RAPID COMMUNICATION

Study on signal intensity of low field nuclear magnetic resonance via an indirect coupling measurement*

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We carry out an ultra-low-field nuclear magnetic resonance (NMR) experiment based on high-T_c superconducting quantum interference devices (SQUIDs). The measurement field is in a micro-tesla range (∼10 μT–100 μT) and the experiment is conducted in a home-made magnetically-shielded-room (MSR). The measurements are performed by the indirect coupling method in which the signal of nuclei precession is indirectly coupled to the SQUID through a tuned copper coil transformer. In such an arrangement, the interferences of applied measurement and polarization field to the SQUID sensor are avoided and the performance of the SQUID is not destroyed. In order to compare the detection sensitivity obtained by using the SQUID with that achieved using a conventional low-noise-amplifier, we perform the measurements using a commercial room temperature amplifier. The results show that in a wide frequency range (∼1 kHz–10 kHz) the measurements with the SQUID sensor exhibit a higher signal-to-noise ratio. Further, we discuss the dependence of NMR peak magnitude on measurement frequency. We attribute the reduction of the peak magnitude at high frequency to the increased field inhomogeneity as the measurement field increases. This is verified by compensating the field gradient using three sets of gradient coils.

Keywords: indirect coupling measurement, superconducting quantum interference devices, low field nuclear magnetic resonance

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1. Introduction

Superconducting quantum interference devices (SQUIDs) possess high magnetic sensitivities and unique frequency characteristics, and they have attracted lots of attention since the 1960s. They can detect extremely weak magnetic signals or electrical signals that can be transferred into magnetic ones. As one of the most important approaches to molecular structure and tissue imaging of matters, the technique of nuclear magnetic resonance and imaging (NMR/MRI) has been commonly used in medicine,[1] biology,[2–5] chemistry,[6] and industry. Because the quality of NMR/MRI such as the resolution and signal-to-noise ratio (SNR), is improved as the strength of the magnetic field increases, high-field MRI systems are commonly used in many fields. But these systems are very expensive and complex, thereby restricting their applications. To overcome these difficulties, the technique of a low-field MRI is proposed (low-field usually refers to the field below 0.5 T). In the last decade, NMR/MRI using the SQUID has been investigated by many groups,[7,8] owing to the high magnetic sensitivity and wide frequency response range (10 kHz∼ 100 kHz for a direct current SQUID) of the SQUID. Recently, the SQUID has been used in the ultra-low-field (ULF) NMR/MRI system (μT to mT).[9–11] It can reduce the cost and complication associated with high magnetic fields. The relative homogeneity of the measurement field in the ULF MRI system is not important compared with in the high-field system. Moreover, we can obtain a narrow spectral line width[12] and pure J-coupling spectrum[13] by this simplified installation. However, the main disadvantage of the ULF NMR method is the very low signal intensity and SNR. It is desirable to make an effort to improve the signal pickup circuit and signal processing in order to obtain an NMR spectrum with high resolution and high SNR in MRI.

In this paper, we introduce the indirect coupling method in the ULF NMR system based on a high-T_c SQUID. This method overcomes two disadvantages of the direct coupling system used previously: one is that the signal is very weak caused by the large distance between the SQUID sensor and the sample; the other is the interference to the SQUID by the magnetic field coil. Our experimental results show that the SNR of the SQUID is much higher than that of a low noise amplifier SR560, and there is a maximum SNR at a specific

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measurement frequency. The reason is discussed.

2. Experiments

Figure 1 shows the layout of a coil system placed in a magnetic field shielding room.[14] The measuring field B_0 is a uniform magnetic field in the z-axis direction, supplied by a pair of Helmholtz coils each with a diameter of 0.6 m. A polarization field B_p ($B_p \gg B_0$) is produced by a solenoid with 2800 turns, which is oriented along the y axis. The pulse coil B_1 is a pair of Helmholtz coils of 0.14-m radius and its axis is along the x axis. The gradient fields are generated by three gradient coils G_x, G_y, and G_z. The direction of gradient field G_z is the same as that of measuring field B_0, whose gradient coil is a pair of Maxwell coils 0.6-m in diameter. While gradient fields of the other two directions (G_x and G_y) arise from two sets of biplane coil pair perpendicular to z-axis direction. The pickup coil, the input coil, the coupling loop, and an SQUID constitute the signal detection system. The pickup coil, a solenoid with 870 turns, is placed around the sample in the same direction as the polarization coil. The input coil and the SQUID are located inside a shielding barrel, which is placed at the corner of the shielding room, far from the magnetic field coils. The power supply of the pulse coil is a signal generator WF1974, while the other coils are supplied by direct current supplies. The controlling system and power supplies of all the coils are placed outside the shielding room.

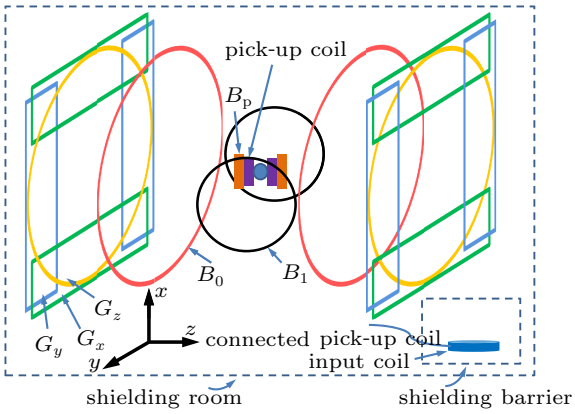


Fig. 1. (color online) Schematic plot of the coil system in an indirect coupling measurement.

Figure 2 shows the signal transport system. The SQUID is separated from the sample which is placed in the center of the polarization coil. To transfer the signal between the SQUID and sample, we employ a resonant circuit composed of a pickup coil with inductance L_1, an input coil with inductance L_2 , a resistor R_2, and a capacitor C. Two relays are used to separate the SQUID from the external coil system before the current in the B_p coil reaches zero in order to protect the SQUID from the current shock. The mutual inductance between the pickup coil and the polarization coil is very large, and thus the rapid decay of the polarization field will produce a large induced current in the pickup coil. The RL loop composed of the resistor R_2 and inductance L_2 can make sure that the induced current drops off to zero rapidly. Then the relays will be closed to connect the pickup coil and input coil, and we can utilize the SQUID to measure the NMR signal of the sample: the free induction decay (FID) signal.

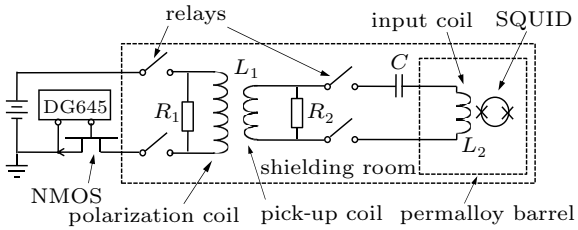


Fig. 2. Diagram of the polarization coil loop and the signal transport system.

3. Results and discussion

In order to study the possibility that the SQUID sensor is replaced by a low noise amplifier, we compare the SNR of pure water by SR560 and by the SQUID. The experimental set-up is shown in Figs. 3(a) and 3(b). Due to the different frequency response characteristics, we measure the SNR at different frequencies. Fixing the capacitance of coupling loop, adjusting the measuring field B_0, and changing the NMR precession frequency of the sample, we obtain a signal-frequency response curve (see the inset in Fig. 3(c)). The frequency corresponding to the signal peak value is exactly the resonant frequency of the coupling loop. Thus, the SNR can be gained through dividing the maximal signal by the average noise voltage. Varying the capacitance of the coupling loop, we obtain a series of datum points in Fig. 3(c). Since the coupling loop in Fig. 3(a) comprises an inductance of the input coil, the maximal SNR from SR560 cannot be coincident with that from the SQUID. Figure 3(c) shows clearly that the maximal SNR of the SQUID is higher than that of SR560 throughout.

However, after the resonant frequency exceeds 3000 Hz, the maximal SNR from the SQUID drops off significantly. This phenomenon cannot fit the theory of traditional NMR, so it needs further investigating. At first, we study the relationship between the noise of system without sample and resonant frequency of the coupling loop. It is found that though the resonant frequency varies with the capacitance of the coupling loop, there is no obvious change in the noise level of the SQUID. Then we consider the case with a sample. Figure 4 shows the curve of signal versus resonant frequency. It is obvious that when resonant frequency is lower than 2860 Hz, the signal intensity increases considerably as frequency increases. However, the signal intensity decreases when the resonant frequency is higher than 2860 Hz. The peak shows that there is an

optimal measurement frequency, which is a key to improving the low field NMR system.

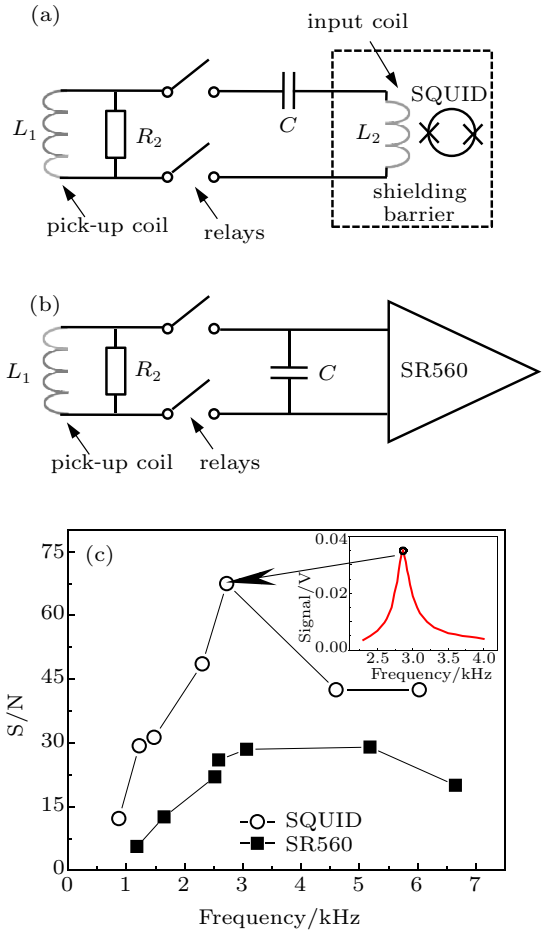


Fig. 3. Schematic diagram of a signal detection system based on (a) SQUID, (b) SR560; (c) dependence of the SNR on the resonant frequency; The inset shows the relationship between signal and measurement frequency.

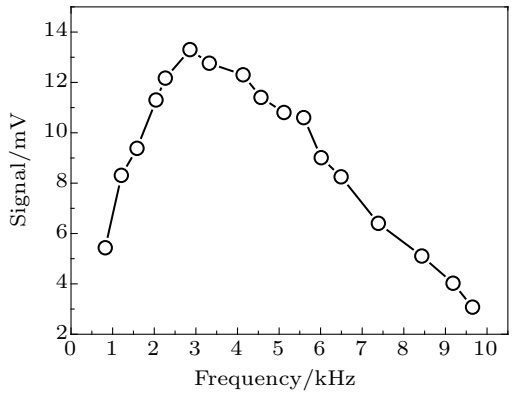


Fig. 4. Dependence of signal intensity on resonant frequency.

In the indirect coupling measurement system, the magnetic signal produced by the SQUID is actually related to the induction current, which is proportional to the induction electromotive force U of the pickup coil. According to Faraday's law of electromagnetic induction, U depends on the changing speed of the magnetic flux Φ through the coil and it is expressed as

$$U = -n\frac{\mathrm{d}\Phi}{\mathrm{d}t}, \tag{1}$$

where n is the number of turns of the coil. The magnetic flux Φ is in proportion to the y component of nuclear magnetic moment M_n of the sample and is given by,

$$\Phi \propto M_{\mathrm{n}y} = M_\mathrm{p}\,\mathrm{e}^{-t/T_1}\,\mathrm{e}^{-t/T_2}\cos(\omega t), \tag{2}$$

where $M_{\mathrm{n}y}$ is the component of M_n along the direction of y axis, M_p is the polarization nuclear magnetic moment caused by the polarization field B_p, T_1 denotes the relaxation time of M_n from the direction perpendicular to B_0 to the direction of B_0, T_2 represents the relaxation time of M_n in the x–y plane, ω is M_n rotational angular velocity along the z direction in the measuring field B_0, i.e., the NMR resonant angular velocity. Inserting formula (2) into Eq. (1), we obtain

$$U \propto nM_\mathrm{p}\omega. \tag{3}$$

Hence the signal intensity is dependent on the number of turns of input coil n, resonant angular velocity ω, and polarization nuclear magnetic moment M_p. This formula can easily explain the left part of the peak in Fig. 4. In order to understand the decrease of the right part of the curve in Fig. 4, the decay of the nuclear magnetic moment should be taken into account. It can be seen from formula (2) that without the polarization field B_p, the signal intensity decays exponentially, which is called the FID signal. After the induced current of the pickup coil caused by the shutdown of the polarizing coil decays to zero, we can measure the FID signal.[15,16] In general, the relaxation time $T_1 \gg T_2$, so we can only consider the effect of T_2. It is known that increasing the gradient field ΔB will reduce the relaxation time T_2 as shown below

$$T_2 \propto \frac{1}{\Delta B} \propto \frac{1}{B_0} \propto \frac{1}{\omega}. \tag{4}$$

Inserting formula (4) into formula (2), we have

$$U \propto nM_\mathrm{p}\,\mathrm{e}^{-\Delta t/T_2}\omega \propto nM_\mathrm{p}\,\mathrm{e}^{-\alpha\omega\Delta t}\omega, \tag{5}$$

where Δt (≈ 0.1 s) is the interval between the shutdown of current in the polarization coil and the moment when the SQUID starts to work, α is a coefficient and should be determined experimentally. Formula (5) shows that we can have maximal signal intensity only at the optimal measurement frequency.

In order to verify the above theory, we measured the FID signals with and without the gradient G_z of the external magnetic field respectively. As shown in Fig. 5, the interval between the maximal signal and the noise level is exactly the relaxation time T_2. When G_z exists, T_2 decreases and so does the signal intensity. It is clear that the magnetic gradient may speed up the decay of signal intensity.

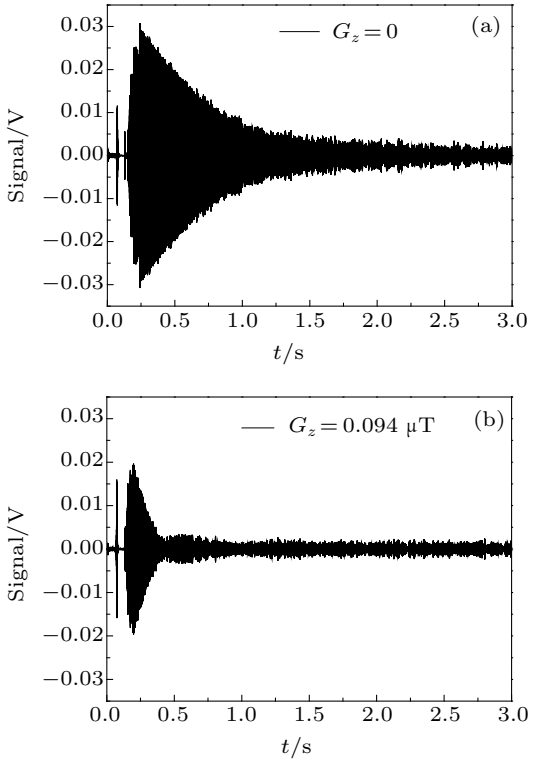


Fig. 5. FID signals with different gradients at $G_z = 0$ (a) and 0.094 μT/m (b).

4. Conclusions

In this paper we measured the FID signal and SNR of pure water using a home-built ULF NMR/MRI system based on a high-T_c SQUID. The indirect coupling method is used to improve the SNR and to protect the SQUID from being interfered by the measuring magnetic field. We compare the sensitivity of the SQUID and that of a low noise amplifier. The result shows that the SNR of SQUID is much higher than that of the low noise amplifier SR560. In addition, there is an optimal measuring frequency in the indirect coupling system. We attribute the reduction of the SNR at high frequency to the inhomogeneity of the measurement magnetic field. Our theory shows that the signal intensity can be improved by strengthening the polarization field B_p or by improving the homogeneity of the measuring field B_0. Moreover, gradient compensation will be a good method to improve the quality of the ULF NMR system.

References

[1] Liao S H, Huang K W, Yang H C, Yen C T, Chen M J, Chen H H, Horng H E and Yang S Y 2010 *Appl. Phys. Lett.* **97** 263701
[2] Volegov P L, Mosher J C, Espy M A and Kraus R H Jr 2005 *J. Magn. Reson.* **175** 103
[3] Volegov P L, Matlachov A N, Espy M A, George J S and Kraus R H Jr 2004 *Magn. Reson. Med.* **52** 467
[4] Matlachov A N, Volegov P L, Espy M A, Stolz R, Fritzsch L, Zakosarenko V, Meyer H G and Kraus R H Jr 2005 *IEEE Trans. Appl. Supercond.* **15** 676
[5] Zotev V S, Matlashov A N, Volegov P L, Urbaitis A V, Espy M A and Kraus R H Jr 2007 *Supercond. Sci. Technol.* **20** s367
[6] Espy M, Flynn M, Gomez J, Hanson C, Kraus R, Magnelind P, Maskaly K, Matlashov A, Newman S, Owens T, Peters M, Sandin H, Savukov I, Schultz L, Urbaitis A, Volegov P and Zotev V 2010 *Supercond. Sci. Technol.* **23** 034023
[7] Greenberg Y S 1998 *Rev. Mod. Phys.* **70** 175
[8] Kumar S, Avrin W F and Whitecotton B R 1996 *IEEE Trans. Magn.* **32** 5261
[9] Möβle M, Myers W R, Lee S K, Kelso N, Hatridge M, Pines A and Clarke J 2005 *IEEE Trans. Appl. Supercond.* **15** 757
[10] Lee S K, Möβle M, Myers W R, Kelso N, Trabesinger A H, Pines A and Clarke J 2005 *Magn. Reson. Med.* **53** 9
[11] McDermott R, Lee S K, Haken B, Trabesinger A H, Pines A and Clarke J 2004 *PNAS* **101** 7857
[12] Clarke J, Hatridge M and Möβle M 2007 *Ann. Rev. Biomed. Eng.* **9** 389
[13] Bernarding J 2006 *J. Am. Chem. Soc.* **128** 714
[14] Li S, Ren Y F, Wang N, Tian Y, Chen Y F, Li J and Zheng D N 2009 *Acta Phys. Sin.* **58** 5744 (in Chinese)
[15] Liao S H, Yang H C, Horng H E, Yang S Y, Chen H H, Hwang D W and Hwang L P 2009 *Supercond. Sci. Technol.* **22** 045008
[16] Qiu L, Zhang Y, Krause H J, Braginski A I and Offenhäusser A 2009 *J. Magn. Reson.* **196** 101