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Bragg prism monochromator and analyser for super ultra-small angle neutron scattering studies

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Abstract. We have designed, fabricated and operated a novel Bragg prism monochromator–analyser combination. With a judicious choice of the Bragg reflection, its asymmetry and the apex angle of the silicon single crystal prism, the monochromator has produced a neutron beam with sub-arcsec collimation. A Bragg prism analyser with the opposite asymmetry has been tailored to accept a still sharper angular profile. With this optimized monochromator–analyser pair, we have attained the narrowest and sharpest neutron angular profile to date. At this facility, we have recorded the first SUSANS spectra spanning wave vector transfers $Q \sim 10^{-6}$ Å^{-1} to characterize samples containing agglomerates up to tens of micrometres in size.

Keywords. Neutron collimation; sub-arcsec collimation; Bragg prism; super ultra-small angle neutron scattering; dynamical diffraction.

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1. Introduction

Super ultra-small angle neutron scattering (SUSANS) experiments involve wave vector transfers $\sim 10^{-6}$–10^{-5} Å^{-1} and hence necessitate monochromatic neutron beams with angular widths less than or of the order of an arcsecond. Bonse–Hart proposal [1] to attain a sharp and a few arcsec wide angular profile by multiple Bragg reflections from a channel-cut single crystal has since been realized [2,3], affording studies at wave vector transfers $\sim 10^{-5}$ Å^{-1}. We have exploited dynamical diffraction of neutrons in single crystal prisms [4–7] to achieve a further reduction in the neutron angular width.

2. Theory

We consider a monochromatic neutron beam incident on a thick single crystal prism in an asymmetric Bragg configuration outside the total reflectivity domain. The

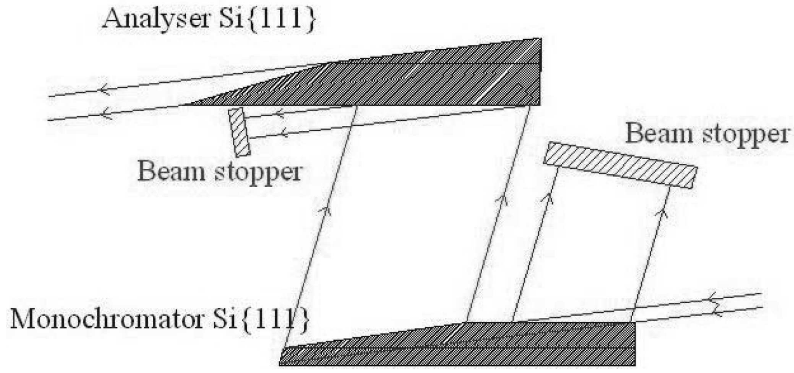

Figure 1. Optimized Bragg prism monochromator–analyser pair.

fraction of neutrons not reflected at the incidence surface, reaches the side face and emerges in two separate beams, one diffracted and the other forward diffracted [7]. Boundary conditions at the entrance and exit faces of the prism completely determine the wave vectors and intensities of the two beams [7,8]. The diffracted beam exits the side face at an angle

$$\delta_{\rm cr}^{H} = -\frac{\chi_H y}{\sin(A-\theta_{\rm B}-\theta_S)\sqrt{|b|}} \times \left\{ \frac{\sin(A+\theta_{\rm B}-\theta_S)}{\sin(2\theta_{\rm B})} - \frac{\sin A(1-\sqrt{1-y^{-2}})}{2\sin(\theta_{\rm B}+\theta_S)} \right\}, \tag{1}$$

up to an additive constant. Here $\theta_{\rm B}$ denotes the Bragg angle, θ_S, the angle between the front face and Bragg planes and A, the angle between the front and side faces. χ_H symbolizes Fourier component of susceptibility for the **H** reflection and $y = (\theta - \theta_{\rm c})/w$ stands for the scaled deviation of the incidence angle, $\theta_{\rm c}$ and w representing the centre and the half-width, respectively of the domain ($|y| \leq 1$) of total reflectivity. The asymmetry factor b equals $-\frac{\sin(\theta_{\rm B}-\theta_S)}{\sin(\theta_{\rm B}+\theta_S)}$. If the angle A lies between $\theta_{\rm B}+\theta_S$ and $\pi - (\theta_{\rm B}-\theta_S)$, an intensity

$$I_H(\delta_{\rm cr}^H) = \left(\frac{\sin\Delta\sin(A-\theta_{\rm B}-\theta_S)}{\sin(\theta_{\rm B}-\theta_S)\sin(A+\Delta)}\right)^2 \frac{\sin(\theta_{\rm B}-\theta_S-\Delta)}{\sin(\theta_{\rm B}+\theta_S+\Delta)} \tag{2}$$

exits the side face in the diffracted beam direction. A judicious choice of the Bragg reflection, its asymmetry, and the apex angle A for the prism can make $\mathrm{d}\delta_{\rm cr}^H/\mathrm{d}\theta$ approach -1. The single crystal prism then produces a neutron beam with sub-arcsec collimation. The analyser can likewise be designed to accept an extremely narrow neutron angular profile. Figure 1 depicts such an optimized Si {1 1 1} monochromator–analyser combination for 5.26 Å neutrons.

A monochromator with $\theta_S = 50.1°$ and $A = 172°$ yields a beam collimated to 0.53 arcsec FWHM. An analyser with $\theta_S = -51°$ and $A = 16°$ is expected to accept a pair of 0.22 arcsec wide neutron peaks separated by 2.13 arcsec. Figure 2 displays these predicted angular profiles, incorporating the appropriate Debye–Waller factor

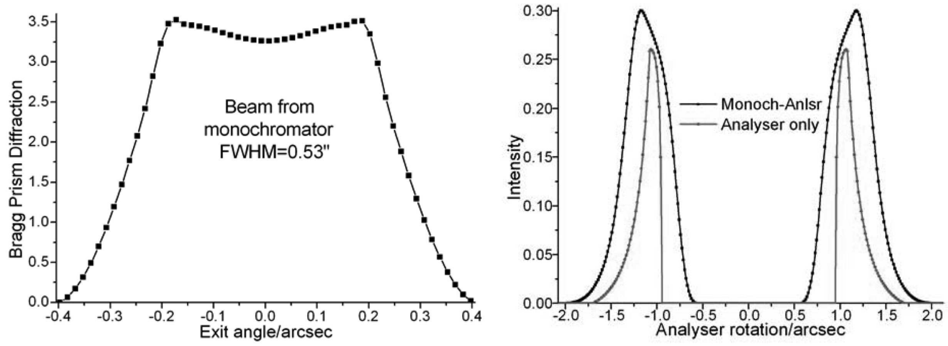

Figure 2. Theoretical monochromated, analyser acceptance and combined angular profiles.

and their convolution, comprising two 0.57 arcsec wide peaks separated by 2.35 arcsec.

3. Experimental

Several monochromators with θ_S close to 50° and one analyser with $\theta_S = -51°$ were fabricated with the specified apex angles at BARC. A $\langle 1\,0\,0\rangle$ single crystal silicon ingot was aligned using $\{1\,1\,1\}$ and $\{2\,2\,0\}$ reflections of 1.2 Å neutrons at the triple axis spectrometer (TAS) in the Dhruva reactor. Cuts at the desired orientations and dimensions were made on this ingot on a Blohm precision grinding machine and subsequent surfacing achieved with a diamond polishing wheel at the Centre for Design and Manufacture (CDM). Final sub-micron polishing and a long (20-min) and slow etching, to remove all residual strains, operations were performed at the Chemical Laboratory of Hahn-Meitner-Institut (HMI) in Berlin.

The experiment was carried out at the V12b double crystal diffractometer set-up of BENSC (HMI) in Berlin. The analyser rotation could be adjusted in two stages; first in 1 arcsec steps with a geared step motor and then with a piezocrystal-driven stage, with the smallest step size of 0.156 arcsec. For 5.26 Å neutrons Bragg reflected from an asymmetric ($\theta_S = 53.5°$) Si $\{1\,1\,1\}$ monochromator, the analyser tilt had to be set to within 9 arcsec. The analyser rocking curve (figure 3) displayed a well-resolved pair of ∼1.6 arcsec wide peaks (circles), in good agreement with theory (triangles), implying successful operation [9] of the analyser as per the design.

A prism ($\theta_S = 50.1°$) was next tested. Direct Bragg reflections, being much stronger and wider than prism diffractions, were first used, facilitating a quick and easy alignment of the analyser. The monochromator was then translated along the incident beam (figure 1) to illuminate the analyser with its prism diffraction. After optimizing the analyser alignment, a cadmium sheet was introduced before the detector to stop Bragg reflected neutrons from the analyser. With these Bragg prism diffractions, the analyser tilt adjustment became even more critical and had to be made in 0.9 arcsec steps. The rocking curve (figure 4) consisting of a pair of

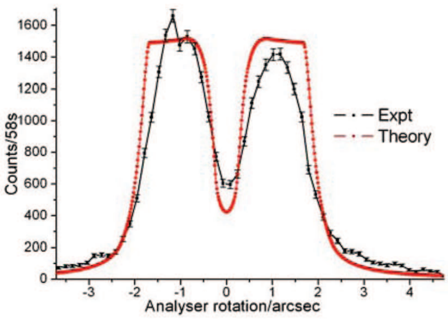

Figure 3. Monochromator Bragg reflection, analyser: Bragg prism.

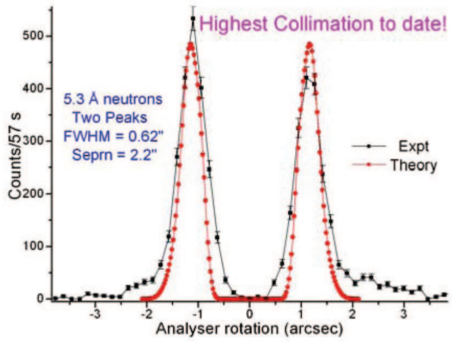

Figure 4. Monochromator and analyser: Bragg prisms.

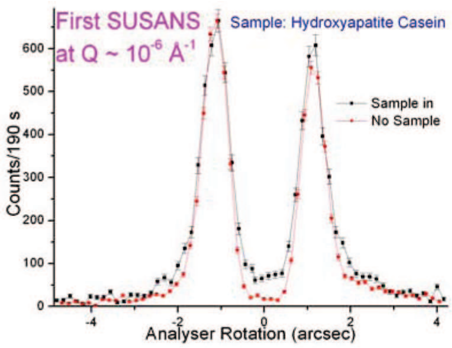

Figure 5. First $Q \sim 10^{-6}$ Å^{-1} SUSANS spectrum.

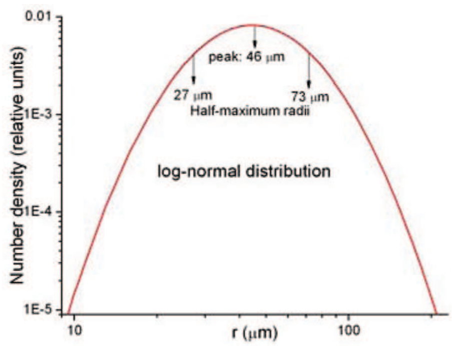

Figure 6. Sphere size distribution inferred from figure 5.

0.62 arcsec wide peaks separated by 2.2 arcsec (squares), is in fair agreement with the theoretical prediction (circles). This constitutes the best neutron collimation attained to date.

This super collimated neutron beam can probe wave vector transfers $Q \sim 10^{-6}$ Å^{-1}. We recorded the first SUSANS spectrum in this Q-range with a hydroxyapatite casein protein sample (figure 5) placed between the monochromator and analyser. The size distribution [10] of spherical agglomerates in the sample inferred from our instrument follows a log-normal distribution with median and FWHM equal to 53 and 46 micrometres, respectively (figure 6) and this instrument is capable of characterizing agglomerates upto 150 micrometres in size.

A rocking curve recorded with an annealed $Fe_{73}Al_5Ga_2P_8C_5B_4Si_3$ sample and scattering length density distribution deduced therefrom are shown in figure 7.

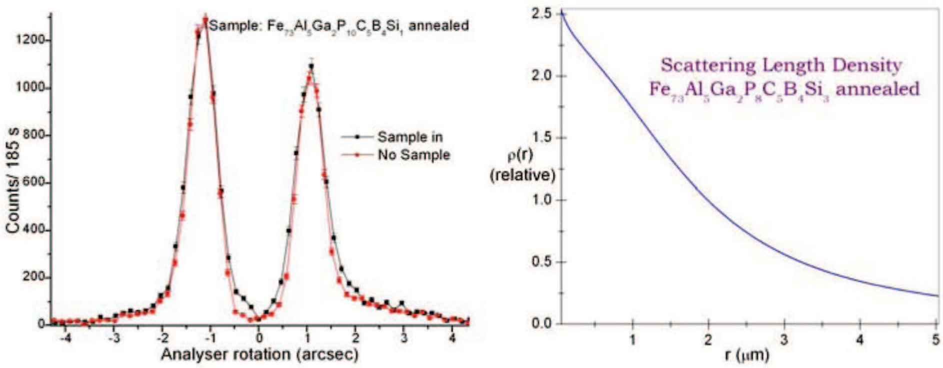

Figure 7. Analyser rocking curve for an iron sample and its scattering length density distribution.

4. Conclusion

We have presented the first sub-arcsec collimation of monochromatic neutrons and the first SUSANS spectra in $Q \sim 10^{-6}$ Å^{-1} regime, demonstrating a capability of studying agglomerates up to 60 micrometres in extent.

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References

[1] U Bonse and M Hart, *Appl. Phys. Lett.* **6**, 155 (1965)
[2] A G Wagh, V C Rakhecha and W Treimer, *Phys. Rev. Lett.* **87**, 125504 (2001)
[3] W Treimer, M Strobl and A Hilger, *Appl. Phys.* **A74(Suppl.)**, S191 (2002)
[4] G Borrmann, G Hilderbrandt and H Wagner, *Z. Phys.* **142**, 406 (1955)
[5] A Authier, *J. Phys. Radium* **23**, 961 (1962)
[6] B W Batterman and H Cole, *Rev. Mod. Phys.* **36**, 681 (1964)
[7] A G Wagh, *Phys. Lett.* **A121**, 45 (1987); **A123**, 499 (1987)
[8] A G Wagh and S Abbas, *Solid State Phys. (India)* **51**, 353 (2006)
[9] A G Wagh, S Abbas, M Strobl and W Treimer, *BENSC Experimenal Reports 2006* (HMI, Germany 2007) p. 22
[10] A G Wagh, V C Rakhecha, M Strobl and W Treimer, *Pramana – J. Phys.* **63**, 369 (2004)