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PROGRESS REPORT ON POLARIZATION OF ^{6}LiD AND ^{7}LiH IRRADIATED WITH HIGH ENERGY ELECTRONS

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A B S T R A C T

After our previous attemps to polarize ^{7}LiH irradiated in liquid argon, with a maximum polarization of 1 % ,the irradiation cryostat has been modified in order to work at higher and well monitored temperature. New irradiations have been achieved on ^{6}LiD and ^{7}LiH at 150 K. We have obtained a polarization of 10 % on the ^{7}LiH sample.The maximum has not been reached because of the slow polarization build-up due to the long relaxation time of the paramagnetic centers. The other characteristics of this polarization seem to be satisfactory for the use in frozen spin mode.We now work to determine accurately the best temperature and dose of irradiation by a systematic study of the paramagnetic resonance of the f-centers.

1 - INTRODUCTION

It was recognised in 1978 [1] that polarized ^{6}LiD [2] could be an excellent material for high energy experiments requiring polarized neutron targets. More recently [3] it has been conjectured that, similarly , polarized ^{7}LiH may have a substantial average polarization of all protons and may thus be a good polarized target for inclusive reactions on polarized protons. Thin chips of both materials (0.5 mm thick) had been successfully polarized by prof.Abragam's group in 1978 .The paramagnetic centers were created by irradiation with low energy electrons (2-3 MeV, 10^{17} e/cm^2).This irradiation technique is unconvenient for large samples required in high energy experiments. However the nature and the characteristics of the paramagnetic centers obtained by the SPSRM are our reference for optimising irradiation with high energy electrons (200-600 MeV) from the Saclay Linear Accelerator (ALS).

2 - IRRADIATION OF ^{7}LiH AT 90 K.

In 1984 we had irradiated sintered powder samples of ^{7}LiH [4] in liquid argon (90 K) with 10^{17} e/cm^2. Our attemps of polarization showed a small growth which stopped after 1 hour at a maximum polarization of about 1 % at 2.5 T and 5 T. The results were reported at the Bad Honnef workshop in 1984 [4].

We have measured the EPR spectra at 10 Ghz of our sintered samples. Comparison with our reference (the small single crystals irradiated by the SPSRM at low energy) has shown that our sample had one order of magnitude less F centers by unit of volume, and that it had a large and sharp resonance of metallic ^{7}Li (1.4 Gauss wide) in the middle of the F center band (85 Gauss wide).

From the existing informations on the strong variation of the number of paramagnetic center and of their nature as a function of the temperature during irradiation [5,6], we concluded that liquid argon temperature was too low. In order to obtain a sufficient number of centers of the correct type, the sample has to be maintained at some higher and well defined temperature. We also concluded that we had to use samples less contaminated by metallic lithium ions which are suspected to interfere destructively with the DNP polarizing processes.

For the sintered powder sample irradiated at 90 K we have made an attempt to suppress the unwanted type of centers by annealings at different temperatures up to room temperature. However the EPR spectra showed no clear evidence for significant modification due to annealing.

3 - IRRADIATION OF ^{6}LID AND ^{7}LIH AT 150 K.

In 1985 we have irradiated new samples of both ^{7}LiH and ^{6}LiD in a variable temperature cryostat.

a) Quality of the sample.

For both materials clean samples free of metallic lithium were prepared directly from the manufactures melting pot. The cooling after synthesis at high temperature results in irregular crystals of sizes ranging from 2 mm to 2 cm. The crystals were broken loose, crushed and filtered to select monocrystallin pieces of about 2 mm. These operations were carried out in pure argon atmosphere, carefully avoiding local heating of the material.

b) Argon gaz cryostat.

We considered initially the use of liquids but there seems to be no appropriate cryogenic liquid in the temperature range 120 K - 150 K. We finally decided to use a forced flow of argon gas at variable temperature. The liquid argon cryostat used in 1984 was modified by installing a turbine driven by a special 12000 rpm. aeronautic quality motor capable of running between -60 °C and +110 °C in vacuum or argon atmosphere (fig. 1). The gas circulates in closed loop in contact with both a cold source provided by liquid nitrogen, and a source of heat provided by beam heating and an additional electric heater. The system can evacuate more than 150 W from the 70 cm^3 sample at any temperature from 80 K to room temperature. During the irradiation the temperature rise of the gazeous argon across the sample was less than 3 K at 150 K, independently of beam intensity variations. The temperature is regulated by feed-back acting on both the nitrogen level and the heater power. Mul-

tiple safety devices retract the entire cryostat out of the beam to avoid overheating in case of failure of one of the components.

In november 1985 we have irradiated 5 samples of each of the two materials simultaneously, at distances from the beam axis between 0 and 15 cm. The relative intensities at the 5 positions were 1, 1/3, 1/10, 1/30 and 1/100, respectively, monitored by nickel foils. The central foil was calibrated against the reading of the Faraday cage immediatly behind our apparatus. In the 5 days irradiation the dose at the center reached 2.5 10^{17} e/cm^2. The beam energy was variable between 200 and 600 MeV and the temperature was chosen at 150 K.

c) Characteristics of irradiated ^{7}LiH.

i) Polarization.

The polarizability of a 4 cm^3 sample of ^{7}LiH irradiated at 2.5 10^{17} e/cm^2 was investigated in the Saturne polarized target apparatus [7] comparing the proton NMR signal to the one at thermal equilibrium. In addition a 10 mm^3 single crystal,irradiated with the same dose, was studied in the SPSRM apparatus at 5 T [2], using the ^{7}Li NMR signal for calibration.

At 2.5 T field we found growth rates of 0.55 and 0.35 % per hour for positive and negative polarizations, respectively. Temperature during polarization was between 200 mK and 400 mK. Because of the very large polarization time, the build up was interrupted after 24 hours. In both cases we have observed no decrease of the growth rate. The positive and negative polarizations had reached 10.5 and 8.5 %,respectively. In spite of a higher field (5 T), the single crystal studied in the SPSRM apparatus showed very similar behaviour.

ii) Density and relaxation time of the pramagnetic centers.

Good polarization growth rate depends critically on sufficient density and short relaxation time of the paramagnetic centers. The relaxation time for the small single crystal has been determined by observing the recovery of the internal field following microwave saturation of the EPR line. The shift of the NMR line was used to observe the change of the internal field due to the paramagnetic centers. The approximately cubic shape of our sample does not allow to determine the density of the paramagnetic centers from the observed shift of the NMR line. For this reason we will in the future irradiate also thin chips having a large demagnetization form factor.

iii) Nuclear zeeman relaxation.

The proton polarization relaxation time was measured for the 4 cm^3 sample at 0.1 T and 50 mK. The observed decay time of 4 to 5 hours is similar to the one for alcohols under the same conditions.

iv) Adiabatic polarization reversal

In the SPSRM apparatus the sample was submitted in the frozen spin mode (100 mK, 5 T) to an Adiabatic Demagnetization in the Rotating Frame (ADRF). Since only one NMR coil was available in the apparatus, the ADRF was performed on the ^{7}Li spins: an RF field of a few tenth of mG was applied in the vicinity of the ^{7}Li resonance, and the magnetic field was swept from -75 G to +75 G from resonance at a rate of 0.5 Gauss/sec. The absolute value of the final polarization was about 60 % of its initial value (itself equal to 4 %).The result can be compared to the one obtained with ^{6}LiD where the initial polarization was substantially higher and where both ^{6}Li and D were reversed simultaneously. In this case the yield of the ADRF was of 75 %.

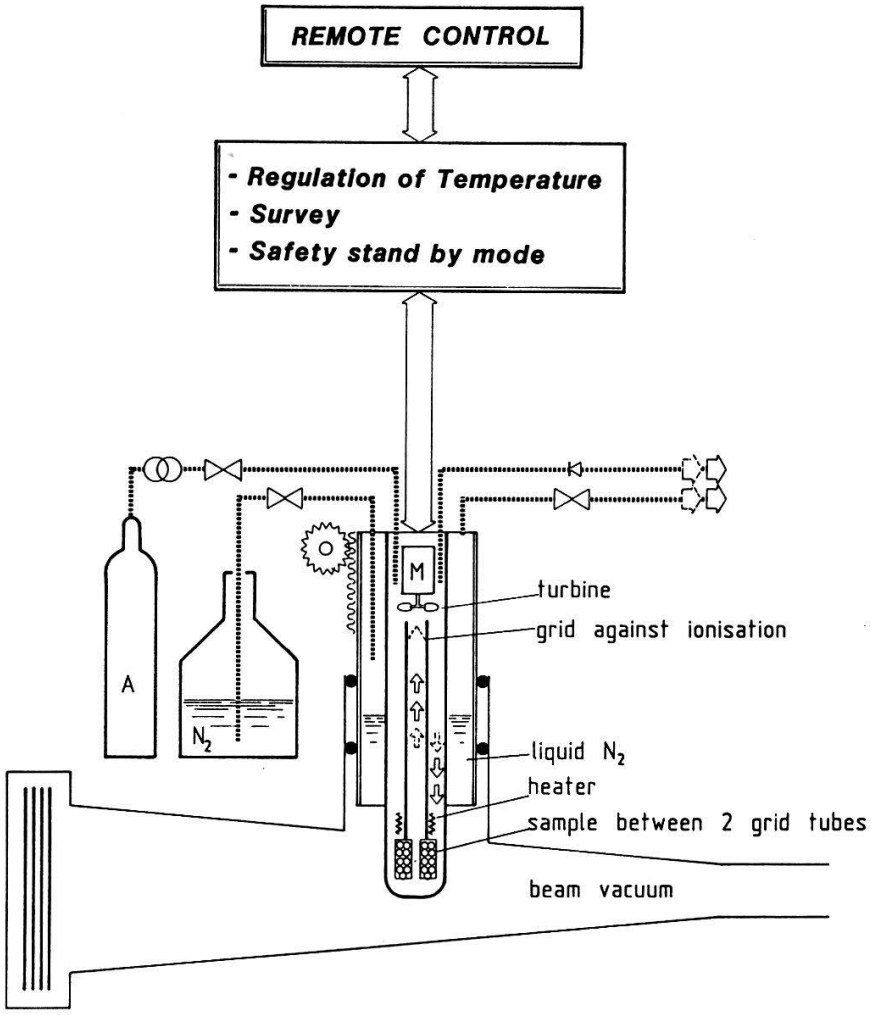

Figure 1. Argon gas cryostat.

d) Characteristics of irradiated ^6LiD.

Simultaneously with ^7LiH , one gram of ^6LiD was installed in a special RF coil (6 turns, high filling factor) connected to a Q-meter at 16 MHz. During the study of the ^7LiH sample, we have observed substantial enhancement of the D and ^6Li signals. The D NMR signal is a single symmetrical peak of 3.5 KHz FWHM. The ^6Li and the D signal at 16.07 and 16.35 MHz, respectively, have similar shape and amplitudes. The single peak of the deuteron signal in ^6LiD, as opposed to double peak pattern in the other deuterated materials, is due to the absence of quadrupolar effect in a crystal with cubic symmetry. Unfortunate breakdown of the apparatus prevented us from measuring the natural signals. The Zeeman relaxation of the ^6LiD polarization at 0.1 T and 50 mK was about 4 hours, similar to the ^7LiH sample.

CONCLUSION

By rising the irradiation temperature from liquid argon to 150 K we have come closer to creating centers of the right kind in sufficient number. We suspect that the slow polarization build-up is related to both the small density and the long relaxation time of our paramagnetic centres. In the future we will try to produce higher densities and faster paramagnetic centres by further increasing the beam doses and by going to even higher temperatures. Any temperature can be set acurately with the present gas cryostat which operates very satisfactorily.

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Rapporteur's report, Session (K), W. Wenckebach:

The interest of the polarization of ^{6}LiD is due to the assumption that the ^{6}Li nucleus can be described to consist of a ^{4}He and a ^{2}D nucleus, so ^{6}LiD contains effectively two polarizable deuterons per molecule. During the discussion it became clear that this assumption is by no means obvious: e.g. ^{14}N cannot be described to consist of ^{12}C and a deuteron, however the nuclear physicists assure us that for ^{6}Li this assumption is correct.

As it has been proved by Abragam's group that small samples of ^{6}LiD can be polarized very well, the present problem is to construct a larger target. Then it is necessary to use high energy (200 MeV) electrons instead of low energy (2 MeV) electrons to create the paramagnetic centres necessary for DNP throughout the sample. Alternatives like γ-rays were tried but did not lead to DNP. At present the results with high energy electrons are promising, but the rate of the DNP is still an order of magnitude slower than in the case of samples irradiated with low energy electrons.