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Comparison of different detectors in low field NMR measurements

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Abstract. Low-field (LF) liquid sample NMR measurements using different detectors are performed at Larmor frequencies \( f_L \) ranging from 300 Hz to 35 kHz. Five different sensors cooled with liquid nitrogen are compared: a wire-wound coil, a printed planar copper coil, both resonant with capacitance \( C \) and read out by a room-temperature amplifier, a high critical temperature \( T_c \) thin film rf (bare) SQUID and two tuned SQUID circuits, one with a wire-wound input coil and another with a high-\( T_c \) superconducting tape coil. The resonant frequency of each \( LC \) circuit is adjusted to \( f_L \). The sensitivity of any \( LC \) circuit is determined by its inductance, quality factor, and \( f_L \). The magnetic field resolution of the sensors was evaluated with a homogenous magnetic field source at different frequencies and with a small source at different positions. We discuss the signal-to-noise ratio of the LF NMR signals recorded by these detectors and find the high-\( T_c \) tape coil to be unsuitable for LF NMR measurement. The homogeneity of the magnetic measurement field is deteriorated by this bulk superconducting object. That effect leads to a dramatic reduction of the decay time of the free induction decay (FID) signal. In contrast, the bare SQUID oriented parallel to the measurement field does not influence the FID significantly.

1. Introduction

Although high-field nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI) have achieved remarkable success, the low- and Earth-field NMR has been used as well since early days [1]. Several detecting sensors have been used, depending upon special demands of different applications. In the case of geophysical research, for example, the simple and convenient Faraday coil is the most common choice [2]. However, in some cases, such as the J-coupling studies at ultralow fields [3], noninvasive prostate cancer detection [4] and for simultaneous magnetoencephalography (MEG) and MRI measurements [5], detection with superconducting quantum interference device (SQUID) is an attractive alternative. Other sensors, such as atomic magnetometers, for example, are still at an early stage of development, in spite of their extreme sensitivity claimed by authors [6].

Qiu \textit{et al.} evaluated a resonant input \( LC \) circuit to improve the sensitivity of a high-\( T_c \) SQUID [7] used as a low-field NMR detector. Two tuned SQUID circuits with different coils were tested in that
paper: one tuned SQUID circuit (TSC1) included a copper coil (of inductance \( L \)) cooled by liquid nitrogen (LN\(_2\)) and inductively coupled to the SQUID. In another tuned SQUID circuit (TSC2), the copper coil was replaced by a high-\( T_c \) tape coil of lower \( L \) \([8]\). Each of these input circuits resonated with capacitance \( C \) of an exchangeable capacitor. Several capacitances were chosen to cover the frequency range of interest.

In this work, the performance of different sensors in low-field NMR measurements has been compared. A wire-wound copper coil (WCC), and a printed planar copper coil (PCC) were both LN\(_2\)-cooled, resonated with a capacitance \( C \), and were directly coupled to a room-temperature (RT) amplifier readout as shown schematically in figure 1 (a). A bare high-\( T_c \) thin film rf SQUID magnetometer, and the two tuned SQUID circuits (TSC1 and TSC2) have been also used as signal detectors. The inductive coupling to the SQUID is shown in figure 1 (b). The sensitivities of all these sensors have been quantitatively evaluated in either a homogeneous field or a small-source field, since in many practical cases, the NMR and MRI signal can be approximated by the field generated by a small source. The spatial sensitivity distribution is of great importance for designing a low-field MRI system, because it determines the number of sensors required to cover a given sample volume.

![Figure 1. Schematics of compared sensors: (a) the resonant Faraday coil connected to a room-temperature amplifier, and (b) a similar LC circuit inductively coupled to the high-\( T_c \) SQUID via mutual inductance \( M \).](image)

The quality factor \( Q \) and the sensitivity of the resonant-type coupling circuit decreases with decreasing the resonant frequency. For Larmor frequencies below 5 kHz, the performances of the bare SQUID and of TSC1 are compared in \([9]\). In this work, we choose the Larmor frequency \( (f_L) \) of 9.3 kHz for our NMR measurements, because at that frequency the sensitivities of the five sensors are comparable. This frequency is also within the \( f_L \) range of SQUID-based low field NMR and MRI \([10]\).

2. Setup
The NMR experiments were performed in a magnetically shielded room. Our setup is schematically shown in figure 2 (a). A 20 mL sample of water was surrounded by a solenoid located beneath a fiberglass cryostat, at the center of a Helmholtz coil pair that determined \( f_L \). The solenoid produced the prepolarizing field \( B_p \) of about 10 mT perpendicular to the measurement field \( B_m \). In the described NMR experiments, the sample was polarized for 6 seconds.
All the sensors evaluated by us are listed in Table 1. The parameters of the coils and of the SQUID magnetometer are shown in Table 2. The printed planar coil contains six layers of identical spiral coils. Figure 2 (b) shows a photograph of one such coil. Its outer diameter is 45 mm. The linewidth and the spacing of the windings are 155 µm each.

**Figure 2.** (a) Schematic diagram of measurement setup. (b) Photo of single layer printed planar coil.

| Sensors | Abbreviation |
|---------|--------------|
| Wire-wound resonant copper coil + RT amplifier | WCC |
| Printed (planar) resonant copper coil + RT ampl. | PCC |
| Bare high-$T_c$ rf SQUID | SQUID |
| TSC 1: WCC (w/o amplifier) + high-$T_c$ rf SQUID | TSC1 |
| TSC 2: High-$T_c$ tape coil + high-$T_c$ rf SQUID | TSC2 |

**Table 2.** Parameters of the coils and rf SQUID with a washer area of 10 x 10 mm².

| Coils          | Number of turns | Inductance (mH) | Resistance (Ω @ 77 K) | $f_i^a$ (kHz) | Mean diameter (mm) | Height (mm) |
|----------------|-----------------|-----------------|-----------------------|----------------|---------------------|-------------|
| Copper wire    | 1600            | 115             | 25                    | 27.8           | 40                  | 11          |
| HTS tape       | 49              | 0.09            | 0                     | $>100^b$       | 36.7                | 4           |
| Printed coil   | $49 \times 6$   | 2.5             | 10.6                  | $>100^b$       | 30                  | 7           |

$a f_i$ is the intrinsic resonance frequency which is determined by the inductance and the parasitic capacitance of the coil.

$b$ The exact value is not obtained because of the bandwidth limitation of the amplifier and the dynamic signal analyzer.

| Loop $(\mu m^2)$ | Inductance (pH) | $B-\Phi$ coefficient $(nT/\Phi_0)$ | White flux noise $(\mu T/\Phi)$ | Field sensitivity $(fT/\sqrt{Hz})$ |
|------------------|-----------------|----------------------------------|--------------------------------|----------------------------------|
| $150 \times 150$ | 225             | 1.85                             | 20                             | 40–50                            |

Each sensor coil and the exchangeable capacitor were positioned at the bottom of the cryostat and cooled with LN$_2$. The rf SQUID was positioned within the coil to ensure inductive coupling. The
readout amplifier (not shown in figure 2 (a)) was held outside of the cryostat, at room temperature. The distance between the bottom of the sensor (coil) and the sample centre was about 2.7 cm. The resonance frequency $f_0$ of each $LC$ circuit was tuned to $f_0$ by changing the value of $C$. The outputs of the Faraday coils were read out by a room temperature amplifier using the operational amplifier OPA627AU as a preamplifier, while the substrate resonator of the rf SQUID was connected to an inductively coupled flux-locked-loop readout electronics [11]. The signals were recorded with a HP 3562A dynamic signal analyzer.

3. Results

3.1 Sensitivity Comparison under Homogeneous Field

The noise of tuned and untuned SQUID detectors and of (tuned) Faraday coils was theoretically calculated in ref. [12]. However, these calculations are only suitable for low-$T_c$ coupled SQUIDs with superconducting pickup and input coils. For a high-$T_c$ SQUID coupling with the surrounding copper coil the noise analysis is more complicated. Consequently, in this study the magnetic field resolutions of our five different sensors are experimentally calibrated using a known homogeneous field. This field is produced by a coil surrounding the outside of the dewar finger. Figure 3 compares the measured resolutions in the frequency range from 300 Hz to 35 kHz.

Below 1 kHz, the bare SQUID is superior to all other sensors because of its frequency-independent sensitivity of $40 - 50 \text{ fT/}\sqrt{\text{Hz}}$. The sensitivity of any $LC$ circuit is related to its impedance $Z = QL\omega_0$, where $\omega_0$ is the angular frequency at resonance. The $Q$ value of an $LC$ circuit increases with increasing frequency, so the sensitivity of the SQUID coupled to WCC resonant coil exceeds that of the bare SQUID above 1 kHz and reaches about $6 \text{ fT/}\sqrt{\text{Hz}}$ at 15 kHz, limited by the Nyquist noise of the Faraday coil at 77 K [12]:

$$S_{B}^{1/2} = \sqrt{4k_B TR / \omega_0 N_W A_c}.$$  

Here, $S_{B}^{1/2}$ is the magnetic field noise produced by a coil with the number of turns $N_W$ and resistance $R$ at temperature $T$; $k_B$ is the Boltzmann constant, and $A_c$ the coil area.

The highest sensitivity between 1 kHz and 25 kHz (about $6 \text{ fT/}\sqrt{\text{Hz}}$) is attained by TSC1, consisting of the SQUID and wire-wound resonant coil. While the coil of TSC2 has lower intrinsic noise, a resolution of $20 \text{ fT/}\sqrt{\text{Hz}}$ is attained in this frequency range, because of the superconducting coil’s much lower number of turns and inductance.

Unlike the intrinsic resonance limitation of WCC (see Table 2a), the PCC with a rather small parasitic capacitance can be used at higher frequencies and reaches a sensitivity of about $4 \text{ fT/}\sqrt{\text{Hz}}$ above 50 kHz, which is also restricted by the Nyquist noise (not shown in figure 3).

![Figure 3. The magnetic field resolution of the five sensors under test; WCC, PCC, bare SQUID, TSC1 and TSC2 at different frequencies.](image-url)
3.2 Dependence of Sensitivity on Small Source Position

One of the development trends in LF NMR is towards LF MRI, the advantages of which are discussed in [10]. Examples are the \(T_1\)-contrast MRI (e.g., the possibility of prostate cancer detection), MEG-MRI combination detection, etc.. The basic principle of MRI is that by applying a gradient magnetic field, the sample image can be represented by a number of slices, whose Larmor frequencies reflect the positions of these slices. Usually one can assume that the magnetization of a sample slice in MRI is equivalent to a small source. However, the finite detection area of a sensor determines the maximal imaging region for a single imaging process. Therefore, the signal-to-noise-ratio (SNR), or the sensitivity of a sensor to a small source would be affected by the source position.

To verify the inference above, a small source is moved from the center to the edge of the dewar finger. A current with frequency of 9.3 kHz flows in a small solenoid which simulates the small source. This solenoid wound with copper wire (diameter \(\phi = 0.2\) mm) has an inner diameter of 2 mm and a length of 50 mm. The applied current generating the magnetic field of the solenoid is adjusted for each sensor, because of their different sensitivities. The central axis of the solenoid is aligned with the direction of the maximum sensitivity of our sensors \((B_s)\), shown in figure 2 (a).

Figure 4 (a) illustrated the relation between the normalized output amplitude and the position of the small source located beneath the dewar. It is clearly seen that the sensitivities of the sensors vary with the change of the source position. These curves can be fit by Gaussian functions (dashed lines). When the SQUID is inductively coupled with the wire-wound coil (TSC1), the larger dimension of the coil compensates the SQUID’s drawback of a small area, and it significantly increases the effective detecting area of the SQUID to the same level as the coil’s detecting area.

To study the relation between the signal loss and the size of the sensors, the normalized amplitude versus position divided by radius \(r\) of each sensor is given in figure 4 (b). Considering the WCC and TSC1, the sensitivities at the edge of the sensors (position 20 mm, radius 20 mm) reduce by about 50%, while the signal amplitudes of PCC (mean radius of about 15 mm) decreases to about 60% at its edge (position 15 mm). When the position of the point source exceeds the diameter of the sensor coil, most part of the signal will be lost. In particular, the signal is attenuated dramatically from \(r\) to \(2r\) for the Faraday-coil-based sensors (PCC, WCC, TSC1). However, for the bare SQUID with a field-to-flux transfer coefficient \(\frac{\partial B}{\partial \Phi}\) of 1.85 nT/\(\Phi_0\), corresponding to an effective radius of about 0.597 mm, the signal amplitude reduces much more gradually. This is because the signal amplitude of an ideal point source is mainly determined by the larger value of the sensor size and the sensor-to-sample distance. In the case of bare SQUID, the sensor size is much smaller than the distance between the sample and the source, so that the SQUID is less sensitive to the position change of the source, while in the case of coils the sensor size dominates the sensor-to-sample distance. This result might be meaningful for the sensors’ configuration in a multichannel LF MRI system [5].

![Figure 4](image.png)

**Figure 4.** (a) Normalized amplitude versus the small source position. The dashed lines are Gaussian fits to the experimental data. The point source is moved from the center to the edge of the cryostat. (b) Normalized amplitude versus the position to coil radius ratio. The same relation for the bare SQUID is shown in the inset.
3.3 FID Signal Comparison
The FID signal from the same sample of 20 mL tap water was recorded by the five detectors separately at $f_L$ of 9.3 kHz, as shown in figure 5. Because of the sudden switch-off of the strong polarizing field $B_p$, ringing is induced in the $LC$ circuit. This ringing may partly mask the FID signal and influence the NMR measurement. To effectively remove the ringing, we developed two Q switch circuits [9], one of which is applied in this experiment.

In figure 3, the sensitivities of the bare SQUID and of PCC are relatively similar, which leads to similar noise content in figure 5 (a) and (b). Traces of figure 5 (c) and (d) with higher signal to noise ratios (SNR) were acquired by the more sensitive WCC and TSC1. At $f_L = 9.3$ kHz, their field resolutions are about 15 fT/$\sqrt{\text{Hz}}$ and 7 fT/$\sqrt{\text{Hz}}$, respectively. Although the sensitivity of TSC2 is higher than that of the bare SQUID, the FID signal recorded by the high-$T_c$ tape coil is almost invisible and the signal is dominated by noise, as shown in figure 5 (e). This is because this coil represents a superconducting bulk in the proximity of the sample. The resulting magnetic field distortion in a certain volume causes the decline of the $B_m$ homogeneity. In contrast, the small thin film SQUID doesn’t influence the signal and the effective relaxation time $T_2^*$ is the same as that recorded by the copper coil.

![Figure 5. The FID signals recorded by the PCC (a), SQUID (b), WCC (c), TSC1 (d) and TSC2 (e).](image)

4. Conclusion
Different detectors which may be used in low field NMR were quantitatively compared in the frequency range from 300 Hz to 35 kHz. Below 1 kHz, the bare SQUID was superior to the other detectors, because of its frequency-independent characteristic. The combination of SQUID and wire-wound copper coil, TSC1, is the best choice between 2 kHz and 25 kHz because of its relatively high sensitivity and large detecting area. To further increase the SNR of the detection, a planar (thin-film) high-$T_c$ coil which would not affect the homogeneity of the measurement field might be also a feasible scheme.

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