Storage of ultracold neutrons in a volume coated with diamondlike carbon


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I. INTRODUCTION

Currently a source of ultracold neutrons (UCN) based on a dedicated spallation neutron source is being developed and built at the Paul Scherrer Institut (PSI). Fast neutrons produced by an up to 8-s-long pulse of the full power PSI proton beam (590 MeV, 2 mA) are thermalized in a 4-m³ tank of heavy water. A cold source with about 30 dm³ of solid deuterium is to operate with a cycle of 4- to 8-s beam on every 800 s. The UCN produced during the pulse are collected in a high-quality storage volume to increase their extraction efficiency. The UCN produced is to be stored in a volume coated with diamondlike carbon. Here, m is the neutron mass, N the scattering center density, b the bound coherent nuclear scattering length, σ the loss cross section per atom of the surface material (i.e., absorption plus inelastic scattering), and v the neutron velocity. Usually, the quality of the surface is described in terms of the critical velocity, \( v_c = \sqrt{\frac{\hbar^2}{m}} \). Candidate materials are beryllium, beryllium oxide, and diamondlike carbon (DLC). Nickel, and particularly isotope pure \(^{58}\)Ni, has a high Fermi potential; it also has a high neutron absorption cross section and is more useful for guides where only a few wall collisions occur. Be and BeO are the established wall materials for storage vessels and have been produced and proven to work (see, e.g., Refs. [3,4]). However, a fundamental drawback is their toxicity, which leads to difficulties in manufacture, use, and disposal. A comparatively new and very attractive alternative wall material for UCN storage is DLC. It has already been tested to some extent for UCN applications (see, e.g., Refs. [5–7]). Theoretically, its loss coefficient, \( \eta \), is less than \( 3 \times 10^{-7} \), although values extracted from measurement lie in the region of \( 10^{-4} \) [5]. The reason for the difference, termed “anomalous losses,” is presently not understood. A Fermi potential of about 260 neV, corresponding to about 60% sp³ fraction in the DLC, is realistically achievable. (Graphite, in comparison, has sp² bonds, lower density, and a potential of about 180 neV.) Diamondlike carbon is widely used in industry and techniques for making high-quality coatings are well established. A detailed systematic study of all relevant characteristics for UCN applications is in progress [8,9].

In this article we give the results of an experimental study of the storage properties of DLC coatings as a function of temperature and of UCN energy up to the maximum storable energy. The experiment measures the lifetime of UCN stored in a DLC coated foil bottle under a variety of conditions. After a storage time, \( t \), the fraction of the UCN remaining is equal to \( N_0 \cdot e^{-t/t_0} \), where \( t_0 \) is the lifetime of UCN stored in the bottle and depends on the neutron lifetime and the reflection characteristics of the walls. The initial number of UCN is \( N_0 \).
For the sake of brevity we refer to $\tau_0$ as the storage time constant.

The loss per bounce values, $\eta$, are extracted from the storage time constants by modeling. The measurements were carried out using a down scaled model (about a factor 25 in cross section while maintaining the original height) of what is believed to be a practical design for a storage vessel for the actual PSI source: clearly, a very low loss coefficient only leads to a long storage time constant in the storage volume if a design with a low fraction of surface defects can be built.

II. DESCRIPTION OF THE EXPERIMENT

The principle of the experiment is to measure the number of UCN surviving selected storage times under various conditions following filling a test bottle to equilibrium density. The test bottle used is of quasi square cross section (see Fig. 1), about $200 \times 200 \text{ mm}^2$, and height about 3000 mm. It is constructed from four independent segments forming a side wall and a quarter of the bottom each [(5) in Fig. 1]. These segments consist of a 100-$\mu$m-thick stainless steel foil, which forms the inner surface, mounted tautly on a suitable support frame. The foils are galvanically coated with nickel and then with about 200–300 nm of DLC using the laser controlled vacuum arc technique [10]. The four segments are tightly clamped together at the edges. The resulting seams only marginally impair the volume-to-surface ratio seen by the stored UCN and hence result in only insignificantly deteriorated storage conditions. However, the design leads to four unavoidable holes at the bottom corners, measured to be about 1 mm$^2$ each (see Fig. 2). The walls are in contact with containers that can be filled with liquid nitrogen so that measurements from room temperature down to 115 K can be made. The bottom of the vessel includes a “boot-lid” type shutter (see Fig. 2) mounted in an assembly that includes the fillets for covering the joins in the bottom parts of the foils. All surfaces that can be seen by UCN are coated with DLC.

The measurements were carried out at the Institut Laue Langevin (ILL) at the PF2 EDM beam position (named after an experiment to measure the electric dipole moment (EDM) of the neutron) using the assembly sketched in Fig. 3. Ultracold neutrons from the turbine enter the test bottle (4) through a neutron guide (1) with a vertical U-shaped chicane (2) and are reflected by a movable mirror (3). When the test bottle reaches equilibrium density (this takes about 40 s), the shutter (7) is closed and the movable mirror repositioned to connect the test bottle directly to the UCN detector (5) mounted vertically below the UCN bottle. After a suitable storage time values between 10 and 500 s were used the shutter (7) is re-opened and the number of UCN remaining in the bottle is counted over the emptying time.

FIG. 1. Sketched top view of the storage volume (“foil bottle”). It is surrounded by liquid Nitrogen containers (1) that were used to cool down the whole storage volume to about 115 K. The bars (2) and bolts (2a) are used to squeeze the individual foil segments (3) together to form a light tight connection. (4) entrance guide with boot-lid shutter (see also Figs. 2 and 3). The shaded area (5) indicates one of the four basic individual segments forming the storage volume.

FIG. 2. (Color online) Photograph of the boot-lid type inner shutter (1) and the fillets (2) after coating with DLC and before mounting in the foil bottle. The diameter of the circular lid in the center is 80 mm. The number (3) denotes the position of the unavoidable hole in the corners of the storage volume not covered by the fillets (2).

FIG. 3. Overview of the experimental setup. UCN are filled from the left through the filling guide (1) and the energy selector “U” (2), which is revolvable around the beam axis. In “filling” position of the switch (3) UCN enter the storage volume (4), while in “emptying” position they fall into the detector (5). A polyethylene absorber (6) allows the spectrum to be cut at a freely selectable upper energy. The boot-lid inner shutter, shown in Fig. 2, is at position (7).
The remaining time is used for background counting. Storage volume is emptied into the detector ("emptying peak" (2)). The counts during the storage show the leakage through the shutter. From 120 s to 174 s the time here is 80 s, from 40 s to 120 s. The counts during the storage when the switch is moved into the emptying position. The storage of the dead volume between switch and storage volume that are released peak visible at 40 s ["fill peak," (1)] comes from the UCN stored in 90–110 neV) as a function of the "detector time." Bin size is 1 s. The parameter surviving various storage times and (ii) extraction of the loss time constants from the variation of the number of UCN, i.e., neutrons with a velocity below \( v_c \) or the maximum energy selected by the absorber, and a residue of neutrons failing the UCN criterion marginally and so having a relatively long lifetime before being lost. Because of intensity limitations, it is better to use short storage times and to fit the measured data to a two component exponential function than to wait several cleaning time constants (see, e.g., Fig. 5). That is, the number of neutrons \( N_t \) remaining after a storage time \( t_i \) of 54 s. Measurement cycles with the same conditions but with different storage times were taken so that the storage time constant could be extracted. Neutron counts over a typical measurement cycle with 80-s storage times were taken so that the storage time constant could be extracted from the measured data.

The wall loss parameter \( \eta \) is related to the time constant for wall loss, \( \tau_\text{tot} \), and thus to the wall loss parameter \( \mu \) through a suitable average of the wall loss probability per bounce for a neutron of kinetic energy \( E \) (\( E \) varies systematically with height \( h \) in the bottle, \( E = E_{\text{max}} - mgh \)) with an angle of incidence \( \Theta \) to the surface and the wall collision frequency \( v(E) \) [2,5,11]:

\[
\frac{1}{\tau_0} = \frac{1}{\tau_\beta} + \frac{1}{\tau_\text{loss}} + \frac{1}{\tau_\mu}
\]

may be extracted from the measured data.

A. Extraction of the storage time constant \( \tau_0 \)

The neutrons detected after each storage time are a mixture of UCN, i.e., neutrons with a velocity below \( v_c \) or the maximum energy selected by the absorber, and a residue of neutrons failing the UCN criterion marginally and so having a relatively long lifetime before being lost. Because of intensity limitations, it is better to use short storage times and to fit the measured data to a two component exponential function than to wait several cleaning time constants (see, e.g., Fig. 5). That is, the number of neutrons \( N_t \) remaining after a storage time \( t_i \) of 54 s. Measurement cycles with the same conditions but with different storage times were taken so that the storage time constant could be extracted. Neutron counts over a typical measurement cycle with 80-s storage times were taken so that the storage time constant could be extracted from the measured data.

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\[
\mu(\Theta, E) = 2\eta \sqrt{\frac{E \cos^2 \Theta}{V - E \cos^2 \Theta}}
\]

\[
\tilde{\mu}(E) = 2 \int_0^1 \mu(\Theta, E) \cos \Theta d \cos \Theta
\]

\[
\frac{1}{\tau_\mu} = \int_0^h v(E_{\text{max}} - mgz) \tilde{\mu}(E_{\text{max}} - mgz) dz
\]

where \( V \) is the Fermi potential for the surface; the frequency \( v(E) \) together with estimates for \( \tau_\text{loss} \) [Eq. (4)] are obtained from modeling the performance of the storage bottle. The energy \( E_{\text{max}} \) is the starting energy of the UCN at \( h = 0 \).

### Table I. Measured storage time constants \( \tau_\text{tot} \) for 115 and 290 K at the selected UCN energies.

<table>
<thead>
<tr>
<th>E (neV)</th>
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<tr>
<td>30–50</td>
<td>242.4 ± 3.2</td>
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<tr>
<td>50–70</td>
<td>204.2 ± 1.9</td>
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<tr>
<td>70–90</td>
<td>184.1 ± 1.7</td>
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<tr>
<td>90–110</td>
<td>195.3 ± 1.9</td>
<td>142.3 ± 1.6</td>
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<td>110–130</td>
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<td>137.3 ± 1.3</td>
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<tr>
<td>130–150</td>
<td>172.6 ± 3.3</td>
<td>130.2 ± 0.6</td>
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<td>150–170</td>
<td>167.6 ± 3.5</td>
<td>128.3 ± 0.7</td>
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<tr>
<td>170–190</td>
<td>149.6 ± 4.2</td>
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<td>190–210</td>
<td>68.9 ± 24.1</td>
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<td>190–230</td>
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<td>210–230</td>
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### III. ANALYSIS AND RESULTS

The data analysis has two parts: (i) extraction of the storage time constants from the variation of the number of UCN surviving various storage times and (ii) extraction of the loss parameter \( \eta \) from the storage time constants.

The storage time constants measured have contributions from several effects, \( \tau_\beta \) for \( \beta \) decay of the free neutron, \( \tau_\text{loss} \) for losses through defects (holes, pinholes, and cracks), \( \tau_\text{leak} \) for neutron leakage past the shutter, and \( \tau_\mu \) from the wall loss. Of these, the effect of leakage past the shutter is measured in the experiment so that a storage time constant, \( \tau_0 \), given by

\[
\frac{1}{\tau_0} = \frac{1}{\tau_\beta} + \frac{1}{\tau_\text{loss}} + \frac{1}{\tau_\mu}
\]

FIG. 4. Typical data during one measuring cycle (energy interval 90–110 neV) as a function of the “detector time.” Bin size is 1 s. The peak visible at 40 s ["fill peak," (1)] comes from the UCN stored in the dead volume between switch and storage volume that are released when the switch is moved into the emptying position. The storage time here is 80 s, from 40 s to 120 s. The counts during the storage show the leakage through the shutter. From 120 s to 174 s the storage volume is emptied into the detector ("emptying peak" (2)). The remaining time is used for background counting.

Data were taken (i) at room temperature (290 K), (ii) during cooling down, and (iii) at 115 K. Height intervals of typically 20 cm for the U and the absorber were chosen to give energy intervals \( \Delta E \) of nominally 20 neV in width. The first interval, however, covered 0–30 neV and the last all stored neutrons with energy of 210 neV and above (energies are with respect to the bottom of the storage volume). The storage time constants for all measured energy intervals and for wall temperatures 290 and 115 K are presented in Table I.

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the time when the bottle shutter is closed and two exponential fit Eq. (8) to the data gives the cleaning time $\tau_1 = (23.5 \pm 1.2) \text{ s}$ and the storage time constant $\tau_0 = (154.8 \pm 2.1) \text{ s}$.

is given by

$$N_i = N_1 \exp(-t_i/\tau_1) + N_0 \exp(-t_i/\tau_0), \quad (8)$$

where the first exponential, with lifetime $\tau_1$, describes the loss of marginally trapped neutrons (cleaning effects) and the second, with lifetime $\tau_0$, describes the loss of the actual UCN.

Three corrections must be applied to the data to extract the loss characteristics of the wall: (i) for the background count rate, (ii) for neutrons lost by leaking past the shutter, and (iii) for loss during the emptying period. For (i) the background rate was measured to be on average $R_{BG} = 0.005 \text{ s}^{-1}$, which is negligible compared to the count rates during a measurement cycle.

The shutter leakage rate (ii) is obtained from the counts $M_j$ measured during the storage time interval; that is, between the time when the bottle shutter is closed and $t_j$ when the emptying of the bottle is initiated. These are the counts in Fig. 4 occurring just after the peak (1), which comes from the neutrons trapped between the movable mirror and the shutter reaching the detector, and the start of the emptying phase (peak 2). As the shutter leakage should be proportional to the neutron density in the bottle, the values require correcting for the intensity reduction over the storage time:

$$N_i \rightarrow N_i + \sum_{j<i} M_j \times e^{-t_j/\tau_0}, \quad (9)$$

where $M_j$ is the count in the time bin at $t_j$ and the sum extends over the storage time. Equation (8) and the relation (9) are solved iteratively. This correction changes the value of $\tau_0$ only marginally.

For (iii) the emptying time constants after shortest (10 s) and longest (up to 300 s) storage times do not change within the associated statistical errors. Corrections resulting from different emptying time constants are hence neglected. The values of the storage time constants as a function of UCN energy and for two DLC temperatures are shown in Fig. 6.

### B. Extraction of the loss coefficient $\eta$

The extraction of values for the wall loss coefficient, $\eta$, essentially requires solving Eqs. (6) and (7) backwards. We use simulations where values for the relevant parameters are sought that best represent the measured data. The start point is to replace the integrals in Eqs. (6) and (7) with quantities averaged over the energy range of the UCN at the bottom of the storage bottle ($E_{\text{max}}$):

$$\frac{1}{\tau_{\mu}} = \bar{v}(E_{\text{max}}) \times \bar{\mu}(E_{\text{max}}). \quad (10)$$

A simulation of the bottle made with the GEANT4 code [12, 13] is used to obtain the wall collision frequencies $v(E_{\text{max}}, h)$ as a function of height in the bottle and for a range of starting energies. These are integrated to give the average wall collision frequency for an UCN starting with energy $E = E_{\text{max}}$ at $h = 0$. An appropriate functional form for $\bar{\mu}(E_{\text{max}})$ may be obtained from Eqs. (6) and (7) (see [5]) and gives

$$\bar{\mu}(E_{\text{max}}) = 2\eta \left[ \frac{V}{s \cdot E_{\text{max}}} \arcsin \frac{s \cdot E_{\text{max}}}{V} - \frac{V}{s \cdot E_{\text{max}}} - 1 \right], \quad (11)$$

where the factor $s (0 < s \leq 1)$ is introduced to allow a suitable average energy for the reflections to be selected. Some values for $\bar{\mu}(E_{\text{max}})$ calculated using Eq. (11) are shown in Fig. 7. Finally, values for $\eta$ and $s$ are determined that lead to Eq. (10) giving the best match to the experimentally found $\tau_0$ vs $E_{\text{max}}$ variations at 115 and 290 K.

For UCN energies $E_{\text{max}}$ below 50 neV, corrections to the wall loss time constant and for the contribution of defects in the DLC surface become significant:

(i) The holes in the bottom: the loss rate is estimated using the ratio of the area of the holes $A_{\text{holes}}$ to the total surface area of the bottle $S(E_{\text{max}})$ seen by the UCN of start
energy $E_{\text{max}}$ times the collision frequency

$$\frac{1}{\tau_{\text{hole}}} \sim \bar{v}(E_{\text{max}}) \times \frac{A_{\text{hole}}}{S(E_{\text{max}})}. \quad (12)$$

(ii) The clamping method of the four edges cannot completely eliminate defects, so an extra area of defect $d_{\text{slit}} \times l_{\text{slit}}$, starting from the bottom of the storage volume, is introduced as an additional fit parameter. (Because of mechanical constraints the present method is different from the one used in [5,7] where slits could be ruled out.)

That is, in the complete UCN energy region measured, matching simulation data to the measured storage time constants is carried out by using

$$\frac{1}{\tau_0} = \bar{v}(E_{\text{max}}) \times \left[ \bar{\mu}(E_{\text{max}}) + (A_{\text{hole}} + d_{\text{slit}} \times l_{\text{slit}}) \times \frac{1}{S(E_{\text{max}})} \right]. \quad (13)$$

C. Results and discussion

The results for the measured storage time constants for various UCN energies and for the DLC walls at 115 and 290 K are presented in Table I and plotted in Fig. 6 together with the results from the simulation. The parameter values that best match the measured data are quoted in Table II. The values for $\eta$, $(1.8 \pm 0.2) \times 10^{-4}$ at 115 K and $(3.1 \pm 0.9) \times 10^{-4}$ at 290 K, are consistent with those of DLC coated aluminum foils measured earlier [5,7]. Because of limited time and design constraints, we did not use any elaborate surface treatment such as baking at 620 K in a helium atmosphere [14] or glow discharge cleaning [15]. It is well known, however, that such procedures may lead to further significant reduction of the loss factors. The total areas of defective surface that best explain the storage time constants are 41 mm$^2$ at 115 K and 44 mm$^2$ at 290 K; a possible distribution between holes and slits is indicated in Table II. We obtained a $\chi^2$/DOF of 1.24 at 290 K and 0.81 at 115 K for 4 degrees of freedom, where the errors on energy averaging and storage time constants are added in quadrature. The flat region around 100 to 160 neV is a hint at a nearly blemish-free storage region.

The simulation fails to model the quite strong decrease of the storage time constant for energies above 180 neV under the assumption of a diamond fraction of the surface coating of more than 60%. The various contributions to the measured storage time constants, which were used for the simulation, are shown in Fig. 8 as a function of UCN energy. The best fit is based on reasonable assumptions for imperfections for the storage volume geometry and measured loss values [16].

Explanation of the data could possibly be obtained by increasing the loss per bounce values. These values, however, are mostly considerably higher than theory predicts. Mechanisms for this “anomalous” loss rate have been proposed, e.g., hydrogen upscattering or UCN localization around holes [17]; however, no satisfactory method of making a reasonable correction to theoretical values is achievable.

A decrease of the critical velocity for the DLC coating from 7.1 to 6.7 m/s does give a possible explanation. Potential causes for the lower critical velocity are, first, imperfections or, second, graphite clusters in the coating. Imperfections can be caused by “dust” particles present on the surface during the coating process. Such particles account not only for holes

![Fig. 7. The averaged loss parameter $\bar{\mu}(E_{\text{max}})$ from simulated wall collision frequencies (points) and resulting best fit using Eq. (11). Input parameters are: (1) $\eta = 2.5 \times 10^{-4}$, (2) $\eta = 1 \times 10^{-4}$, for both fits $s = 0.3$.](image1)

![Fig. 8. Simulated energy dependent loss rates. (1) Total loss rate $1/\tau_0$, (2) loss rate due to wall material $1/\tau_{\mu}$, (3) loss rate due to slits, and (4) loss rate due to holes in the bottom $1/\tau_{\text{loss}}$.](image2)
made during cooling down.

FIG. 9. The loss coefficient $\eta$ for UCN in the energy interval 170–190 neV as a function of temperature. The measurements were made during cooling down.

in the nickel layer but likely also account for holes in the DLC surface. A defective surface area of $\sim 5 \times 10^{-4}$ has been observed (see, e.g., Ref. [16]). Graphite clusters have typically a size of 300–1000 nm and cover a surface fraction similar to the hole area. They lead to an increased loss probability because they have a lower Debye temperature and hence a higher inelastic scattering cross section. Additionally, low-density graphite is known to be hydrophillic [18], contributing directly to the losses through absorption and adsorption of hydrogen. Both graphite and steel have a Fermi potential of ∼180 neV. Thus, we cannot distinguish between these two possible explanations for the drop in storage time constant above 180 neV.

Finally, the storage time constants for the neutron energy band 170–190 neV were measured at 15 temperatures during cooling of the bottle. The resulting values for the loss parameter $\eta$ are shown plotted against temperature in Fig. 9. The values are in good agreement with those in [16], where data up to an UCN energy of 90 neV have been presented. The observed temperature dependence of the storage time is another hint at the presence of hydrogen at the surface (see also [16]).

For the UCN source under construction at PSI we conclude that DLC is a suitable wall coating for the intermediate storage volume. Improved performance of the DLC coatings are expected following further development of the coating process and surface cleaning procedures. Because of its much bigger size, a more favorable surface-to-volume ratio and hence a reduced influence of mechanical imperfections on the corners or shutters are expected in the storage volume at the PSI source.

ACKNOWLEDGMENTS

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