Development of Focusing Neutron Small-Angle Scattering Spectrometer in Serpong, Indonesia for Macromolecular Structure Investigation

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The 36-meter small-angle neutron scattering (SANS) spectrometer in BATAN Serpong which utilizes thermal neutron produced from a 30 MW of multi-purposes G. A. Siwabessy reactor (RSG-GAS) is a pinhole or conventional SANS spectrometer. The performance of the spectrometer has been improved by implementing a focusing device system, a stack of 40 MgF$_2$ biconcave lenses, for gaining the neutron intensity up to 2.5 times and maintaining its resolution with a neutron wavelength of 5.7 Å. The minimum momentum transfer $Q_{\text{min}}$ of 0.002 Å$^{-1}$ can be achieved.

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I. INTRODUCTION

A small-angle neutron scattering (SANS) technique has been pointed out as a major tool for characterizing materials which provides the information about the fundamental structures in nanometer scale, 1–100 nm. The 36-meter SANS BATAN spectrometer (SMARTer) which is a pinhole or conventional SANS spectrometer installed at the end of 49 meter guide tube of the 30 MW multi-purpose G. A. Siwabessy reactor (RSG-GAS) in Serpong, Indonesia [1]. In the last several years, this spectrometer has been revitalized and gradually developed with the intention to improve its performance [2–4]. Along with upgrading and enhancing the performance, the spectrometer has been also utilized for basic research in limited performance, i.e. intensity and minimum momentum transfer $Q_{\text{min}}$ [5–7].

Neutron intensity or flux at the sample position, the accessible $Q_{\text{min}}$, resolution and the instrument background are the key characteristics of the SANS spectrometer. High neutron intensity is required for the speed of experiments and dynamics measurements, providing information on detailed structures in non-equilibrium before reaching the equilibrium state. Meanwhile $Q_{\text{min}}$ determines the largest length scale that can be probed by the instrument. When a larger neutron wavelength is employed in measurement it will expand to reach a lower $Q_{\text{min}}$ and higher resolution, on the contrary the neutron intensity reduced. Therefore, for obtaining higher neutron intensity and resolution as well at a large neutron wavelength without cold neutron facility, the neutron source has to be as close as possible to the sample or using the large pinhole collimation geometry. This circumstance is very difficult to be achieved by a pinhole SANS spectrometer without losing its resolution and gain the intensity. Moreover, the RSG-GAS reactor operates regularly only at half its maximum power and there is no cold neutron facility available. Instead of developing and installing a cold neutron source facility for gaining and attaining the neutron intensity at
a larger neutron wavelength, a focusing beam system using material lens or magnetic lens could possibly be developed and then applied at the spectrometer [8, 9]. Focusing of the neutron beam by inserting material lens like as MgF$_2$ lens was achieved and succeeded by S. M. Choi of NIST [10] and now has been implemented at other SANS spectrometers, such as SANS-J of JAEA (Japan Atomic Energy Agency) [11], SANS-U of the University of Tokyo [12], SANS-I of PSI (Paul Scherrer Institut) [13], Quokka of ANSTO (Australian Nuclear Science and Technology Organisation) [14].

In this paper, we report the mapping of the neutron flux or intensity at several configurations, analytical calculation on designing, implementing and performing the focusing system using material MgF$_2$ lens at the SANS BATAN spectrometer.

II. METHODS

II-1. Neutron flux measurement

Mapping the neutron flux at various configurations such as collimation length, neutron wavelength has been carried out using gold foil method through neutron activation analysis (NAA) measurement. The flux measurements were also performed at the end of neutron guide or before the velocity selector (MVS) and after the selector or before the collimation system.

Highly pure gold foils with 5-mm in diameter were mounted at the cadmium plate and then exposed with neutron beam for a certain time at different positions as the function of collimation length and neutron wavelength. The measurements which have been performed at before and after the velocity selector positions corresponding respectively to the intensity of white and monochromatized neutron beams at several neutron wavelengths, i.e. 5.7 Å, 3.9 Å, and 3.2 Å. The neutron flux at sample position as the function of neutron wavelength, i.e. 3.2, 3.9 and 5.7 Å and collimation length, i.e. 1.5, 4, 8, 13 and 18 m was also measured.

The gamma-ray from the activated gold foils after exposed by neutrons was detected using an HPGe detector gamma-ray spectrometer, accumulated and then analyzed using a multi-channel analyzer (MCA). The efficiency of HPGe detector, irradiation time, cooling or decay time, radioactive half-time of $^{198}$Au isotope, number of atoms and neutron cross section data are certainly required to calculate the activity of the activated $^{198}$Au. Then the neutron flux at SANS spectrometer with several setting configurations can be determined.

II-2. Direct beam profile and its intensity

The divergence of direct beam and its intensity were obtained directly using a 2-dimensional position sensitive detector (2D-PSD) with $\sim$ 8.5 mm spatial resolution. The pinhole collimation geometry of spectrometer was setup with a fixed collimation length and detector position, $L_1 = L_2$ configurations. The source aperture size $A_1$ was fixed, $\varnothing = 20$ mm, while sample aperture $A_2$ size was varied, $\varnothing = 20$ mm and 10 mm. The measurements at several collimation length and detector position configurations, i.e. 1.5, 4, 8, 13 and 18 m, and using a neutron wavelength of 5.7 Å were performed. The schematic diagram of the setup geometry of pinhole SANS spectrometer performed in this experiment is shown in
Fig. 1 (a)–(b). Theoretically, the beam size at the detector on pinhole collimation geometry is defined by the source and sample apertures \( A_{1,p} \) and \( A_{2,p} \), and the distances of \( L_1 \) and \( L_2 \). The optimal pinhole condition for a symmetric instrument setup \( L_1 = L_2 \) is \( A_{1,p} = 2A_{2,p} \), for which the beam profile at the detector is triangular base width of \( B_P = 2A_{1,p} \). However, for focusing geometry configuration using different size of source apertures is shown in Fig. 1 (c)–(e). Beside the distances of \( L_1 \) and \( L_2 \), the beam size at the detector on focusing collimation geometry is defined by the source aperture \( A_{1,p} \), number and specification of lenses.

**FIG. 1:** Beam collimation geometry for SANS. (a) Pinhole collimation geometry \( A_{1,p} = 2A_{2,p} \), (b) Pinhole collimation geometry \( A_{1,p} = A_{2,p} = 20 \) mm, (c) Focusing lens geometry \( A_{1,L} = A_{2,L} = 20 \) mm, (d) Focusing lens geometry \( 2A_{1,L} = A_{2,L} \), (e) Focusing lens geometry \( 4A_{1,L} = A_{2,L} \). \( N \) is the number of lenses. \( L_1 \) and \( L_2 \) are source-to-sample distance (collimation length) and sample-to-detector distance, respectively.

### II-3. Analytical calculation for focusing device

In addition to neutron intensity measurement, analytical calculation for implementing focusing material (MgF\(_2\) lens) was carried out firstly. In this calculation, the number of
lens which depends on the neutron wavelength and the radius curvature of the lens have to be considered. Those parameters are very important to determine the design and the number of lenses which correspond to the focal length of lens:

\[ f_0 = \frac{R}{2(1 - n)} = \frac{R}{\xi} = \left( \frac{R}{\rho b_c} \right) \left( \frac{\pi}{\lambda^2} \right), \]  

(1)

where \( R \) is the radius curvature of the lens and \( \xi \) is the refractive index decrement that corresponds to \( \rho \) the atomic density, \( b_c \) the bound coherent scattering length of an atom and \( \lambda \) the neutron wavelength.

\[ \xi = \left( \frac{\lambda^2}{\pi} \right) \rho b_c. \]  

(2)

From the references [10, 15], \( \rho b_c \) can be calculated for a specific neutron wavelength to obtain the refractive index decrement, \( \xi \). Here, we have calculated that the refractive index decrement \( \xi \) of SANS BATAN spectrometer with a neutron wavelength \( \lambda \) of 5.7 Å is \( 5.17 \times 10^{-5} \). Then the focal length of a single lens \( f_0 \) with a radius of curvature \( R = 25 \) mm is about 484 meter.

\[ f_0 = \frac{R}{\xi} = \frac{2.5 \times 10^{-2}}{5.17 \times 10^{-5}} \approx 484 \text{ meter}. \]  

(3)

However, to reduce the focal length it definitely requires a linear array of \( N \) lenses and it is given as

\[ N = \frac{f_0}{f} \]  

(4)

The imaging condition can be calculated using Gaussian optics by \( 1/f = 1/L_1 + 1/L_2 \) where \( L_1 \) and \( L_2 \) are respectively the distance from the source to the lens and from the lens to the detector. Therefore with a symmetrical instrument setup \( L_1 = L_2 = 18 \) meter, then \( 1/f = 1/18 + 1/18 = 1/9 \) or \( f = 9 \) meter (focal length). Finally, the number of lenses that has to be implemented at SANS BATAN spectrometer with a neutron wavelength \( \lambda \) of 5.7 Å at a focal length of 9 meter is about 54 lenses.

\[ N = \frac{f_0}{f} = \frac{484}{9} = 53.7. \]  

(5)

III. RESULT AND DISCUSSION

III-1. Neutron flux measurement

The flux of the white beam at the end of neutron guide is \( 6.57 \times 10^8 \text{ cm}^{-2} \text{ s}^{-1} \). After the velocity selector which is defined as 0 m of collimation length depends on the wavelength, Fig. 2. The neutron flux are \( 2.7 \times 10^7, 2.3 \times 10^7 \) and \( 1.2 \times 10^7 \text{ cm}^{-2} \text{ s}^{-1} \) for 3.2 Å, 3.9 Å
and 5.7 Å, respectively. Those results showed that the flux of monochromatized neutron decreases by magnitude of one compared to the white beam. On the other hand, as the collimation length increases from 1.5 m to 18 m, the neutron flux decreases exponentially by magnitude of one. In total, the intensity of monochromatized neutron such as at 5.7 Å certainly decreases at least by magnitude of two using the longest collimation length configuration, 18 m.

FIG. 2: The neutron intensity profiles after the velocity selector (MVS) at various neutron wavelength and at sample position as a function of collimation length. The pinhole collimation geometry was setup using 30 mm in diameter.

III-2. Direct beam profile and its intensity

The profile and its intensity of the direct beam divergence from pinhole collimation geometry in 2- and 1-dimensional are shown in Fig. 3 and 4. In average, the size of beam spot $B_P$ with 20 & 10 mm aperture configurations (Fig. 1 (a)) is c.a. 15 pixels which refer to $\sim$ 70–80 mm in diameter, Fig. 3 (a). While, by employing 20 & 20 mm aperture configurations (Fig. 1 (b)) the size of a beam spot is about 100 mm in diameter, Fig. 3 (b). Those results indicated that at fixed neutron wavelength, collimation length $L_1$, detector position $L_2$ and source aperture size $A_{1,p}$, the size of beam spot is depending on the size of the sample aperture $A_{2,p}$. It is clearly shown from Fig. 4 that at the same wavelength, the resolution which corresponds to the full-width half maximum, FWHM of the beam spot increases by reducing the sample aperture size. In the meantime, the intensity goes down to about 1/4 to 1/5 times.

From the above results it can be concluded that in gaining high neutron intensity will be reducing in the resolution. For that reason, a focusing system has to be designed and then developed for obtaining the intensity and the resolution as higher as possible. The implementation of a focusing device using optical lens has affected the neutron intensity by magnitude of two and also improving the $Q_{\text{min}}$ range of the conventional pinhole SANS spectrometer [8, 10, 11, 15].
FIG. 3: The patterns of direct beam at 2D-PSD with the collimation length $L_1 = L_2$ where indicated at the graphs. The pinhole setup (a) $A_{1,P} = 2A_{2,P}$, (b) $A_{1,P} = A_{2,P}$ and a neutron wavelength $\lambda = 5.7\AA$.

FIG. 4: The resolution of direct beam at 2D-PSD in 1-dimensional patterns. The pinhole setup, neutron wavelength, collimation length, detector position, and resolution are indicated at the figures.
III-3. Focusing system development and implementation

Analytical calculation for designing focusing lens has been completed as the requirement of minimum number of lenses is 54 with a neutron wavelength of 5.7 Å and focal length of 9 meter. We have performed the experiment of focusing system using 40 MgF$_2$ biconcave lenses.

Three collimation geometry configurations have been performed for examining the focusing system implementation, Fig. 1 (a)–(c). The collimation length and detector position were fixed at $L_1 = L_2 = 18$ meter. A conventional pinhole geometry was setup with a fixed source aperture or pinhole $A_{1,L} = 20$ mm and two different sample apertures $A_{2,L} = 20$ and 10 mm, Fig. 1 (a) and 1 (b). Meanwhile, a focusing geometry for employing 40 MgF$_2$ biconcave lenses was setup with fixed source and sample apertures $A_{1,L} = A_{2,L} = 20$ mm, Fig. 1 (c). All measurements were performed using a neutron wavelength of 5.7 Å and the results are shown in Fig. 5–7.

Two other setting configurations have also been performed for investigating the resolution of the focusing system, Fig. 1 (d)–(e). The collimation length and detector position were fixed at $L_1 = L_2 = 18$ meter with a neutron wavelength of 5.7 Å. A stack of 40 MgF$_2$ biconcave lenses was setup with a fixed sample aperture size $A_{2,L} = 20$ mm and varied source aperture sizes, $A_{1,L} = 10$ and 5 mm. Based on the schematic diagram of the focusing lens geometry with $L_1 = L_2 = 18$ meter and focal length $f = 9$ meter, Fig. 1 (c)–(e), the size of perfectly focused beam at the detector is the same as the source size, $B_L = A_{1,L}$. It means that the beam size at the detector is independent of the sample

![Image](image_url)

**FIG. 5:** Image of the direct beam on 2-dimensional detector at a symmetric instrument setup of $L_1 = L_2 = 18$ meter with a neutron wavelength of 5.7 Å. The collimation geometry configurations and the average of beam size are described on the picture that corresponded to Fig. 1 (a)–(c).
Focusing lens geometry with 40 arrays of biconcave lenses and $A_{1,L} = 10 \text{ mm}$; $A_{2,L} \varnothing = 20 \text{ mm}$

FIG. 6: Image of the direct beam on 2-dimensional detector at a symmetric instrument setup of $L_1 = L_2 = 18 \text{ meter}$ with a neutron wavelength of 5.7 Å. (a) $A_{1,L} = 10 \text{ mm}$ (b) $A_{1,L} = 5 \text{ mm}$. The collimation geometry configurations and the average of beam size are described on the picture that corresponded to Fig. 1 (d)–(e).

Focusing lens geometry with 40 arrays of biconcave lenses and $A_{1,L} = 5 \text{ mm}$; $A_{2,L} \varnothing = 20 \text{ mm}$

(a) Beam size $\varnothing \sim 70 \text{ mm}$

(b) Beam size $\varnothing \sim 55 \text{ mm}$

FIG. 7: The angular resolution of direct beam at various collimation geometries with $L_1 = L_2 = 18 \text{ m}$ and neutron wavelength $\lambda$ of 5.7 Å. A stack of 40 MgF$_2$ biconcave lenses were used for a focusing SANS system. (a) Comparison of pinhole and focusing collimation geometry (b) Comparison of focusing collimation geometry at different source aperture size.

aperture size. Hence, the samples size up to the size of lens opening can be used to increase neutron intensity without affecting the beam size at the detector.

It is clear from Fig. 5 that the size of direct beam at the detector is depending on the collimation geometry, pinhole or focusing lens at a fixed collimation length. The pinhole collimation geometry with $A_{1,P} \varnothing = 20 \text{ mm}$ and $A_{2,P} \varnothing = 10 \text{ mm}$ including $L_1 = L_2 = 18 \text{ m}$,
Fig. 5 (a) is defined as a standard configuration for SANS experiment. Optimum resolution and intensity can be achieved at this configuration whereas the diameter of the beam spot is c.a. 77 mm, Fig. 5 (a). The intensity increases approximately 4 times by changing the sample aperture size \( A_{2,P} \) from \( \varnothing = 10 \) mm to 20 mm, Fig. 7 (a). However, this setting configuration affected the resolution as the diameter of the beam spot becomes larger, c.a. 105 mm. The implementation of 40 MgF\(_2\) biconcave lenses has an effect on the resolution and intensity as well, Fig. 5 (c). The resolution is quite similar with the resolution of the sample aperture size \( A_{2,P} \varnothing = 10 \) mm configuration, Fig. 1 (b), while the intensity is about 20% less than the intensity of the sample aperture size \( A_{2,P} \varnothing = 20 \) mm configuration, Fig. 7 (a).

On the other hand, the resolution of the focusing collimation geometry can be enhanced by reducing the source aperture, \( A_{1,L} \) from \( \varnothing = 10 \) mm to 5 mm, Fig. 6 (a)–(b). The size of focused beam spot decreases from 83 mm into 55 mm in diameter by reducing the source aperture. This reduction is also improving the minimum of momentum transfer \( Q \) by a relation

\[
Q_{\text{min}} = c_{L}k \frac{A_{1,L}}{2L_{2}}.
\]  

where \( k = 2\pi/\lambda \) and \( c_{L} \) is a smearing factor which includes detector resolution, chromatic aberration, gravity effect and any scattering by the lenses. From this relation it can be found out that to reach \( Q \) minimum with sufficient intensity at focusing collimation geometry, the source aperture size has to be reduced. The \( Q_{\text{min}} \) that can be achieved from focusing geometry configuration is 0.002 Å\(^{-1}\) which corresponds to about 3.000 Å of size dimension. This \( Q_{\text{min}} \) range will be suitable for investigating the macromolecular structure of biological materials such as protein complex, virus, etc.

In the typical condition, the size of focused beam spot should be similar with the size of the source size geometry \( A_{1,L} \). From the measurement data, the size of beam spot is much larger than the size of the source size geometry. The difference result between the experimental data and theoretical calculation can be justified from the focal length of 40 lenses instead of 54 lenses. The focal length of 54 lenses at 5.7 Å is 9 meter, while using 40 lenses is 12 meter. This discrepancy caused the focused beam image at the same detector position is much bigger than the calculated ones. Other contributions in enlarging the image size of the beam spot are coming from the de-magnified of the aperture \( A_{1} \) and the chromatic aberration of the effective lens aperture and the wavelength spread of the neutron \( \Delta\lambda/\lambda \). In this case, the \( \Delta\lambda/\lambda \) of 5.7 Å is about 10%.

Beside the beam size, the beam intensity with pinhole and focusing collimation geometries, \( I_{P} \) and \( I_{L} \) can also be calculated. Those intensities are proportional to

\[
I_{P} \propto \frac{A_{1,P}^{2} \times A_{2,P}^{2}}{L_{1}^{2}} \quad \text{and} \quad I_{L} \propto \frac{A_{1,L}^{2} \times A_{2,L}^{2}}{L_{1}^{2}} T_{L}.
\]

The size of the focused beam at the detector \( B_{L} \) for \( L_{1} = L_{2} \) is the same as the source size \( B_{L} = A_{1,L} \). The beam size \( B_{L} \) is independent of the sample aperture \( A_{2,L} \). Therefore, one
can use sample sizes up to the lens diameter to increase the intensity $I_L$ without increasing the beam size on the detector or, equivalently, without reducing the $Q$ resolution of the instrument. The gain $G$ of intensity at the sample is given by

$$G = \frac{I_L}{I_P} = \frac{A_{1,L}^2 \times A_{2,L}^2}{A_{1,P}^2 \times A_{2,P}^2} T_L, \quad (8)$$

where $T_L$ is the transmission of lenses. From our setting configuration, pinhole and focusing collimation geometries, the aperture sizes were $A_{1,P} = 20$ mm, $A_{2,P} = 10$ mm, $A_{1,L} = 20$ mm, $A_{2,L} = 20$ mm, the wavelength $\lambda = 5.7 \text{ Å}$, the collimation and sample-to-detector distance $L_1 = L_2 = 18$ m, the total intensity gain factor $G$ can be calculate using Equation (8).

$$G = \frac{(20)^2 \times (20)^2}{(20)^2 \times (10)^2} \times 0.63 = 2.52$$

The calculated total intensity gain factor $G$ using those above setting configurations is $G = 2.52$ that similar with the result from the measurement data, Fig. 7 (a). While by reducing the size of a source aperture from 20 mm to 10 mm or from 10 mm to 5 mm with focusing collimation geometry the intensity decreases theoretically by factor of 4. The experimental result is also comparable with this theoretical calculation, Fig. 7 (b).

IV. CONCLUSIONS

It was demonstrated the performance of focusing system at the conventional SANS BATAN spectrometer by employing an array of refractive optical MgF$_2$ biconcave lens. Up to 40 MgF$_2$ biconcave lenses have been employed and installed easily at the new extended collimator system. The parasitic background scattering is negligible and the finite transmission is also tolerable, > 60%. The scattering intensity on focusing collimation system at SANS BATAN spectrometer increases by factor of 2.5 and also allowing to employ a larger sample (larger sample pinhole) up to the size of the lens as compared with the pinhole collimation SANS setup without focusing lens.

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