Recent Development of a 36 meter Small-Angle Neutron Scattering BATAN Spectrometer (SMARTer) in Serpong Indonesia

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Abstract. The 36 meter small-angle neutron scattering (SANS) spectrometer BATAN (SMARTer) in Serpong, Indonesia has been revitalised for several years. The work on replacing, upgrading and improving the control system and the experimental method were conducted in order to setup the spectrometer back in operation. Two main personal computers, one for handling and controlling the mechanical system and another one for acquiring neutron data were employed at the spectrometer. The standard and established SANS data reduction and analysis programs, such as GRASP and NIST Igor have been implemented to subtract the raw scattered neutron data with the backgrounds and then analyse the corrected data. The scattering data of ferrofluids samples, Fe\textsubscript{3}O\textsubscript{4} and MnZnFe\textsubscript{2}O\textsubscript{4} have been obtained using SANS spectrometers in BATAN Serpong, Indonesia and HANARO-KAERI, Republic of Korea for inter-laboratory comparison and investigation of proposed research interest. The results were comparable from both scattering data analysis.

1. Introduction
Small-angle neutron scattering (SANS) is a powerful technique for characterizing the static and dynamic-structures of materials in the nanometer scale range of 1 – 500 nm. Information on the average size and its distribution, spatial correlation, as well as shape and internal structure of materials can be obtained from SANS scattering intensity profiles. The quantitative analyses on number or volume density from investigated structures in the surrounding medium can be determined from the absolute scale of scattering intensity. Recently, SANS becomes a valuable technique for characterization in many areas of research, i.e. alloys, ceramics, magnetic, polymers, colloids, vesicles, including biological materials such as proteins, viruses, etc.

A 36 meter SANS spectrometer owned by the National Nuclear Energy Agency of Indonesia (BATAN) was constructed in the neutron-guide hall (NGH) of the 30 MW G. A. Siwabessy research reactor in 1992\textsuperscript{1}. It is the second largest SANS spectrometer that operates in the Asia-Pacific region nowadays and detail specification of this instrument has been reported elsewhere\textsuperscript{2,3}. Nevertheless, the spectrometer was not well utilized until 2004 due to a shortage in staff members, instrument failures and an undefined long-term research program. A five years in-house work plan was proposed in 2005
to replace, change and upgrade gradually the electronics and mechanics system, computer software of the instrument.

The original main computer IBM PS2/70 for data acquisition and control system running on AIX (Advanced Interactive eXecutive) was getting out-of-date and entirely halted at the end of 2003 then the computer finally crashed. Due to the unavailability of the syntax codes for running and controlling all the motor drivers as well as for acquiring the neutron counts from 2-dimensional detector, a new main control system is definitely required. Modifying the data acquisition program based on the time-to-digital converter (TDC) system from the original syntax codes provided by the National Laboratory Risø, Denmark had to be worked out first for acquiring the neutron counts from the detector. In the meantime, a new motor controller program and its hardware were designed and developed to handle motor drivers of the instrument which is controlled by a dedicated computer for device control via ISA (Industry Standard Architecture) interface connection. Shortly, when the new control system has been implemented, re-alignment of the collimation system, detector movements, and beam stop adjustment works were also accomplished. Those initial developments on data acquisition and control system were applied in 2006 for running the SANS experiments.

Seeing as the number of SANS experiments and the research interest using SANS BATAN spectrometer (SMARTer) significantly increased in the last few years, expanding the applicable conditions and performance of instruments as well as experimental methods are demanded. A major development has been made by improving the data acquisition system, employing the established SANS data reduction and analysis, upgrading the control system and designing a focusing lens device. In this paper, we report the latest development and the inter-laboratory result of SMARTer on magnetic fluids or ferrofluids samples.

2. Instrument development

2.1. Replacement of the SMARTer control system

In 2005 the original main computer IBM PS2/70 which run on the AIX operating system was replaced by the IBM compatible computer running on Windows for handling the motor driver system of the instrument. The 17 stepping motors for pinholes, collimators/neutron guides, beam stop and 2-
dimensional main detector movements are controlled by the computer through ISA interface connection. Another dedicated computer running on Linux was also set up for acquiring the neutron counts from the 2-dimensional detector via General Purpose Interface Bus (GPIB) connection. Figure 1 shows the current and in progress development works (underlined) relationship between the devices and the control system of SMARTer. The feedback from encoder of the system is still read by the dedicated computer for device control via the original RS232C interface connection through the CPU system of control panel.

A programmable remote control written in Visual Basic was employed in the device control computer for handling the setting configurations of spectrometer. This program is able to display all experimental settings and command menus of spectrometer such as 2-dimensional detector position, pinhole setting, collimator/neutron guide positions, beam stop position and beam narrower setting. The display of the command menu and the current status of the experimental setting configuration are shown in Figure 2.

A microcontroller based on the new control system for SMARTer is now being designed and developed via RS232 (serial) interface connection in order to accommodate and handle more motors or devices. The additional motor devices such as for beam shutter, automatic sample exchanger together with the temperature control for sample environment system are being implemented into the new control system. Recently, a new programmable remote control is also established on the computer for device control and it can also be controlled directly from the computer for data acquisition via a Local Area Network (LAN), Figure 1. The advantage of this new system offers that the SANS experiment based on preset count is able to be run automatically and continuously for ordered samples with a set of sequencers. The experiment can also be monitored from the staff room using a dedicated computer for monitoring and data analysis.
2.2. Real time data acquisition and modifying the raw data format

The previous data acquisition system running on Linux and using a command text mode has been replaced recently by the new one running on Windows with some additional mode applications. The new system which is provided in command window mode is set up for handling the neutron counts from 2-dimensional detector and monitor detector for preset count measurement, respectively via GPIB and serial interface connections, Figure 1. This newest system is also dealing with the computer for device control for automatic and continuous experimental settings via a LAN interface. The additional mode applications of the new system includes 2-dimensional pattern of a real-time data acquisition in linear and log-scales, a command window menu for input parameter of experimental settings, run and experimental numbers for identifying the running experiment and file names, and the format of the output data file, Figure 3.

Figure 3. The command menu (left) and the experimental setting display (right) of the new real-time data acquisition system of SMARTer

The standard and established data reduction and analysis package programs have to be applied in order to verify the experimental data of SMARTer for inter-laboratory comparison with other established laboratories. For that reason, the output data file of SMARTer can be saved into HANARO and ILL (the Institut Laue-Langevin) data format. A NIST (the National Institute of Standards and Technology) Igor data reduction \(^{10}\) modified by T.H. Kim of KAIST (Korean Advance Institute of Science and Technology), Republic of Korea is able to subtract the data of SMARTer in HANARO format, while GRASP (Graphical Reduction and Analysis SANS Program) \(^{11}\) is able to subtract the data of SMARTer in D22 format of ILL. Other SANS spectrometers such as D11 (ILL), SINQ_SANS I and II (PSI, the Paul Scherrer Institut); V4 (HMI, the Helmholtz-Zentrum Berlin), NG7 & NG3 (NIST), etc. were also supported by GRASP. A NIST Igor data analysis is regularly utilised for analysing the corrected data of SMARTer for solution system such as micellar solution, colloids, polymers and proteins \(^{12}\).

A set of experiment on ferrofluids samples was examined using SMARTer and HANARO SANS spectrometer. The data was treated using GRASP and then analysed using NIST Igor data analysis for inter-laboratory comparison.

2.3. Focusing collimation

The simulation and analytical calculation for developing a focusing lens collimation of the SANS spectrometer has been carried out. Here, 50 biconcave MgF\(_2\) lenses are required to be implemented for increasing the neutron intensity at the sample position and also for covering a lower momentum transfer \(q\) range. The specification of lens is as follows: the curvature radius = 25.0 mm, the center...
thickness = 1.00 mm, and the outer diameter = 40.0 mm. This calculation is appropriate for the sample to detector distance = 9 m symmetrical optical arrangement or 18 m collimation length with 20 mm \( \phi \) (diameter) sample aperture and a neutron wavelength = 0.57 nm setting configurations. Focusing of the neutron beam by insertion MgF\(_2\) biconcave lens system was achieved and succeeded by S.M. Choi of NIST\(^{13}\) and now has been implemented at other SANS spectrometers, such as SANS-J of JAEA (Japan Atomic Energy Agency)\(^{14}\), SANS-U of the University of Tokyo\(^{15}\), SANS-I of PSI (Paul Scherrer Institut)\(^{16}\), Quokka of ANSTO (Australian Nuclear Science and Technology Organisation)\(^{17}\).

3. Experiment for inter-laboratory comparison

The improvement of the experimental method was demonstrated on ferrofluids samples, Fe\(_3\)O\(_4\) and MnZnFe\(_2\)O\(_4\). Those samples were performed using HANARAO SANS spectrometer for inter-laboratory comparison. The detector was set at 4 m from the sample position and shifted laterally for 0.2 m. This setting covers a momentum transfer \( q \) range of 0.007 < \( q \) (Å\(^{-1}\)) < 0.15 using a neutron wavelength of 6.38Å and sample aperture, \( \phi \) = 12 mm. Detail of the specification of this spectrometer is reported elsewhere\(^{18}\). While in order to cover similar \( q \) range, the detector of SMARTer was positioned at 4 and 8 m with the neutron wavelength of 4.91 Å and sample aperture, \( \phi \) = 12 mm. The 2-dimensional pattern of ferrofluids samples are shown in Figures 4a, 4c for ferrofluids Fe\(_3\)O\(_4\) and Figures 5a, 5c for ferrofluids MnZnFe\(_2\)O\(_4\). Those scattering data was then subtracted from the backgrounds using GRASP and averaged radially into a 1-dimensional scattering pattern, Figures 4b, 4d and Figures 5b, 5d. All experimental data were then analysed using a NIST Igor data analysis.

A theoretical fractal calculation was fitted on the experimental data of ferrofluids sample of Fe\(_3\)O\(_4\) taken using SMARTer as well as HANARAO SANS, Figures 4b and d. The calculation is in a good agreement with the experimental data. From the fitting result it can be obtained that a primary particle of magnetite has a radius of c.a. 50 Å which is a nature of the magnetic particle size with a single magnetic domain (superparamagnetic). The mass fractal dimension \( D \) related to the structure of the fractal dimension is a positive constant applies only in the regime \( \xi >> q^{-1} >> a \), where \( a \) is a typical chemical or bond distance related to local structure and \( \xi \) is the correlation length or average diameter of a scatter. Here, we obtained that the mass fractal dimension is close to 1 which indicates the magnetite particle of Fe\(_3\)O\(_4\) aggregated to form a long-chain or fiber-like structure. A log-normal distribution spherical model calculation of dilute solution was fitted on the experimental data of MnZnFe\(_2\)O\(_4\) sample. The best fitting values are given as follow: the median radius of a primary particle of magnetite is c.a. 58 Å with the size of distribution \( \sigma \) is c.a. 0.12 – 0.14 which implies that the magnetic particle is entirely monodisperse. The experimental result from SMARTer is definitely as good as the result from HANARAO SANS which is analysed by applying the same SANS data reduction and analysis package programs.

4. Conclusion

The SANS BATAN spectrometer, SMARTer has been developed for improving its performance. The latest major developments were as follow (1) Replacement of a new user-friendly data acquisition system with some additional mode applications (2) Upgrading the control system for handling the device motor control using parallel interface connection (3) Setup the SANS data reduction and analysis using established package programs (4) Initiating a focusing SANS development using optical lenses. As described above, the spectrometer is now remarkably more powerful and useful for carrying out many experiments in materials science and biology research.

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Figure 4. The corrected neutron data on ferrofluids Fe$_3$O$_4$ sample in 1- and 2-dimensional profiles were taken using SMARTer: a-b and HANARO SANS: c-d.

Figure 5. The corrected neutron scattering data on ferrofluids MnZnFe$_2$O$_4$ sample in 1- and 2-dimensional profiles were taken using SMARTer: a-b and HANARO SANS: c-d.
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