Polarization analysis in small-angle neutron scattering with a transportable neutron spin filter based on polarized protons

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Abstract. We present the status of development of a novel neutron spin filter based on the strong spin dependence of the neutron scattering on protons. Using photo-excited triplet states for the dynamic nuclear polarization (DNP) process, proton polarization values of 80% can now be achieved in pentacene doped single crystals of naphthalene at a field of 0.36 T in a simple helium flow cryostat. Careful sample preparation lead to extremely long polarization decay times under moderate conditions that allowed us to develop a transportable device. The filter is polarized in the lab under well controlled conditions and is then transferred to the neutron beam line where it can be operated during several days with almost constant polarization while requiring only a minimum of equipment. We describe briefly the main features of the spin filter and its use as a spin analyzer in a small-angle neutron scattering (SANS) experiment probing the magnetic structure of a nanocrystalline soft magnetic material. Furthermore the procedure for performing the background and spin leakage corrections of the neutron data is outlined in detail.

1. Introduction

In their classical experimental study Moon, Riste and Koehler [1] introduced the one dimensional polarization analysis and demonstrated the polarization dependence of nuclear and magnetic scattering, which allows the unambiguous separation of magnetic scattering from nuclear scattering. Only by a polarization analysis of the scattered neutrons, measuring the so called spin-flip process, which is free of coherent nuclear SANS and purely magnetic, one gets access to the magnetic scattering vector (Halpern-Johnson vector) [2] perpendicular to the neutron polarization. This information is crucial for the understanding of the magnetic response of magnetic materials in intermediate non saturating fields [3], as well as the unique spin structure of exotic non-collinear magnetic phases e.g. skyrmions [4]. Longitudinal polarization analysis (POLARIS) [5] in small-angle neutron scattering (SANS) experiments became only recently possible with the development of ³He spin filters [6] that allow the neutron spins from a divergently scattered beam to be assayed.

We have developed an alternative neutron spin filter [7] based on the strongly spin dependent scattering of neutrons on polarized protons [8]. The protons in a naphthalene single crystal are
polarized with a recently developed dynamic nuclear polarization (DNP) method \cite{9,10} allowing the construction of a compact device that is suitable for longitudinal polarization analysis in magnetic SANS \cite{11}. Unlike polarized $^3$He, polarized protons are not sensitive to stray magnetic fields, so that the filter can be placed close to the sample under investigation and a large angular acceptance is achieved even with a small filter cross section. Very recently we have realized a transportable device that is operated similar to an ex situ $^3$He spin filter.

This short note summarizes the most recent advances of our transportable triplet spin filter and describes its application for the polarization analysis in SANS of a nanocrystalline soft magnetic material. More details on the filter can be found in \cite{12}.

\section*{2. Transportable neutron spin filter based on polarized protons}

The neutron polarization process with polarized protons is based on scattering and not absorption, as is the case for polarized $^3$He. The working principle of a polarized proton spin filter is based on the fact that the singlet cross section for neutron-proton scattering is much higher than the triplet cross section \cite{13}. Neutrons with spin anti-parallel to the proton polarization will be much stronger scattered, while neutrons with spin parallel to the proton polarization are preferably transmitted. The relevant cross-sections are such that a polarized solid of a few mm thickness is sufficient to yield a high analyzing power over a broad wavelength range. Details on the principles of neutron spin filtering with protons and the corresponding cross-section values as well as a comparison of its efficiency with $^3$He are given in \cite{14}.

As base material for our spin filters we use high quality naphthalene single crystals doped with $\sim 2 \times 10^5$ mol/mol d-pentacene that are grown in a self-seeding vertical Bridgman technique \cite{15}. Cubic samples of typically 4 to 5 mm side length are then cut out of the large single crystals, while the top/bottom planes correspond to the crystal ac-plane. They are polarized by dynamic nuclear polarization (DNP) that uses short-lived optically excited triplet states of pentacene \cite{16} using a home built apparatus \cite{17}. Recently we have reached a record value of 80\% proton polarization at a field of 0.36 T using a simple helium flow cryostat for cooling. Properly tuned, the DNP process can run unattended until saturation is reached. Then the triplet exciting laser and the microwaves are switched off and the polarization is ”frozen”. The polarization decay times are very long even at temperatures around 80 K, so that the cryostat with the polarized filter can be demounted from the DNP apparatus and transported conveniently to the nearby neutron scattering facility. A small holding field of $\sim 20$ mT is sufficient to prevent any polarization loss until the device is installed in the electro-magnet on the neutron instrument and cooling is established again. The whole transport procedure takes about 30 min. The filter is then operated with a minimum of equipment, essentially a helium dewar, a temperature controller and a cw-NMR system to monitor the polarization. The latter is also used to reverse the proton polarization by adiabatic fast passage with an efficiency of above 99\%.

Of course the decay of the polarization during a typical several days neutron experiment decides on the ultimate feasibility of the adopted ex situ scheme. The relaxation depends on the filter temperature and the applied field. The latter is determined by the neutron experiment as the sample under investigation and the spin filter are placed at short distance in the same magnetic field provided by the electro-magnet \cite{11}. Ideally, the apparatus should be capable to operate at fields down of a few 10 mT, as required e.g. for SANS experiments probing the magnetism in bulk ferromagnets in the non saturated state. As the helium flow cryostat cooling the filter can be conveniently operated at any temperature down to about 6 K, the polarization decay can be strongly limited. With the recent progress in filter crystal preparation we achieve proton polarization relaxation times longer than 700 h at a field of 20 mT and a temperature of 6 K. This value increases significantly with the applied field \cite{12}. Thus the filter performance is stable over many days and no complicated time dependent corrections are necessary (see below).
3. Polarization analysis of magnetic SANS of FINEMET

In the following we describe the use of the transportable triplet spin filter and the data analysis procedure in the study of an example material, the Fe-based nanocrystalline soft magnetic material FINEMET (Fe$_{73.5}$Si$_{15.5}$B$_7$Nb$_3$Cu$_1$) [18]. Similar materials have been previously studied with unpolarized and polarized neutrons [19, 20].

3.1. Experimental setup and data taking

The experiment was conducted at the SANS-I instrument at SINQ at PSI [21]. A scheme of the experimental set up is given in Fig. 4 of [11]. The incoming beam was monochromatized by a velocity selector to a wavelength of $\lambda = 0.6$ nm with a spread of $\Delta \lambda / \lambda = 10\%$ and polarized with a V-shaped Fe/Si supermirror transmission polarizer to 0.98 at the collimations used. The neutron polarization could be reversed by means of an adiabatic spin flipper with an efficiency of 0.99. The detector was set at 6 m from the sample with 6 m beam collimation. The naphthalene spin filter crystal of $5.7 \times 4.9 \times 4.4$ mm$^3$ size doped with $2.5 \times 10^5$ mol/mol d-pentacene was polarized in the laboratory and then transported to the neutron scattering facility and installed in the electromagnet at the SANS instrument. A $4 \times 4$ mm$^2$ cadmium aperture, which is placed inside of the cryostat at a distance of 14 mm from the filter crystal, defined the analyzer cross section. A stack of 10 sheets of FINEMET, resulting in a sample thickness of about 0.2 mm, was mounted on a holder on the outer vacuum tube of the analyzer cryostat at a distance of 32 mm to the analyzer. The external magnetic field of $H = 0.88$ T provided by the electromagnet also served to preserve the analyzer polarization. The field was perpendicular to the incident neutron beam in the plane of the sample and sufficient to saturate the soft magnetic material.

With the experimental setup, it is possible to measure four intensities, $I_{\uparrow P}(Q)$, $I_{\downarrow P}(Q)$, $I_{\uparrow N}(Q)$, $I_{\downarrow N}(Q)$, where $\uparrow$ and $\downarrow$ correspond to the two neutron spin states selected by the spin flipper and $P$ and $N$ to positive or negative analyzer polarization. As is well illustrated in Figure 1, these experimental intensities will be, due to the imperfection of the spin filter, the spin flipper and the beam polarizer, a mixture of the four partial scattering intensities we are interested in, which are proportional to the elastic differential POLARIS cross sections [3, 20]. These are two non-spin-flip (nsf) quantities $S_{\uparrow\uparrow}$ and $S_{\downarrow\downarrow}$ and two spin-flip (sf) quantities $S_{\uparrow\downarrow}$ and $S_{\downarrow\uparrow}$, from which the micro magnetic properties of the sample under investigation can be obtained [3]. Here the vertical arrows in the subscripts refer to the neutron spin state before (left) and after (right) the scattering. Often a “$-+$” notation is used that relates to the neutron magnetic moment directions, which might cause confusion, as the neutron magnetic moment is anti-parallel to the spin direction while the proton magnetic moment (the analyzer) is parallel. The notations are related by simply replacing $\uparrow$ by $-$ and $\downarrow$ by $+$, so that

$$
\begin{bmatrix}
S_{\uparrow\uparrow} \\
S_{\downarrow\uparrow} \\
S_{\uparrow\downarrow} \\
S_{\downarrow\downarrow}
\end{bmatrix} =
\begin{bmatrix}
S^{--} \\
S^{+-} \\
S^{-+} \\
S^{++}
\end{bmatrix}.
$$

In a typical experiment a set of data is first measured for one analyzer state, e.g. $I_{\uparrow P}$, $I_{\downarrow P}$ together with the corresponding analyzer backgrounds, where the transmissions are monitored regularly in between the scattering runs. Afterwards the analyzer polarization is reversed by adiabatic fast passage (AFP) [22], which is well known in NMR and is also applied for $^3$He spin filters [23]. Then the other two channels are measured. The need to correct for the analyzer background, i.e. the isotropic incoherent neutron-proton scattering of the spin filter itself, is a principle disadvantage compared to a $^3$He spin filter, which just absorbs one neutron spin state. However, this correction can be straightforwardly applied. Due to the extremely long proton spin lattice relaxation time of the naphthalene filter, its transmission does hardly change over the course of an experiment. In fact, the spin filter keeps the same performance over several
Figure 1: Concept of the SANS experiment with the polarization analysis. In this example we show the case where both, the incident neutrons and the analyzer have a positive polarization. The resulting intensity on the detector is labelled as $I_{\uparrow P}$.

days of measurement, so that no time dependent corrections are necessary as is the case for $^3\text{He}$ cells [24].

3.2. Polarization and transmission dependent corrections of the neutron data

As already mentioned above, on the detector we will measure a mixture of the scattering of the sample and the analyzer of all four components (see figure 1), which need to be related to the four partial scattering intensities of the sample $S_{\uparrow\uparrow}$, $S_{\uparrow\downarrow}$, $S_{\downarrow\uparrow}$ and $S_{\downarrow\downarrow}$. To disentangle the four spin channels a leakage correction analogous to [25] needs to be performed. However by conducting measurements with the four different states of analyzer and spin flipper, we obtain a complete set of equations that allows us to determine the scattering cross sections:

$$
\begin{bmatrix}
    I_{\uparrow P} \\
    I_{\downarrow P} \\
    I_{\uparrow N} \\
    I_{\downarrow N}
\end{bmatrix}
= \mathcal{M}^A
\begin{bmatrix}
    S_{\uparrow\uparrow} \\
    S_{\uparrow\downarrow} \\
    S_{\downarrow\uparrow} \\
    S_{\downarrow\downarrow}
\end{bmatrix},
$$

where

$$
\mathcal{M}^A =
\begin{bmatrix}
    (p_n T_{\uparrow P}) & (1 - p_n) T_{\uparrow N} & (p_n T_{\downarrow P}) & (1 - p_n) T_{\downarrow N} \\
    (p_n + f - 2p_n f) T_{\uparrow P} & (p_n + f - 2p_n f) T_{\downarrow P} & (p_n + f - 2p_n f) T_{\uparrow N} & (p_n + f - 2p_n f) T_{\downarrow N} \\
    (1 - p_n) T_{\uparrow P} & (1 - p_n) T_{\downarrow P} & (p_n T_{\downarrow N}) & (p_n T_{\uparrow N}) \\
    (p_n + f - 2p_n f) T_{\uparrow N} & (p_n + f - 2p_n f) T_{\downarrow N} & (p_n T_{\downarrow N}) & (p_n T_{\uparrow N})
\end{bmatrix},
$$

is the transmission matrix of the analyser, where e.g. $T_{\uparrow P}$ is the transmission of neutrons with spin $\uparrow$ through a positively polarized analyzer. Here, $p_n$ is defined as the percentage of neutrons with spin parallel to the guide field and $f$ as the percentage of neutrons that are flipped when the flipper is active [25]. They are related to the beam polarization $P_n$ and the flipper efficiency $F$, respectively by $P_n = 2p_n - 1$ and $F = 2f - 1$. Typical values are $P_n = 0.98$ and $F = 0.99$. Notice, the quantities denoted with $S$ or $I$ are detector images and in our case matrices of 128 $\times$ 128 intensity values, while quantities denoted with $T$ are numerical values.

However, equation (2) is a simplification of the process as it only considers the scattering of the sample and the transmission of the analyser, without taking into account the homogeneous incoherent scattering of the analyser producing a background. Therefore (2) needs to be
extended to include the spin-dependent scattering of the analyzer:

\[
\begin{bmatrix}
I_{\uparrow P} \\
I_{\downarrow P} \\
I_{\uparrow N} \\
I_{\downarrow N}
\end{bmatrix}
= \mathcal{M}^A_T
\begin{bmatrix}
S_{\uparrow\uparrow} \\
S_{\downarrow\uparrow} \\
S_{\uparrow\downarrow} \\
S_{\downarrow\downarrow}
\end{bmatrix}
+ \mathcal{M}_T^S
\begin{bmatrix}
T_{\uparrow\uparrow} \\
T_{\downarrow\uparrow} \\
T_{\uparrow\downarrow} \\
T_{\downarrow\downarrow}
\end{bmatrix}
+ \text{"double scattering"}
\tag{4}
\]

where

\[
\mathcal{M}_T^S =
\begin{bmatrix}
(p_n + f - 2p_n f)S_{\uparrow P} & (1 - p_n)S_{\downarrow P} & (p_n + f - 2p_n f)S_{\uparrow N} & (1 - p_n)S_{\downarrow N} \\
(p_n + f - 2p_n f)S_{\uparrow P} & (1 + 2p_n f - p_n)S_{\downarrow P} & (p_n + f - 2p_n f)S_{\uparrow N} & (1 - p_n)S_{\downarrow N} \\
(p_n + f - 2p_n f)S_{\uparrow P} & (1 - p_n)S_{\downarrow P} & (p_n + f - 2p_n f)S_{\uparrow N} & (1 + 2p_n f - p_n)S_{\downarrow N}
\end{bmatrix}
\tag{5}
\]

is the scattering matrix of the analyzer. Analogous to above, \(S_{\uparrow P}\) e.g. is the scattering of neutrons with spin ↑ by a positively polarized analyzer, while \(T_{\uparrow\uparrow}\) and \(T_{\downarrow\downarrow}\) denote the sample transmission and depolarization of the spin ↑ component of the incident neutron beam. Additionally a small fraction of the neutrons is scattered by both the sample and the analyzer. The scattering by the analyzer is per se incoherent thus a second scattering just contributes to the background. We have experimentally determined that the scattering of the analyzer within the SANS detection area is less than 1% of the transmission, which means that the “double scattering” term is less than 1% of the first term in (4) and is therefore neglected in our analysis. Notice, the scattering of the analyzer contains a spin-independent part originating from all other background sources.

Since the scattering processes are detector images and hard to numerically evaluate in a matrix, we rewrite (4) in the form

\[
\begin{bmatrix}
I_{\uparrow P} \\
I_{\downarrow P} \\
I_{\uparrow N} \\
I_{\downarrow N}
\end{bmatrix}
= \mathcal{M}^A_T
\begin{bmatrix}
S_{\uparrow\uparrow} \\
S_{\downarrow\uparrow} \\
S_{\uparrow\downarrow} \\
S_{\downarrow\downarrow}
\end{bmatrix}
+ \mathcal{M}_T^S
\begin{bmatrix}
S_{\uparrow P} \\
S_{\downarrow P} \\
S_{\uparrow N} \\
S_{\downarrow N}
\end{bmatrix}
, \tag{6}
\]

where \(\mathcal{M}_T^S\) is the transmission matrix of the sample given by

\[
\mathcal{M}_T^S =
\begin{bmatrix}
m_T^S & 0 \\
0 & m_T^S
\end{bmatrix}
\tag{7}
\]

and \(m_T^S\) and 0 are 2 \times 2 matrices

\[
m_T^S = \begin{bmatrix}
(p_n + f - 2p_n f)T_{\uparrow\uparrow} + (1 - p_n)T_{\downarrow\uparrow} & (p_n + f - 2p_n f)T_{\uparrow\downarrow} + (1 - p_n)T_{\downarrow\downarrow} \\
(p_n + f - 2p_n f)T_{\downarrow\uparrow} + (1 + 2p_n f - p_n)T_{\uparrow\downarrow} & (p_n + f - 2p_n f)T_{\downarrow\downarrow} + (1 + 2p_n f - p_n)T_{\uparrow\downarrow}
\end{bmatrix}
. \tag{8}
\]

The transmission values of the sample and hence the elements of the transmission matrix \(\mathcal{M}_T^S\) can be obtained from a measurement of the total transmission \(T_{\text{tot}}\) through the sample and the analyzer, given by

\[
\begin{bmatrix}
T_{\uparrow\uparrow}^{\text{tot}} \\
T_{\downarrow\uparrow}^{\text{tot}} \\
T_{\uparrow\downarrow}^{\text{tot}} \\
T_{\downarrow\downarrow}^{\text{tot}}
\end{bmatrix}
= \mathcal{M}_T^S
\begin{bmatrix}
T_{\uparrow\uparrow} \\
T_{\downarrow\uparrow} \\
T_{\uparrow\downarrow} \\
T_{\downarrow\downarrow}
\end{bmatrix}
, \tag{9}
\]

where the analyzer transmission \(\mathcal{M}_T^A\) can be directly measured without sample.

We can now solve (6) to obtain the four partial scattering intensities

\[
\begin{bmatrix}
S_{\uparrow\uparrow} \\
S_{\downarrow\uparrow} \\
S_{\uparrow\downarrow} \\
S_{\downarrow\downarrow}
\end{bmatrix}
= \mathcal{M}_T^A^{-1}
\begin{bmatrix}
I_{\uparrow P} \\
I_{\downarrow P} \\
I_{\uparrow N} \\
I_{\downarrow N}
\end{bmatrix}
- \mathcal{M}_T^S
\begin{bmatrix}
S_{\uparrow P} \\
S_{\downarrow P} \\
S_{\uparrow N} \\
S_{\downarrow N}
\end{bmatrix}
, \tag{10}
\]
where the analyzer scattering intensities \( S_{\uparrow P}, \) etc.) can also be directly obtained from background measurements.

In the approximation of \( P_n = f = 1, \) (3) and (7) simplify to

\[
M_A^T = \begin{bmatrix}
T_{\uparrow P} & 0 & T_{\downarrow P} & 0 \\
0 & T_{\uparrow P} & 0 & T_{\downarrow P} \\
T_{\uparrow N} & 0 & T_{\downarrow N} & 0 \\
0 & T_{\uparrow N} & 0 & T_{\downarrow N}
\end{bmatrix}
\]

and

\[
M_S^T = \begin{bmatrix}
T_{\uparrow\uparrow} & T_{\uparrow\downarrow} & 0 & 0 \\
T_{\downarrow\uparrow} & T_{\downarrow\downarrow} & 0 & 0 \\
0 & 0 & T_{\uparrow\uparrow} & T_{\uparrow\downarrow} \\
0 & 0 & T_{\downarrow\uparrow} & T_{\downarrow\downarrow}
\end{bmatrix}
\]

Furthermore, in case the sample does not depolarize the beam, \( T_{\uparrow\downarrow} = T_{\downarrow\uparrow} = 0 \) and \( M_S^T \) becomes diagonal and (10) reduces to

\[
\begin{bmatrix}
S_{\uparrow\uparrow} \\
S_{\downarrow\downarrow} \\
S_{\uparrow\downarrow} \\
S_{\downarrow\uparrow}
\end{bmatrix} = M_A^{T^{-1}} \begin{bmatrix}
I_{\uparrow P} - T_{\uparrow\uparrow}S_{\uparrow P} \\
I_{\downarrow P} - T_{\downarrow\downarrow}S_{\downarrow P} \\
I_{\uparrow N} - T_{\uparrow\uparrow}S_{\uparrow N} \\
I_{\downarrow N} - T_{\downarrow\downarrow}S_{\downarrow N}
\end{bmatrix}
\]

Notice, we did not assume any symmetry between positively and negatively polarized analyzer. Actually both equations, (4) and (6) can be separated according to the analyzer polarization and also solved separately. This means we don’t require the polarization of the two analyzer states being the same, and one could even use two different analyzer crystals for the measurements.

Figure 2: Result of the spin leakage correction. In the experimental scattering image \( I_{\downarrow P} \) (left figure) the small spin-flip term is hidden by the strong \( \sin^2\theta \) non spin-flip term [20], which is responsible for the broad band at the top and the bottom. This band is completely removed with the correction and the at magnetic saturation predicted \( \sin^2\theta\cos^2\theta \) spin-flip term appears (right). Notice, the scales of the two plots are arbitrary and not related, since only the relative analyzer transmission is used in the analysis. The range of momentum transfers roughly corresponds to \( 0.1 \text{ nm}^{-1} < Q < 0.7 \text{ nm}^{-1} \).
3.3. Results
The above procedure has been applied to disentangle the four scattering channels in the case of
the FINEMET experiment. Figure 2 well illustrates the effect of this leakage correction for one
of the spin-flip channels. Even though the characteristic $\sin^2 \theta \cos^2 \theta$ spin-flip signal [20] is not
visible in the “raw” experimental detector image, it is nicely retrieved with the data treatment.

Similarly we have obtained all four scattering channels as shown in figure 3. The non spin-
flip channels have a $\sin^2 \theta$ shape while the spin-flip channels have a $\sin^2 \theta \cos^2 \theta$ shape. The
result is in excellent agreement with the theory [3] as well as a previous study of a similar
FeCr nanocrystalline alloy in saturation [20]. This experiment demonstrates that our neutron
spin filter, though presently still limited in the flipping ratio to below 5, can reveal the small
spin-flip signal of a magnetic scattering. This owes to the fact that the filter performance is
stable over extremely long time, so that no relaxation corrections are required. Furthermore the
transmission values necessary to perform the leakage correction can be very precisely measured.

![Figure 3: Corrected intensity plots of four spin channels by SANS polarization analysis on the
nanocrystalline alloy FINEMET. The range of momentum transfers roughly corresponds to 0.1
nm$^{-1}$ < $Q$ < 0.7 nm$^{-1}$.](image-url)
4. Outlook and conclusion

We have demonstrated that a neutron spin filter based on protons polarized by triplet DNP is perfectly suited to be used as spin analyzer in SANS experiments investigating magnetic properties of nanocrystalline materials, which so far have been be studied using cells of polarized $^3$He gas. The triplet spin filter is compact and works in inhomogeneous fields. Recent improvements in the preparation of the naphthalene filter crystals lead to very long relaxation times, which allowed to implement an \textit{ex situ} scheme for the filter operation. The filter is polarized in the laboratory and then transported to the nearby neutron scattering facility, where it is installed on the SANS instrument. A record proton polarization of 80\% has been measured on the neutron beam. To keep this high polarization, the filter crystal is cooled by a simple helium flow cryostat and a small magnetic field is applied. At a field of 20 mT, a proton spin lattice relaxation time of more than 700 h has been achieved, which will get considerably longer at higher magnetic fields. The filter thus provides a constant analyzing performance over a several day long neutron experiment so that time dependent corrections are unnecessary and a straightforward leakage correction can be applied to the SANS raw data. The spin-flip scattering of a nanocrystalline alloy FINEMET sample could be well revealed even with a flipping ratio of below 5. This limit is addressed by a new system that is presently under development that allows to polarize large crystals up to almost cm$^3$ size [14].

Acknowledgements

We wish to thank W.Th. Wenckebach for many helpful discussions. This work was supported by the Swiss National Science Foundation grant 200021,165496.

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