Applications of $^3$He neutron spin filters on the small-angle neutron scattering spectrometer SANS-J-II

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Abstract. A polarized $^3$He neutron spin filter has been applied to polarization analysis on the small-angle neutron scattering spectrometer SANS-J-II at JRR-3. Measurements were taken on silver behenate, which has several coherent peaks in the small-angle region with a background of spin incoherent hydrogen scattering. Here we demonstrate that the coherent and spin incoherent scattering were successfully separated by the polarization analysis using the $^3$He spin filter on the instrument.

1. Introduction

Polarization analysis is a powerful technique to distinguish magnetic scattering from nuclear scattering and to separate spin incoherent scattering from nuclear coherent scattering. In the polarization analysis on a small-angle neutron scattering instrument, $^3$He neutron spin filters are useful because of their broad angular acceptance, which is limited only by the solid angle subtended by the $^3$He cell. Also, the $^3$He cell contributes little scattering in the small-angle region. The $^3$He neutron spin filters use very high spin-dependent neutron capture cross-sections of $^3$He gas and high $^3$He nuclear spin polarization is required for the scattering experiments. Recent progress on $^3$He polarization techniques enables us to use $^3$He neutron spin filters in a small-angle scattering instrument [1,2]. In Japan, a high $^3$He polarization technique based on spin-exchange optical pumping (SEOP) was established by Ino et al [3, 4] and our $^3$He neutron spin filter development group in Japan is improving on this technique [5, 6]. Current circumstances mean that it is now possible to obtain a supply of polarized $^3$He gas for the neutron scattering experiment in Japan. In this paper, we report on the applications of $^3$He neutron spin filters on the neutron small-angle scattering spectrometer
SANS-J-II at JRR-3 and demonstrate that $^3$He neutron spin filters are now available on the instrument. As a test sample, we have chosen silver behenate [7, 8] and tried to separate spin incoherent hydrogen scattering from coherent scattering of the sample.

2. Experiment

2.1 Spectrometer and experimental setup

The experiment was performed with the small-angle neutron scattering spectrometer SANS-J-II at JRR-3. In the spectrometer, polarization devices are fully installed and include an Fe/Si polarising supermirror, a two-coil π-flipper, a radio-frequency gradient π-flipper and a supermirror analyzer [9-12]. We used the polarizing supermirror to get a polarized incident neutron beam. The spin of the beam was flipped by a two-coil flipper. The scattered neutron beams were detected by 25 cm x 25 cm high-angle $^3$He detectors. The arrangement of the experimental apparatus is illustrated in Figure 1(a). We used a 4 cm diameter, sealed spin-exchange cell made of GE180 glass [13]. It was placed in a 54 cm long and 12 cm inner diameter solenoid coil. In order to get the maximum possible coverage of the scattering angle region, the sample was placed 17 cm behind the cell in the solenoid coil, as shown in Fig.1(b). The spin filter cell was filled with 6.7 atm-cm of $^3$He gas, which was chosen considering the neutron wavelength of the spectrometer, 6.5 Å. The spin-exchange cell was pumped up at our optical pumping station, and then, it was transported to SANS-J-II. To avoid unwanted scattering from the glass cell and the solenoid coil, the inside of the coil and the space around the cell were covered by B$_4$C rubber.

![Experimental apparatus](image)

Figure 1. Arrangement of experimental apparatus.

2.2 Sample

In this experiment, we have chosen silver behenate as the test sample. This is a crystalline long-chain silver carboxylate, CH$_3$;(CH$_2$)$_n$ COOAg. There is a (001) long-period spacing of 58.38 Å, which results from a tail-to-tail alignment of two silver behenate molecules. Due to the presence of order, the material is used for low-angle calibration of X-ray and neutron diffractometers in a $Q$-range of order 10$^{-1}$Å$^{-1}$. In the previous SANS experiment by Gilles et al., which was performed with an unpolarized neutron beam, an intensive first peak,
comparatively small 2nd and 3rd peaks, and a constant background component were observed. The ratio of these components is 1.00 : 0.12 : 0.06 : 0.29. The aim of this experiment is to subtract this background component, which would originate from spin incoherent hydrogen scattering.

3. Results and discussion

3.1. Neutron small-angle scattering for unpolarized and polarized beams

Figure 2 shows the small-angle neutron scattering profiles of silver behenate, which was obtained from an unpolarized neutron beam. The sample was placed in the solenoid coil, but the $^3$He neutron spin filter was not. The sample stage was fixed at 10°. The pattern is similar to that of Gilles et al., in which a strong first peak, weak second peak and constant background component are observed. The ratio of the first peak, the second peak and the background component is 1.00 : 0.1 : 0.23. This is consistent with the result measured by Gilles et al.

![Figure 2](image-url)

**Figure 2.** The small-angle neutron scattering of silver behenate, obtained from an unpolarized neutron beam.

Figure 3 shows the result obtained from a polarized neutron beam. To obtain the data, the absorption of the sample, the background measured by a scattering from the empty sample cell, and the sensitivity of the detector were corrected. There was a large loss in intensity compared to the measurement using an unpolarized beam. However, the difference in scattering pattern is clearly observed between “spin flipper on” and “off”: there are two peaks and a constant $Q$-independent component for “spin flipper on”, while there is only the constant $Q$-independent component for “spin flipper off”. From the result, it is found that the scattering for “spin flipper on” represents the scattering when the $^3$He polarization is parallel to incident neutron polarization, while scattering for “spin flipper off” represents scattering when the $^3$He polarization is antiparallel to the incident neutron polarization.
3.2. $^3$He polarization

The $^3$He polarization has been checked by measuring the transmittance through the $^3$He filter during the experiment. The initial $^3$He polarization was 34% and the relaxation time was 57 h. These values were less than those we could expect from previous measurements [14]. Because of the fast decay in $^3$He polarization, we expect that there was a disturbance in the magnetic environment during the experiment.

3.3. Separation of spin incoherent scattering from nuclear coherent scattering

The imperfection of spin-filtering by the $^3$He spin filter has to be corrected considering the incident neutron polarization and the transmission of neutrons through the $^3$He spin filter [15, 16]. For the incident neutron beam, the polarization efficiency of the supermirror polarizer is better than 0.99 and the flipping efficiency of the two-coil flipper is almost unity [12]. Therefore, we assume here that the polarization of the incident neutron beam is unity for both “spin flipper on” and “off”. In this case, the observed number of neutrons scattered by the sample for the incident neutron beam polarization parallel (anti-parallel) to the $^3$He polarization, $N_+$ ($N_-$), is expressed as:

$$N_+ = N_{nsf}T_+ + N_{sf}T_-$$
and

$$N_- = N_{nsf}T_- + N_{sf}T_+$$

where $T_+$ ($T_-$) is the transmission of the $^3$He analyzer for neutron spin parallel (antiparallel) to the $^3$He polarization. $T_+$ and $T_-$ are given by

$$T_+ = \exp\{-\sigma_0(1-P_{He})\rho l\}$$
and

$$T_- = \exp\{-\sigma_0(1+P_{He})\rho l\}$$

$\sigma_0$, $P_{He}$, and $l$ are the cross-section of $^3$He and its polarization, respectively.
where $\sigma$ is the absorption cross-section for unpolarized neutrons, $\rho$ is the number density of the $^3$He gas, and $l$ is the thickness of the $^3$He neutron spin filter. For $^3$He polarization, $P_{\text{He}}$, we used the value obtained in section 3.2.

From the above equations, we obtain:

$$N_{\text{nsf}} = \frac{1}{4} \exp(-\sigma_0 \rho l) \{ (N_e + N) / \cosh(\sigma_0 P_{\text{He}} \rho l) + (N_e - N) / \sinh(\sigma_0 P_{\text{He}} \rho l) \}$$

$$N_{\text{sf}} = \frac{1}{4} \exp(-\sigma_0 \rho l) \{ (N_e + N) / \cosh(\sigma_0 P_{\text{He}} \rho l) - (N_e - N) / \sinh(\sigma_0 P_{\text{He}} \rho l) \}$$ (3)

Using the relationship between non-spin-flip scattering, spin-flip scattering, coherent scattering and spin-incoherent scattering [17, 18]:

$$N_{\text{nsf}} = \frac{1}{3} N_e = N_c$$

$$N_{\text{sf}} = \frac{2}{3} N_e$$ (4)

where the subscripts refer to coherent (c) and spin-incoherent (si) scattering. $N_e$ and $N_{\text{sf}}$ are finally written as:

$$N_e = \frac{1}{8} \exp(\sigma_0 \rho l) \{ (N_e + N) / \cosh(\sigma_0 P_{\text{He}} \rho l) + 3(N_e - N) / \sinh(\sigma_0 P_{\text{He}} \rho l) \}$$

$$N_{\text{si}} = \frac{3}{8} \exp(\sigma_0 \rho l) \{ (N_e + N) / \cosh(\sigma_0 P_{\text{He}} \rho l) - (N_e - N) / \sinh(\sigma_0 P_{\text{He}} \rho l) \}$$ (5)

Figure 4 is the result of separation of the coherent and incoherent scattering components, in which these corrections are made. Although the data are scattered, the ratio of the intensity between the first peak and the incoherent component is about 1.00 : 0.23. This is consistent with the value obtained from an unpolarized neutron beam.

Figure 4. Separation of coherent and incoherent scattering for silver behenate.
4. Summary
We have applied $^3$He spin filters to polarization analysis on the small-angle neutron scattering spectrometer SANS-J-II. For the silver behenate test sample, the spin incoherent hydrogen scattering was separated from coherent scattering. From the results, it was shown that $^3$He spin filters can contribute to the upgrade in the focusing and polarized small-angle neutron scattering spectrometer, SANS-J-II. This method is applicable to other protonated polymers or biological samples where the presence of massive spin-incoherent scattering from hydrogen can hinder the determination of the coherent scattering. Also, we expect that this demonstrative experiment encourages further polarization analysis with other neutron scattering instruments at JRR-3 and J-PARC (Japan Proton Accelerator Research Complex). In the present experiment, the decay of $^3$He polarization was faster than expected. This could be caused by the conditions in the magnetic environment being insufficient and should be improved for our subsequent studies.

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