Double Bent Crystal Monochromator for High Resolution Neutron Powder Diffraction

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Abstract

Recent results of focusing and reflectivity properties of the dispersive double-bent-crystal monochromator have shown that it could be succesfully used in high resolution neutron diffraction experiment. By using a standard polycrystalline sample of α-Fe, the resolution of the diffraction performance in the vicinity of the scattering angle $2\theta_S=90^o$ for the neutron wavelength λ= 0.162 nm was tested in detail. It has been found that for thin (1.3 mm) bent second crystal the angular resolution represented by *FWHM* of diffraction profiles was $1x10^{-3}$ rad for 211 and 200 reflections and about $3x10^{-3}$ rad for 220 reflection.

Key words: neutron diffraction, dispersive monochromator, powder diffraction

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I. INTRODUCTION

It is well known that due to lower neutron monochromatic current, nondispersive double-crystal (DC) monochromators ((1,-1) setting) are rarely used for neutron scattering instrumentation (e.g. HB-1A Triple-Axis Spectrometer at High Flux Isotope Reactor in ORNL). Namely, it is valid for mosaic crystals having the peak reflectivity $r<1$. Then, for the DC monochromator the neutron monochromatic current is proportional roughly to r^2. The situation is even much worse in the case of dispersive double-crystal (1,+1). DC monochromator with two parallel identical crystals in (1,-1) setting has some advantages. It produces monochromatic exit beam running parallel to incident white beam. For the change of the neutron wavelength such monochromator does not need a movable (rotating) monochromator arm and the position of the sample is fixed. Moreover, neutron instruments equipped with such DC monochromators are generally characterized by lower background detector signal. Nevertheless, in the case of mosaic crystals, there is commonly accepted opinion that the DC setting is not convenient for neutron monochromatisation due to a low monochromatic neutron current and thus a low luminosity of the scattering instrument. It is really true. However, it is not known that some dispersive DC monochromator arrangements with crystals either in (1,-1) or (1,+1) would be permanently employed even though some attempts were already carried out (Popovici and Yelon, 1995). On the other hand nondispersive as well as dispersive DC settings are quite common at synchrotron sources. As to neutron scattering, however, in the case of bent perfect crystals (e.g. Si or Ge) the situation is rather different and some advantages of the DC setting can be found. First, one should consider that when the curvature of the crystals is not drastic, the reflection probability $r_{1,2}$ of both bent perfect crystals can be close to 1 and there is a small and acceptable loss of neutrons due to the double reflection process (Mikula *et al.*, 1984; Mikula *et al.*, 1985). Namely, it is valid when neutrons of $\lambda > 0.2$ nm are used (Kulda, 1984; Mikula *et al*., 1994). First experimental results related to this dispersive DC setting and its possible use in residual strain/stress measurements were published in ref. (Mikula *et al*., 2012) in which excellent reflectivity and focusing properties fully confirmed promising future of the unconventional DC setting. In this paper we present further experimental results obtained by testing dispersive double bent crystal (1,-1) setting Si(111) + Si(220) in relation to possible exploitation in powder diffractometry.

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II. EXPERIMENTAL ARRANGEMENT

For this experiment, similarly to the previous experiments (Mikula *et al.*, 2012), we used testing neutron diffractometer installed at the 10 MW medium power research reactor LWR-15. The diffractometer operates at the fixed neutron wavelength of λ=0.162 nm, which is provided by a cylindrically bent perfect Si(111) crystal of the dimensions of 200x40x4 mm^3 (length x width x thickness) with the fixed radius of curvature of $R_1 \approx 10$ m, which was not changed during the experiment. Schematic sketch of the diffractometer performance is drawn in figure 1. The testing diffractometer can operate in two or three axis mode. The second bent perfect Si(220) monochromator was in fact always a sandwich of four slabs, from which either four slabs or two slabs or even one were operating and other supplementary slabs were of another cut. Therefore, the sandwich had all the time the same thickness, which helped us to avoid possible errors in determination of Si(220) curvature coming from the construction of the bending device. The dimension of each slab in the sandwich was 200x40x1.3 mm^3 (length x width x thickness). By using a sandwich instead of a single piece of the same thickness a large range of curvatures without a danger of breaking could be used. Even in the case of diffraction studies with one Si(220) slab, a sandwich of four slabs was used when three additional slabs in the bending device had a different orientation (the longest edge was parallel to [001] vector) and did not participate in the diffraction process. For neutron detection either a point detector or neutron Imaging Plate (IP) were used. The sample could be put on the third axis of the instrument, which is at the distance of 50 cm from the Si(220) sandwich. The distance between the Si(111) and Si(220 crystal was fixed and equal to 180 cm. No Soller collimator was installed before and after Si(111) crystal. The focal length of the bent Si(111) crystal was about 130 cm and the convergent rays of the extracted monochromatic beam crossed about 50 cm in front of the Si(220) sandwich (see figure 1).

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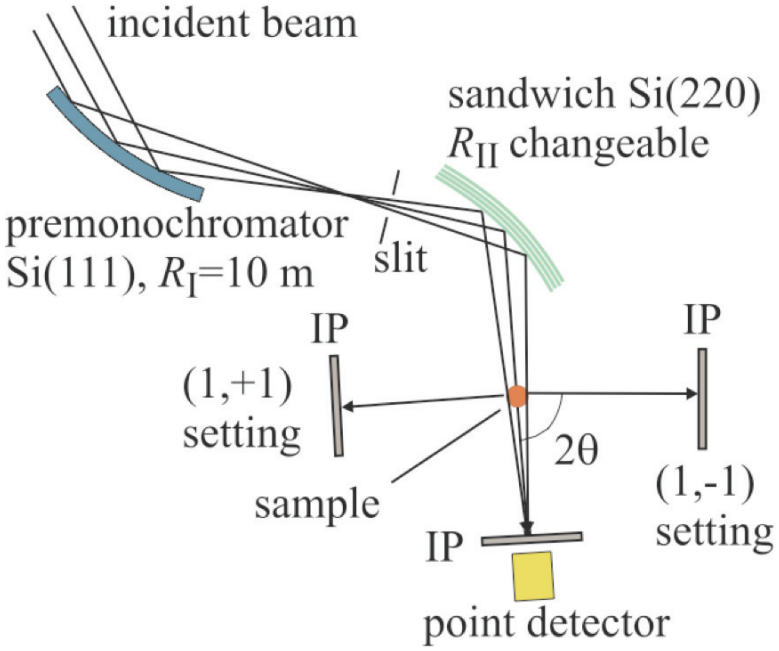


Figure 1. Schematic sketch of the diffraction performance as used for the experimental investigations.

III. EXPERIMENTAL RESULTS

First of all, the rocking curves of the double bent crystal monochromator and the intensities of the monochromatic beam were tested. Figure 2 shows the related experimentally obtained dependencies for different types of Si(220) sandwiches in symmetric reflection geometry. Even though the setting Si(111)+Si(220) is dispersive the *FWHM* of the rocking curves is not large and is practically given by $\Delta\lambda$ and $\Delta\theta$ distributions of the premonochromated beam coming from the bent Si(111) crystal.

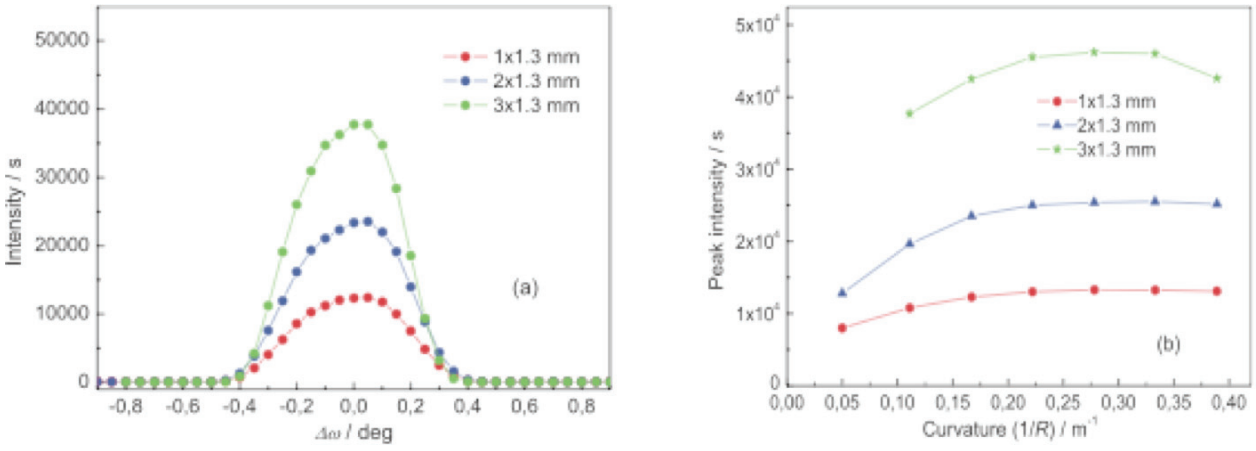


Figure 2. Examples of the rocking curves of the double crystal Si(111)+Si(220) setting for the radius of curvature R=6 m ($1/R$ = 0.167 m^{-1}) – (a) and the dependence of the peak intensities of the rocking curves on the curvature of the sandwich – (b).

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Then, profiles of the diffracted momochromatic beam obtained by the double diffraction process were studied when imaged by the Imaging Plate at different distances from the bent Si(220) sandwich for different radii of curvature *R*. During the extensive experimental studies two widths of the incident beam from the bent Si(111) crystal of 5 mm and 18 mm were used (two widths of the slit from figure 1). In this paper we will deal only with the results related to the slit width of 5 mm. Figure 3 shows an example of two such profiles for the Si(220) sandwich consisting of four slabs bent to the radius of *R*=3 m. It can be seen from figure 4 that for one slab in the sandwich focusing provides a monochromatic beam having *FWHM* of the profile of about 1 mm.

Inspection of the figures 3 and 4 reveal the following properties which has been common also for other sandwiches with two or one operating Si(220) slabs:

1. There is a strong focusing effect.
2. Focusing can be easily manipulated in a large range of curvatures.
3. Mutual orientation error of the slabs in the sandwich is very low which results in a homogeneous distribution of the monochromatic beam.

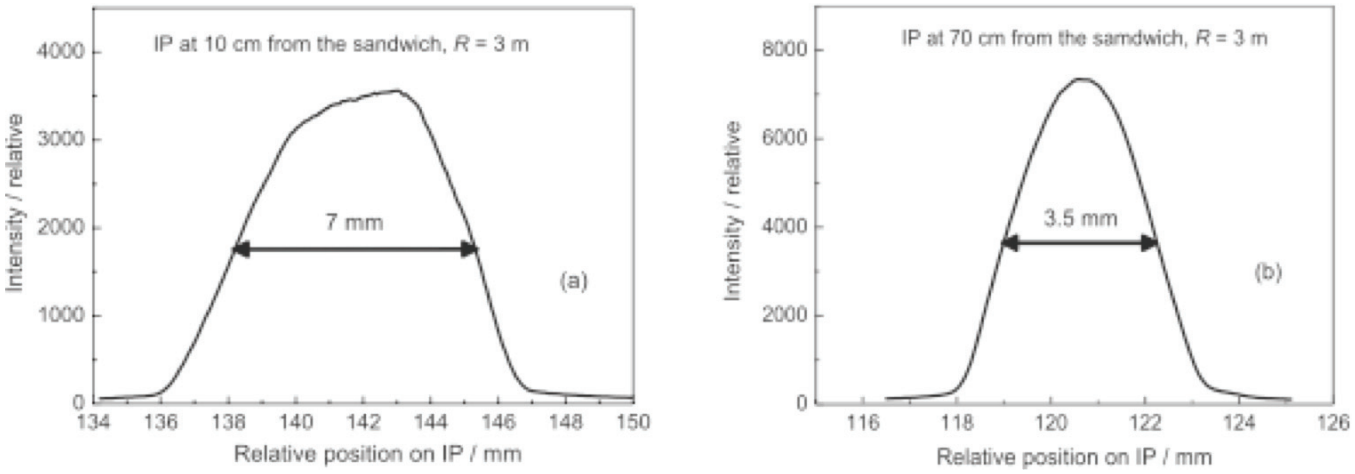


Figure 3. Examples of the intensity distribution profiles of the monochromatic beam imaged at two different distances from the Si(220) sandwich consisting of four slabs of the thickness of 1.3 mm.

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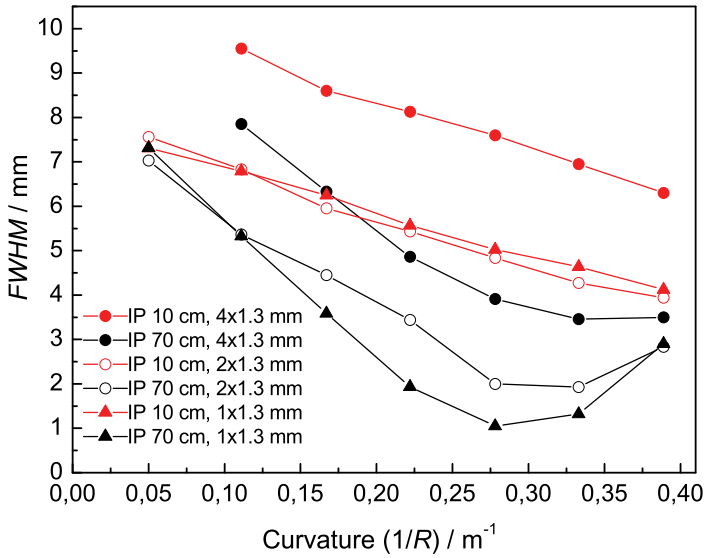


Figure 4. *FWHM* of monochromatic beam distribution from the bent sandwich as a function of curvature taken at two distances from the sandwich containing 1, 2 or 4 slabs of the thickness of 1.3 mm for symmetric reflection geometry of the sandwich.

As a next step, diffraction profiles from a well annealed standard α-Fe solid polycrystalline pin of 2 mm diameter situated on the third axis of the diffractometer were taken by IP installed at the distance of 45 cm from the α-Fe pin. The profiles were taken for different curvatures of the Si(220) sandwich. As to the imaging, we were limited to using IP which has namely excellent spatial resolution (50 μm x 50 μm) but rather poor detection efficiency, We focused our attention mainly on α-Fe(211) diffraction profiles taken at the scattering angle 2θ of about 88^{o}. Together with the α-Fe(211) diffraction profiles, several α-Fe(200) (2θ=69^{o}) and α-Fe(220) (2θ =106^{o}) profiles were also imaged. Moreover, two imaging plates could be used simultaneously: first at the scattering angle $+2\theta$ for (1,-1) setting and the second one at the scattering angle -2θ for (1,+1) setting (see figure 1). Figure 5 shows an example of diffraction profiles of the α-Fe(211) reflections obtained by diffraction on a standard polycrystalline pin of the diameter of ϕ=2 mm for (a) – (1,-1) setting, (b) - (1,-1) setting when using one Si(220) bent slab (R=6 m). Then, figure 6 displays the related dependences of *FWHM* of the imaged diffraction profiles on the curvature of the sandwich α-Fe(211) diffraction profiles and figure 7 displays similar dependencies when using the Si(220) sandwich consisting of 4 slabs.

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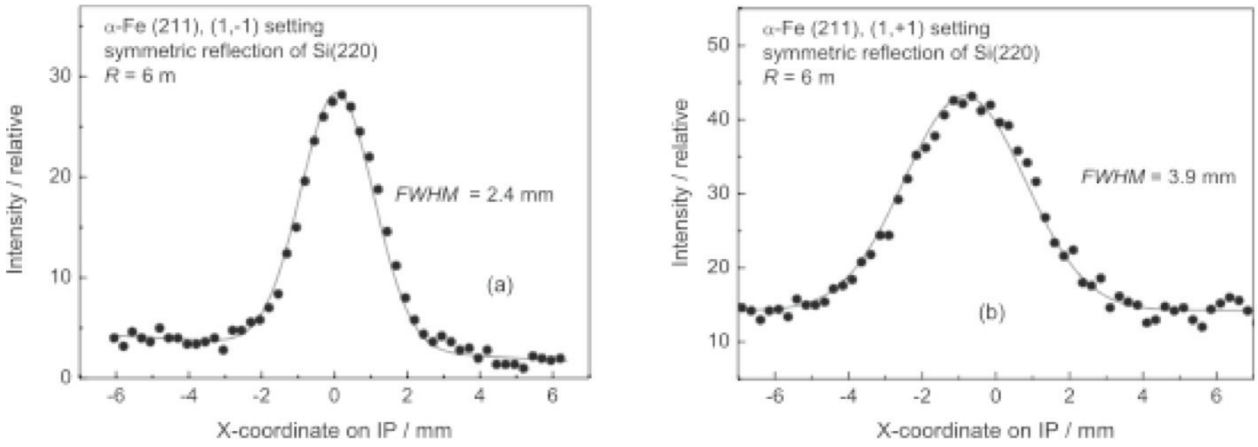


Figure 5. Examples of profiles related to α-Fe(211) reflections obtained by diffraction on a standard polycrys-talline pin of the diameter of ϕ=2 mm for (a) – (1,-1) setting, (b) – (1,+1) setting.

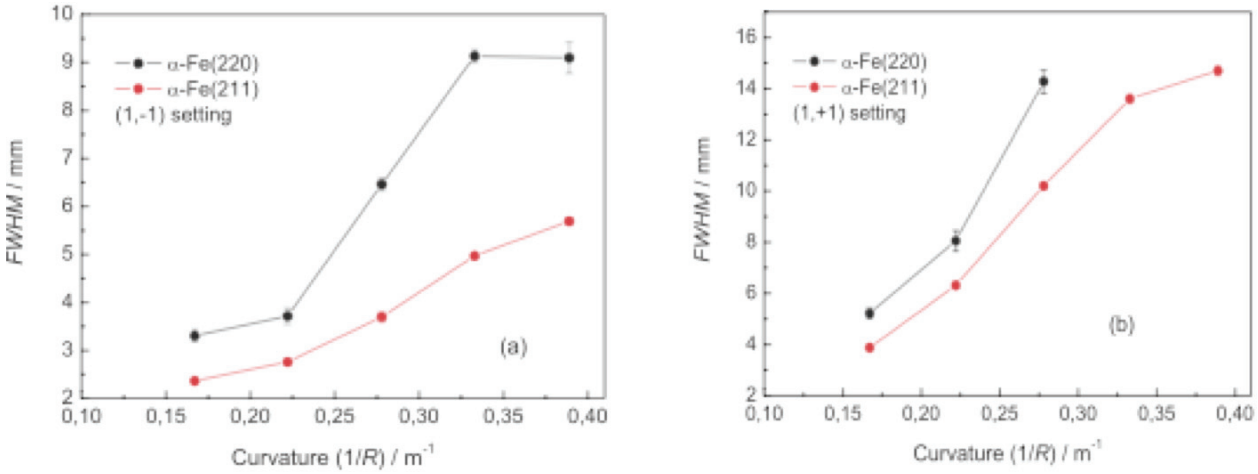


Figure 6. *FWHM* of the diffraction profiles imaged by IP situated at the distance of 45 cm from the α-Fe pin of the diameter of ϕ=2 mm for different curvatures of one Si(220) slab:
(a) – (1,-1) setting, (b) – (1,+1) setting.

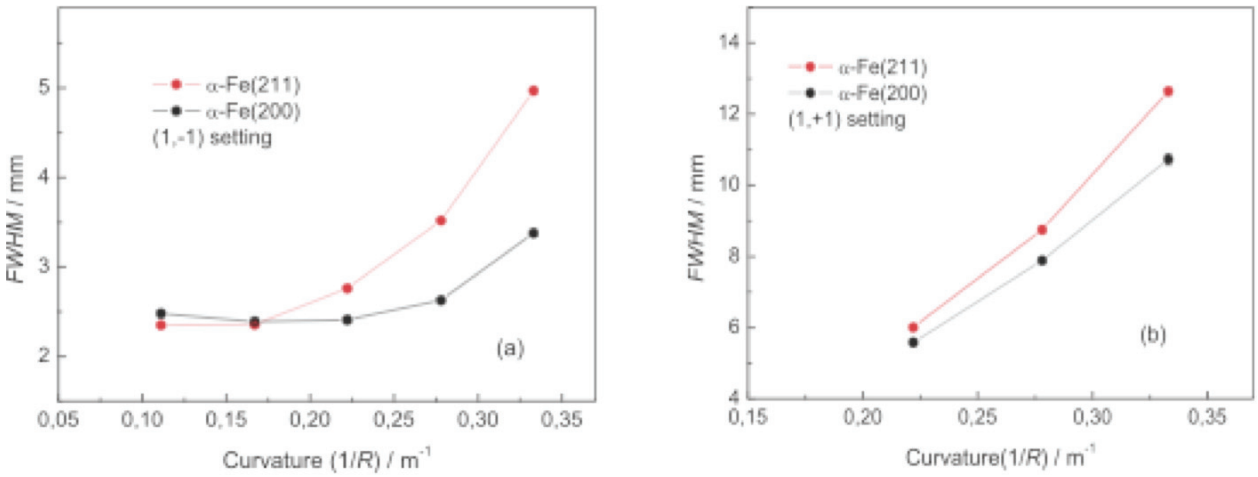


Figure 7. *FWHM* of the diffraction profiles imaged by IP situated at the distance of 45 cm from the α-Fe pin of the diameter of ϕ=2 mm for different curvatures of the sandwich with four Si(220) slabs: (a) – (1,-1) setting, (b) – (1,+1) setting.

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IV. DISCUSSION

Inspection of figures from 5 to 7 reveals that the best resolution is obtained at smaller curvatures below 0.25 m^{-1}. However, the minimum *FWHMs* of the spatial distributions of the focused diffracted beams are observed at larger curvatures above 0.2 m^{-1}. This result can be expected, because for smaller curvatures the extracted monochromatized beam has the spread $\Delta\lambda/\lambda$ as well as $\Delta\theta$ smaller. It means that the combination Si(111)+Si(220) is more dispersive. In the practical experiment some compromise In practice, a compromise choice of curvature should be made. It should be noticed that the *FWHMs* of the spatial distribution of the focused diffracted beams depends on the distance from the Si(220) sandwich. Inspection of figures from 5 to 7 thus reveals that the beams diffracted from the α-Fe pin have very low divergence $\Delta(2\theta_S)$ of about $1x10^{-3}$ rad for 211 and 200 reflections and about $3x10^{-3}$ rad for 220 reflection, respectively. Such property of the beam can be effectively exploited in microstrain studies in polycrystalline materials from diffraction profile analysis. On the other hand, the excellently focused and luminous beam can be eventually used in diffraction experiments under an extreme external load requiring samples of small dimensions.

For finding an optimum performance of the diffractometer employing dispersive DC focusing monochromator, there are still some open questions which could be answered by Monte Carlo simulations. Namely, an employment of a thinner Si(111) crystal which can open the door for obtaining a highly focused beam of sub millimeter width at the focal point could be interesting.

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