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High-sensitivity cooled coil system for nuclear magnetic resonance in kHz range

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In several low-field Nuclear Magnetic Resonance (LF-NMR) and surface nuclear magnetic resonance applications, i.e., in the frequency range of kHz, high sensitivity magnetic field detectors are needed. Usually, low-T_c superconducting quantum interference devices (SQUIDs) with a high field sensitivity of about 1 fT/Hz$^{1/2}$ are employed as detectors. Considering the flux trapping and operational difficulties associated with low-T_c SQUIDs, we designed and fabricated liquid-nitrogen-cooled Cu coils for NMR detection in the kHz range. A cooled coil system consisting of a 9-cm diameter Cu coil and a low noise preamplifier was systematically investigated and reached a sensitivity of 2 fT/Hz$^{1/2}$ at 77 K, which is 3 times better compared to the sensitivity at 300 K. A Q-switch circuit as an essential element for damping the ringing effects of the pickup coil was developed to acquire free induction decay signals of a water sample with minimum loss of signal. Our studies demonstrate that cooled Cu coils, if designed properly, can provide a comparable sensitivity to low-T_c SQUIDs. *© 2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4901964]

I. INTRODUCTION

Several applications for biological or geophysical measurements require high-sensitivity magnetic field detectors in the frequency range of kHz. For example, nuclear magnetic resonance (NMR) at low measurement field B_m (LF) with a Larmor frequency f_L in the kHz range offers some advantages compared with high-field (HF) NMR.[1] At lower fields, the spin-lattice relaxation time T_1 is more material dependent than the high field cases, resulting in improved T_1-contrast imaging.[2] Furthermore, the high homogeneity of the Earth's magnetic field (~0.05 mT) increases the amplitude and duration of free induction decay (FID) signal, allowing for surface nuclear magnetic resonance (SNMR) detection of groundwater down to 100 m.[3]

An effective way to improve the signal-to-noise ratio (SNR) of LF-NMR measurements is to increase the sensitivity of pickup sensors, e.g., to utilize superconducting quantum interference devices (SQUIDs). Liquid-nitrogen-cooled (high-T_c) SQUIDs, with a field resolution of 30–50 fT/Hz$^{1/2}$ and liquid-helium-cooled (low-T_c) SQUIDs with a field resolution of about 1 fT/Hz$^{1/2}$ have been employed for various applications in LF-NMR.[4–7] Recently, low-T_c SQUIDs were also introduced as the detection sensor for a SNMR experiment.[8]

Although low-T_c SQUIDs yield the best field sensitivity, they are not commonly applied because of their susceptibility to flux trapping, and the need to be cooled down to liquid He temperature, as well as the operation difficulty. Therefore, more robust and easier-to-use magnetic field sensors with a comparable sensitivity are needed. In the kHz range, a *LC* resonance circuit consisting of a pickup coil (with inductance *L*) and a capacitor (*C*) is a good alternative. Matlashov *et al.* reported that a *LC* circuit with Cu coils at room temperature (300 K) reached a field resolution of 20 fT/Hz$^{1/2}$.[9] Other reports suggest that high-*Tc* SQUIDs, which coupled with cooled *LC* system (77 K), could obtain sensitivity less than 10 fT/Hz$^{1/2}$.[10,11] The above systems should be capable of performing NMR and magnetic resonance imaging (MRI).[12]

In the work presented here, we propose to use a cooled *LC* circuit with a Cu coil at 77 K as the only signal detector in the kHz range. Actually, this *LC* circuit and a preamplifier noise figure determine the system sensitivity. The coil with different turn numbers and different preamplifiers were investigated. The field sensitivities of 2 fT/Hz$^{1/2}$ at 77 K and 6 fT/Hz$^{1/2}$ at 300 K were demonstrated, when employing a flat coil with a diameter of 9 cm and preamplifier AD745 (Analog Devices Inc.).

II. OPTIMIZATION OF LIQUID-NITROGEN COOLED COIL SYSTEM

A. Noise sources

To obtain the lowest system noise, the thermal noise of the coil V_T and the preamplifier's input noise consisting of the voltage noise V_n and the current noise I_n should be considered. The system's voltage noise (V_{sys}) is then described by Eq. (1):

$$V_{sys} = \sqrt{V_n^2 + (I_n Z)^2 + (Q\sqrt{4k_B T R_s})^2}, \qquad (1)$$

where Q denotes the quality factor of the resonance circuit, $Z \approx Q\omega L$ the impedance at the Larmor frequency ω, k_B the Boltzmann's constant, T the coil temperature, and R_s the dc resistance of the coil. Q is an important parameter for the

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TABLE I. Parameters of the Cu pickup coils.

N	R_{300} (Ω)	R_{77} (Ω)	L (mH)	Q_{77} (2.07 kHz)	Q_{77} (6.07 kHz)
90	3.9	0.49	1.25	15.7	35.2
180	7.9	0.99	4.92	28.8	68.5
270	11.6	1.46	10.83	51.9	95.3
360	15.7	1.95	19.72	61.5	98.8
450	19.5	2.44	35.66	74.7	98.3
540	23.4	2.93	54.21	77.1	96.2
630	27.3	3.41	66.25	81.1	99.1

pickup coil's sensitivity. An effective method to improve the field sensitivity is to cool the pickup coil, thus reducing its resistance, i.e., enhancing $Q = \omega L/R_s$.

According to Faraday's law of induction, the transfer coefficient of the resonance circuit can be described as

$$\partial V/\partial B = QNS\omega, \tag{2}$$

where N denotes the number of turns of the coil and S the average detection area.

From Eqs. (1) and (2), we can obtain the system field sensitivity B_n and its power $B_n{}^2$, expressed by Eqs. (3) and (4), respectively,

$$B_n = \frac{V_{sys}}{(\partial V/\partial B)}, \tag{3}$$

$$B_n{}^2 = \frac{V_n{}^2}{(Q(T)NS\omega)^2} + \left(\frac{L}{NS}\right)^2 I_n{}^2 + \frac{4k_B T R_s(T)}{(NS\omega)^2}, \tag{4}$$

where $Q(T)$ and $R_s(T)$ denote the Q and R_s values at temperature T, respectively. Equation (4) contains three parts of noise contributions. Two of them, i.e., the first and third term, are temperature dependent because R_s reduces (i.e., Q increases) monotonically with decreasing T. Different preamplifiers with different V_n and I_n values lead to a minimum of the system noise at different N, when the coil diameter is fixed.

B. Calculation of sensitivity using different preamplifiers

At 3 kHz, the skin depth δ of copper is 0.42 mm and 1.18 mm at 77 K and 300 K, respectively. Therefore, an enameled copper wire with a wire diameter of 0.4 mm was selected to neglect skin depth effects. Uniformly wound and layered coils with an average diameter of 9 cm and a height of 1.8 cm were used. The number of coil layers was varied from 2 to 14, corresponding to a change of N from 90 to 630 (45 turns/layer). The coil parameters are shown in Table I.

Q values were measured at 3.07 kHz at 300 K and 77 K, for different numbers of layer. Q *vs.* N curves are shown in Figure 1. As expected, the Q value at 77 K is always higher than that at 300 K due to the reduced resistance of the coil (Table I). There are two ranges for the behavior of Q *vs.* N: (i) a linear range ($Q \approx \omega L/R_s$), in which Q increases linearly with increasing N, and (ii) a transition range ($Q < \omega L/R_s$), in which Q increases slowly with N and finally saturates due to the large parasitic capacitance of the coil at larger N. The maximum Q value at 77 K is 87, while it is about 35 at 300 K.

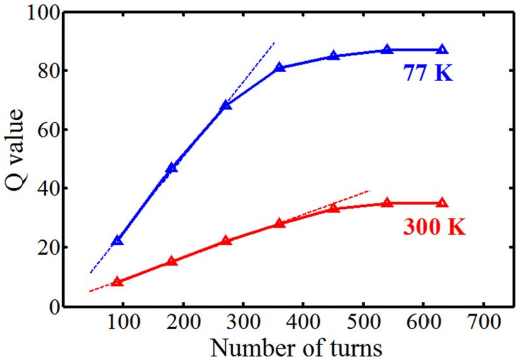

FIG. 1. Measured Q *vs.* number of turns of the pickup coil at 77 K and 300 K when $f_L = 3.07$ kHz.

To optimize the combination of coil parameters and preamplifiers, we investigated system noise contributions from different preamplifiers. We chose two low-noise operational amplifiers, i.e., AD797 (Analog Devices Inc.) and AD745. AD797 has a voltage noise $V_n = 0.9$ nV/Hz$^{1/2}$, which is the lowest among commercial operational amplifiers, and a relative higher current noise of $I_n = 2.0$ pA/Hz$^{1/2}$ at 2 kHz; in contrast, AD745 yields a higher $V_n = 2.9$ nV/Hz$^{1/2}$, and a very low $I_n = 6.9$ fA/Hz$^{1/2}$.

Figures 2(a) and 2(b) show the total noise power of the cooled coil system ($PB_n = B_n{}^2$) for AD797 and AD745 at 77 K, respectively, which consists of preamplifier voltage noise (PV_n), current noise (PI_n), and thermal noise (PT_n). Experimentally measured Q values (Figure 2) and Eq. (4) were used for the calculations. For AD797 (Figure 2(a)), a minimum of $PB_n = 33$ fT^2/Hz appears at $N = 90$, where $PI_n \approx PV_n$. When $N > 90$, PI_n increases rapidly, and dominates PB_n, while PV_n and PT_n decrease to become negligible. For AD745 (Figure 2(b)), the PI_n contribution is so small that it can be neglected. PB_n is initially dominated by PV_n, and then both by PV_n and PT_n, reducing with increasing N. At $N = 180$, $PV_n = PT_n$, and PB_n is twice PV_n. When $N > 180$, PT_n starts to dominate PB_n, indicating that the contribution of preamplifier noise can be neglected. The minimum value of PB_n reaches about 0.9 fT^2/Hz at $N = 630$, which is 40 times lower than that of AD797.

Figures 2(c) and 2(d) show the noise power of the pickup coil system at 300 K using the two preamplifiers mentioned above. The minimum value of PB_n reaches about 175.5 fT^2/Hz for AD797 at $N = 270$, and about 27.7 fT^2/Hz for AD745 at $N = 630$. The above results indicate that AD745 leads to a smaller system noise than AD797 both at 77 K and 300 K at optimum numbers of coil turns.

C. Coil sensitivity measurement using preamplifier AD745

Figure 2(e) shows a comparison of the calculated and measured system sensitivity B_n at 77 K and 300 K using the preamplifier AD745 at 3.07 kHz. The noise measurements were done inside a magnetically shielded room made of 2 layers of Mumetal and 1 layer of aluminum. The preamplifier's outputs were averaged using a dynamic signal analyzer (HP35670A). For the coils at 300 K, the measured B_n are

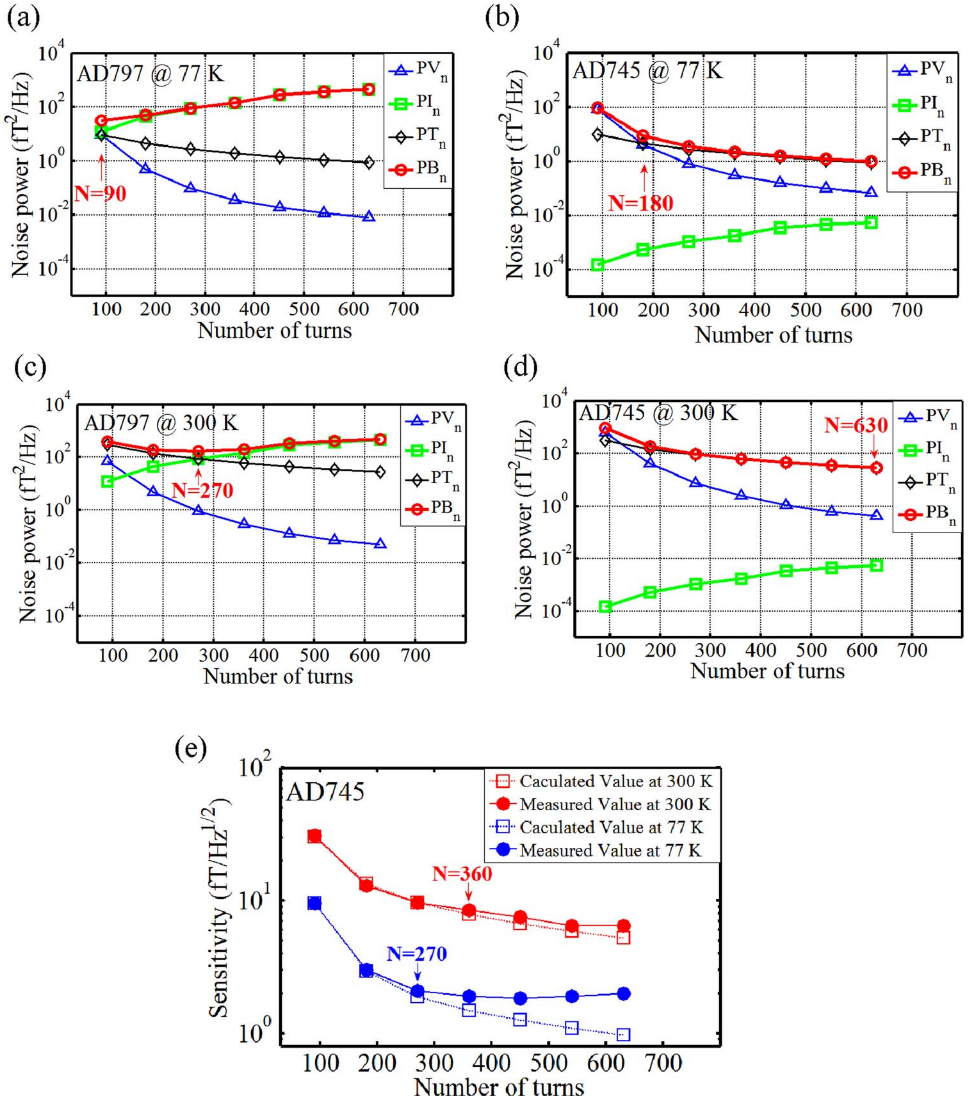

FIG. 2. Measured and calculated sensitivity of the pickup coil system at f_L = 3.07 kHz. Noise power $B_n{}^2$ calculated for preamplifier AD797 ((a) and (c)) and AD745 ((b) and (d)) at 77 K and 300 K, respectively. (e) Comparison of system sensitivities between calculated values and measured data at 77 K and 300 K with AD745.

consistent well with the calculated data, though there is a minor discrepancy for $N \geq 360$ at 300 K. At 77 K, a deviation between the measured and calculated sensitivities starts at $N = 270$ and increases with the increasing N. The discrepancy between calculated and measured values at large N seems to originate from several effects; the eddy current noise of the metallic coil,[13] the eddy current noise of the shielded room, and the residual environmental noise inside the shielded room. When the measurement position of the pickup coil was changed inside the shielded room, the total system noise changed appreciably. The practical minimum B_n obtained is about 2 fT/Hz$^{1/2}$ at 3.07 kHz and at 77 K, while it increases to 6 fT/Hz$^{1/2}$ at 300 K. Both of these sensitivities are comparable to the sensitivity of low-T_c SQUIDs, though the coil's pickup area is larger than that of the SQUIDs.

Based on the results of the comparison of AD797 and AD745 described above, we chose AD745 as the preamplifier in the frequency range from 2 to 6 kHz for measurements at 77 K. Larmor frequencies in this frequency range are typically used for both LF-NMR and SNMR. The measured Q values are shown in Table I. The sensitivities of the cooled coils are limited to 2 fT/Hz$^{1/2}$ for all the frequencies we tested, possibly due to the residual noises inside the shielded room (Figure 3). When f_L is increased, number of turns necessary to achieve 2 fT/Hz$^{1/2}$ sensitivity is reduced. The reduction of N is helpful to minimize the ringing effect of the pickup coil system as we will discuss in Sec. III. As a result, we choose $N = 450$ for 2.07 kHz, $N = 270$ for 3.07 kHz, and $N = 180$ for 6.07 kHz in the Q-switch experiments.

III. Q-SWITCH FOR SUPPRESSING RINGING EFFECTS

In all the experiments described here, the sample was polarized by B_p of 10 mT for 6 s. When using the LC circuit in NMR experiments with B_p, a transient ringing effect, however, is encountered just after B_p is switched off. Such ringing masks the FID signals and delays the starting time

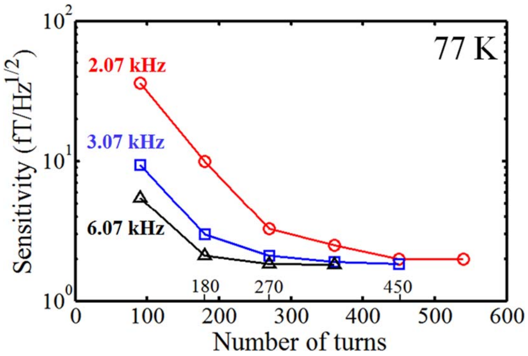

FIG. 3. Measured system sensitivities depending on the number of pickup coil turns for 3 different frequencies using AD745.

of data acquisition.[14,15] The measured ringing duration τ_r is determined by $\tau_r = Q/(\pi f_L)$.[16] To solve this problem, we employed the Q switch circuit. This circuit consisting of one FET (e.g., BF246B) connected parallel to the LC circuit serves as a time-variable damping resistance. The resistance between drain and source of FET, R_{DS}, is controlled by on the gate voltage, V_{GS}, of FET. During the prepolarizing time, R_{DS} is small to remain low Q of LC circuit. After B_p is switched off, R_{DS} should be gradually increased, in order to damp the ringing energy. Once the ringing is ended, it is started to record the FID signal. The principle of Q-switch was described in Dong *et al.*, 2009 in more details.[17] Also, the time constant τ_q for Q to recover to its maximum value should be adjusted to minimize τ_r and to start the data acquisition with minimum loss in the signals. As shown in Figure 4, τ_r was measured for different N at different frequencies. The black curves show the measured ringing signals without Q-switch. By introducing Q-switch, τ_q was successively reduced until the ringing was completely suppressed. At $f_L = 2.07$ kHz ($N = 450$), a ringing time $\tau_r = 50$ ms was observed (Figure 4(a), black curve). The ringing was completely suppressed by the Q-switch with a $\tau_q = 18$ ms, see red curves in Figure 4(a). Correspondingly, the ringing at 3.07 kHz which lasted $\tau_r = 22$ ms ($N = 270$) was suppressed by a Q-switch with $\tau_q = 10$ ms (Figure 4(b)). At 6.07 kHz, a ringing time of $\tau_r = 12$ ms was observed ($N = 180$), and suppressed by a Q-switch by $\tau_q = 3$ ms (Figure 4(c)). Thus, a lower f_L with a higher Q leads to a longer τ_r.

IV. MEASUREMENT SETUP AND EXPERIMENTAL RESULTS IN LF-NMR

To demonstrate the effectiveness of cooling and of the Q switch circuit, a simple LF-NMR experiment was carried out in the magnetically shielded room. The schematic configuration is shown in Figure 5. It mainly consists of five parts: (i) a Helmholtz coil pair for the generation of the measurement field $B_m = 72.22\ \mu$T, which determines $f_L = 3.075$ kHz; (ii) A two-layer solenoid (wire diameter 0.4 mm, inductance 4.98 mH and dc resistance 13.4 Ω at room temperature) surrounding the water sample was employed to generate B_p with 10 mT A^{-1}. A 20-ml tap water sample is located in the center of the Helmholtz coil pair; (iii) a cooled coil system for measuring FID signals. The pickup coil with a diameter of 9 cm is cooled to liquid nitrogen temperature (77 K) using a styrofoam container; (iv) a Q-switch circuit to damp the ringing effects of the pickup coil (described in Sec. III); (v) data sampling and sequence controller for B_p, Q-switch and signal acquisition using a software. To improve SNRs, the sequence of measurements can be repeated and the acquired FID signals are averaged, thus reducing both the white- and Gaussian-noise contributions. The sensing directions of the pickup coil, B_m and B_p are perpendicular to each other.

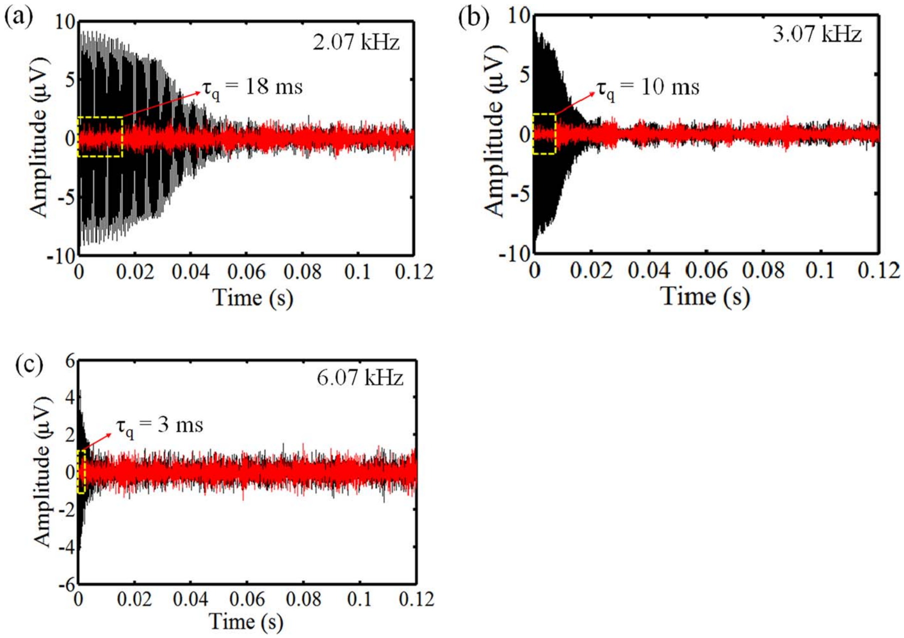

FIG. 4. Damping of ringing effects using the Q switch circuit from 2 to 6 kHz. (a) At $f_L = 2.07$ kHz, $\tau_r = 50$ ms, $\tau_q = 18$ ms; (b) At $f_L = 3.07$ kHz, $\tau_r = 22$ ms, $\tau_q = 10$ ms; (c) At $f_L = 6.07$ kHz, $\tau_r = 6$ ms, $\tau_q = 3$ ms. The coils are at 77 K. The yellow dotted lines outline the minimum time constant τ_q for each frequency.

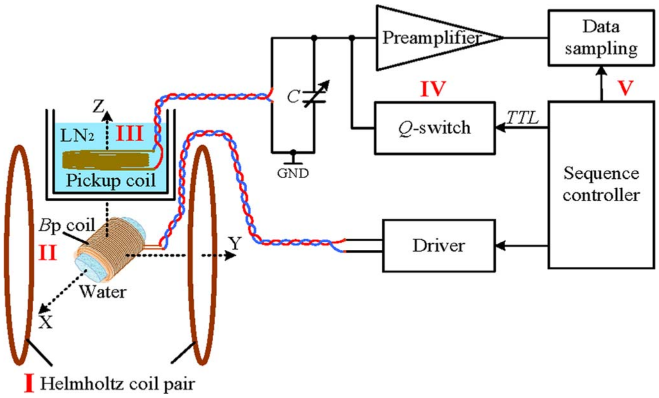

FIG. 5. Schematic diagram of the LF-NMR system using a cooled coil.

The optimized cooled coil with 270 turns ($Q = 70$) at 77 K as described in Sec. II, and room temperature coil with 540 turns ($Q = 35$) were used for signal detection. Thus, the transfer coefficients for both coils, i.e., amplitude of the FID signals, are nearly the same at a certain f_L according to Eq. (2). The bandwidths Δf of the LC resonant circuit are obtained to be 44 Hz at 77 K and 88 Hz at 300 K, respectively, where $\Delta f = f_L/Q$. In the case of $B_p = 10$ mT, FID signals from a 20-ml tap water sample at $f_L = 3.075$ kHz were recorded by the pickup coil systems at 77 K and 300 K. As shown in Figure 6, a clear FID signal with high SNR was observed in the time domain at 77 K. In contrast, the FID signal recorded with the 300 K coil is noisy. The observed improvement in SNR by a factor of 3.0 by cooling, agrees well with the three-fold enhancement of the coil's field sensitivity upon cooling.

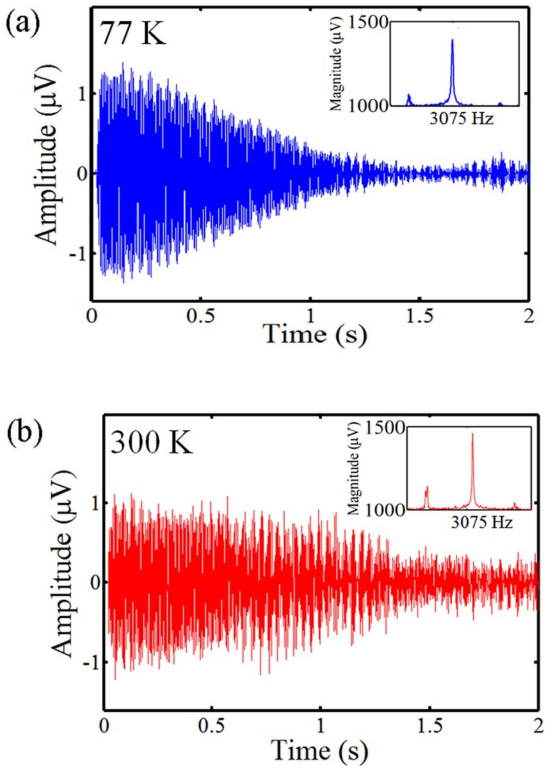

FIG. 6. Measured FID signals and its corresponding spectra (inset) when $B_p = 10$ mT at 77 K (a), and 300 K (b). All data were averaged 32 times at $f_L = 3075$ Hz.

V. CONCLUSION

By the optimization of the total noise power which consists of the thermal noise of the coil and the preamplifier's input noise, we obtained a low noise level of 2 fT/Hz$^{1/2}$ from the cooled copper coil system, which is comparable to low T_c SQUIDs. Considering that the optimum sensitivity of 2 fT/Hz$^{1/2}$ is limited by the environmental noise in the shielded room, a better sensitivity of the cooled coil system can be expected if the shielding effectiveness of the shielded room is improved. By using a Q-switch technique, we could remove the ringing effects in the resonance circuit within several ms. Measurements of FID signals obtained with a pickup coil optimized at 77 K show that the SNR is increased by 3.0 times upon cooling of the pickup coil.

Compared with high-sensitivity SQUIDs, our study enables a simpler and more economic NMR measurement for biological and geophysical applications with a comparable sensitivity in the kHz range. Apart from NMR detection, the cooled coil system can also be applied to MRI acquisitions. However, MRI may display intensity artifacts if the coil's 3 dB bandwidth Δf is less than the image acquisition bandwidth.[12] To solve this problem, additional resistance in parallel with the coil is necessary for damping Q, i.e., enhancing Δf, although sacrificing some sensitivity of the cooled coil system.

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