Abstract. 3D vector polarisation analysis called also SNP (Spherical Neutron Polarimetry) is a powerful method for the detailed investigation of complex magnetic structures. The precise control of the incoming and scattered neutron polarisations is essential for this technique. Here we show an instrumental setup, that was recently implemented on the new single crystal diffractometer POLI-HEiDi at the FRM II for performing SNP experiments using two $^3$He spin filters for the production and for the analysis of the neutron polarisation. The design and optimisation procedure for the used spin filter cells are presented. Methods for in-situ measurements of the incoming polarisation as well as the particularities of the using two spin filters and corrections for the time dependent relaxation are discussed. Statistical precision of 1% has been achieved for the measurements of the polarisation matrix under the real experimental conditions using described cells and applying proposed correction method for the data.

1. Introduction

Gaseous $^3$He neutron Spin Filter Cells (SFC) are simple transmissions devices, with predictable characteristics, that do not require any special optical adjustments on a neutron beam. Since polarized nuclei of $^3$He possess a very high spin-dependent neutron absorption efficiency over a wide range of energies, the $^3$He SFC can be used as a broadband neutron polariser or analyser, with the possibility to optimise its efficiency for nearly all neutron wavelengths. The use of $^3$He SFC allows for the decoupling the processes of neutron reflection and polarisation, unlike the case of the Heussler crystal polariser. The straight line transmission of the SFC allows the use of a focussed beam and gives a homogenous polarisation over the entire beam cross section. Up to now these advantages have been mostly used to produce SFC analysers especially for small angle neutron scattering or reflectometry [1, 2, 3]. However in the combination with hot neutrons SFC is a particularly efficient polarising device. According to the calculations [4] the combination of the focused Cu [220] crystal monochromator with $^3$He spin filter with 70% $^3$He polarisation will increase the efficiency of the instrument in the production of the hot polarised neutron beam by a factor of 1.7 compared to the Heussler crystal monochromator. Exactly this design has been employed in the new polarised diffractometer POLI-HEiDi. The advantage of this technique is a better resolution and a high flux of the polarised short wavelength neutrons on the sample. The drawback is a degradation of the incident neutron beam polarisation and analysing efficiency of the analyser with time. Really, when using one SFC as an analyser the measured resulting polarisation is proportional to the time dependent analysing efficiency of the cell. Therefore, precise knowledge of cells parameters allows for straightforward corrections of the data for this time dependence. However, when using two SFC as polariser...
and analyser in the same time, the measured resulting polarisation is proportional to a product of two independent time functions, so that the correction is only possible if polarisations of the $^3\text{He}$ gas in the polariser and analyser cells are measured independently. A way how to perform this is presented in the Sect. 4. In the Sect. 3 we describe the optimisation of cell’s parameters. Important to note that using the optimal cells parameters in order to maximise the figure of merit $Q$ and not the polarising efficiency $P_n$ lead to better results regarding the precision of experimental measurements. In the present report we demonstrate that by using the optimal SFC, the precise control of the incoming polarisation and accurately performed corrections for the time dependent depolarisation, the measurements of the polarisation matrices with the required precision are implemented on the new diffractometer POLI-HEiDi.

2. POLI-HEiDi diffractometer

The polarisation of the neutron beam can be described in terms of the classical vector and can be tuned in the frame of the instrument coordinate system. 3D vector polarisation analysis known also as Spherical Neutron Polarimetry (SNP) is a technique where all components of the scattered polarisation vector are measured in turn for three different directions of the incoming polarisation vector. Determining the relationship between the directions of incident and scattered polarisations gives access to the 16 independent correlation functions involved in the most general nuclear and magnetic scattering process. Generally, this allows for the determination of the direction of the magnetic interaction vectors of magnetic structures. For those structures, in which nuclear and magnetic reflections coincide in reciprocal space, SNP leads to the determination of the amplitude of the magnetic interaction vectors and hence to the magnetisation distribution [5]. SNP using third generation zero-field polarimeter CRYOPAD [6] have been recently implemented and successfully tested on the new single crystal diffractometer POLI-HEiDi at the FRM II [7,8]. The unique feature of the POLI-HEiDi compared to other instruments performing SNP is that both neutron polarisation production and analysis are done with $^3\text{He}$ spin filters. The parameters of the used spin filters are optimised in order to achieve the best performance in measuring polarisation at a given wavelength. Special DECPOL-like magnetostatic cavities

![Figure 1. (colour on-line). Instrument layout of the SNP - setup on POLI-HEiDi.](image-url)
have been constructed and built for the housing the cells during the experiment. The layout of the instrument is shown in the Fig. 1. Non-polarised focused monochromatic neutron beam (different wavelengths are available) from the HEiDi monochromator is coming from the right in the picture (see [9] for more details).

3. Spin filter cells

It has been shown in [10] that the statistical accuracy in the determination of the scattered beam polarisation is directly proportional to the square root of the quality factor $Q$, of the spin filter. The $Q$ value is denoted in terms of neutron polarisation $P_n$ and transmission $T_n$ as:

$$Q = P_n^2 T_n.$$  \hspace{1cm} (1)

The maximisation of $Q$ leads to the optimisation of the spin filter parameters. Usually spin filter cells are characterised by their opacity $O$ denoted as:

$$O = 0.0732 \cdot \frac{p}{\text{bar}} \cdot \frac{l}{\text{cm}} \cdot \frac{\lambda}{\text{Å}} = 0.0732 \cdot O',$$  \hspace{1cm} (2)

where $p$ is the pressure of the $^3\text{He}$ gas in the filter at 20°C, $l$ is the length of the neutron path through the gas, and $\lambda$ is the neutron wavelength. According to [11] the optimum opacity $O'_m$ at given $P_{He}$ is:

$$O'_m = \frac{1}{P_{He}} \cdot \text{arctanh} \left( \frac{1}{2P_{He}} + \sqrt{\frac{1}{4P_{He}^2} + 2} \right) \cdot \frac{\text{bar cm Å}}{0.0732}.$$  \hspace{1cm} (3)

Taking $P_{He} = 0.7$ as a realistic mean value that is routinely delivered by the HELIOS filling station at the FRM II [12], one obtains a simple expression for the optimal opacity of the used spin filters:

$$p\text{[bar]} \cdot l\text{[cm]} \cdot \lambda\text{[Å]} \approx 26.$$  \hspace{1cm} (4)

For a certain wavelength, the only parameters to optimise are the length of the cell $l$ and the gas pressure $p$. In the following, we will define the meaningful limits for both. It is worth noting, that we considered so far optimal conditions for the case $P_{He} = \text{const}$. However, after a cell had been detached from a filling station, the $P_{He}$ in the cell on the neutron instrument decreases

![Figure 2](image-url)  \hspace{1cm} \text{Figure 2. Pressure dependence of } T_1 \text{ in a surface relaxation free cell situated in a magnetic field with transversal gradient of } 5 \times 10^{-4} \text{ cm}^{-1}.  \hspace{1cm} (4)
exponentially with the time constant $T_1$ (relaxation time): 

$$P_{He}(t) = P_{He}(0) \exp\left(-t/T_1\right).$$  \hspace{1cm} (5)

Therefore, beside geometrical parameters of the cell, $T_1$ is another essential parameter for the spin filter. Three major relaxation mechanisms are generally accepted: 1) surface relaxation on a filter walls, 2) relaxation due to a magnetic field gradient $\nabla \left| B_z \right|/B_0$, in a magnetostatic cavity, 3) relaxation due to the dipole-dipole interaction inside the polarised gas. The last two mechanisms are pressure dependent. Considering an ideal surface relaxation free cell, $T_1$ can be written as:

$$\frac{1}{T_1[h]} = 1.7 \times 10^4 \cdot (\nabla \left| B_z \right| / B_0)^2 \frac{1}{p} + \frac{p}{750}.$$  \hspace{1cm} (6)

By taking for the field gradient a realistic value of $5 \times 10^{-4}$ cm$^{-1}$ one obtains the pressure dependence of $T_1$ shown in Fig. 2. It is easy to see that $T_1$ reaches the maximum at the pressure close to 2 bar. We assume now that the same cell will be used in experiments with different neutron wavelengths and the optimization will be done by tuning the pressure. If we accept that the reduction of $T_1$ due to pressure tuning should not overcome 10% of $T_1^{max}$, then we come to the conclusion that the optimal pressure region expands from 1.3 to 3 bar for a gradient of $5 \times 10^{-4}$ cm$^{-1}$.

Figure 3. (colour on-line). 130 mm long and 60 mm in diameter SFC used at POLI-HEiDi. The cells are made of high purity quartz glass and coated inside with metallic Cs. Small Cs droplets can be observed in the lower part and in the appendix of the cell on the left. The dark spots inside the cell on the right are Cs sub-oxides, which are liquid at room temperature.

Now the length of the cells requested for the operation with neutrons with wavelengths from 0.55 Å to 1.17 Å can be easily determined from Eq. 4. The following practical limitations have to be considered as well: a) the geometry of the polariser and analyser cells should be
the same (exchangeability), b) the space available at the instrument for the polariser magnetostatic cavity is limited to max. 50 cm - this means that cells should be as short as possible to achieve a high magnetic field homogeneity. Taking into account all mentioned criteria, cells with length $l = 13$ cm have been produced for the POLI-HEiDi. The cell diameter should be large enough to cover whole incoming beam reaching the sample position as well as the complete scattered beam reaching the detector. Considering the beam spot at the sample position of about 30 mm in diameter and the convergent incoming beam, cells of 60 mm in diameter have been designed. Typical cells used on POLI-HEiDi are shown in Fig. 3. Fig. 4 presents the calculated quality factor of our cells for two different filling pressures at $P_{He}$ = 0.7. Maximising of the Q for almost all available on HEiDi monochromator wavelengths is possible using the same cell by tuning the pressure.

![Figure 4](image)

Figure 4. Quality factor of the used cells calculated for two different pressures and $P_{He}$ = 0.7. The filled data points correspond to available discrete wavelengths, the connecting lines are the eye-guides showing the wavelength dependent behaviour.

The cell windows are made from the same type of fused silica material as the cell body and are 3-4 mm thick in order to withstand the pressure up to 3 bar. They are welded to the cells body. The neutron transmission of empty cells $T_0$, measured for the used wavelengths is about 0.86-0.88 depending on the window thickness. Relaxation times $T_1$ measured in different cells after the preparation varies between 100 h and 200 h, when $T_1$ up to 260 h have been measured in some of the cells. $T_1$ of any particular cell can also vary with time depending on the number of cell fillings, slow Cs oxidation, magnetic history of the cell, etc.

4. Polarisation measurements and time-dependent corrections

In the preceding sections we discussed following advantages of the $^3$He SFC: high efficiency for hot neutrons, easy adjustment for the different wavelengths, flexibility regarding beam geometry, etc. The importance of the $T_1$ has been also mentioned. Time dependency of the polarising (analysing) efficiency of the filters represents a main drawback of the technique requiring additional corrections, which lead to some decrease of the statistical accuracy of the measurement. Since the precise knowledge of the incoming and scattered beam polarisation is essential for the SNP, in the following we will describe briefly the correction procedure applied to the data measured on POLI-HEiDi. We note that the accurate error analysis of the correction procedure is not the subject of the present report and it will be treated.
The correction is possible in principle due to the fact that the filter's performance follows the known theoretical predictions. In order to apply them, two conditions should be satisfied for each measurement. First, the precise knowledge of the filter parameters such as opacity and $T_1$ is required both for polariser and analyser. Second, a time mark should be assigned to each measured experimental value.

We denote with $n^+$ and $n^-$ the counting rates for the neutrons with the parallel and antiparallel direction of the polarisation vector with respect to the magnetic field in the analyser, respectively. The experimentally measured asymmetry $A$ has the meaning of the product of scattered neutron beam polarisation $P'$ and analysing efficiency of the analyser $A_n$:

$$A = P' A_n.$$  

Measuring $n^+$ at the time $t_1$ and $n^-$ at the time $t_2$ ($t_2 - t_1 << T_1$), we define the scattered beam polarisation at the mean time $t_m = (t_1 + t_2)/2$, $P'(t_m)$ as:

$$P'(t_m) = \frac{n^+(t_1) - n^-(t_1)}{n^+(t_1) + n^-(t_2)} \cdot \frac{1}{A_n(t_m)}.$$  \n
In SNP, the usual experimental strategy is to measure the scattered beam polarisation $P'$ when the incident polarisation $P$ is set alternatively along $x$, $y$ or $z$ axis. This determines the polarisation matrix. The matrix element $P'_{ij}$ gives the $i$th component of the scattered polarisation vector when the incident polarisation is in the $j$th direction. Using the time marking and the same time scale for the polariser and analyser, the precise measurement of $P'_{ij}$ is getting possible. The incident polarisation $P(t_m)$ at the moment $t_m$ can be precisely calculated using transmission measurements from two neutron monitors, placed before and after the polariser (see Fig. 1). Transmission of the polariser $T$ is a simple normalised ratio between the two monitors:

$$T(t) = T_0^p P_{e} - O_p \cosh(O_p P_{He}^p(t)).$$  

where $T_0^p$ is the transmission of the empty polariser cell, $O^p$ is the opacity of the polariser cell defined by Eq. 2, $P_{He}^p(t)$ is the time dependent polarisation of $^3$He gas in the polariser cell defined by Eq. 5. Fitting the experimental transmission data (Fig. 5) using Eq. 11 results in precise values for the $^3$He initial polarisation and $T_1$ of the polariser cell. The refined values for the presented experiment are shown in the inset of Fig. 5: $P_{He}^p(0) = 0.7363(3)$, $T_1^p = 93.77(22)$ h. In Fig. 6 the calculated time-dependent polarisation of the incoming beam is shown for the data plotted on Fig. 5. The measurement of the scattered polarisation matrix element $P'_{ij}$ according to Eq. 9 requires a precise knowledge of the time-dependent analysing efficiency $A_n(t)$:

$$A_n(t) = \tanh (O^p P_{He}^{o}(0) \exp(-t/T_1^s),$$  \n
where \( O^a \) is the opacity of the analyser cell, \( P^{He^a}_0(0) \) is the polarisation of the \(^3\)He gas at the time moment zero (initial gas polarisation), \( T^a_1 \) is the relaxation time constant of the analyser.

Figure 5. Time-dependent transmission of the polariser cell measured using two transmission monitors situated before and after the polariser. The result of the least squares fit of \(^3\)He initial polarisation and \( T^a_1 \) according to Eqs. 5 and 11, are given in the inset.

Using known \( O^a \) the problem reduces to the determination of the initial polarisation and \( T^a_1 \) in the analyser. One can use different ways to do this. First, by analogy with the procedure described for the polariser, one can measure transmission of the analyser cell with the incident non-polarised beam. This has been done in our first SNP experiment with two spin filters using MuPAD [12], where the polariser cell had to be removed temporarily from the incoming beam to perform the measurement only with the analyser cell. This is not technically foreseen in the actual POLI-HEiDi design, therefore it could be only done for two time points: before the measurement when the polariser cell is not yet installed into the incoming beam and after the measurement, when the polariser cell is removed from the beam for the replacement with the “fresh” polarised gas. Knowing transmissions over a definite time interval and assuming that depolarisation follows the form given by Eq. 5, one can calculate \( P^{He^a}_0(0) \) and \( T^a_1 \) using an expression analogous to Eq. 11. However, as shown in practice, the values derived only from two measurements have a rather high uncertainty.

Another method that allows time-dependent corrections to measured data is based on the transmission measurement of the partially polarised beam according to expression derived in Ref. [13]:

\[
T^a_\tau(t) = T^a_0 \exp(-O^a) \cosh(O^a P^{He^a}_\tau(t)) \left[1 + P^\tau_\eta(t) \tanh(O^a P^{He^a}_\tau(t))\right],
\]

(13)

where \( P^\tau_\eta(t) \) is the time-dependent polarisation of the incident beam (see Fig. 6). Although measurements in arbitrary number of points is possible and the subsequent least square fit will improve the accuracy of the refined parameters, this is nevertheless an indirect method, which also includes the uncertainty in the calculation of \( P^\tau_\eta(t) \). Moreover, the transmission measurements should be done in the direct beam or at least on a pure nuclear Bragg reflection in order to be sure that the polarisation of the incoming beam is not changed due to the interaction with the sample.
Time dependent measurements of non-magnetic Bragg reflection permits, however, a direct measurement of $A_n(t)$. In this particular case $A_n(t)$ can be easily found by normalising the measured asymmetry (Eqs. 7 and 8) to the incoming polarisation measured earlier. Taking the predefined $O$' to be a fixed parameter and fitting $A_n(t)$ to measured data with Eq. 12, one obtains the required parameters of the analyser cell: $P_{He}(0)$ and $T_1$. Fig. 7 shows an example of the time-dependent analyser efficiency calculated for the set of experimental data presented in Fig. 5 and 6 measured with a nuclear Bragg peak. Due to high counting rates in nuclear Bragg peaks, the total time spent for additional measurements necessary for the correction parameter determination is less than 5% of the total measurement time.

**Figure 6.** Time-dependent polarisation of the incoming beam $P(t)$ derived from the transmission data in Fig. 5. The thick line in the graph includes the error bars.

**Figure 7.** Measured time dependent analysing efficiency of an analyser cell. The result of the least squares fit gives the $^3$He initial polarisation and $T_1$ according to Eqs. 5 and 12. The reason for the unusually low $T_1$ observed in the DECPOL [8] in this measurement is a wrong current setting in the coils.
5. Results and conclusion

Measured and corrected polarisation matrices for two Bragg reflections [200] and [005] are presented in Table 1. The measurement have been done on a HoNi$_2$B$_2$C superconducting sample at 4.2K at the POLI-HEiDi diffractometer using the described setup and neutrons with the wavelength of 0.87 Å. The Bragg peak [200] from this sample is a purely nuclear one and thus a singular diagonal matrix has been measured with a high precision. Conversely, the [005] peak is a purely magnetic reflection, and rotation through 180° for the $P_{xx}$ component is seen. Other diagonal components are totally depolarised that suggests the presence of the equally populated 180° magnetic domains in the structure. All diagonal and off-diagonal components could be determined with the precision of 1% using the described correction procedure.

Table 1. Polarisation matrices for two different Bragg peaks from a HoNi$_2$B$_2$C sample “as measured” and corrected for $^3$He time relaxation.

| Bragg peak     | $P_{in}$ / $P_{out}$ | Asymmetry as measured | Corrected polarisation matrix |
|----------------|----------------------|-----------------------|------------------------------|
|                |                      | x                     | y               | z               | x                     | y               | z               |
| 200 nuclear    |                      | 0.730(3)              | 0.003(5)         | 0.009(5)        | 0.99(1)               | -0.00(1)         | 0.01(1)         |
| 005 magnetic   |                      | -0.652(12)            | 0.004(11)        | -0.032(11)      | -1.00(3)              | 0.01(1)          | -0.06(1)        |

The SFC with the optimised parameters are employed to produce and analyse neutron polarisation at the new polarised diffractometer POLI-HEiDi (RWTH Aachen) at FRM II. The use of SFC assures a high resolution and the optimal efficiency for hot neutrons. Spherical neutron polarimetry using the third generation zero-field polarimeter CRYOPAD for the investigation of complex magnetic structures is implemented on POLI-HEiDi. The correction procedure for the time-dependent polariser (analyser) efficiency has been developed and successfully applied.

Acknowledgements:
The work has been supported by the German Federal Ministry for Education and Science (BMBF) through the projects 03HE7AAC and 03HE6AA3.

References:
[1] Lelievre-Berna E et al. 2005 Physica B: Condensed Matter 356 141.
[2] Keiderling U, Wiedenmann A, Rupp A, Klenke J and Heil W 2008 Meas. Sci. Technol. 19 034009
[3] Chen W C, Erwin R, McIver J W, Watson S, Fu S B, Gentile T R, Borchers J A, Lynn J W, and Jones G L, 2009 Physica B: Condensed Matter 404 2663
[4] Lelievre-Berna E and Tasset F, 1999 Physica B: Condensed Matter 267 21
[5] Brown P J, 2005 Spherical Neutron Polarimetry, in Neutron Scattering From Magnetic Materials, ed. Chatterji T
[6] Lelievre-Berna E, Anderson I and Guérard B , Eds., 2002 Proceedings of SPIE 4785 112
[7] Hutanu V, Meven M and Heger G, 2007 Physica B: Condensed Matter 397 135
[8] Hutanu V, Meven M, Lelievre-Berna E and Heger G, 2009 Physica B 404 2633
[9] http://www.frm2.tum.de/wissenschaftliche-nutzung/diffraction/poli-heidi/index.html.
[10] Tasset F and Ressouche E, 1995 Nucl. Instr. and Meth. A 359 537
[11] Surkau R et al. 1995 Nucl. Instr. and Meth A 384 444
[12] Hutanu V, Masalovich S, Meven M, Lykhvar O, Borchert G and Heger G, 2007 Neutron News 18 14
[13] Hutanu V, Janoschek M, Meven M, Böni P and Heger G 2009 Nucl. Inst. and Meth. A 612 155
[14] Masalovich S, Nucl. Inst. and Meth. A 581 791