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NUCLEAR MAGNETIC RESONANCE IN POTARIZED NUCLEI

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NUCLEAR MAGNETIC RESONANCE IN POLARIZED NUCLEI E. Matthias and R. J. Holliday Lawrence Radiation Laboratory University of California Berkeley, California

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The nuclear ferromagnetic resonance of 60 Co in Fe has been detected by observing the effect of a rf field on the angular distribution of the γ -radiation emitted by polarized 60 Co nuclei at temperatures of about 0.03°K. At resonance, rf induced transitions partially randomize the Boltzmann distribution, which is set up by the low temperature and the large hyperfine field for Co in Fe, in the 60 Co ground state. This has a destructive effect on the angular distribution of the emitted γ -rays, which define the detected by observing the counting rate at a specific angle as a function of frequency. The enhancement of the rf amplitude in a ferromagnet¹ was necessary to keep the rf power at a sufficiently low level in order to avoid a rapid warm up of the sample.

This experiment was part of a more general program of our group in which we are exploring the possibilities of performing NMR experiments in radioactive states, using the emitted radiation pattern to detect the resonance. From the detection point of view all methods can be used that-involve emitted radiation with an anisotropic angular distribution (or correlation) which can be destroyed by the rf field at resonance. This selects three different types of experiments: 1) Nuclear orientation at low temperatures; 2) Angular correlations; 3) Angular distributions produced by various kinds of nuclear reactions. Apart from the different experimental techniques the three methods apply to different life⁻ time regions of the nuclear state. For short lifetimes ($\tau_{\rm N} \leq 10^{-6}$ sec) both

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2) and 3) can be used. For lifetimes between 1^{-6} sec and 1 sec only method 3) would apply, and for states with $\tau_N > 1$ sec low 'emperature orientation is applicable. The given time limits are approximate and might in some cases change as much as an order of magnitude. In a remarkable experiment of type 3) Sugimoto et al.² proved that the polarization of nuclei produced by a nuclear reaction can be preserved up to 10 sec or longer at room temperature and subsequently used to detect the nuclear magnetic resonance. The resonance destruction of an angular correlation was also shown to be possible³ by making use of the ferromagnetic enhancement of the rf amplitude. An experiment of type 1) has in principle been suggested by Bloembergen and Temmer⁴ in 1953, but to our knowledge, no successful experiment has been carried out along this line. The reason for this was probably the large heating effect of the rf field.

-2.

In an attempt to investigate the suggestion of Bloembergen and Temmer further, we chose to again take advantage of the rf amplitude enhancement in a ferromagnetic sample. For polarized foils the rf field, H_1 , is amplified according to the relation¹ 2 $H_1^{eff} = (1 + H_{hf}/H_0)H_1$, where H_0 is the polarizing external field and H_{hf} is the hyperfine field. In this way, it was possible to keep the externally applied rf power sufficiently low so that an adequate warm up of the sample was obtained. For the first experiment of this type we chose radioactive ⁶⁰Co for which both the magnetic moment and the hyperfine field in iron are known accurately, thereby making the search for the resonance easier, as we anticipated a fairly small linewidth. ⁶⁰Co emits two γ rays of 1.173 and 1.332 MeV in cascade both of which have negative anisotropy and were therefore taken together in the window of the single channel analyzer to give a larger total counting rate. The low temperature $(0.02^{\circ}K)$ was reached by adiabatic demagnetization of a chrome-alum-glycerine slurry. The sample was in thermal contact with this salt by means of copper fins which reached into the demagnetization salt.⁵ The sample was a thin $(\sim 10^{-4} \text{ cm})$ Fe foil about 3 x 3 mm² onto which the activity was electroplated. The activity was then diffused (alloy I at 950°C for 80 hr, alloy II at 950°C for 48 hr) into the foil and soldered with Wood's metal onto the copper fins. The polarizing field, H₀, was parallel to the plane of the foil and was produced by a pair of Nb rings located on either side of the foil. The rf field, H₁, oriented parallel to the plane of the foil but perpendicular to the polarizing field H₀ was fed in by a 2 turn coil in the He bath around the cryostat. The angular distribution of γ rays emitted from polarized nuclei is given by

$$W(\theta) = \sum_{k \text{ even}} B_k U_k F_k P_k (\cos \theta)$$

with the usual definition of the coefficients B_k , U_k , and F_k .^{5,6} The orientation parameters B_k are functions of the Boltzmann distribution, depending on the magnetic moment, μ , effective magnetic field, H_{eff} , and the temperature. Resonance induced transitions consequently alter the orientation parameters B_k which is observed as a change in radiation intensity at a fixed angle θ . This effect can be seen in the upper part of Fig. 1 where the warm up curve for $\theta = 0^{\circ}$ is shown as a function of frequency, which was varied at constant time intervals. The resonance effect is clearly visible at about 165.8 MHz. To check whether this peak is really a frequency effect we measured the warm up curve for the same temperature interval and the same rf amplitude but at a fixed frequency off resonance. The result was negative as shown in the lower

part of Fig. 1. To improve statistics we added together the results of different demagnetizations and the final result for two different alloys is shown in The data in the left part of this figure (alloy I) are the sum of four Fig. 2. demagnetizations, in which the frequency range was scanned twice per demagneti-The data on the right (alloy II) are the result of two demagnetizations zation. with one frequency scan after each. Although the effect at 90° is smaller the opposite sign compared with O^O is apparent. The observed line widths of about 0.8 MHz (alloy I) and 0.5 MHz (alloy II) is of the same order of magnitude as observed in nuclear ferromagnetic resonance. The slightly larger line width of the resonance in alloy I is in all probability caused by a larger inhomogeneity of the polarizing field, $H_{
m o}$, due to the fact that alloy I was larger. Also, the second frequency scan after a demagnetization sees a slightly displaced resonance compared to the first scan which is probably due to a change of the trapped flux in the Nb rings. To draw any conclusions from the observed line width one would have to use a better controlled external polarizing field with greater homogeneity. This would also provide the possibility of observing the change of the resonance frequency as a function of the external field and thereby measuring the g factor of the nuclear state directly.

The average value of the resonance frequency taken from both alloys is 165.75 ± 0.15 MHz. With a magnetic moment of $\mu = 3.754\pm0.008$ n.m.⁷ this gives an effective magnetic field of $|H_{eff}| = 289.6\pm0.7$ kGauss. Assuming that the demagnetization factor for a thin foil is negligible we obtain after correcting for the polarizing field (1.0±0.5 kGauss) the value

 $|H_{if}| = 290.6 \pm 0.9 \text{ kGauss}$

for the internal magnetic field of very dilute Co in Fe (less than 1 in 10^3 atoms) at 0.03 K. This agrees well with the value of 289.7 kGauss measured by NMR for 1% Co in Fe⁸ and 4 to 17% Co in Fe⁹.

To conclude it can be said that the radiative detection of NMR in polarized nuclei is proved to be possible. Compared to conventional nuclear polarization measurements⁶ this method offers at least an order of magnitude higher accuracy which also makes it possible to determine the nuclear g factor directly by varying the external polarizing field. Further, it is independent of any precise knowledge of the temperature scale. It should be noted that the conventional nuclear orientation technique using γ rays measures an energy $|\mu \cdot H_{pff}|$ while the present method gives a frequency $|g H_{eff} \mu_N/\hbar|$. Combining both techniques would determine the spin of the nuclear state.¹⁰ Also with regard to magnetic hyperfine fields of impurities in ferromagnetic lattices this technique will prove very valuable. Aside from the accuracy it measures to a good approximation the hyperfine field at zero degrees and is capable of working with extremely small concentrations. A great number of radioactive isotopes and isomeric levels are accessible to this technique, in particular in connection with isotope separators which provide a way to shoot isotopes into a lattice in cases where chemical procedures fail.

Acknowledgments

We are grateful to Professor D. A. Shirley for stimulating discussions and his great interest in this work. The success of these experiments was dependent on the excellent nuclear orientation facilities of his laboratory. Helpful discussions with Professor A. M. Portis are gratefully acknowledged.

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FOOTNOTES AND REFERENCES

This work was performed under the auspices of the U.S. Atomic Energy Commission. A. M. Portis and R. H. Lindquist, in "Magnetism," Ed. by G. T. Rado and H. Suhl, (Academic Press, Inc., New York, 1965), Vol. II A, p. 357.

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FIGURE CAPTIONS

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Fig. 1. Warm up curves for 60 Co in Fe (alloy II) at $\theta = 0^{\circ}$. In the upper part the frequency was varied with time and the resonance effect appears at 165.8 MHz. The lower curve was taken with a fixed frequency off resonance but with the same rf amplitude as in the upper case.

Fig. 2. Resonance effect at 0° and 90° for two different alloys (I and II) of 60 Co in Fe.



Fig. 1



Fig. 2

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