Application of Spin-echo NMR to Gradient Magnetic Field Measurement

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Abstract

The possibility of magnetic field measurements of high-gradient fields using spin-echo NMR is demonstrated by spin-echo simulation. Detailed discussion on this possibility and preliminary experimental results on the observation of the spin-echo signal in a gradient field with $48G/cm$ at $2.3kG$ will be described.

1 Introduction

Nuclear magnetic resonance by a CW-rf field (CW-NMR) is widely used for precise measurements of magnetic field strength. The static magnetic field strength H_0 is measured by observing the NMR absorption signal at the resonance frequency $\omega = \gamma H_0$ caused by the resonant precession of nuclear spins, where γ is given by the Larmor frequency of the nuclear magnetic moment divided by H_0. For protons, $\gamma = 2\pi \times 42.58MHz/10kG$. However, it is well known that CW-NMR is not usable for gradient fields because of NMR saturation due to the narrow resonance linewidth (on the order of $\sim mG$). On the other hand, spin-echo NMR in a magnetic field with a small gradient field superposed is widely used for MRI (Magnetic Resonance Imaging) in medicine and biology. The MRI precedent seems to suggest some possibilities for the use of spin echo in a high-gradient field. We will discuss spin-echo NMR in a high-gradient field such as a quadrupole field in an accelerator based on simulation, and demonstrate the preliminary observation of the spin-echo signal in a gradient field.

2 Simulation of spin echo

In materials, the precession of the macroscopic nuclear magnetization $\boldsymbol{\mu} = \mu_x \boldsymbol{e}_x + \mu_y \boldsymbol{e}_y + \mu_z \boldsymbol{e}_z$ associated with nuclear spins in the magnetic field of $\boldsymbol{H} = \boldsymbol{H}_0 + \boldsymbol{H}_1(t)$ is described by the Bloch equation, where $(\boldsymbol{e}_x, \boldsymbol{e}_y, \boldsymbol{e}_z)$ is a set of unit vectors defining the laboratory coordinate (x, y, z). $\boldsymbol{H}_0 = H_0 \boldsymbol{e}_z$ is a strong static field along the z-direction and $\boldsymbol{H}_1(t) = 2H_1 \cos\omega t \cdot \boldsymbol{e}_x$ is a small rf field perpendicular to $\boldsymbol{H}_0$ with frequency ω. As is well known, in the rotating frame (x', y', z') which rotates around z-axis at the rf frequency, the Bloch equation is expressed as [1]:

$$\frac{d\mu_{x'}}{dt} = -\Delta\omega\mu_{y'} - \frac{\mu_{x'}}{T_2}, \quad (1)$$

$$\frac{d\mu_{y'}}{dt} = \Delta\omega\mu_{x'} + \omega_1\mu_{z'} - \frac{\mu_{y'}}{T_2}, \quad (2)$$

$$\frac{d\mu_{z'}}{dt} = -\omega_1\mu_{y'} - \frac{\mu_{z'} - \mu_0}{T_1}. \quad (3)$$

In the rotating frame, the rf field is expressed by a static field $H_1 \boldsymbol{e}_{x'}$. The Bloch equation represents the spin precession about the effective static field $\boldsymbol{H}_{eff} = H_1 \boldsymbol{e}_{x'} - (\Delta\omega/\gamma)\boldsymbol{e}_{z'}$, where $\Delta\omega = \omega - \gamma H_0$ is the difference between the rf field frequency and the precession frequency γH_0 of the nuclear spin about the static field $\boldsymbol{H}_0$. $\omega_1 = \gamma H_1$ $(\omega_1 << \omega)$ is the precession frequency about $H_1 \boldsymbol{e}_{x'}$ in the rotating frame μ_0 is the thermal equilibrium nuclear magnetization along $\boldsymbol{H}_0$. T_1 is the relaxation time of μ_z, and T_2 the relaxation time of μ_x and μ_y. Hereafter we consider the case of $T_1 = T_2$ which corresponds to NMR in a liquid like water doped with paramagnetic ions. If the rf frequency is adjusted to γH_0, i.e. at resonance, $\boldsymbol{\mu}$ precesses slowly about the x'-axis. Therefore, applying the rf field during $\Delta t = \pi/\gamma H_1 \equiv \Delta t_{180}$, the so-called "180° pulse", at the resonance, the direction of $\boldsymbol{\mu}$ with initial direction along $\boldsymbol{H}_0$ is reversed. In the case of $\Delta t = \Delta t_{180}/2 \equiv \Delta t_{90}$, the rf pulse is called the "90° pulse", where the direction of $\boldsymbol{\mu}$ changes to the $-y'$-direction. Dividing the NMR sample into small segments labeled by j, the total nuclear magnetization $\boldsymbol{M}$ is given by $\boldsymbol{M} = \Sigma_j \boldsymbol{\mu}_j$, where $\boldsymbol{\mu}_j$ is the nuclear magnetization of the j-th segment.

In a non-uniform magnetic field, as is well known, the spin-echo signal is created by manipulating the 90° pulse and the 180° pulse. The 90° pulse is applied to the thermal equilibrium nuclear spin system where all spins are in the direction of $\boldsymbol{H}_0$ to change their directions in the perpendicular plane to $\boldsymbol{H}_0$. After turning off the 90° pulse, the spins precess about $\boldsymbol{H}_0$. Since the precession frequency about $\boldsymbol{H}_0$ has a spread of $\gamma\Delta H$ due to the effective spread of the field strength ΔH in the sample, the phase coherency of the spin precession vanishes and the transverse component M_T of $\boldsymbol{M}$ also vanishes after the coherency time $T_2' = 1/\gamma\Delta H$ of the precession phase. Here $T_2' < T_2$ is assumed. After applying the 180° pulse, the coherency of the precession phase recovers and M_T also recovers. This recovery of $\boldsymbol{M}$ is called the spin echo. Arranging a detection coil enclosing the NMR sample to detect M_T, the magnetic induction signal of $\boldsymbol{M}$ is observed across the coil. When the 180° pulse is applied sequentially, we have the sequential spin echoes as shown in figure 1 where the echo signal peak decreases with the relaxation time T_2. The Bloch equation in the rotating frame means that this concept of spin rotation is valid in the vicinity of the resonance limited by $|\Delta\omega| < \omega_1$. It should be noted that the spin echo is expected in any field distribution, including quadrupole fields, since the spin echo occurs in a localized area of the sample even for $\Delta H > \omega_1$.

In order to estimate the spin echo signal in a magnetic field with high gradient, we have to solve the Bloch equation numerically. So we reduce the differential equations to the difference equation to track the spin precession,

$$\mu'_{n+1} = \tilde{T}\mu'_n - \begin{pmatrix} \mu_{x'n}/T_2 \\ \mu_{y'n}/T_2 \\ (\mu_{z'n}-\mu_0)/T_1 \end{pmatrix}\Delta t, \qquad (4)$$

where $\mu'_n = (\mu_{x'n}, \mu_{y'n}, \mu_{z'n})$ and $\Delta t = t_{n+1} - t_n$. The matrix elements of $\tilde{T} = (T_{ij})$ are given by

$$T_{11} = \alpha^2 + (1-\alpha^2)\cos\omega'\Delta t,$$
$$T_{12} = -T_{21} = -\beta\sin\omega'\Delta t,$$
$$T_{13} = -T_{31} = -\alpha\beta(1-\cos\omega'\Delta t),$$
$$T_{22} = \cos\omega'\Delta t,$$
$$T_{23} = -T_{32} = -\alpha\sin\omega'\Delta t,$$
$$T_{33} = \beta^2 + (1-\beta^2)\cos\omega'\Delta t,$$

where $\alpha = \omega_1/\omega'$, $\beta = \Delta\omega/\omega'$ and $\omega' = \sqrt{\Delta\omega^2 + \omega_1^2}$. The 1st term of Eq. 4 represents the spin precession around the effective field H_{eff} in the rotating frame, and 2nd term expresses the relaxation.

For the spin echo simulation 1000 μ's are tracked by Eq. 4. Figure 1 shows an example of the simulation in a low gradient field, supposing $T_1 = T_2 = 20ms$, $H_1 = 5G$, and the maximum field deviation ΔH_0 in the NMR sample is $\pm 1G$. In this example all spins are expected to contribute to the spin echo because $\Delta H \approx \Delta H_0/2 < H_1$, and T_2' is expected to be $\sim 75\mu s$. These features are reproduced in figure 1.

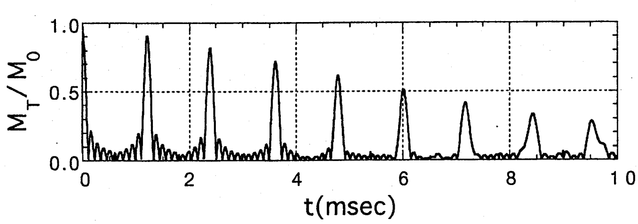

Fig. 1 Simulation result of M_T in non-uniform magnetic field with $\Delta H_0 = \pm 1G$.

3 Field measurement by spin echo

In order to investigate the possibility of gradient field measurements by spin echo, we will discuss the expected resolution based on simulations. We denote the field gradient by H' and the sample size along the direction of the field gradient by d. The spin echo occurs in the rf frequency interval of $\gamma H'd$, so we can not identify the field strength within the interval $H'd$. Therefore we have to limit the size of the NMR sample to within $d \sim \delta H/H'$ to restrict the field deviation in the sample to within the required resolution δH. For example, $d \sim 0.01mm$ is required for a resolution of $\delta H = 1G$ at a gradient of $H' = 1kG/cm$, which is the typical field gradient of quadrupole magnets used in accelerators. However, a sample size larger than $\sim 0.1mm$ is desired to overcome difficulties in sample making and to avoid degradation of the S/N ratio of the spin-echo signal. One of the methods to overcome this difficulty is the detection of the precession frequency using an NMR sample with size limited to $d \sim 2H_1/H'$. At this sample size, the frequency difference $\Delta\Omega = \omega - \omega_0$ between the 180°-pulse frequency ω and the precession frequency ω_0 of M depends on the magnetic field averaged over the NMR sample as shown in figure 2 and figure 3, where $T_1 = T_2 = 20ms$, $H_1 = 5G$ and $d = 0.1mm$ are assumed. Figure 2 shows the simulation result of M_T and the frequency difference $\Delta\Omega$ at an average field strength in the NMR sample of $\overline{H}_0 = \omega/\gamma$. Figure 3 shows the case where $\overline{H}_0 = \omega/\gamma + 1G$. From these results it is suggested that the average field strength $\overline{H}_0$ in the NMR sample can be identified by measuring the rf frequency at $\Delta\Omega = 0$. Here, it should be remarked that the observed $\Delta\Omega$ does not coincide with $\omega - \overline{H}_0/\gamma$ except where $\Delta\Omega = 0$.

As shown in figure 2 and figure 3, only the first 2-3 echo signals in the sequential echo train may be used for signal processing for the field measurement because we have remarkable decoherence between μ's and considerable degradation of the echo signal quality after the first 2-3 echos in this high-gradient field.

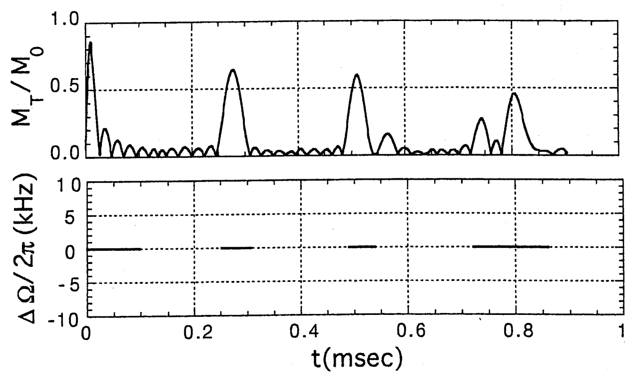

Fig. 2 Simulation result of M_T in the gradient field, where the field deviation in the NMR sample is $\pm 5G$ and $\overline{H}_0 = \omega/\gamma = 10kG$.

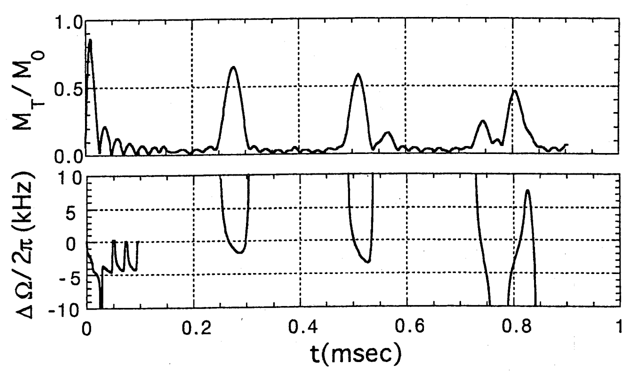

Fig. 3 Simulation result of M_T in the gradient field, where the field deviation in the NMR sample is $\pm 5G$ and $\overline{H}_0 = \omega/\gamma + 1G$.

4 S/N ratio of spin-echo signal

To increase the S/N ratio we assume that the detection coil with the inductance L is an inductive element of a resonant circuit with quality factor Q. And we assume $L \approx 0.7\mu H$, corresponding to the inductance of a coil with diameter $D = 10mm\phi$, length $l = 10mm$ and number of turns $N = 10$. The amplitude of the voltage induced in the detection coil by M is given by

$$v = \pi^2 \omega_0 M_T \eta Q N D^2 . \quad (5)$$

The frequency difference $\Delta\Omega$ can easily be detected using a synchronous detector such as a double-balanced mixer with the reference signal at the rf frequency ω. Supposing $M_T \approx \chi_0 H_0$, $\chi_0 = 3\times10^{-10}$, $H_0 = 10kG$, $\eta \approx 10^{-3}$, and $Q = 20$, a signal voltage of $v \approx 20\mu V$ is expected, where χ_0 is the static magnetic susceptibility of the nuclear spin system and η is the filling factor of the NMR sample in the coil volume.

In a high-gradient field, since the time duration of the spin-echo signal, i.e. the coherence time of the spin precession, is $\sim 2/\gamma H_1$ ($<< T_2$) for an NMR sample size of $d \sim 2H_1/H'$, a signal detection bandwidth of $\Delta f > \Delta f_s$ is required for the echo signal observation, where $\Delta f_s \approx \gamma H_1/4$ is the spectrum width of the echo signal. For example at $H_1 = 5G$, the pulse width of the echo signal is $\sim 30\mu s$ and a bandwidth of $\Delta f > 33kHz$ is required. At this bandwidth the thermal noise $v_n = \sqrt{4kTQ\omega_0 L\Delta f}$ gives a poor S/N ratio of $S/N < 15$ and the identification of $\Delta\Omega = 0$ becomes impossible. For the determination of $\Delta\Omega$ within the frequency error δf, we need a S/N ratio satisfying $S/N > (\Delta f_s/\delta f)^2/2$. This condition requires a signal-averaging technique with accumulating n echo signals where the S/N ratio is proportional to $\sqrt{n}$. Supposing a detection bandwidth of $\Delta f = 33kHz$, the number of signals accumulated is required to be $n > 1600$ at $H_0 = 10kG$ for an expected frequency error of $\delta f < 1kHz$ corresponding to a field strength error of $\delta H < 0.23G$. The above discussions indicate the possibility of high-gradient field measurement by spin-echo NMR with a resolution of $\sim 10^{-5}$. Finally it should be remarked that careful design of the NMR sample shape and the detection coil will be required to obtain the S/N ratio needed for the field measurement since the expected resolution depends on the sample size d along the direction of the gradient and the filling factor η.

5 Preliminary observation of spin-echo signals in a gradient field

As the first step of spin-echo observation in a high-gradient field, a preliminary observation has been done in a medium gradient field using a commercially available spin-echo detector (E-35/Echo Electric. Corp.). This detector can be used only at a fixed frequency of $10MHz$, which corresponds to a field strength of $2.35kG$ for proton NMR. The magnetic field with gradient was produced by a spare of the small steering magnets for the KEKB accelerator with $0.5mm$ thick iron plates attached on the pole face. Figure 4 shows an example of the field profile measured by a hole probe. The proton spin-echo signal in water doped with $MnCl_2$ in the gradient field is shown in figure 5, where 32 oscilloscope sweeps were accumulated. Though we have considerable leakage of the 180° pulse due to some mistuning of the detector, the spin-echo signal is clearly observed in the field with a gradient of $H' = 48G/cm$. In this experiment the rf field amplitude of the 180° pulse is estimated to be $H_1 = 4G$ from the pulse width of $30\mu sec$ required to flip spins. Taking account of g, H_1 and the sample size ($\sim 3mm\phi \times 5mm$), it is expected that about 50% of spins in the NMR sample contributed to the spin echo. From a view point of sample making, a thin rubber sample will be a candidate for the NMR sample for gradient field measurement. Although, at very preliminary observation, we observed the proton echo signal in a small rubber sample in a uniform field, we need systematic investigation with the rubber sample.

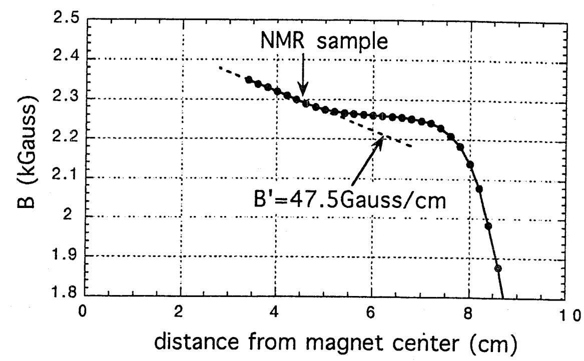

Fig. 4 Magnetic field profile of the test magnet.

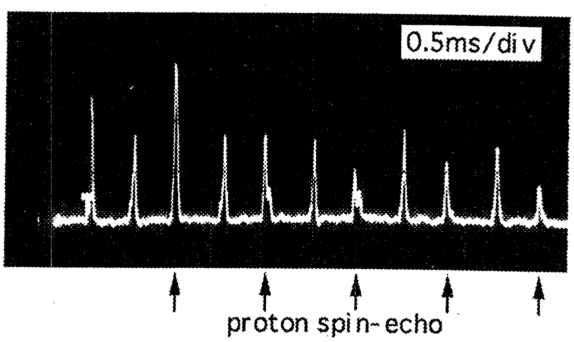

Fig. 5 The proton spin-echo signal in the gradient field with $H' = 48G/cm$ at $2.3kG$.

6 Conclusions

The simulation suggests the possibility of field measurement using spin-echo NMR in a high-gradient field such as the quadrupole field of an accelerator magnet. Limiting the NMR sample size to $d \sim 0.1mm$ and using the signal averaging technique, we can expect a resolution of $\sim 10^{-5}$ for the gradient of $H' \sim 1kG/cm$ at $\sim 10kG$. In the preliminary experiment we observe a clear spin-echo signal of protons in the gradient field with $H' = 48G/cm$ at $2.3kG$. The development of a spin-echo detector with frequency sweep is underway.

References

[1] A. Abragam, "The Principle of Nuclear Magnetism", Oxford, 1961.