DEUTERON AND PROTON POLARIZATIONS IN IRRADIATED MATERIALS

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In a continuing study of irradiated polarized target materials, various samples have been subjected to irradiations with the 19 MeV electron beam at the MIRF facility at NIST. Previously, deuterated materials were investigated and the polarization results reported. In this paper we report on further investigations with deuterated materials and a first look at proton polarizations under the same conditions.

1. Introduction

It has long been recognized that irradiation of materials for polarized targets is an alternative means of producing paramagnetic radicals to that of chemical doping. [1] However, for a long time, the proton polarizations obtained with irradiated materials were not competitive with the values obtained with chemical dopants and thus irradiated materials tended not to be used in particle scattering experiments. With the development of irradiated ammonia the situation changed; proton polarizations of $> 95\%$ were routinely achieved and ammonia was used as a polarized target material in many experiments. Deuterated ammonia was later found to need first, a "warm" irradiation at 80 - 90 K, followed by a "cold", usually *in situ,* irradiation at $\lesssim$ 1K, in order that deuteron polarizations of about 50% could be realized. This is about double that typically obtained with chemically doped materials. The historical development of ammonia as a polarized target material was recently discussed by W. Meyer [2]. Even as irradiated ammonia was being recognized as a viable polarized target material, data on irradiated ^{6}LiD had been published showing that deuteron polarizations of 70% were possible [3] at high fields (6.5 T) and low temperatures (100 -

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$\sim$300 mK). However, for many reasons, it took about twenty years before the LiD (or LiH) was used in a polarized target scattering experiment [4]. The main drawback to these materials is that relatively high doses ($\geq 10^{17}$ charged particles cm^{-2}) have to be applied to the putative polarized target, involving a substantial committment of resources. But recently, it was found that deuterated butanol, when irradiated to a level of 10^{15} electrons cm^{-2}, gave deuteron polarizations of 50 -70%, with a dilution refrigerator[5].

This paper reports on studies of polarization for various irradiated materials. The intent is to do a systematic study of polarization obtained at 1 K, by varying the dose and magnetic field, of various hydrogen and deuteron rich materials. Some materials were studied in the past, in a rather scattershot way, and these will be re-evaluated in a more systematic manner.

2. Irradiations

The irradiations are being carried out at the MIRF facility at NIST[a] in Gaithersburg, MD. This is an electron Linac which can operate at energies from 7 MeV to 32 Mev and currents of 100 μA. Material irradiations take place at 19 MeV and currents ranging from 4 μA to 10 μA with the beam focussed to cover the target cross section.

Irradiation doses are usually quoted as particles cm^{-2} incident on the face of the irradiation dewar. However, because of the difference in mechanical construction, size of target and the like, the actual dose deposited in the target material itself may vary by at least a factor of two among the different set-ups. For the moment we also quote the dose in incident electrons cm^{-2}, but are studying the energy deposited in the material itself with the EGS4 program.

3. Polarization Results

The results of the first irradiations with deuterated material have been reported[6] and show polarization results at 1 K for d- butanol and CD_2, together, for comparison, with the polarizations of d-butanol doped with TEMPO and also EDBA. The main conclusions are:

(1) the optimal dose for d-butanol is between 2 and 3 10^{15} electrons cm^{-2}.

[a]The Medical-Industrial Radiation Facility
http://physics.nist.gov/MajResFac/mirf/mirf.html

(2) over a limited range, the polarization for CD_2 is increasing with dose, but is always smaller than that for d-butanol under the same conditions and about the same as for TEMPO doped d-butanol.
(3) For both d-butanol and CD_2 the polarization increases with magnetic field, reaching over 60% and over 34%, respectively at 6.55 T
(4) d-butanol + D_2O + EDBA did not polarize at all at 5.0 T and 6.55 T, consistent with other measurements [7].
(5) At 5.0 T and 6.55 T, the negative polarization is less in magnitude than the positive polarization, in contradistinction to measurements at 2.5 T.
(6) The high polarizations quoted were reached in $1\frac{1}{2}$ to 2 hours.

3.1. *New Results*

In the past two months, various materials, both hydrogenous and deuterated, were irradiated at NIST. All except one of the polarizations reported here were measured at 5 T and 1 K, the usual conditions for operating our polarized target for particle scattering experiments. The deuteron polarizations were estimated using the asymmetry in the NMR signal but thermal equilibrium calibrations were made for the proton.

3.1.1. *Deuterated Samples*

D-butanol(C_4D_9OD) A sample of d-butanol was irradiated to 2.5 10^{15} electrons cm^{-2}, approximately the optimal dose, from the data quoted above.

The result is shown in Fig. 1. Polarizations of +50.5% and -47.6% were obtained with a build up time of about two hours. Also shown are the polarizations obtained with the 2 and 3 10^{15} samples of reference[6], after storage under liquid nitrogen for about 9 months. These polarizations were obtained after build up times of from three to six hours. About 97% of the ultimate polarization is achieved after 3 hours.

The 2 10^{15} sample was also polarized at 6.55 T and achieved a value of over 60%, in agreement with the data published in reference[6].

CD_2 The two original samples were re-irradiated because an order for fresh material had not arrived by the time of the irradiation. The original 1. 10^{15} irradiation was increased to 5 10^{15} electrons cm^{-2} and the original 2 10^{15} irradiation was increased to 10^{16} electrons cm^{-2}. The polarizations

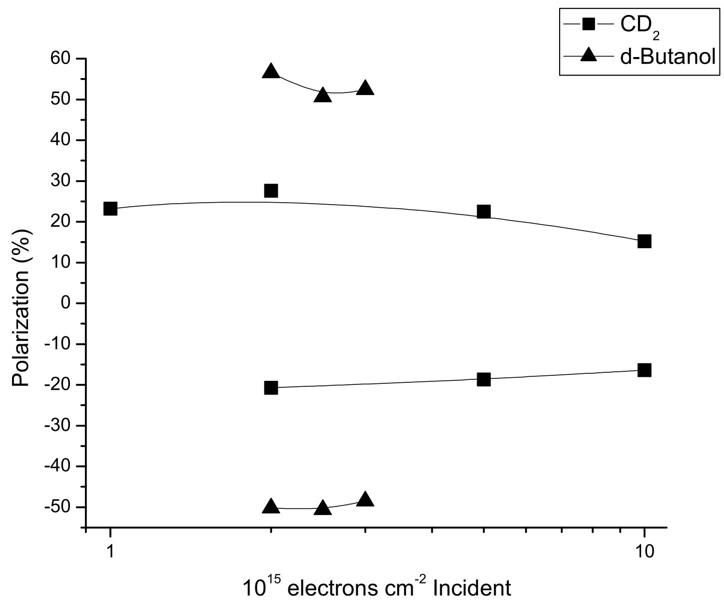

Figure 1. Deuteron polarization (%) for deuterated butanol and deuterated polyethylene (CD_2) as a function of beam dose (10^{15} electrons cm^{-2}).

achieved, as a function of dose, are shown in Fig. 1.

3.1.2. *Hydrogenous Samples*

Figure 2 shows the polarizations of borane ammonia, CH_2 and butanol as a function of beam dose

Borane Ammonia (BH_3NH_3) Borane Ammonia has been investigated in the past, irradiated *in situ* at 1 K with a 20 GeV electron beam[8] and in a reactor with the sample under LN_2 [9]. Polarizations of about 30% were obtained at 5 T in a 1K refrigerator after a total irradiation of about 10^{15} electrons cm^{-2} and after annealing. The reactor irradiated material gave a polarization of 18% in a dilution refrigerator and field of 2.5 T. Because of the different irradiation and polarizing conditions, these two results cannot be easily compared.

The Borane Ammonia was obtained from Aldrich Chemicals in powder form and, for irradiation and polarization, was pressed into thin discs of

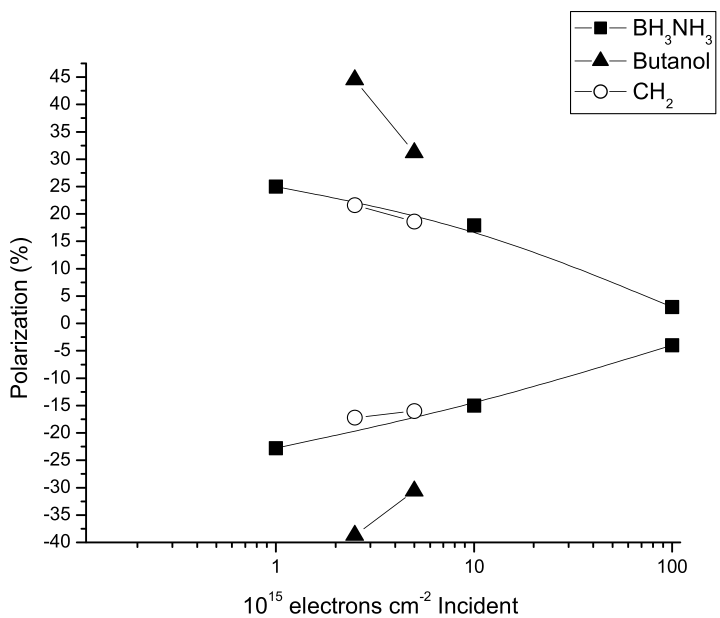

Figure 2. Proton polarizations (%) for borane ammonia (BH_3NH_3), Polyethylene (CH_2) and butanol (C_3H_9OH) as a function of beam dose (10^{15} electrons cm^{-2}).

about 1 cm in diameter. As seen from Fig. 2 the value of the polarization achieved is inversely proportional to dose. From its behaviour, the 10^{17} electron cm^{-2} sample was obviously highly overdosed. The trend is still for increasing polarization with lower doses, but it does not appear likely to reach very high values, with the best obtained being 25% at the lowest dose of 10^{15} electrons cm^{-2}.

Polyethylene (CH_2) The polyethylene used was high density (HDPE), obtained from Goodfellow, in the form of 3 mm pellets. Previous polarization measurements have been inconsistent, because of variations of the grade of polyethylene used and the conditions under which the irradiations were carried out. However none have been very encouraging.

Over a limited range of beam dose, the proton polarizations in polyethylene closely track those obtained with borane ammonia, reaching a value of 21.6% at a dose of 2.5 10^{15} electrons cm^{-2}.

Butanol(C_4H_9OH) The results obtained with butanol are much more encouraging, than with the previous two examples. A dose of 2.5 10^{15} electrons cm^{-2} led to a proton polarization of close to 45%, up from 31% at a dose of 5 10^{15}, with a build up to the maximum value in about an hour. The negative polarizations have the same trend as the positive, though with lower values.

As a comparison, with the same apparatus, butanol, doped with 2.5 10^{19} spins/ml of TEMPO was polarized to 75% and -80%.

4. Summary

Irradiated deuterated butanol has clearly the best polarization performance when compared to any other deuterated material/radical combination, with the possible exception of trityl doped d-butanol[10] which has not been polarized under our conditions. The range of optimal doping has been established and the polarization increases with magnetic field, unlike some material radical combinations [7]. It is a very good candidate for many scattering experiments.

Deuterated polyethylene has a moderate response to dose, with the best polarization between 25 and 30%; yielding a possible target material under some circumstances. However there is no reason to continue the study of CD_2 any further, under these conditions.

For the hydrogenated samples, butanol again gives the best response and could well go to high polarizations with lower doses.

Both CH_2 and borane ammonia have a similar, moderate response to dose, getting better with progressively lower doses. It will be worthwhile to see what improvement will arise from irradiations at less than 10^{15} electrons cm^{-2}.

The materials studied so far show their best performance at doses of around 10^{15} rather than 10^{17} electrons cm^{-2} which is necessary for ammonia and lithium hydride. Thus runs of minutes rather than hours are required. The radiation damage resistance at low temperatures is unknown for these material/radical combinations.

5. Acknowledgments

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