Polarization analysis on the LET cold neutron spectrometer using a $^3$He spin-filter: first results

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Abstract. We report on the design and construction of a neutron polarization analyzer based on a $^3$He spin filter for the LET spectrometer at the ISIS facility. Its commissioning completes the uniaxial polarized mode of the instrument. Besides the $^3$He spin filter cell, the analyzer consists of a set of electromagnetic coils to provide a uniform magnetic field, and a collimator to eliminate background from the sample environment and analyzer infrastructure. The $^3$He cell is separated from the LET main tank vacuum by means of a gas tank, which permits rapid cell changeovers and guarantees safe operation. In addition to the analyzer hardware, software has been written to reduce and correct polarized data. The successful operation of the polarized mode was realized for the separation of coherent and incoherent dynamics in an organic solar cell polymer blend.

1. Introduction

Time-of-flight neutron spectroscopy is a technique used to probe the dynamics of condensed matter over a broad range of space ($1 - 100$ Å) and time ($10^{-4} - 10$ ns). It is typically used to map coherent quasi-particle excitations such as phonons and magnons, as well as to investigate local motions such as ionic diffusion. Frequently, however, the dynamics of interest are hidden beneath other scattering contributions; for example, the quasi-elastic scattering signal arising from ionic diffusion of Li$^+$ or Na$^+$ cations in battery electrolytes is usually dwarfed by the elastic scattering from the remainder of the lattice (1). In these situations, longitudinal polarization analysis – where the scattered polarization of a neutron beam is analyzed along the initial polarization direction – can be used to separate the contributions to the total neutron scattering cross section, i.e. the coherent nuclear, magnetic, and nuclear-spin incoherent cross sections (2). One of the main obstacles to realizing longitudinal polarization analysis on a time-of-flight spectrometer is the large solid angle of a typical detector array which has to be covered by the polarization analyzer. This renders conventional technologies such as polarizing supermirrors and crystals costly or impractical, making hyper-polarized $^3$He spin filters as the method of choice (3; 4; 5; 6).

In this paper we describe the design, construction and testing of a $^3$He spin filter-based analyzer for uniaxial polarization analysis on the LET time-of-flight spectrometer at the
ISIS Neutron and Muon Source, UK. We will begin by reviewing our previous work on the characterization of the polarized primary spectrometer in section 2 (7), before discussing the analyzer hardware, comprising a $^3$He cell, holding field coils, a radial oscillating collimator, and supporting infrastructure in section 3. Following this, we will consider the problem of correcting for the finite polarizing efficiency of the polarizer, neutron spin flipper, and analyzer, and provide a description of the resulting data reduction work-flow in section 4. Finally, we present preliminary results from the first polarized experiment on single-particle and collective motions in a solar cell polymer blend in section 5.

2. Overview and previous work

LET is a direct geometry cold neutron time-of-flight spectrometer located on the second target station (TS2) at ISIS. A liquid H$_2$ moderator and focusing guide system provide a high flux of neutrons in the energy range $\sim 1 - 30$ meV (8). Multiple incident energies $E_i$ are selected from this spectrum by a series of five disc choppers [Figure 1]. All of these $E_i$ can be measured simultaneously using so-called “repetition rate multiplication” (9). Another key feature of LET is its large detector array, providing $\sim \pi$ st of continuous detector coverage.

The polarized incident beam on LET is generated by a two-channel “V-cavity” polarizer, located approximately 2.5 m upstream of the sample position (10). This is immediately followed by a precession coil neutron spin flipper and guide fields leading up to the sample tank. In a previous paper (10) we have shown that the polarizer delivers an energy-independent neutron polarization of $\sim 96\%$ for $E_i = 1 - 9$ meV, with a transmission relative to the unpolarized incident beam of $\sim 40\%$ at 9 meV and $\sim 35\%$ at 1 meV, respectively. The flipper was found to be $\sim 99\%$ efficient between 2 and 9 meV at constant current.

3. Analyzer design and implementation

Our highest priority in selecting the type of polarization analyzer for LET was to take maximum advantage of the instrument’s large, continuous detector coverage. $^3$He spin filters were therefore...
identified as the best option given the cost and practical difficulties associated with wide-angle supermirror devices. The main design criteria for the analyzer on LET were:

- Full coverage of the solid angle of the LET detector array.
- No shadowing of the detector by e.g. the holding field coils or their supporting framework.
- Ensuring that field inhomogeneities do not contribute noticeably to $^3\text{He}$ depolarization by achieving a transverse relative field gradient $\nabla B(\langle B \rangle/|B| > 10^{-3} \text{ cm}^{-1}$ in the entire volume filled by the $^3\text{He}$ cell.
- $^3\text{He}$ cell changeovers that are rapid (< 5 min) and reproducible.
- Minimization of the background scattering from the sample environment, the $^3\text{He}$ cell and its surrounding infrastructure, by using an integrated radial (oscillating) collimator.
- Easy correction of the data for the time-dependent analyzing efficiency and transmission, by continuous and precise monitoring of the $^3\text{He}$ polarization.

Based on these criteria, the resulting conceptual design and its integration within the instrument is shown in Figure 1 (7) and a cross section of the detailed design is shown in Figure 2(a).

Starting from the sample position and moving radially outwards; the analyzer insert consists of a cryostat with non-magnetic tails to which a $^3\text{He}$ cell whose size matches the solid angle of the detector is mounted via a shelf; the cryostat and cell are both located inside a gas tank which is in turn installed in the LET main vacuum tank. Separating the $^3\text{He}$ cell from the main tank vacuum facilitates rapid cell changeovers by removing the need for pumping the very large (40m$^3$) volume of the latter after each change. To further speed these up, the cryostat and cell can be lifted out of the tank using a set of guide rails (Figure 2(b)), reducing the time for each replacement to around 5 minutes. The gas tank can be filled with any inert gas (typically Ar), increasing the maximum operating pressure of the $^3\text{He}$ cell at the expense of only a small increase in background.

Moving to the outside of the gas tank (and thus to the inside the LET main vacuum tank), the scattered beam is covered by a radial oscillating collimator, which defines a 20 mm cylindrical volume at the sample position, outside which the scattering is collimated away. This is a crucial part of the design given the large amount of material the beam travels through following scattering; in sequence, the cryostat tails, the inner and outer $^3\text{He}$ cell walls, and the gas tank. Located above and below the collimator are the holding field coils, which consist of a main and a compensation pair; these provide a highly homogeneous magnetic field for the $^3\text{He}$ cell. The final element is the direct beam monitor (Not shown in Figure 2(a)), located directly behind the collimator, which tracks the time-dependent polarization of the instrument, and hence allows for correction of the data. The completed analyzer insert is shown in Figure 2(b). We will now provide some additional detail on the design and construction of the cell, the coils, and the collimator.

3.1. $^3\text{He}$ cell

The dimensions of the so-called “banana cell” spin filter used on LET were chosen to optimize the analyzing power and transmission of the cell in the wavelength range of the polarizer, namely $3 – 10 \text{ Å}$. The inner radius of the cell is 50 mm to accommodate the outer cryostat tail, with an outer radius of 120 mm and 5 mm thick walls to provide 6 bar cm of $^3\text{He}$ pressure length at 1 bar pressure; this results in a polarization and transmission of 92% and 28%, respectively, at a neutron energy of 3.3 meV, given a $^3\text{He}$ polarization of 70%. To cover the vertical angle of the LET detector, the height of the cell is 170 mm. The cell is constructed from fused quartz (Heraeus HSQ300 amorphous SiO$_2$), which has the advantages of high neutron transmission, ready availability, and high purity. Scattering from the quartz produces a broad, diffuse background which is mitigated almost entirely by the radial oscillating collimator (see
Figure 2. (a) Cross-section of the analyzer, including the holding field coils (1), the cryostat (2), the gas tank (3), the sample position (4), the $^3$He cell (5), and the radial oscillating collimator (6). (b) The assembled analyzer insert with cryostat removed. The rails at the top (7) facilitate rapid changeover of the $^3$He cell by allowing the cryostat to be removed and replaced reproducibly.

$^3$He spin filters are often coated internally with alkali metals such as caesium or rubidium to both neutralize surface paramagnetic surface ions, and reduce adsorption of $^3$He on the cell walls (12). This minimizes depolarizing interactions, thereby improving the characteristic cell lifetime $T_1$ over which the $^3$He polarization decays, $p_{^3He} = \exp\left(-t/T_1\right)$. In order to coat the banana cell, 2 g of caesium were inserted using a pipette in an argon-filled glove-box, and the cell was baked overnight at $\sim$200°C to allow the caesium to vaporize and coat the interior surfaces. Immediately after construction, the LET cell had a lifetime of 5 hours, which improved to 23 hours after caesium coating. Another attempt was made to improve the lifetime of the cell by baking it further, but a small leak in the cell valve caused slow oxidation of the caesium over the period of $\sim$24 hours. The oxidized cell used for the test in section 5 had a resulting $T_1 = 12$ hours. At the time of writing, the cell is being re-coated with caesium after a thorough cleaning. A second cell is also under preparation using a different cleaning protocol.

To facilitate real-time monitoring of the $^3$He polarization, a monitor is located in the path of the transmitted beam, behind the collimator. A small pinhole is drilled in the beam-stop to allow sufficient intensity through to this monitor to measure the flipping ratio of the transmitted beam. This is necessary to allow corrections for the time dependent analyzing power of the $^3$He spin filter [see section 4].

3.2. Coils

The optimization of the coil geometry was performed by simulated annealing, and is outlined in (7). The final design consists of a main pair of coils with an outer diameter of 695 mm and 300 turns and a counter-wound compensation pair with an outer diameter of 416 mm and 69 turns. This arrangement permits large coil separations, 334 mm and 410 mm for the compensation and main pair, respectively, providing the desired large vertical opening angle of $\pm 28.1^\circ$. Furthermore, the transverse relative field gradient $\nabla B(\mathbf{B})/|\mathbf{B}|$ (with respect to the average field direction) was predicted to be better than $8 \times 10^{-4}$ cm$^{-1}$ over the entire volume of
the cell (13), ensuring a field-gradient $T_1 > 1000$ hours, around an order of magnitude greater than the wall relaxation $T_1$ of a typical $^3$He cell. The coils provide a field of $|B| = 11$ G at a current of 2.5 A.

The magnetic field homogeneity of manufactured coils was tested by measuring the $T_1$ of a cylindrical $^3$He cell of known wall relaxation lifetime $T_1 = 190$ hours. The contribution to the $^3$He relaxation from field inhomogeneities was found to be negligible. As an additional measurement of the field quality, the relative transverse field gradient was mapped using a Hall probe mounted to a XYZ translation stage, and found to be $< 1 \times 10^{-3}$ (upper limit defined by the instrumental sensitivity) across the entire cell volume. Testing of the coil heating under vacuum revealed only a slight increase in temperature to $\sim 40^\circ$C at 2.5 A.

3.3. Collimator

As mentioned above, a key feature of our analyzer design is the inclusion of a radial oscillating collimator to eliminate background from the sample environment and analyzer infrastructure. To illustrate the need for a collimator, we simulate the backgrounds from the cryostat tails, gas tank, and $^3$He cell with either no sample or a quartz rod of diameter 10 mm at the sample position using the UNION component (14; 15) in the McStas package (16). The simulations without a sample indicate the structure of the background, while those with the quartz rod provide an indication of the relative magnitudes of the signal and background. The results for $E_i = 3$ meV and no quartz rod are shown in Figures 3(a-c), and the results with the quartz rod are shown in Figures 3(d-f). Scattering from the front and back of the gas tank and the cryostat tails produce the “V”-shaped background features in Figure 3(a), while the cell causes the inelastic features between 0.2 and 0.6 meV. The scattering from the quartz rod introduces some secondary reflections which pass the collimator in the simulation, however these are eliminated in the experimental case using cadmium foil which was not included in the simulation. Several additional features are observed above the Bragg cutoff of Al at $\sim 6$ meV, but are not shown here.

Given the spatial constraints imposed by the gas tank and the compensation coils, the radial oscillating collimator has an inner diameter of 266 mm and an outer diameter of 410 mm, with blades spaced every $3^\circ$. It is thus optimized for a sample diameter of 20 mm. The blades are double-coated with $10 \mu$m of absorbing $^{10}$B, which results in a transmission of $< 0.1\%$ for background neutrons. The overall transmission from the sample volume is 80%. The simulations show that the reduction in the background is 87% of the integrated intensity across the detector. Crucially, the remaining background is concentrated at small angle and on the elastic line, which means it does not interfere with the inelastic scattering. A comparison between the simulation and experimental background data shows reasonable agreement, with the small excess of scattering at small angles being caused by a slightly misaligned beamstop. The agreement is also good for the quartz sample, except for some background features in the simulations which were eliminated using absorbing cadmium screens in the experiment. The signal to noise ratio in the measured spectrum is better than 1000:1, granted for a large and strongly scattering sample.

4. Polarization corrections

In a polarized neutron experiment imperfections in the polarizing optics introduce a degree of “leakage” between polarization channels. If the magnitude of this leakage is similar to the signal to be measured via polarization analysis, it will obscure the signal. This is addressed by quantifying the inefficiency of the polarizing devices and correcting the number of counts in each polarization channel accordingly.

Polarization corrections on LET are carried out using the instrumental flipping ratio $R$, which is defined as,
Figure 3. LET spectra illustrating background reduction due to the radial oscillating collimator, \( E_i = 3 \) meV: (a) Simulated - no collimator, no sample; (b) Simulated - with collimator, no sample; (c) Experiment - with collimator, no sample; (d) Simulated - no collimator, quartz rod sample; (e) Simulated - with collimator, quartz rod sample; (f) Experiment - with collimator, quartz rod sample. Simulated backgrounds are all on the same logarithmic intensity scale, however the real backgrounds have been arbitrarily scaled for qualitative visual comparison. The scattered intensity from a quartz rod at sample position is \( > 10^3 \) times greater than the background intensity in both simulation and experiment. Some features of the simulated background have been mitigated in the experimental case using cadmium foil which was not included in the simulation model.

\[
R = \frac{N^\uparrow}{N^\downarrow} \tag{1}
\]

where \( N^\uparrow, N^\downarrow \) are the counts in a detector in each polarization channel when the polarization of the beam is not modified by the sample. The scattered intensity in this detector from the sample of interest can then be corrected using (17),

\[
I^\uparrow_{\text{corrected}} = I^\uparrow + \frac{1}{R - 1}(I^\uparrow - I^\downarrow), \tag{2}
\]

where \( I^\uparrow, I^\downarrow \) is the intensity in a detector in each polarization channel. This correction method is valid in the limit that the incident polarizing efficiency is equal in each channel, i.e. the flipper efficiency \( f \approx 1 \).

For a \(^3\)He analyzer, \( R \) has both a geometrical and time dependence which may be written as the product of the polarizer and analyzer efficiencies,

\[
\frac{R - 1}{R + 1} = P \cdot \tanh(O \cdot p_{\text{He}}(t) \cdot g(\theta, \alpha)), \tag{3}
\]

where \( P \) is the incident polarizing efficiency, \( O \) is the in-plane opacity of the \(^3\)He analyzer (which is a function of the straight through pressure length of the cell and the neutron wavelength),
Figure 4. Polarised data correction workflow for LET. All of the required calculations are implemented using the MANTID data reduction package.

$p_{He}(t)$ is the time dependent $^3$He polarization, and $g(\theta, \alpha)$ is a factor representing the deviation of the average neutron path length through the analyzer in the direction $(\theta, \alpha)$ where $\theta$ and $\alpha$ are the azimuthal and polar scattering angles respectively. The $^3$He polarization decays exponentially with time with a characteristic lifetime $T_1$,

$$p_{He}(t) = p_{He}(t_0) \cdot \exp\left(-\frac{t-t_0}{T_1}\right).$$

(4)

The form of $g(\theta, \alpha)$ depends upon the geometry of the sample being measured. In the cylindrically symmetric geometry of LET, $g(\theta, \alpha)$ reduces to a function of the out of plane scattering angle, $g(\alpha)$. For small samples, $g(\alpha)$ is closely approximated by $\sec(\alpha)$ (exact in the limit of a point sample), however for larger samples this is increased by a multiplicative constant that can be obtained from numerical simulation. For example, the path length increases by $\sim 3.2\%$ for a 40mm diameter cylindrical sample and $\sim 0.7\%$ for a 20mm diameter cylindrical sample.

In order to obtain values for $p_{He}(t_0)$ and $T_1$, a monitor is placed behind the sample position to measure $R$ in the transmitted beam. Because of its non-linear form and three fitting parameters, fitting a flipping ratio time series to Equation 3 directly is unreliable. Instead, $P$ is obtained by fitting the out of plane scattering angle dependence of $R$ (measured across the whole detector using a diffuse scatterer like quartz, which does not affect the polarization of the beam) - a problem involving only two parameters. Fitting the time dependence is then simplified to fitting an exponential decay to

$$p_{He}(t) = \frac{1}{O} \cdot \text{atanh}\left(\frac{1}{P} \cdot \frac{R - 1}{R + 1}\right),$$

(5)

where $g(\theta, \alpha) = 1$ as $R$ is being measured using the straight through beam. Once $p_{He}(t_0)$, $T_1$, and $P$ have been determined, $R$ can be calculated for any time $t$ and detector pixel $(\theta, \alpha)$. This
Figure 5. Out-of-plane angular dependence of the product of the polarizer and analyzer efficiency for several values of $^{3}$He analyzer polarization (legend) at a constant polarizer efficiency $P = 0.96$, simulated in McStas. With the flux of LET, this quality of data would require around 3 hours of measurement to obtain. The overlaid black lines show fits to the geometrical dependence of this product using the sec($\alpha$) approximation. These fits yield values of $p_{He}$ of, from top to bottom, 59.5±0.6%, 52.2±0.3%, 45.0±0.1%, 37.4±0.1%, 30.0±0.1%. The fits also yield $P = 96.6 \pm 0.7\%$ (averaged over the fits for each value of the $^{3}$He cell polarization), consistent with the simulated incident neutron polarization.

The workflow is summarized in Figure 4. An optional extra step to reduce the uncertainty in $O$ is to determine it directly from a transmission measurement of a depolarized cell. We again stress that this procedure is only strictly speaking valid for $f = 1$, and that deviations $\delta f$ in the flipper efficiency will introduce errors of $O(\delta f^2)$ (for small $\delta f$) in the correction. This will mainly be relevant in the case of samples with either a very large or small flipping ratio.

To demonstrate the feasibility of this correction procedure, McStas simulations of the polarised option on LET were carried out. In Figure 5, the flipping ratio product in Equation 3 is shown as a function of $\alpha$ for several values of the $^{3}$He analyzer polarization. Also shown are fits to the geometrical dependence of the data using the secant approximation to $g(\alpha)$ for a 20mm radius cylindrical sample.

All of the above procedures are integrated in the MANTID data reduction package at no extra burden to the user compared with the unpolarized data reduction.

5. First results
The design and setup described was tested on a solid polymer-fullerene blend poly(3-hexylthiophene) (P3HT) and [6,6]-Phenyl-C$_{61}$ butyric acid methyl ester (PCBM), which shows promise as the photoactive layer in organic photovoltaic cells. The efficiency of photoconversion in this type of device has previously been linked to the dynamics of the polymer side-chains, which are thought to be strongly affected by the blending process (18). Indeed, previous quasielastic neutron scattering experiments have shown that blending appears to cause a slowing
Figure 6. Normalized and Q-integrated spectra for both the 1:1 P3HT:PCBM blend and pure P3HT taken at 430 K and resolutions of 0.084 meV (top) and 0.041 meV (bottom).

down of the polymer side-chain dynamics (19; 20; 21; 22). Unambiguously resolving whether this apparent slowing down is simply due to an increase in coherent scattering in the blend versus the pure polymer is not trivial, however, given the relatively large coherent contribution (∼10%) to the scattering after blending. To resolve this issue, polarized time-of-flight spectroscopy was carried out on the D7 instrument, and found to show no clear coherent contribution to the dynamics for either the pure polymer or the blend within the statistics or energy resolution (∼0.1 meV) of the experiment (20).

Here, the dynamics in both pure P3HT and a 1:1 P3HT:PCBM blend were investigated at higher resolutions, up to 0.025 meV, using polarized quasielastic neutron scattering on LET. A set of incident energies $E_i = 3.01$ meV, 1.73 meV, and 1.01 meV were selected by the LET chopper system, yielding resolutions of 0.084 meV, 0.041 meV, and 0.025 meV at the elastic line, respectively. The neutron spin flipper was operated in white-beam mode, where the current is ramped as $\propto 1/t$ in time to flip all $E_i$ in a pulse. Although it was not possible to unambiguously determine the flipper efficiency without a second flipper installed, we estimate that $f > 98\%$ for all $E_i$. The $^3$He analyzer cell used was that described in section 3, which had a very short lifetime of $T_1 = 12$ hours, and the initial polarization of the $^3$He cell was typically around 56 – 60%. Polarization corrections were carried out according to the procedure outlined in section 4; the consistency of the data with that reported previously, and the nearly zero coherent scattering away from the elastic line for the polymer blend both provide confidence in the accuracy of the
correction procedure.

Both the pure P3HT and 1:1 P3HT:PCBM samples were prepared as described previously (20), and loaded in annular aluminium sample cans. These were attached to a non-magnetic sample stick with two cartridge heaters placed in a copper block above the sample. Spectra were taken at 10 K for resolution, 330 K and 430 K. Despite the relatively poor polarization and $^3$He lifetime, good quality data could be collected in 6 hours for each temperature. This is especially notable given the small coherent cross section.

The coherent (predominantly collective) and incoherent (single particle) contributions to the scattering were separated from the corrected spin-flip and non-spin-flip intensities, $I_{\text{corrected}}^{\uparrow\uparrow}$ and $I_{\text{corrected}}^{\downarrow\uparrow}$, using

$$I_{\text{coh}} = I_{\text{corrected}}^{\uparrow\uparrow} - \frac{1}{2} I_{\text{corrected}}^{\downarrow\uparrow},$$

$$I_{\text{SI}} = \frac{3}{2} I_{\text{corrected}}^{\uparrow\uparrow}.$$

The normalized spectra integrated over the $Q$-range $0.8 - 1.6 \, \text{Å}^{-1}$ are shown for both samples at a temperature of 430 K and resolutions of 0.084 meV and 0.041 meV in Figure 6. Both resolutions reproduce the previous result for the incoherent scattering, which contained a sharp ($\sim 0.1$ meV) and broad ($\sim 1$ meV) Lorentzian quasi-elastic component. The former is, however, much more clearly resolved than before due to the better resolution of the present measurement. Interestingly, our data suggest a significant coherent quasi-elastic contribution in the pure P3HT sample, not observed in previous measurements (20). This presumably originates from the polymer side-chain back-bone. The disappearance of this component in the blend therefore implies that the slowing down proposed previously is a real phenomenon. A more complete analysis of the data will be reported in a future publication.

6. Conclusion

We have presented the design and commissioning of a polarized neutron analyzer on the LET spectrometer at the ISIS facility. The most important features of our design are:

(i) the large vertical size of the spin filter and opening angle of the coils, permitting access to the entire LET detector

(ii) the radial oscillating collimator, which eliminates nearly all of the background from the sample environment and $^3$He cell

(iii) the rapid changeover of the $^3$He cell, facilitated by the gas tank and a system of rails

(iv) the user-friendly data reduction protocol, based on the use of the vertical resolution and continuous monitoring of the $^3$He cell transmission.

The commissioning of the analyzer, and thus the completion of the polarized mode on LET, culminated in a successful test experiment on the solar cell polymer blend P3HT:PCBM. This illustrates the power of LET for polarized quasi-elastic scattering on non-magnetic samples. The application of LET to magnetic samples will be reported in a future publication.

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