Ultra-Low Field SQUID-NMR using LN$_2$ Cooled Cu Polarizing Field coil

K Demachi, S Kawagoe, S Ariyoshi and S Tanaka
Toyohashi University of Technology, 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580, Japan

E-mail address: tanakas@ens.tut.ac.jp

Abstract. We are developing an Ultra-Low Field (ULF) Magnetic Resonance Imaging (MRI) system using a High-Temperature Superconductor superconducting quantum interference device (HTS rf-SQUID) for food inspection. The advantages of the ULF-NMR (Nuclear Magnetic Resonance) / MRI as compared with a conventional high field MRI are that they are compact and of low cost. In this study, we developed a ULF SQUID-NMR system using a polarizing coil to measure fat of which relaxation time $T_1$ is shorter. The handmade polarizing coil was cooled by liquid nitrogen to reduce the resistance and accordingly increase the allowable current. The measured decay time of the polarizing field was 40 ms. The measurement system consisted of the liquid nitrogen cooled polarizing coil, a SQUID, a Cu wound flux transformer, a measurement field coil for the field of $47 \mu$T, and an AC pulse coil for a 90°pulse field. The NMR measurements were performed in a magnetically shielded room to reduce the environmental magnetic field. The size of the sample was $\phi 35$ mm x L80 mm. After applying a polarizing field and a 90°pulse, an NMR signal was detected by the SQUID through the flux transformer. As a result, the NMR spectra of fat samples were obtained at 2.0 kHz corresponding to the measurement field $B_m$ of $47 \mu$T. The $T_1$ relaxation time of the mineral oil measured in $B_m$ was 45 ms. These results suggested that the ULF-NMR/MRI system has potential for food inspection.

1. Introduction
One of the advantages of Ultra-low field NMR/MRI (ULF NMR/MRI) is that the restriction of the magnetic field homogeneity is moderated because the magnetic field is much lower than that of a traditional MRI. Therefore the size of the ULF MRI is smaller and it can be made at a lower cost [1-2]. Moreover, using a modified inversion recovery technique, enhancement of the contrast with $T_1$ dispersion at a specific part of a tissue in a wide range of fields is possible [3].

Since our previous ULF MRI food inspection experiment system used a permanent magnet, which provided a polarizing field, the magnet was placed well away from the measurement position preventing the leakage field, which degrades the measurement field homogeneity; in this case, a sample transportation mechanism was equipped [4-5]. Therefore, it is difficult to measure the NMR signal of a sample with short relaxation time, such as fat, because of the longer transportation time.

In the paper, we demonstrate a ULF SQUID-NMR system using a 77 K cooled Cu wound polarizing coil to measure the fat, which has a short relaxation time without transportation.
2. Experimental setup and Measurement Method

2.1. ULF SQUID-NMR system using LN$_2$ cooled Cu polarizing field coil

Figure 1(a) shows a schematic of the ULF SQUID-NMR system. The main system consists of the following units: Helmholtz type measurement field ($B_m$) coil, an AC pulse ($B_{AC}$) coil, a polarizing field ($B_P$) coil, a pick up coil and an NMR spectrometer (Kea$^2$, Magritek, New Zealand). A specially designed LN$_2$ Dewar with a room temperature bore dimension of $\phi 50$ mm × L 230 mm was prepared. The polarizing coil was installed in the Dewar; the pickup coil was placed at the center of the room temperature bore. A SQUID was separately cooled by liquid nitrogen in an aluminium Dewar (8 $\ell$), which was surrounded by a three-layer mu-metal cylinder with a 2-mm thickness in order to attenuate environmental magnetic noise. The detected signal was transferred to the SQUID via a flux transformer. The NMR measurements were performed in a magnetically shielded room to attenuate the Earth's magnetic field.

![Diagram of ULF SQUID-NMR system](image)

**Figure 1.** Experimental setup. (a) Schematic drawing of the ULF SQUID-NMR using Cu polarizing coil. (b) Cross sectional view of the measurement system.

Figure 1(b) shows the cross sectional view of the measurement system, which involves $B_P$ coil, $B_m$ coil and $B_{AC}$ coil. The $B_P$ coil and the $B_m$ coil were coaxially aligned; thus the $B_P // B_m$ configuration can avoid any signal losses during a nonadiabatic switching event [6]. The pickup coil was arranged orthogonally to the $B_{AC}$ coil and the $B_m$ coil. The HTS rf-SQUID and driving electronics used in the experiment are made by Jülicher SQUID GmbH in Germany. The $B_{AC}$ coil and the $B_P$ coil were connected to an AC power amplifier (R&K, A1.5K030-4040R) and a pulsed magnet power supply (Kudo-denki, Japan, 30 V × 50 A), respectively.

The signal was filtered (Stanford Research System, SR650, HPF: 1.5 kHz, LPF: 3.8 kHz) and recorded by Kea$^2$ with amplifier (Kea$^2$ internal receiver amp, gain: +40 dB). The Kea$^2$ also controlled the sequence for $B_P$, $B_{AC}$ and the reset pulse of the SQUID.
2.2. Cu wound flux transformer

Figure 2(a) shows the detail of the flux transformer, which consists of a pickup coil and an input coil. The NMR signal from a sample was detected by the pickup coil. Then, the signal was transferred to the HTS rf-SQUID via the input coil, with which the number of turns was set to 950. The flux transformer circuit was electrically opened by a mechanical relay during the polarization so that the SQUID was not exposed to the strong magnetic field.

Figure 2(b) shows the schematic drawings of the two different pickup coils. One was a round shape solenoid coil and was prepared for a “Small” plastic canister with a capacity of 5.6 ml; the other was a racetrack shape solenoid coil and was prepared for a “Large” glass bottle with a capacity of 50 ml. The number of turns of each coil was 205 and 200, respectively.

The measured mutual inductance between the SQUID and the input coil was 15.5 nH at 77 K. The noise profiles of the SQUID were measured in both cases of the SQUID itself and with the flux transformers. As shown in Figure 2(c) the noise increased by a factor of two to three when the flux transformer was coupled. The white noise of the SQUID itself was approximately 100 fT/Hz$^{1/2}$.

![Figure 2](image-url)

**Figure 2.** (a) Schematic of flux transformer. (b) Round shape and racetrack shape pick up coil. (c) Noise spectrum density of the SQUID itself and with flux transformer measured in the magnetically shielded room.

2.3. $LN_2$ cooled Cu polarizing field coil

Although the Cu wound polarizing coil has a drawback, producing joule heat due to an electric resistance, there are some advantages, such as having no residual magnetic field which may become the origin of measurement field inhomogeneity or a quench event when changing the coil current. Another advantage is that the resistance of the coil wire can be reduced at 77 K by a factor of 1/6 compared with that at room temperature. As a result, it is possible to increase the allowable current with low loss comparing with water-cooled Cu coils. Specifications of the $B_P$ coil are listed in Table 1.

The $B_P$ coil was installed inside the specially designed $LN_2$ Dewar so that it can effectively polarize the sample in the room temperature bore. The wire diameter of the coil was set as $\phi$1.8 mm to harmonize the impedance of the coil with the specification of the power supply.
Table 1. Parameters of polarizing coil.

|                     | Design       | Experimental |
|---------------------|--------------|--------------|
| Inner Diameter      | 80 mm        | 80 mm        |
| Outer Diameter      | 134 mm       | 130 mm       |
| Length              | 80 mm        | 80 mm        |
| Wire Diameter       | $\phi1.8$ mm | $\phi1.8$ mm |
| Number of Turns     | 660          | 687          |
| Resistance [R.T.]   | 1.5 $\Omega$ | 1.7 $\Omega$ |
| Resistance [77 K]   | 0.259 $\Omega$ | 0.278 $\Omega$ |
| Inductance          | 29.9 mH      | 32.5 mH      |
| Central Magnetic Field | 313 mT@50 A | 313 mT @50 A |

Figure 3 shows the measured field distribution in the X-Z plane at the sample position. It was found that 300 mT can be obtained in a range of 30 mm $\times$ 30 mm when a current of 50 A is applied; a current-magnetic field conversion coefficient at the sample center can be calculated as 6.26 mT/A. When turning off the current, no large ringing was observed in the magnetic field, and the measured decay time of the magnetic field was 40 ms.

Figure 4 shows the change of the voltage across the $B_T$ coil while keeping a constant current of 50 A. The voltage increased up to 30 V in 90 sec associated with the increase of coil resistance due to the Joule heat. This voltage is in the upper limit of the rating of the power supply. Therefore, when the polarization time is supposed to be 5 sec each, a total of 18 sets of continuous measurements are possible. When the current was reduced to 35 A, 200 times of the measurement could be performed because of less Joules heat. Therefore, regarding the $T_1B_m$ measurement, a current of 35 A passed to reduce the heat generation of the $B_T$ coil.

![Figure 3. Magnetic field distribution in X-Z plane.](image1)

![Figure 4. Time trace of the applied voltage.](image2)
3. Result and Discussions

3.1. NMR measurement of water and fat

The pulse sequence for the NMR signal measurement is shown in Figure 5. Firstly, the measurement field $B_m$ of 47 $\mu$T was applied and kept constant until the end of the measurement. Then the sample was polarized by the $B_p$ for 5 s. In this period the flux transformer circuit was electrically opened so that the SQUID was not exposed to the strong polarizing field. After the $B_p$ current had dropped sufficiently, the $B_p$ circuit was disconnected by a mechanical relay. Subsequently, a 90° pulse was applied to rotate the magnetization of the sample. We note that $B_p$ delay time is the time between the turn-off edge of the $B_p$ field and the turn-on edge of 90° pulse. The $B_p$ delay time of 50 ms was selected in the NMR measurements, while it was varied in the following $T_1$ measurements. After closing the flux transformer circuit and setting the SQUID, the NMR signal was then detected by the SQUID via the flux transformer. The output signal of SQUID was acquired by Kea² through the filter for 512 ms.

![Figure 5. Pulse sequence for NMR signal measurement.](image)

Figure 6 (a) shows the FID trace of a "Small" sample containing water of 5.6 $m\ell$ and a "Large" sample containing water of 50 $m\ell$. Since the signal was down converted with a local frequency of 1.99 kHz, the period of the oscillation was 0.1 sec, which corresponds to the Larmor frequency of 2.0 kHz. The signal intensity of the large sample was three times larger than that of the smaller one. However the $T_2^*$ relaxation time of the large sample is much smaller than that of the smaller one. This may attribute to the inhomogeneity of the measurement field. The spectrum of each small and large sample is shown in Figure 6(b). The NMR spectrum of each water sample shows the peak at 2.0 kHz corresponding to the measurement field strength of 47 $\mu$T. The half-value width of large sample was wider than that of small sample. Additionally, both samples had an asymmetric peak spread, which showed the tail on the lower frequency side. The reason for the asymmetry was considered to be a fluctuation of the $B_m$ current due to the voltage induced by of the polarizing field switching.

Mineral oil (Johnson’s, baby oil) and lard were prepared as samples. As for the lard, both a solid state and a liquid state, melted by heat were used. They were filled in the large sample container,
which was easier than in the small one to exchange specimens. The spectra of those samples are shown in Figure 7. There were peaks for the mineral oil and liquid lard but no peak for the solid lard. The intensities of the peaks were smaller than that of the water sample as shown in Figure 6(b) because of the difference of the material.

![Figure 6. NMR signal of the water samples. (a) FID signal of each large and small sample. (b) NMR spectra.](image)

![Figure 7. NMR spectra of the fat samples.](image)

![Figure 8. Dependence of the amplitude on B_P delay time.](image)

3.2. $T_1$ measurement in $B_m$

It was reported that images of gel phantoms with $T_1$ weighted contrast demonstrated dramatic enhancement in low field [3]. To investigate whether the enhancement of $T_1$ contrast could be applied to fat with short relaxation time, $T_1$ relaxation time in the measurement field $B_m$ was measured. The NMR signal was measured by changing the $B_P$ delay time, which was the time between the turn-off edge of the $B_P$ field and the turn-on edge of the $90^\circ$ pulse as shown in Figure 5. When the $B_P$ delay time was longer than the relaxation time $T_1$, it was hard to distinguish the signal from the noise since the signal intensity became extremely low. Therefore, the signal of the mineral oil can be observed until only a $B_P$ delay time of 100 ms, while that of the water sample can be taken until 2000 ms as shown in Figure 8. The values were normalized after integration in the bandwidth of 10 Hz at the peak frequency since the shape of each spectrum is different and the spectrum has some area. The $T_1$ relaxation time was calculated from the time constant of the exponential decay by fitting the plots of
the NMR observed signal. As shown in the Figure, it was found that the $T_1$ relaxation time of the mineral oil sample was 45 ms, which was much shorter than $T_1$ of water sample, 2.6 s.

4. Conclusions
In this study, we developed a ULF SQUID-NMR system using an LN$_2$ cooled Cu polarizing field coil aiming for food inspection. In the system, the NMR signal was detected by the HTS rf-SQUID with the Cu wound flux transformer. The handmade polarizing coil was able to continuously apply a polarizing field of 300 mT at maximum. It was demonstrated that the NMR signal of fat was successfully measured in the system. The results suggested that the ULF SQUID-NMR/MRI system has potential for food inspection.

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