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Very long nuclear relaxation times of spin polarized helium 3 in metal coated cells

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Abstract

We obtained very long relaxation times T_1 of up to 120 h for the nuclear polarization of an optically pumped helium 3 gas. The glass containers were internally coated with metallic films such as bismuth or cesium. These findings will have applications in the field of helium magnetometers and polarized targets.

1. Introduction

Optically pumped helium cells at low pressure (~ a few torr) [1] have been in use for a long time as precision magnetometers in low field applications such as geophysics [2]. More recently this technique has been further developed in Caen [3] with the additional goal of extending it also towards gyroscopic devices, as considered in Ref. [4]. In fundamental physics experiments, polarized ^{3}He targets play an important role as a substitute of spin polarized targets of neutrons at rest, because the single neutron of the ^{3}He nucleus carries most of its spin and magnetic moment. After a first attempt along these lines [5], a Canadian group devoted great efforts to produce dense polarized targets by mechanical compression [6]. After the development of powerful infra-red lasers for the optical pumping of helium [7], a breakthrough was obtained in the compression technique [8] resulting in a novel measurement of the neutron electric form factor by electron scattering [9].

The alternative method to achieve dense polarized ^{3}He targets is by spin exchange with optically pumped Rb [10], which recently led to precise measurement of the spin structure function of the neutron at SLAC [11]. Polarized ^{3}He targets as broadband spin filters for neutron beams [12] is another important field of application which is opening now [13,14,15].

In all these experiments, it is an essential issue to obtain long relaxation times T_1 in order to achieve and maintain large nuclear polarizations of the samples. The most important relaxation source usually results from the interaction with the walls of the cell, provided the magnetic field is sufficiently homogeneous [1] and the pressure does not exceed a few bars (at 300 K), in which case the dipole–dipole interaction among ^{3}He atoms can play a dominant part. Hence wall relaxation of ^{3}He has been exten-

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sively studied already by several groups [1,16,17], including measurements in the low temperature regime [18]. A number of materials, mainly glasses [16] and also copper [5], were tested as cell walls. In addition, various metallic surfaces were examined by introducing metallic samples into the cell [17]. Typically, pyrex cells of 5 cm diameter give T_1 values of the order of 1 h at room temperature, whereas aluminosilicate glasses such as Corning 1720 or Schott Supremax are known to give an order of magnitude more [16]. In conclusion, metals were found to give poorer results than aluminosilicate glasses. The basic mechanisms of wall relaxation are thought to be related to the interaction with paramagnetic electrons in the walls, the ^{3}He atoms being either adsorbed at the surface or diffused inside [16].

Although the details of all these processes have never been completely cleared up, in view of the crucial importance of this problem we have decided to carry on new investigations on relaxation with internally coated cells, motivated by the success of the coating technique at low temperatures [18,19]. This research followed two guiding lines:

(i) To coat the inner walls with diamagnetic metals such as bismuth, which can be evaporated as very pure films and should prevent permeation of helium atoms because of their compact lattice structure.

(ii) To try cesium coating at room temperature, having in mind the very small adsorption energy (2.3 K) of ^{3}He on cesium coated glass as well as the relatively long relaxation times measured in the low temperature regime [19]. This effect is due to the very weak attractive potential of the helium atom to the cesium surface [21], which in turn explains the recent discovery of the nonwetting behaviour of superfluid helium on cesium surfaces [22].

2. Cell preparation

The present experiments were performed both on sealed-off glass cells at low pressure of a few millibars, such as commonly used for ^{3}He magnetometers, and on high pressure targets of a few bars prepared by the compression method as described in Ref. [20].

The coated cells were prepared according to the following procedure. The cells of 6 cm diameter were blown out of Pyrex or Supremax glass 8409 (supplied by Schott) and rinced with distilled water. They were then baked for two days at a temperature of about 400°C under vacuum down to 10^{-8} mbar produced by a sealed-on vacuum ion pump. After that the inner surface was further cleaned by running repeatedly a bright discharge in ^{4}He gas. Then the metal intended for coating was distilled from a side arm into the cell kept at room temperature, until a visible almost homogeneous metallic film covered up the cell walls. Then the side arm was sealed off, leaving a small transparent area at that place in the final cell that could be used to pass through the optical pumping beam. We chose metals that could be distilled at temperature below the transformation point of the glass. The purity of the substance is typically better than 99.9%. The case of cesium is somewhat different because its melting point (27°C) is close to ambient temperature and results in the formation of droplets rather than homogeneous mirrors. These droplets were usually rolled later on over the whole surface; this procedure is thought to leave a very thin film of cesium on the walls, established anyway at the saturated vapour pressure (10^{-7} mbar at room temperature).

Concerning low pressure cells, pure ^{3}He gas (grade 5 chemical purity) was filled in at a pressure of 6 torr after passing two further purification stages, namely a volume getter ST707 (supplier SAES Milano) at 250°C followed by a liquid nitrogen trap. Finally the cells were sealed off and ready for direct optical pumping and relaxation measurements.

In contrast the high pressure cells were conceived refillable and separable from the filling station which provided the compressed polarized ^{3}He gas [20]. Hence they were closed off by an ordinary glass valve, tightened by Apiezon H vacuum grease and connected to the filling station by a KF glass flange. As a consequence of this concept we had to compromise on the purification procedure. First the valve had to be kept out of the oven during baking, second the gas introduced through the compressor could not be assigned the same level of purity as for the low pressure case, because it flew through unbaked components of the compressor after the above mentioned purification stages. Therefore one hoped for the additional profit to the metal coatings through their gettering action.

3. Relaxation measurement procedure

The polarization was achieved by laser optical pumping of the 2^3S_1 state of ^{3}He with high power LNA lasers [7]. The low pressure cells were directly pumped by laser while running a weak discharge in the gas, which was turned off when the T_1 measurements started. The initial polarization of order 40%, was similar in all cells. The high pressure cells were filled with gas already polarized at about 30% through the compressor described in Ref. [20]. In the latter case no discharge ever touched the coating. We measured the relaxation times T_1 by NMR techniques. For the NMR measurements the cells were immersed in a magnetic field of 20 G at the center of a long solenoid of 2 m in length and 0.25 m in diameter, providing a field homogeneity of better than 1 mG/cm on the axis at the center.

The magnetization was monitored by free induction decay signals induced by $\pi/60$ pulses of a radiofrequency field B_1 at the Larmor frequency (66 kHz at 20 G). The destructivity of each pulse was thus as small as 1.4×10^{-3}. Signals were detected by a pair of pickup coils as part of a resonance circuit. Care was taken, in particular at high pressure, to prevent potential maser oscillation, which would alter the T_1 measurement. This was ensured by reducing the Q factor of the pickup coils. In all cases, we checked that the free induction decay signals decreased exponentially and with a transverse relaxation time T_2 independent of the magnetization. Also precautions were taken to shield the cell from external randomly fluctuating fields that could both spoil the signal to noise ratio and cause parasitic relaxation. For this purpose the whole solenoid was wrapped into a 2 mm thick aluminium foil (the skin depth is 0.3 mm at 66 kHz).

The decay of the longitudinal magnetization was periodically monitored, typically every hour, for at least one characteristic time constant. A fit to the exponential decay gave a value of T_1 with a very good precision (1% or less) (see Fig. 1). We report in Table 1 several values of T_1 measured under these conditions for a number of cells made of various glasses, uncoated or coated with different metals. The sealed cells at low pressure could be easily tried many times and showed no significant evolution after weeks on the shelf. The high pressure cells had to be refilled on the compression machine with clean freshly polarized gaz. In some cases several tries were made with different cells of the same sort (same glass, same metallic coating, same pressure) (see Table 1, lines 3 and 13) for checking the reproducibility of the measurements. It is typically of the order of 20 to 30% and appears to depend on several parameters difficult to control, such as the

Table 1

Measurements of the nuclear relaxation time T_1 of polarized ^{3}He gas contained in different glass cells internally coated with various metals

Line	Glass	Coating	^{3}He pressure	T (h) measured	Number of tested cells	Type of cell
1	pyrex	no	6 torr	1.4	1	sealed
2	pyrex	cesium	6 torr	68	1	sealed
3	supremax	no	6 torr	10–15	2	sealed
4	supremax	magnesium	6 torr	6.2	1	sealed
5	supremax	antimony	6 torr	7.2	1	sealed
6	supremax	zinc	6 torr	12	1	sealed
7	supremax	lead	6 torr	26	1	sealed
8	supremax	bismuth	6 torr	44	1	sealed
9	supremax	cesium	6 torr	72	1	sealed
10	pyrex	rubidium	2.3 bar	26	1	refillable
11	pyrex	bismuth	2.3 bar	48	1	refillable
12	supremax	bismuth	2.3 bar	50	1	refillable
13	supremax	cesium	2.3 bar	90–120	3	refillable
14	supremax	bismuth + cesium	2.3 bar	65	1	refillable
15	ultrasil	cesium	2.3 bar	33	1	refillable

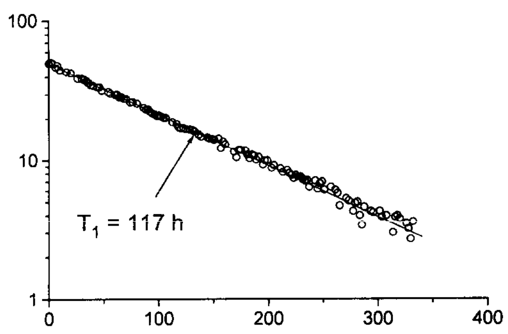

Fig. 1. Amplitude of NMR signals (in arbitrary units) monitoring the ^{3}He magnetization, plotted in log scale as a function of time (in hours). ^{3}He pressure: 2.2 bar; cell volume: 110 cm^3; cell walls: supremax coated with cesium; initial polarization: 30%.

percentage of uncoated area. Earlier findings that the cleaning procedure plays a crucial role were also confirmed here.

4. Deduction of wall relaxation times

In order to deduce from these measurements the relaxation rate $1/T_1^{\mathrm{wall}}$ by the walls, one has to evaluate the other relaxation sources in each case. One can decompose $1/T_1$ into three components,

$$\frac{1}{T_1} = \frac{1}{T_1^{\mathrm{wall}}} + \frac{1}{T_1^{\mathrm{gr}}} + \frac{1}{T_1^{\mathrm{dd}}}, \tag{1}$$

where T_1^{gr} is the relaxation induced by the diffusive motion of the atoms through magnetic field gradients [22,23], T_1^{dd} the relaxation produced by magnetic dipole–dipole interactions occurring between atoms in binary collisions [25,26].

The influence of field gradients, initially studied in Ref. [23], can be expressed according to Ref. [24]. In the present regime, the values of the pressure, of the dimensions of the cell and of the Larmor frequency are such that only the macroscopic diffusion motion of the atoms plays a role in the relaxation by the gradients resulting in the formula

$$\frac{1}{T_1^{\mathrm{gr}}} = \alpha \left(\frac{\mathrm{d}\bar{B}_{\mathrm{t}}}{\mathrm{d}r} \frac{1}{B} \right)^2 \frac{1}{p}. \tag{2}$$

In formula (2) $\mathrm{d}\bar{B}_{\mathrm{t}}/\mathrm{d}r$ represents the transverse field gradient averaged over the cell volume, p the pressure, α is a constant depending on the geometry. Assuming that the cell is a perfect sphere and taking into account only the first mode of diffusion, one finds, using Ref. [24] and numerical values given in Ref. [25],

$$\alpha = 1.8 \times 10^3 \ \mathrm{cm}^2 \ \mathrm{bar} \ \mathrm{h}^{-1}. \tag{3}$$

For a $(\mathrm{d}\bar{B}_{\mathrm{t}}/\mathrm{d}r)/B$ of 10^{-4} cm^{-1}, which is a sensible order of magnitude, one obtains $T_1 \sim 430$ h for $p = 6$ torr.

The intrinsic dipole–dipole relaxation has been studied by several authors [6,26,27]. If one takes the most recent estimate (Ref. [27], formula (2)), one gets at room temperature

$$\frac{1}{T_1^{\mathrm{dd}}(\mathrm{hour})} = \frac{P(\mathrm{bar})}{817}. \tag{4}$$

Formula (2) shows that at large pressure ($p \sim$ several bars) the contribution of the field gradient terms in formula (1) can be neglected. Conversely formula (3) shows that at low pressure (6 torr) the dipole–dipole relaxation is negligible.

An estimate of T_1^{gr} is necessary at low pressure. The explicit value given by formula (2) cannot be used easily, first because the real cell geometry is not spherical, due to side arms, second because the field gradient varies considerably over its volume. For more precision we measured the relaxation time T_1 when moving the cell along the z-axis of the solenoid. We also measured independently the field gradient as a function of z. A fit to the data was made using the functional dependence of formula (2) and two adjustable parameters, α and T_1^{wall}, as shown in Fig. 2 and discussed in Ref. [28]. We deduced from the fit that, at optimum position, T_1^{gr} is 530 h. This value is in the same range as given by formulas (2) and (3) for a perfect sphere, using $(\mathrm{d}\bar{B}_{\mathrm{t}}/\mathrm{d}r)/B_{\mathrm{t}} = 10^{-4}$ cm^{-1} (the value of the field gradient measured at the center of the solenoid). T_1^{gr} thus contributes approximately 10% to the shortest measured relaxation rate at low pressure.

An estimate of T_1^{dd} is necessary at high pressure. Formula (4) indicates that T_1^{dd} is 330 h at 2.5 bars. This number should be taken with some care. The values given in Ref. [27] are theoretical and have no

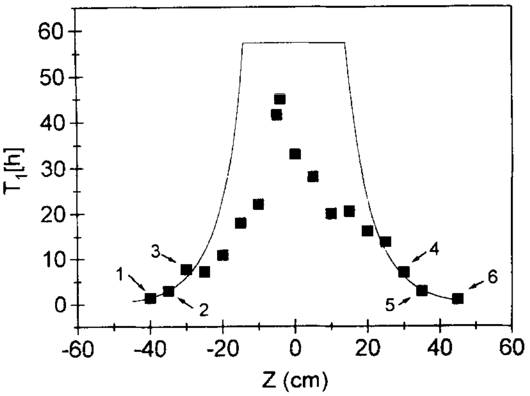

Fig. 2. Squares: nuclear relaxation time T_1 measured with a supremax cell coated with bismuth and filled with 6 torr of ^{3}He, as a function of the position z of the cell moved along the z-axis of the solenoid. The curve is a fit to the data using field gradient measurements (see text), with $\alpha = 1.95 \times 10^3$ cm^2 bar h^{-1}, $T_1^{\text{wall}} = 50$ h. The fit is adjusted on points 1, 2, 3, 4, 5, 6 where the field gradient relaxation dominates.

error bar. Results of Refs. [26,27], both incorporating the latest He–He interaction potential, basically agree with each other and predict the same $1/\sqrt{T}$ temperature dependence for $1/T_1^{\text{dd}}$. But the comparison between experimental and theoretical results in Ref. [27] is not very precise, and the experimental conditions differ from our case. We plan to study the pressure dependence of T_1 in a future work. In short the dipole–dipole relaxation contributes approximately 35% to the smallest relaxation rate measured at high pressure ($1/T_1 = 120$ h^{-1}) and less in the other cases.

5. Results

The values given in Table 1, labelled by the line number, can be analyzed in several ways if one compares results in experimental conditions differing only by one factor.

(a) *Non coated cells of different glasses* (lines 1 and 3). As already found by other authors [16,6], pyrex walls give a much shorter T_1^{wall} than supremax ones (roughly 10 times for our sealed cells). The reason could be that pyrex shows much a larger permeation for helium atoms than aluminosilicate glasses at room temperature. When the atoms penetrate the glass substrate, the probability that they are disoriented by paramagnetic centers in the substrate increases [16]. We observed a large spread of measured T_1 values from cell to cell, certainly influenced by the preparation procedure. The longest T_1 value that we recorded is 15 h, a factor of 2 shorter than reported in Ref. [16].

(b) *Cells of the same glass with different coatings* (lines 3 to 9 and 12 to 14). Comparing supremax cells with different metallic coatings, we observed a significant improvement in the case of lead, rubidium, bismuth and cesium, whereas magnesium, antimonium and zinc coatings resulted in comparable or even shorter values of T_1, as compared to the non-coated case. Obviously the large improvement was obtained with either bismuth or cesium. This confirmed the initial ideas of this work that the bismuth coating, as a compact diamagnetic film, would prevent permeation of the atoms to the substrate as well as paramagnetic relaxation, whereas cesium would efficiently repell the atoms from the surface. In view of these results we tried to combine these two properties by superposing cesium on top of bismuth in the same cell. The further increase in T_1 that we hoped for was not observed (compare line 14 to 12 and 13). This could be due to the creation of an alloy between the two metals, which visibly dissolved the homogeneous coating after some time and formed islands.

The large increase of T_1 from bare to cesium coated glass is in disagreement with the observation of Ref. [6], where an increase of T_1 by less than a factor of 2 was reported for pyrex cells.

We also observed a large improvement by rubidium coating on pyrex (see lines 1 and 10). This is not too surprising in view of the close chemical and physical similarities of rubidium with cesium. This explains in retrospect why the relaxation times, observed in the alternative polarization method using spin exchange with Rb, have consistently shown longer values [27] than in the direct pumping experiments using uncoated cells.

(c) *Cells of different glasses with the same coating* (lines 2, 13, 15). Comparing cesium coating on different substrates, one finds an increase of T_1 from ultrasil to pyrex and from pyrex to supremax. Similarly uncoated cells gave longer T_1 values for supremax than for pyrex. This proves that the substrate still plays a role in the relaxation, indicating that the cesium coating (presuming complete coverage) is not completely tight against the permeation of atoms.

(d) *Sealed compared to refillable cells.* The present results show little difference between the sealed-off cells and the refillable ones when the same glass and the same coating were used (compare lines 2 and 11, 9 and 13, 8 and 12). However the filling procedure was substantially different, with a better cleaning for the sealed-off cells. Even successive refillings of the same cell with the compressor, without intermediate refreshment of the coating, gave similar T_1 values in the range of the numbers in line 13. This feature, which is appealing in view of applications, may be attributed to the gettering effect of the coating. This interpretation is confirmed by observations in the discharge in our low pressure cells, which gain spectral purity a few hours after their preparation.

6. Discussion

Any attempt towards a microscopic interpretation of these measurements is necessarily very complex. It involves the adsorption of helium atoms on the surface of the coating, the permeation through it, the relaxation of their spins (located either in the coating or in the substrate) by the residual paramagnetism of electrons at the Fermi surface of the conduction band, and by nuclear dipole–dipole coupling. Too little is known on the physical properties of both the coating and the glass substrate and their interaction with the He atom and its nuclear dipole moment for building a quantitative model of the relaxation processes.

Early investigations of the temperature dependance of T_1 for uncoated glass walls have at least qualitatively revealed two features [23]; (1) Relaxation of atoms adsorbed to the surface dominates at lower temperature and decreases at higher temperature in proportion of the adsorption time (this is the case for aluminosilicate glass). (2) Relaxation in the bulk material increases with temperature due to enhanced diffusion (this is the case for pyrex).

The adsorption time T_{ad} of He on a Cs surface varies according to the Frankel law,

$$T_{ad} = \tau_0 \exp(E_{ad}/kT). \tag{5}$$

The adsorption energy E_{ad} is very small ($E_{ad} \sim 2.3$ K according to Ref. [19]). This means that at room temperature T_{ad} reaches its minimum value τ_0 ($\tau_0 \sim 10^{-13}$ s). Under these conditions, the surface relaxation time T_1^{wall} may be written approximately as [29],

$$T_1^{\text{wall}} = \gamma^2 \langle H_1(t)^2 \rangle \tau_0^2 / T_0, \tag{6}$$

where T_0 is the average time of flight between successive wall collisions, γ the gyromagnetic ratio of ^{3}He and $H_1(t)$ the fluctuating field on the Cs surface. With $T_1 \approx 100$ h and $T_0 \simeq 10^{-4}$ s Eq. (6) would yield for the microscopic random field on the surface an average value of

$$\langle H_1(t)^2 \rangle^{1/2} \simeq 6 \text{ T}, \tag{7}$$

which is by orders of magnitude too large to be realistic. Hence surface relaxation is not responsible for the observed relaxation times in Cs coated cells, unless one evoques surface trapping by some unknown impurities. Rejecting this unprobable alternative one concludes that bulk relaxation in the Cs coating and/or in the underlaying glass substrate dominates.

In the latter case one could expect that the ratio of relaxation times measured in cesiated supremax and pyrex cells equals the ratio measured in uncoated cells (which is of order 10, as can be read from Table 1). However, after coating it is close to 1. Hence the role of the substrate in the residual relaxation is largely reduced. This finding did not come unexpected since alkali atoms are known to fill the holes in the structure of glasses and hence inhibit diffusion.

Aside of the special case of the alkalis, bismuth provides definitely longer relaxation times than other metal coatings. This finding seems to corroborate at least qualitatively our initial intuitive intention to try an essentially diamagnetic metal. But it needs more detailed investigations in order to clear up the residual relaxation mechanisms.

Already now, these coated cells have proved to be very useful [30] and open up many possibilities of applications in different fields [31]. We mention that a group in Caen confirmed our results with cesium coated cells and immediately applied this technique to their work on ^{3}He NMR magnetometry [32]. The possibilities in nuclear physics are under investigation. A target cell filled with 2.5 bar of 25% polarized ^{3}He has been exposed in a test experiment to a 10 μA electron beam from the MAMI accelerator at Mainz for 4 h without showing any appreciable sign of additional spin relaxation caused by the beam. In another test experiment, the same cell was inserted into the thermal neutron beam ($\lambda = 1$ Å) of the Mainz TRIGA-reactor producing a polarization of 35% of the transmitted beam. In both cases, the cell was detached from the filling station, which greatly facilitates experiments.

Investigations along these lines will continue in order to minimize relaxation further and to trace its residual sources. In particular the temperature dependence of T_1 has to be studied systematically.

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