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Performance of the COMPASS polarized target dilution refrigerator

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Abstract

The dynamic nuclear polarization (DNP) of ⁶LiD requires 1 mW/g or more microwave power in the beginning of the process. With the material of 350 g, this gives more than 350 mW in total for the COMPASS polarized target. A temperature around 300 mK or below is needed for an efficient polarization. These low temperatures can only be achieved with a dilution refrigerator designed to operate with a ³He flow of 100 mmol/s. In order to keep the polarization in the frozen mode, temperatures of about 65 mK are used with typical magnetic relaxation times of more than 1400 h at 0.42 T and of more than 15000 h at 2.5 T. Low lattice temperature is important in achieving high nuclear polarization. The base temperature is limited by the heat brought to the mixing chamber by the inlet ³He and by radiation and conduction of heat.

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PACS: 29.25.Pj; 07.20.Mc

Keywords: Polarized target; Dilution refrigerator; COMPASS; Cooling power

1. Introduction

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The large horizontal dilution refrigerator for the twin polarized target of the COMPASS experiment has been used since 2001 [1]. The dilution

^{0168-9002/\$ -} see front matter \odot 2004 Elsevier B.V. All rights reserved. doi:10.1016/j.nima.2004.03.164

refrigerator was originally built for the SMC experiment at CERN [2]. Dilution refrigeration is the only practical method for cooling the target material since temperatures below 100 mK are required to slow down sufficiently the spin-lattice relaxation. The lattice temperature directly connects to the maximum polarization that can be achieved [3]. A high cooling power is needed during the DNP process with microwave power with the material of 350 g is required at the beginning of the DNP process since normally 1 mW or more of microwave power per gram of ⁶LiD material is needed [4,5].

2. Dilution refrigerator

Fig. 1 shows a simplified diagram of the COMPASS dilution refrigerator. The mixing chamber is made of glassfiber-enforced epoxy with 0.6 mm wall thickness. It has a length of 1600 mm and a diameter of 70 mm. In order to ensure uniform cooling inside the mixing chamber, the incoming ³He is fed through 40 holes drilled in a



Fig. 1. The COMPASS dilution refrigerator: (1) liquid helium buffer dewar, (2) separator, (3) evaporator, (4) still, (5) main heat exchanger, (6) mixing chamber, (7) microwave cavity, (8) magnet liquid helium vessel, (9) thermal screen, (10) ³He pumps, (11) ⁴He pumps, and (12) ⁴He recovery line. Black (Blank) arrows show ³He (⁴He) flow routes.

CuNi tube at the bottom of the mixing chamber. A heater wire is wound on this tube. The step sintered heat exchanger has a surface area of 12 m^2 . The mixing chamber is surrounded by a cylindrical copper microwave cavity of 208.5 mm diameter. The cavity is divided axially into two compartments, upstream and downstream, by a thin microwave stopper made of copper foil. The cavity screen is cooled down to 3 K by a pumped ⁴He flow. A ³He pumping system with 8 Root's blowers in series, Pfeiffer Vacuum GmbH, with a pumping speed of $13500 \text{ m}^3/\text{h}$ is used. The amount of ³He gas used for the operation of the dilution refrigerator is 1400 l at standard temperature and pressure. A volume of 9200 l of ⁴He gas is mixed with ³He gas. On the other hand, the dilution refrigerator consumes 15-20 l/h of liquid helium for evaporator, separator, and the screen lines during operation. The liquid helium consumption rate depends on the ³He flow rate. The ³He flow rate is controlled with an electric heater installed in the still. The heater is made of a stainless-steel strip having a surface area of 0.58 m^2 . The typical ³He flow rate is 30– 100 mmol/s measured by a flow meter and a quadrupole mass spectrometer, Balzers OMS 311. The quadrupole mass spectrometer was used for measuring the mixing ratio of 3 He and 4 He. The temperature measurements below 1 K are done with ruthenium oxide (RuO) and carbon resistors (Speer 220 Ω) that are read by 4-wire AC resistance bridges, RV-Elektroniikka Oy AVS-46. Two RuO and three carbon resistors are installed in the mixing chamber. The locations of these thermometers in the mixing chamber are shown in Fig. 2.

3. Paramagnetic resonance spectrum with two carbon resistors

In order to implant paramagnetic centers the ⁶LiD material was irradiated by the 20 MeV electron beam of the injection linac at the Bonn University [6]. The paramagnetic resonance spectra were observed by using TTH1 and TTH2 carbon resistors as bolometers, keeping the microwave frequency at 70.210 GHz, the microwave



Fig. 2. (a) Upstream target cell, (b) downstream target cell, (c) inside bore of the magnet, (d) microwave cavity, (e) cavity end window, (f) microwave guide, (g) microwave stopper, (h) mixing chamber, (i) target holder, (j) ³He feeding tube. Thermometers: ① TTH4 is a RuO calibrated by Scientific Instruments, Inc. ② TTH1 and ④ TTH2 are Speer 220 Ω calibrated by ³He vapor pressure at Dubna. ⑤ TTH6 is a RuO and ⑥ TTH7 is a Speer 220 Ω . TTH6 and TTH7 are not calibrated for 100 mK.



Fig. 3. Paramagnetic resonance spectrum of ⁶LiD at high polarization measured by bolometric method. The solid (dashed) line is the upstream (downstream) resonance at the polarization of +56% (-47%). The increase of the value of resistances represents the decrease of the temperature.

power constant at $\sim 10 \text{ mW}$ in both cells and using a magnetic field sweep speed of 0.024 mT/s (Fig. 3). Since carbon resistors TTH1 and TTH2 also absorb microwaves, an accurate temperature could not be measured. Nevertheless, a temperature of about 100 mK in the mixing chamber was estimated from the microwave power.

The decrease of the thermometer temperature value reflects the microwave absorption by the paramagnetic centers in the material. The measured spectra show the separation of the main Table 1

The polarization *P*, and the microwave status of the frequency and the power with increasing polarization during DNP for each cell ~ 175 g of material at 2.506 T.

P (%)	P > 0		P<0	
	f (GHz)	<i>p</i> (mW)	f (GHz)	<i>p</i> (mW)
0	70.190	200	70.285	200
~ 30	70.210	100	70.265	100
~ 40	70.220	50	70.255	50
~ 50	70.230	25	70.245	25

broad peaks by a few mT which corresponds to the microwave frequency of ~100 MHz. The optimum microwave frequency of the positive (negative) polarization shifted from 70.190 GHz (70.285 GHz) to 70.230 GHz (70.245 GHz) with increasing polarization up to 50% during DNP process at 2.506 T (see Table 1). This total shift of 80 MHz is almost consistent with the separation of the spectrum peaks [7].

4. Optimization of the ³He flow for DNP

The cooling power of 350 mW at \sim 300 mK in the mixing chamber was obtained with a ³He flow of 100 mmol/s at the beginning of the DNP process. During DNP the temperature in the mixing chamber decreases slowly as the optimum microwave power is reduced with increasing polarization. After reaching \sim 50% polarization in both cells, the total microwave power is set to around 50 mW (Table 1). The lowest temperature with 50 mW cooling power is obtained by tuning the ³He flow with the still heater power (Fig. 4). It was found that the minimum temperature of 125 mK with a cooling power of 45 mW was obtained with a ³He flow of 50 mmol/s.

5. Frozen spin mode

Once the microwave was switched off the dilution refrigerator cooled down to 55 mK. The energy deposit of the 190 GeV muon beam with an intensity of 2×10^8 muons/spill was about 1 mW



Fig. 4. The mixing chamber temperature as a function of the ³He flow. The values in the figure represent the cooling powers. The wider shadow area represents the condition for the DNP mode and the narrower area is for the frozen spin mode.

on average in the target cells during beam on. The beam spill cycle was 16 s. The mixing chamber stabilized at 65 mK during beam on. It was sufficient to keep the nuclear spins in *frozen spin mode* with relaxation times of more than 15 000 h at 2.5 T and of more than 1400 h at 0.42 T. For comparison, a relaxation time of ~ 150 s was measured, just after switching off the current of the superconducting magnet.

The frozen spin mode is essential for the spin reversal operation by rotation of the magnetic field and for the transverse polarization data taking mode. This spin reversal was performed every 8 h in order to cancel the systematic error due to different spectrometer acceptances of the two target cells and due to the time dependent variation of the spectrometer efficiency.

6. Cooling power

The enthalpy balance for the mixing chamber can be established as

$$\dot{n}_{3}[H_{\rm d}(T_{\rm mc}) - H_{\rm c}(T_{\rm mc})] \\= \dot{n}_{3}[H_{\rm c}(T_{\rm ex}) - H_{\rm c}(T_{\rm mc})] + \dot{Q} + \dot{Q}_{\rm leak} + \dot{Q}_{\mu}.$$
(1)

 $H_{\rm c}$ is the enthalpy of ³He in a concentrated phase and $H_{\rm d}$ is the enthalpy of ³He in a diluted phase. $T_{\rm ex}$ is the inlet ³He temperature of the mixing chamber and $T_{\rm mc}$ is the outlet temperature. \dot{n}_3 is the ³He flow rate. The cooling capability which is expressed by the left-hand side of Eq. (1) is produced by the transfer of ³He from the concentrated phase to the diluted phase, called *heat of mixing*. $\dot{Q}_{\rm leak}$ and \dot{Q}_{μ} represent the heat leak and the energy deposit of muon beam (~1 mW). The cooling power \dot{Q} is used to precool the warmer liquid ³He coming from the heat exchanger, and to compensate for the heat leak and the energy deposit of muon beam [8]. A term on ⁴He flow is not considered in this equation.

The cooling power \dot{Q} can be obtained from Eq. (1),

$$\dot{Q} = \dot{n}_3 [H_d(T_{\rm mc}) - H_c(T_{\rm ex})] - \dot{Q}_{\rm leak} - \dot{Q}_{\mu}.$$
 (2)

Heat radiation, heat conduction from the microwave cavity, and conduction by gas particles which remain in the vacuum space between the mixing chamber and the cavity are the main candidates of the heat leak. A total heat leak \dot{Q}_{leak} of 4.5 mW was estimated for the COMPASS dilution refrigerator. The dominant source was the heat conducted from the microwave cavity, through the microwave stopper, and through the thermal isolation providing Teflon tubes to the outside of the mixing chamber. This specific heat leak was conservatively estimated to be at most 4.3 mW. In practice, this heat leak was measured to be 2.3 mW in the *one-shot* mode. The inlet ³He line is closed and no warm inlet ³He exists in the one-shot mode, i.e. $\dot{n}_3[H_c(T_{ex}) - H_c(T_{mc})] = 0$ in the right side of Eq. (1). When $T_{\rm mc} < 40$ mK, the heat of mixing can be described as $\dot{n}_3[H_d(T_{mc}) - H_c(T_{mc})] = 84\dot{n}_3 \cdot T_{mc}^2$ [8]. This heat of mixing is used only for the heat leaks at the lowest temperature. Therefore, from Eq. (1) the heat leak \dot{Q}_{leak} can be expressed as

$$\dot{Q}_{\text{leak}} = 84\dot{n}_3 \cdot T_{\min}^2 \tag{3}$$

with $\dot{Q}_{\mu} = 0$ and $T_{\min} < 40$ mK, where T_{\min} represents the minimum temperature of the mixing chamber. \dot{n}_3 was 30 mmol/s. A minimum temperature of 30 mK in the one-shot mode was measured without the muon beam (Fig. 5).

If the heat exchanger has a perfect performance the T_{ex} becomes equal to T_{mc} . This is the ideal performance of the dilution refrigerator. It means



Fig. 5. The temperature in the mixing temperature as a function of the time in the one-shot mode. The solid (dashed) line is for the upstream (downstream).

that the geometrical surface area of the heat exchanger would be infinite or the ³He flow rate was zero. In other words, the maximum cooling power of the dilution refrigerator is reached at the limit of $T_{\text{ex}} = T_{\text{mc}}$. The temperature of the inlet ³He, i.e. the heat exchanger, can be estimated to be 150 mK with Eq. (2) when the mixing chamber temperature is 65 mK [9].

The difference of the cooling power in the upstream and downstream target cells was observed in the 2003 run by using TTH1 for upstream and TTH2 for downstream. The temperature difference was ~ 100 mK at the beginning of the DNP process. However, the temperature difference was negligible in the frozen mode.

Considering the cooling power normalized by ³He flow rate, the performance for each stream of the dilution refrigerator becomes obvious. Assuming that 50% of ³He in the circulation line flows to the upstream, the two lines of the normalized cooling powers of the upstream and downstream parts are split in Fig. 6. It means that the cooling power per mole of ³He is different in the upstream and downstream part. The dashed line shows the ideal performance, i.e. $T_{\text{ex}} = T_{\text{mc}}$ and $\dot{Q}_{\text{leak}} = \dot{Q}_{\mu} = 0$, calculated with enthalpy data of Ref. [9].

However, both lines should follow each other closely since the dilution refrigerator should have the same cooling power per mole of ³He in the upstream and downstream part. Therefore, ³He circulation of 70% (30%) in the circulation



Fig. 6. • (\triangle) represents the upstream (downstream) cooling power normalized by \dot{n}_3 as a function of $T_{\rm mc}$ on the assumption that 50% of ³He in the circulation line flows to the upstream. The dashed line represents the theoretical ideal line.



Fig. 7. • (\triangle) represents the upstream (downstream) cooling power normalized by \dot{n}_3 as a function of $T_{\rm mc}$ on the assumption that 70% of ³He in the circulation line flows to the upstream cell.

line is estimated for the upstream (downstream), see Fig. 7.

The difference of the ³He flow concentration in the upstream and downstream part can explain the averaged difference of the maximum polarizations between both target part. A 2–3% difference of the polarization in the upstream and downstream cell that was observed in the 2003 run as shown in Table 2 [10].

If the dilution refrigerator had had the same cooling power for the both cells then the same value of the positive (or negative) polarization would be obtained for the upstream and downstream part. Table 2

The deuteron polarizations obtained with ^{6}LiD in the COMPASS polarized target in 2003

	Upstream	Downstream
Positive polarization	+57%	+54%
Negative polarization	-51%	-49%

The positive and negative polarization in the upstream cell is higher than those in the downstream cell. Usually the positive polarization of 6 LiD that can be achieved is higher than the negative polarization [11].

7. Conclusion

The COMPASS dilution refrigerator shows a good performance around 300 mK which is the DNP starting point. Although the dilution refrigerator has a heat leak of a few milliwatts to the mixing chamber, a temperature below 60 mK can be achieved regularly. This is sufficient to keep the nuclear spins with relaxation times of more than 15 000 h at 2.5 T and of more than 1400 h at 0.42 T. The difference of cooling power between upstream and downstream target cells is explained by the fact that the 70% of the ³He flow went to the upstream part. It explains the 2-3% difference in maximum polarization.

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