) in one position, it is not the case in the other position as shown in figure 2. The reason why the resolution property is opposite in two diffraction positions is that

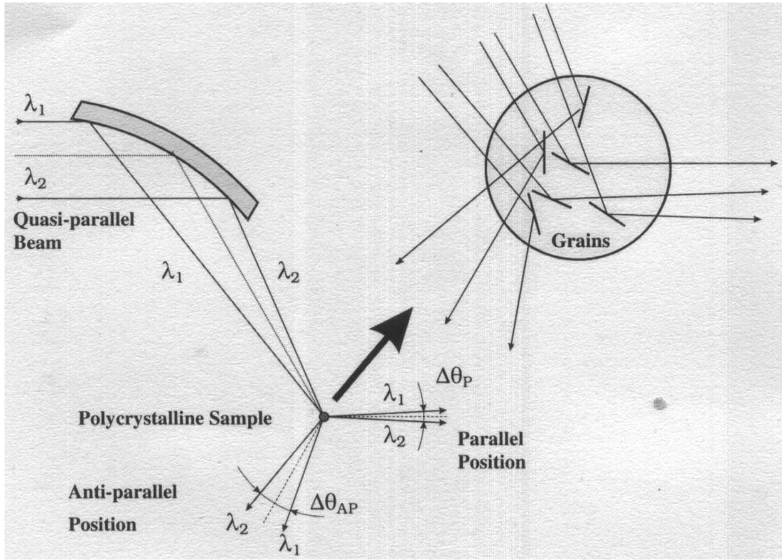

Figure 2. Schematic representation of the resolution property in parallel and anti-parallel diffraction positions.

$(\Delta\lambda)_{\text{anti-parallel}} = \lambda_2 - \lambda_1 = -(\lambda_1 - \lambda_2) = -(\Delta\lambda)_{\text{parallel}}$ and thus simultaneous satisfaction of the condition in eq. (4) in both parallel and anti-parallel positions is impossible.

$$\Delta\lambda(R_{\text{opt}}) = -\cot\theta_S(\Delta\theta_S)_{\text{parallel/anti-parallel}}. \tag{4}$$

The angle χ_b shown in the MSR (figure 1b) is an important parameter which represents characteristics of the diffracted beam.

$$\tan\chi_b = \Delta k'_b/\Delta k''_b = [R\sin\beta/(R\sin\beta - b)]\tan\theta_B. \tag{5}$$

In the case of $\chi_b = 90^\circ$, the lengths of k_b in figure 1b are nearly same through the whole region of Δk element. It means that all neutrons diffracted by BPC have the same wavelength, i.e., $\Delta\lambda = 0$. This situation is called monochromatic focusing (MF) condition and it can be written as eq. (6), obtained from eq. (5) by setting the denominator to zero.

$$R_{\text{MF}} = b/\sin\beta. \tag{6}$$

By changing R, $\Delta\theta_{\text{P}}/\Delta\theta_{\text{AP}}$ could be varied from the value less than unit to the one larger than unit. The condition for $\Delta\theta_{\text{P}}/\Delta\theta_{\text{AP}} = 1$ is another description of the MF condition.

2. Experimental result and discussion

The aim of this experiment was to demonstrate the versatility of the monochromatic focusing for the powder diffraction measurement. All experiments have been carried

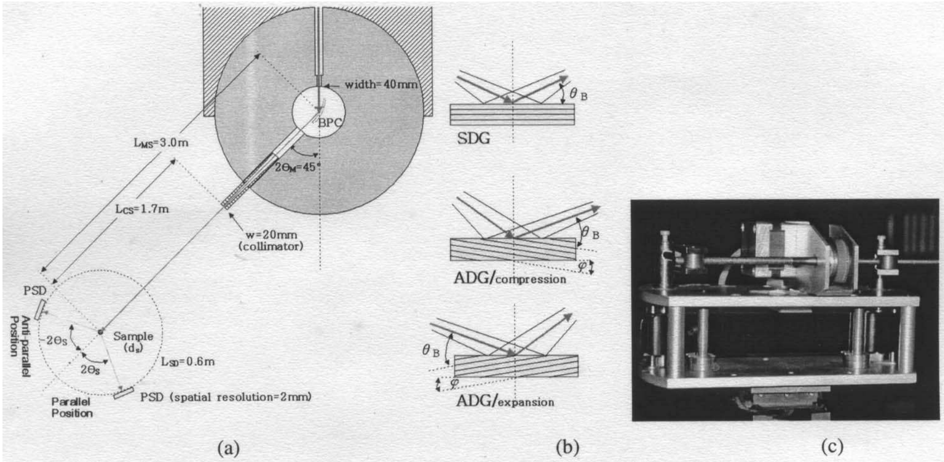

Figure 3. (**a**) Experimental set-up at ST1 beam port of HANARO, (**b**) three diffraction geometries, SDG ($\varphi = 0$), ADG/compression ($\varphi > 0$) and ADG/expansion ($\varphi < 0$), of the BPC monochromator and (**c**) the bending mechanism used for the experiment.

out at the test station beam port (ST1) of HANARO (the designed power is 30 MW and the operated power was 24 MW) in Korea Atomic Energy Research Institute.

Experimental set-up and three diffraction geometries of the BPC monochromator are shown in figure 3. SDG, ADG/compression and ADG/expansion mean the symmetric diffraction geometry, the asymmetric compression diffraction geometry and the asymmetric expansion diffraction geometry, respectively. The take-off angle $2\theta_M$ (or $2\theta_B$) = 45° and distances between the monochromator, sample and detector are $L_{\rm MS} = 3.0$ m and $L_{\rm SD} = 0.6$ m which were given constraints. Since the distance $L_{\rm SD}$ was too short, the resolution property obtained from the measurement was not so good compared with a conventional high resolution powder diffractometer. However, it would be enough to show the resolution characteristic of the monochromatic focusing and could be improved by placing the detector far away. Diffraction peaks from the polycrystalline copper rod (diameter 4.5 mm) were measured by a two-dimensional position sensitive detector (2-D PSD, 200 mm (W) × 200 mm (H), spatial resolution = 2 mm).

As shown in figure 3b, three typical diffraction geometries were chosen to investigate the bending radius dependence of the FWHM and the resolution ($\Delta d/d$) of powder diffraction. The resolution ($\Delta d/d$) data were calculated from the FWHM data. Monochromator crystal in SDG experiment was Si(2 2 0) slab (thickness = 3.0 mm) and that in ADG/compression and ADG/expansion was a special cut of silicon slab, Si(1 1 1)–9.5° (thickness = 3.8 mm). Considering the quite short distance between the sample and detector, $L_{\rm SD} = 0.6$ m, the resolution would be better than results in figure 4 if $L_{\rm SD}$ increases.

MF radii of three experiments $R_{\rm MF}$ are 7.84 m, 13.9 m and 4.46 m, respectively in the order of figures 4a–c and the last one (4.46 m) is beyond the breaking limit of the BPC slab. As R approaches $R_{\rm MF}$, the resolution property becomes similar

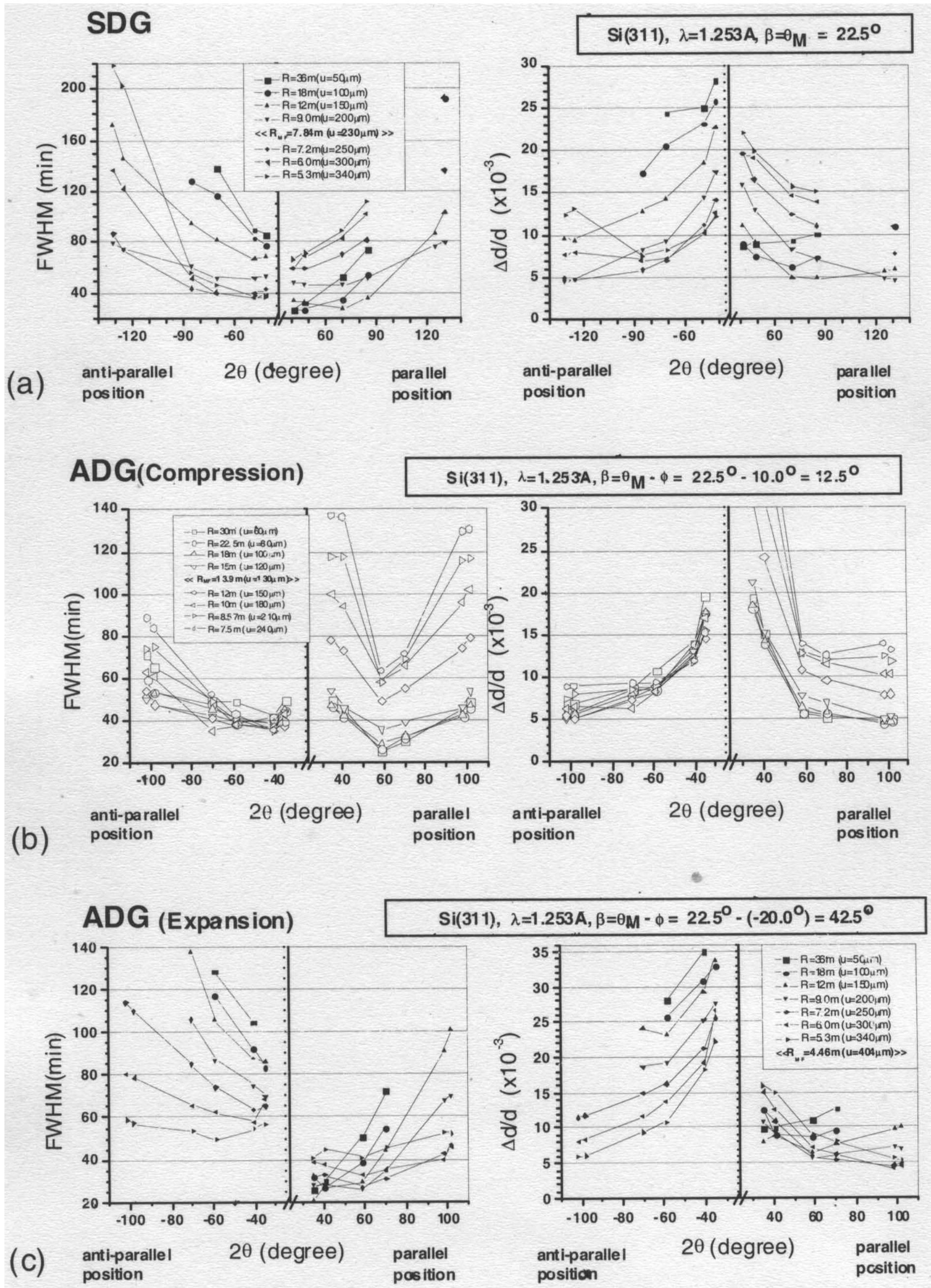

Figure 4. Experiment data of FWHM and $\Delta d/d$ from three diffraction geometrics, (**a**) symmetric, (**b**) asymmetric/compression and (**c**) asymmetric/expansion diffraction. When the bending radius approach R_{MF}, the FWHM and $\Delta d/d$ become nearly same (symmetric) in both parallel and anti-parallel diffraction positions.

in both positions and the angular dependence of $\Delta d/d$ also becomes more flat in two positions (figure 4).

To obtain equal resolution characteristics in the parallel and the anti-parallel positions, the θ–λ correlation should be small or symmetric along the beam axis. One simple case of the small θ–λ correlation is the FAD (fully asymmetric diffraction) geometry [9,10]. But in the FAD geometry, the intensity is smaller and the resolution is worse than other geometries due to the narrow width of the beam incident on the crystal slab and the large effective mosaicity. Previous study [11] shows that a dispersive setting of the double crystal (mosaic and BPC) monochromator which has also a small θ–λ correlation can be used for high resolution neutron strain/stress diffractometer using two diffraction positions (parallel and anti-parallel) near $2\theta_S = \pm 90^\circ$ at the appropriate bending radius.

In the present study, it is the case having symmetric θ–λ correlation, we are demonstrating the possibility of the simultaneous use of both diffraction positions in a wide angular region using BPC mochromator at the MF condition by showing experimental results shown in figure 4. Since R_{MF} is inversely proportional to $\sin\beta$, the smaller the β the larger the R_{MF} (i.e. the easier bending) and it is necessary to use thick crystal which is easy to be broken with a small bending to increase the signal intensity. Therefore the ADG/compression geometry ($\beta > \theta_B$) is the best candidate for the BPC monochromator at the MF condition (MF-BPC) in a practical sense.

3. Conclusion

We have demonstrated the equal and quite flat resolution property of powder diffraction in both the parallel and the anti-parallel positions by using BPC monochromator at the MF condition. By adapting the MF-BPC, one can make simultaneous measurements in both sides of the diffraction positions, parallel and anti-parallel and thus one can increase the measurement efficiency or extend the measurement capacity if the physical spaces of both positions are enough. One application is the simultaneous measurements using the same detector system on both sides, and which can increase measurement efficiency twice. Another application is the combination of a wide scan detector system (e.g., multi-tube detectors) for the whole pattern measurement and a position sensitive detector (PSD) for the measurement of detailed information during the whole scan and it can extend the measurement capacity.

Another possibility of the application of the MF-BPC monochromator could be as a monochromator of triple-axis spectrometer because the monochromatic beam is the very 'monochromatic', $\Delta\lambda \approx 0$, without the decrease of the intensity.

Acknowledgements

The authors are grateful to the in-house collaborators, Mr B H Choi, Mr Y H Choi, Mr K P Hong, Dr M K Moon and Dr V T Em for the experimental preparations and to Ms H S Oh for the numerous data processing. This work was supported by the Korean Ministry of Science and Technology through the National Nuclear R&D Program and the Scientist Visiting Program between Korea and Czech Republic. In Czech Republic the Bragg diffraction optics investigations are supported by the grants of GA-CR (No. 202/03/0891), GA-CAS (No. A1048003) and COST-OC P7.003.

References

[1] G E Bacon, *Neutron diffraction* (Clarendon Press, Oxford, 1975) pp. 101–111
[2] F Rustichelli, *Nucl. Instrum. Methods* **74**, 219 (1969)
[3] T Riste, *Nucl. Instrum. Methods* **86**, 1 (1970)
[4] J Klaus, *J. Appl. Crystallogr.* **8**, 361 (1975)
[5] J Kulda and P Mikula, *J. Appl. Crystallogr.* **16**, 498 (1983)
[6] J Saroun, *J. Appl. Crystallogr.* **27**, 80 (1994)
[7] P Mikula, V Wagner and R Scherm, PTB-N-17, Braunschweig (1994)
[8] V Wagner, P Mikula and P Lukas, *Nucl. Inst. & Meth. in Phys. Res.* **A338**, 53 (1994)
[9] P Mikula, P Lukas and R Michalec, *J. Appl. Crystallogr.* **20**, 428 (1987)
[10] P Mikula, J Kulda, P Lukas, M Vrana and V Wagner, *Nucl. Inst. & Meth. in Phys. Res.* **A338**, 18 (1994)
[11] P Mikula, M Vrana, Y N Choi, S A Kim, H S Oh, K H Sung, C H Lee, M R Muslih and N Nimura, *Proceedings of the 7th World Conference on Neutron Radiography*, Roma, Italy (2002)