Development and application of a $^3$He Neutron Spin Filter at J-PARC

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A B S T R A C T

We are developing a neutron polarizer with polarized $^3$He gas, referred to as a $^3$He spin filter, based on the Spin Exchange Optical Pumping (SEOP) for polarized neutron scattering experiments at Materials and Life Science Experimental Facility (MLF) of Japan Proton Accelerator Research Complex (J-PARC). A $^3$He gas-filling station was constructed at J-PARC, and several $^3$He cells with long spin relaxation times have been fabricated using the gas-filling station. A laboratory has been prepared in the MLF beam hall for polarizing $^3$He cells, and compact pumping systems with laser powers of 30 W and 110 W, which can be installed onto a neutron beamline, have been developed. A $^3$He polarization of 85% was achieved at a neutron beamline by using the pumping system with the 110 W laser. Recently, the first user experiment utilizing the $^3$He spin filter was conducted, and there have been several more since then. The development and utilization of $^3$He spin filters at MLF of J-PARC are reported.

1. Introduction

A $^3$He spin filter is a neutron polarization device in which polarized $^3$He gas is enclosed in a special glass cell that does not contain boron. $^3$He spin filters are widely used to polarize neutrons and analyze neutron spins in neutron scattering experiments because of its capabilities to polarize neutrons in broad energy range and cover wide solid angle [1–6]. Since $^3$He nuclei have a strongly spin-dependent neutron absorption cross section, neutrons passing through polarized $^3$He gas are polarized. $^3$He nuclei are polarized using laser techniques known as Spin Exchange Optical Pumping (SEOP) [7] or Metastability Optical Pumping (MOP) [8]. In SEOP, $^3$He gas, $^2$H$_2$ gas, and pure rubidium or rubidium and potassium are encapsulated within a glass cell. The $^3$He or Rb-K cells are heated to around 170 °C or 220 °C, respectively, to evaporate the alkali metals, and circularly polarized laser light irradiates the cell. Unpaired electrons of alkali atoms are polarized by optical pumping, and the spins of the electrons are exchanged with those of $^3$He nuclei by hyperfine interaction. A diode laser array with output power on the order of 100 W and a wavelength of 794.7 nm, which corresponds to the D$_1$ absorption line of Rb, is typically employed. The $^3$He polarization can be improved by using Rb-K compared to the pure Rb case because of the high optical pumping efficiency of Rb-K [9]. When the laser irradiation is stopped, the $^3$He polarization exponentially decays due to non-uniformity of the surrounding magnetic field, collisions of $^3$He atom with each other, and collisions of $^3$He atoms with impurities in the glass cell and a glass wall.

Ex-situ and in-situ methods are used for $^3$He spin filters with SEOP. In the ex-situ method, a $^3$He cell is polarized in a pumping station away from a neutron beamline, transferred in a magnetic cavity to keep the $^3$He polarization, and installed to a neutron beamline. Since the $^3$He polarization decays during a neutron experiment, the $^3$He cell needs to be periodically exchanged with another polarized $^3$He cell for long-running experiments. However, the advantage of using ex-situ method is that it can be used for a neutron beamline with limited space, and the installation of the instrument is straightforward. This is beneficial for spallation neutron sources, which generally have small experimental spaces. As there are many experiments with short
For details of the technique for optically polarized methods for their versatile utilization of $^3$He spin filters at MLF of J-PARC. In this paper, we report development and utilization of $^3$He spin filters at MLF of J-PARC.

2. Gas-filling station

As mentioned in the previous section, a clean gas-filling station and fabrication process without impurities are important for a $^3$He cell with long relaxation time. The first gas-filling station was constructed in 2018 at J-PARC. The schematic diagram of the gas-filling station is shown in Fig. 1. All the gas lines are wrapped with heaters for vacuum bake-out. An ultimate pressure of the gas-filling station was $5 \times 10^{-8}$ Pa after baking at 150 °C for a few days.

Boron-free aluminosilicate GE180 glass is widely used for the glass cell of a $^3$He spin filter due to a small wall relaxation effect and low permeability of $^3$He [4,19–21]. Our fabrication process of $^3$He cells is similar to those of NIST, JCNS, and ORNL [19–21]. A cylindrical-shaped glass cell made of GE180 is attached to a glassware made of Pyrex glass, which is referred to as “string”, (Fig. 2). The glass cell and the string are rinsed with neutral detergent, pure water, acetone, and alcohol before connecting to the gas-filling station. Rubidium and potassium ampoules are also rinsed with acetone and alcohol. After connecting the string to the gas-filling station, the ampoules are put in the string without breaking the ampoules, and the string is sealed off with a gas torch. The ampoules are broken in a nitrogen atmosphere by dropping hammers made of an iron rod covered with glass. The string and the glass cell are baked out for a week at 200 °C and 400 °C, respectively. In the next step, rubidium and potassium are distilled to retorts at 200 °C and 220 °C, respectively, by using the heaters. After that, the glass tube parts containing ampoules and hammers are pulled off with a gas torch. The glass cell and the string are baked out again for one week, and then the alkali metals in the retorts are distilled to the glass cell. Finally, $^3$He and N$_2$ gases purified using GC50 getters (SAES Getters) are filled to the glass cell, and the glass cell is sealed off by placing a gas torch at the Pyrex part. When encapsulating the gases above 1 atm, the glass cell is submerged in liquid nitrogen to keep the pressure inside the glass cell below 1 atm while sealing. So far, nine $^3$He cells have been fabricated with $^3$He pressures of 3.1 atm using the gas-filling station at J-PARC, and four of them have found to have spin relaxation times over 150 hours. A list of these $^3$He cells and a photograph of Sekichiku cell are presented in Table 1 and Fig. 3, respectively.

| Name   | Dimensions [mm] | $^3$He pressure [atm] | $T_i$ [h] |
|--------|-----------------|-----------------------|-----------|
| Chidori | $\phi 45 \times 75$ | 3.1                    | 201       |
| Karigane| $\phi 45 \times 75$ | 3.1                    | 180       |
| Sekichiku| $\phi 60 \times 60$ | 3.1                    | 175       |
| Hanabishi| $\phi 40 \times 90$ | 3.1                    | 165       |

Table 1. List of $^3$He cells using rubidium and potassium with long relaxation times at J-PARC.
3. NMR and pumping systems

3.1. $B_0$ coil and NMR system

To keep the $^3$He polarization, a coil with double magnetic shields, referred to as $B_0$ coil, is mainly used for the laser pumping [22]. Fig. 4 shows a cross-sectional view of the $B_0$ coil and a photograph of its interior. The coil is consisting of a main solenoid, two compensation coils, and two side coils. The dimensions of the $B_0$ coil are 29.2 cm in diameter and 36.8 cm in length. The $B_0$ coil was designed using the finite element method to make the field gradient less than $5 \times 10^{-4}$ T/cm in a cylindrical region with a diameter of 10 cm and a length of 10 cm at the center of the coil. A magnetic field of 1.5 mT is produced to keep the $^3$He polarization. Frequency-sweep Adiabatic Fast Passage (AFP)-NMR was employed to flip the $^3$He spin as well as to evaluate the $^3$He polarization [17]. A cosine winding is used as a drive coil to apply an oscillating magnetic field to the $^3$He cell. The $^3$He precession signal is detected by a pick-up coil located under the $^3$He cell. The coils were installed inside the $B_0$ coil as shown in Fig. 4. The polarization loss due to spin flip was measured as $3.8 \times 10^{-5}$ per flip. The $^3$He cell is heated by electrical heating using a rubber heater with a power of 100 W wound around the drive coil during optical pumping.

3.2. Pumping station at MLF

We have set up a laboratory in the MLF beam hall to polarize $^3$He cells. $^3$He cells are polarized at the laboratory can be provided to neutron beamlines in MLF. A pumping system using an air-cooling laser module with a laser power of 30 W [23] has been installed in the laboratory (Fig. 5). The dimensions of the pumping system are 60 cm × 60 cm × 40 cm, and it is used for both in-situ and ex-situ methods. Using a volume Bragg grating (VBG) element in the laser module, the center wavelength of the laser is tuned to 794.7 nm with a line width of 0.2 nm in Full Width at Half Maximum (FWHM). The laser light is circularly polarized using a quarter-wavelength plate installed in the module. The circularized laser light from the laser module is reflected by dielectric multi-layer coated quartz mirrors and irradiates the $^3$He cell. The temperature at the cell surface is kept about 170 °C for a pure Rb cell and about 210 °C for a cell with rubidium and potassium during optical pumping. A $^3$He polarization of ~70% is achieved with this laser system. A polarization measurement system using Electron Paramagnetic Resonance (EPR) [24] is also equipped.

3.3. Pumping system using a fiber laser

In order to achieve higher $^3$He polarization, we have developed a separate pumping system using a fiber laser made by DILAS Diode Laser, Inc. (M1F4S22-794.7 [0,6]-100C-IS9), as shown in Fig. 6. The pumping system using this fiber laser has been constructed at another laboratory in the J-PARC research building, about 30 m away from the MLF experimental hall. This fiber laser is an air-cooled diode laser with a maximum output power of 110 W. The center wavelength is tuned to 794.7 nm with a line width of 0.4 nm in FWHM using a VBG element. An optical fiber with an internal diameter of 400 μm is connected to the laser shield box. An unpolarized laser beam from the fiber exit is split into two paths by a polarized beam splitter. The laser beams are circularly polarized with quarter-wavelength plates. The polarized laser beams irradiate both sides of a $^3$He cell. The degree of circular polarization in each path was measured using a polarimeter as more than 98% at the $^3$He cell position. The dimensions of the pumping system are 70 cm × 70 cm × 40 cm, and it can also be used for the in-situ method. The pumping system is currently used only for the ex-situ method, and will be installed in the laboratory at MLF in the near future after following a safety review.

4. Performance evaluation of the $^3$He spin filter

We conducted an experiment at the NOBORU beamline [11,25], which is a test neutron beamline for general purpose, to evaluate the $^3$He polarization for the Chidori cell, which has the longest $T_1$ as shown in Table 1. The $^3$He cell was polarized using the pumping system with the fiber laser at J-PARC research building. Time evolution of the $^3$He polarization $P_{^3He}(t)$ during optical pumping can be described as

$$P_{^3He}(t) = P_{^3He}^0 (1 - \exp(-t/\tau)), \quad \text{(1)}$$

where $t$ is the time from the start of optical pumping, $P_{^3He}^0$ is the $^3$He polarization at $t \to \infty$, and $\tau$ is a polarization build-up time. The time evolution of the $^3$He polarization in this experiment is shown in Fig. 7, and the build-up time $\tau$ was 3.5 hours. For reference, the typical build-up time is 6.5 hours when the pumping system with 30 W laser is used.

After the $^3$He polarization was saturated, the $^3$He cell, the $B_0$ coil, and the NMR system were transferred to the NOBORU beamline in MLF while applying a magnetic field to the $^3$He cell using a battery to keep the $^3$He polarization. Neutron transmission was measured using a Gas Electron Multiplier (GEM) detector [26] for the polarized and unpolarized $^3$He cell as well as for an empty glass cell with the same
Fig. 5. (a) Compact pumping system which can be installed on a neutron beamline. The optical system has been constructed in the laser shield box. (b) Air cooling laser module. The laser power is 30 W.

Fig. 6. (a) Schematic diagram of the pumping system using a fiber laser. (b) Photograph of the pumping system. The optical system was constructed in the laser shield box.

Fig. 7. Time evolution of the $^3$He polarization measured by AFP-NMR during optical pumping. The $^3$He polarization saturates at about 24 h after the start of the laser irradiation. The temperature at the surface of the cell was 205 °C during optical pumping.

dimensions. The number of the transmitted neutrons through the $^3$He cell is described as [27]

\[ N_0 T_{\text{cell}}(\lambda) \exp(-\rho d \sigma(\lambda)) \cosh(P_{^3\text{He}} \rho d \sigma(\lambda)), \]  

(2)

where, $N_0$ is the number of incident neutrons, $T_{\text{cell}}$ is the transmission of the cell windows, $\sigma(\lambda)$ is the detection efficiency of the GEM detector, $P_{^3\text{He}}$ is $^3$He polarization, $\rho$ is the number density of $^3$He nuclei, $d$ is the thickness of $^3$He gas, $\sigma(\lambda)$ is the absorption cross section of the $^3$He nucleus for unpolarized neutrons, and $\lambda$ is the wavelength of incident neutrons, which is calculated from the time of flight. The ratio of transmitted neutrons for the polarized to unpolarized $^3$He cell $N_{\text{pol}}/N_{\text{unpol}}$ is described as

\[ \frac{N_{\text{pol}}}{N_{\text{unpol}}} = \cosh(P_{^3\text{He}} \rho d \sigma(\lambda)). \]  

(3)

The product of the $^3$He gas pressure and thickness $\rho d$ was obtained from the measurement of the ratio of transmitted neutrons for the unpolarized $^3$He cell to the empty glass cell as 20.8 ± 0.1 atm-cm. The measured ratios of $N_{\text{pol}}/N_{\text{unpol}}$ are plotted against the neutron wavelength in Fig. 8 along with the curve of best fit using Eq. (3) with $P_{^3\text{He}}$ as a free parameter. The $^3$He polarization was measured to be 85.3 ± 0.5%. The uncertainty comes mostly from the measured $\rho d$.

The neutron wavelength dependences on the neutron polarization is obtained using the following equation:

\[ P_n = \sqrt{1 - \frac{T_0^2}{T^2}}, \]  

(4)

where $P_n$ is the polarization of neutrons passed through the polarized $^3$He cell, $T$ is the transmission of polarized $^3$He cell, and $T_0$ is the transmission of the unpolarized $^3$He cell. The transmissions $T_0$ and $T$
The pumping system prior to 2020 were conducted using the pumping system with the 30 W laser used for the method in the experiments conducted in 2020. The pumping system with the 30 W laser as shown in Fig. 5. The in-situ system with a \(^3\)He cell of 17 atm-cm and the ex-situ system with \(^3\)He cell of 11 atm-cm were used as a neutron spin polarizer and analyzer, respectively. The dimensions of the \(^3\)He cells were 35 mm in diameter and 55 mm in length for both. A coil with a 0.35 mm-thick ring-shaped magnetic steel core was placed between the polarizer and the analyzer as a sample. A two-dimensional RPMT neutron detector [32] was set after the analyzer. The magnetic field applied to the sample was controlled with the current sent to the coil. Two-dimensional images of the neutron polarization were obtained, and obvious differences in the spin rotations of neutrons were observed as the wavelength dependence of the neutron polarization in two current conditions, \(I = 1.42\) A and \(I = 0\) A. This result indicates that it is possible to perform an experiment for magnetic imaging with \(^3\)He spin filters at J-PARC.

Furthermore, a study for magnetic atomic resolution holography with polarized neutrons using a \(^3\)He spin filter is currently underway. Two user experiments for this study were conducted at NOBORU in 2019–2020.

### 5.2. BL04 ANNRI

BL04 ANNRI [11,33] is a neutron beamline used for studies of nuclear science such as nuclear data for nuclear technology, astrophysics, and quantitative analyses. A germanium detector assembly using 22 high-quality germanium crystals is installed on this beamline to measure the \((n, \gamma)\) reaction. Measurements of the \((n, \gamma)\) reaction for the study of fundamental symmetry violation in nuclei have been conducted as a user experiment using a \(^3\)He spin filter [34–36]. A \(^3\)He spin filter with the ex-situ system was used to polarize epithermal neutrons to measure the \((n, \gamma)\) reaction with polarized neutrons at the 0.74 eV neutron resonance of \(^{139}\)La. A lanthanum sample was placed at the center of the germanium detector assembly, and the emitted \(\gamma\)-rays from the sample were detected by the germanium detectors. The \(^3\)He cell, solenoid, and compensation coils with a magnetic shield made of permalloy were installed between the detector and a neutron collimator (Fig. 10). The magnetic field used to keep the \(^3\)He polarization was perpendicular to the neutron beam in order to obtain a vertically polarized neutron beam. The magnetic shield had two openings in the path of the neutron beam to avoid a decay of the neutron polarization. Neutron polarization was held by the magnetic field produced with the guide magnets to a lanthanum target. A \(^3\)He cell using pure Rb with a pressure thickness of 19.3 atm-cm and dimensions of 50 mm diameter and 70 mm length was used. The \(^3\)He cell was polarized with the 30 W laser system at MLF, and the \(^3\)He polarization at the start of the measurement was typically 60\%. The relaxation time of the \(^3\)He polarization was 130 h. In 2019, as a result of three separate measurements using the \(^3\)He spin filter, a significant spin-dependent angular distribution of \(\gamma\)-rays from the 0.74 eV neutron resonance of \(^{139}\)La was observed [36].

Furthermore, a \((n, \gamma)\) reaction measurement using polarized epithermal neutrons to obtain nuclear data for reactor science is being planned, and the development of a \(^3\)He cell for epithermal neutrons is an ongoing process.

### Table 2

| No. | Name       | Purpose of beamline | Type of \(^3\)He spin filter |
|-----|------------|---------------------|-------------------------------|
| 04  | ANNRI      | Nuclear reaction    | ex-situ                       |
| 05  | NOP        | Fundamental physics | ex-situ                       |
| 06  | VIN ROSE   | Spin echo           | ex-situ                       |
| 10  | NOBORU     | General purpose     | ex-situ, in-situ              |
| 15  | TAIKAN     | SANS                | ex-situ, in-situ              |
| 17  | SHARAKU    | Reflectometer       | in-situ                       |
| 18  | SENUJ      | Single crystal diffraction | ex-situ                  |
| 22  | RAISE      | Pulsed neutron imaging | in-situ                   |

### 5. Trial and user experiments using \(^3\)He spin filter

We have carried out several trial experiments with in-situ and ex-situ SEOP methods at neutron beamlines of MLF [28–31]. In 2017, the \(^3\)He spin filter was first supplied to a user experiment. Although no user experiment was conducted in 2018, five user experiments were performed in 2019–2020. Neutron beamlines, where we conducted experiments using \(^3\)He spin filters, are listed in Table 2. In this section, we report on experiments performed on typical beamlines at MLF using \(^3\)He spin filters, including the results already reported. The experiments prior to 2020 were conducted using the pumping system with the 30 W laser, while the pumping system with the 110 W laser was used with ex-situ method in the experiments conducted in 2020. The pumping system with the 30 W laser used for the in-situ experiments described in this section will be also replaced by that with the 110 W laser in the near future.

#### 5.1. BL10 NOBORU

A trial experiment for magnetic imaging was carried out [28] with in-situ and ex-situ systems using \(^3\)He cell with pure Rb and the 30 W laser as shown in Fig. 5. The in-situ system with a \(^3\)He cell of 17 atm-cm and the ex-situ system with \(^3\)He cell of 11 atm-cm were used as a neutron spin polarizer and analyzer, respectively. The dimensions of the \(^3\)He cells were 35 mm in diameter and 55 mm in length for both. A coil with a 0.35 mm-thick ring-shaped magnetic steel core was placed between the polarizer and the analyzer as a sample. A two-dimensional RPMT neutron detector [32] was set after the analyzer. The magnetic field applied to the sample was controlled with the current sent to the coil. Two-dimensional images of the neutron polarization were obtained, and obvious differences in the spin rotations of neutrons were observed as the wavelength dependence of the neutron polarization in two current conditions, \(I = 1.42\) A and \(I = 0\) A. This result indicates that it is possible to perform an experiment for magnetic imaging with \(^3\)He spin filters at J-PARC.

Furthermore, a study for magnetic atomic resolution holography with polarized neutrons using a \(^3\)He spin filter is currently underway. Two user experiments for this study were conducted at NOBORU in 2019–2020.

#### Fig. 8. Ratio of the numbers of transmitted neutrons for the polarized to the unpolarized \(^3\)He cell. The red line shows the best fit. The error bars show the statistical uncertainty.

#### Fig. 9. Neutron wavelength dependences of neutron polarization (open circles) and neutron transmission (filled circles).

are described as

\[
T_0 = \frac{N_{\text{pol}}}{N_{\text{cell}}}, \quad T = \frac{N_{\text{pol}}}{N_{\text{cell}}},
\]

where \(N_{\text{cell}}\) is the transmitted neutrons for the empty glass cell. The neutron wavelength dependence of the neutron polarization and the neutron transmission are shown in Fig. 9. Each measurement took 15 min. The relaxation time \(T_1\) on the beamline was 170 h, which was measured with AFP-NMR.

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implies that diffusion of $^3$He gas does not affect the MIEZE signal and a $^3$He spin filter can be used as a second analyzer in the MIEZE spectrometer. Furthermore, in 2020, an experiment to measure the spin dynamics of ferrofluid was conducted by using a $^3$He spin filter as shown in Fig. 12. The $^3$He polarization of 72% and relaxation time of 120 h was achieved on this beamline in the experiment, and the analysis is currently underway.

5.5. BL15 TAIKAN

BL15 TAIKAN [11,40] is a small and wide angle neutron scattering instrument. We conducted a trial experiment to measure coherent and incoherent scattering cross sections from hydrogen contained material by analyzing scattered neutrons using the in-situ system [30]. Supermirrors were installed upstream of the beamline as a neutron polarizer, and the $^3$He spin filter was used as a spin analyzer. Silver behenate was used as the sample, and the $^3$He cell with pure Rb was placed 120 mm downstream of the sample. The diameter of $^3$He cell was 35 mm, the length was 55 mm, and the gas pressure was 3 atm. A stable $^3$He polarization of 68% was kept during the experiment. Scattered neutrons reaching a part of the small-angle detector bank were spin analyzed. The coherent and incoherent scattering components were successfully separated by analyzing the scattered neutrons. Although the trial experiment was successful, the solid angle of the $^3$He cell was inadequate and the pressure–thickness was not suitable for the neutron wavelength used in TAIKAN. In order to cover a larger solid angle, an ex-situ SEOP system using a larger $^3$He cell, which is to be placed 10 mm downstream of the sample, is being prepared (Fig. 13). A new $^3$He cell with rubidium and potassium has been fabricated with a diameter of 60 mm, a thickness of 40 mm, and a gas pressure of 1.5 atm for cold neutrons. More trial and user experiments will be carried out in 2020.

5.6. BL21 NOVA

Experiments using $^3$He spin filters at BL21 NOVA [11,41], which is a total diffractometer, are also planned. NOVA is equipped with large solid angle neutron detectors. The detectors and a sample are installed in a large vacuum chamber. We are planning to install two ex-situ systems upstream of the beamline and around the sample to perform polarized neutron scattering experiments. The schematic view of the ex-situ systems at NOVA is shown in Fig. 14. The ex-situ system for the polarizer is installed at the upstream of the beamline, and the neutron polarization is held by the magnetic field produced with a guide coil to the sample. The scattered neutrons by the sample are analyzed with two $^3$He cells. This ex-situ system for the analyzer which can be installed to the vacuum chamber is developed as shown in Fig. 15. A sample is suspended from the top of the vacuum flange. The neutrons scattered by the sample are analyzed with two $^3$He cells placed at a distance of 20 mm from the sample. The stage for the $^3$He cells can be rotated.
so that the scattered neutrons of any angle can be analyzed. The $^3$He polarization is kept using the Helmholtz coil. The $^4$He cell with the dimensions of $460 \times 40$ mm is placed at $10$ mm downstream of the sample to cover a large solid angle.

Fig. 14. Schematic view from the top of the ex-situ systems at NOVA. A neutron beam is polarized by the $^3$He cell installed upstream of the beamline, and the neutron polarization is held by the magnetic field produced with the guide coil to the sample. The scattered neutrons by the sample are analyzed with two $^3$He cells.

6. Conclusion

Development of $^3$He spin filters for user experiments are being carried out at MLF at J-PARC. The in-situ system using a $30$ W laser has been developed, and several trial experiments using $^3$He cells with pure Rb have been conducted. Recently, a gas-filling station has been constructed at J-PARC and several high quality $^3$He cells using rubidium and potassium were fabricated. Additionally, the pumping system using a $110$ W fiber laser was developed. High $^3$He polarization of $85\%$ was achieved on a neutron beamline using the $^3$He cell with rubidium and potassium and the pumping system using the fiber laser. The first user experiment utilizing a $^3$He spin filter took place in 2017, and five user experiments were conducted in 2019–2020. These experiments are beginning to yield scientific results. Preparations are underway for further user experiments using $^3$He spin filters.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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