

Low-temperature nuclear magnetic resonance with a dc SQUID amplifier

M. R. Freeman, R. S. Germain, and R. C. Richardson
Laboratory of Atomic and Solid State Physics, Cornell University, Ithaca, New York 14853

M. L. Roukes
Bell Communications Research, Crawford's Corner Road, Holmdel, New Jersey 07733

W. J. Gallagher and M. B. Ketchen
IBM Thomas J. Watson Research Center, Yorktown Heights, New York 10598

(Received 18 October 1985; accepted for publication 23 November 1985)

We report the detection of nuclear magnetic resonance at 4 K and 1.9 MHz with a receiver based on a thin-film dc superconducting quantum interference device. The noise temperature of the spectrometer is 300 mK, limited by the second stage amplifier.

An optimized nuclear magnetic resonance spectrometer operating at room temperature typically has a noise temperature of order 100 K,¹ meaning that the device noise of the amplifier chain equals the Johnson noise from the probe when the latter is cooled to 100 K. Noise matching to colder probes has been achieved through use of a number of different cryogenically cooled amplifiers, all of which have different limits of applicability. Commercial rf and dc SQUID's operating in a feedback mode have been used to detect nuclear magnetic resonance (NMR) at frequencies below 100 kHz,²⁻⁴ and cold field-effect transistors (FET's) have attained noise temperatures of order 5 K at megahertz frequencies.⁵ This letter reports the use of a dc SQUID small-signal amplifier as the front end of a receiver in an effort to noise match to pulse NMR probes cooled to millikelvin temperatures. The technique is similar to that used recently to detect nuclear quadrupole resonance.⁶

A schematic of the measurement system is shown in Fig. 1. Part (a) of the figure illustrates the input circuit. The NMR cell consists of crossed Helmholtz coils wound on accurately orthogonal forms. At 2 MHz, the isolation between the coils is 60 dB. Crossed diodes, traditionally placed in the transmitter line to block rf gate feedthrough, are mounted at 4 K to reduce the possibility of interference from the room coupling into the excitation coil. The attenuation of the cold diodes drops to less than 1 dB for rf levels greater than 2 V rms. A damping resistor in parallel with the transmitter coil is necessary to prevent the ringdown after the rf pulse from dominating the recovery time of the receiver. The static magnetic field is produced by a superconducting solenoid wound on a brass coil form, capped at both ends to make an rf tight shield around the cell. The transmitter line runs the length of the cryostat as triax, converting to twisted pair at a penetration of the cell enclosure.

The optimal circuit for coupling an NMR signal into a SQUID, which senses current, is a series tuned tank. In general, one wants the quality factor Q to be as large as possible without impinging on the bandwidth requirements of the experiment. The noise temperature of the detector is minimized when $Q\rho \approx 1$, the exact condition being somewhat dependent on device-specific parameters. Here $Q = \omega(L_p + L_{in})/R$, and ρ represents the fractional inductance contributed by the SQUID input coil, $\rho = L_{in}/(L_p + L_{in})$. L_p , L_{in} are the inductances of the Helmholtz pickup coil and

SQUID input coil, respectively, and R is the total dissipation in the tank circuit. This prescription for determining the coupling of the pickup coil to the SQUID results in a balance of the contributions of voltage and circulating current fluctuations to the overall device noise. We have presented a brief discussion of the proper choice of component values elsewhere.⁷

An essential component of the input loop is a limiter to prevent large currents from flowing in the SQUID input coil during the rf pulse. For an early circuit without a limiter, we estimated that our largest rf pulses would induce emf's of 1 mV across the SQUID, far below the threshold for any physical damage. However, we found that even much smaller pulses significantly alter the bias point of the device, presu-

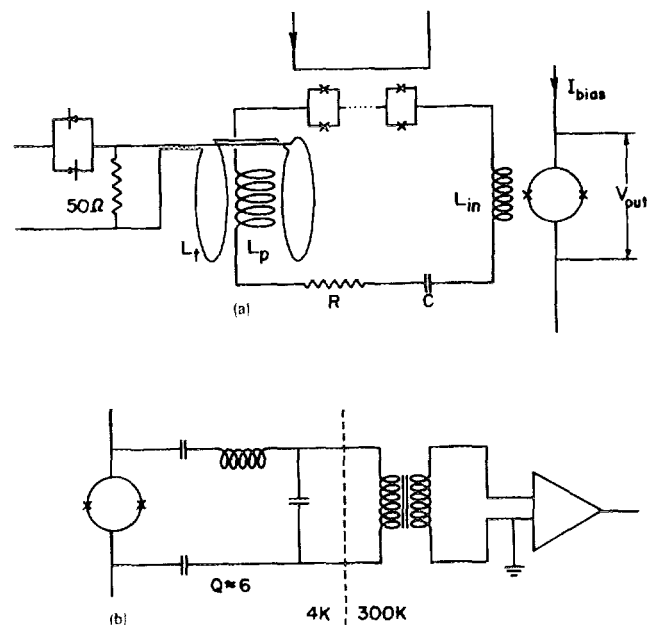


FIG. 1. (a) Series tuned input to the SQUID, shown with the low-temperature components of the transmitter circuit. The spin system is inside the pickup coil L_p , which is a Helmholtz pair perpendicular to the transmitter coil L_t . The superconducting interferometer array consists of 26 devices and is modulated by a single control line. (b) SQUID output impedance is matched to a room-temperature amplifier (Trontech W50-ATC) using a cooled, tuned transformer. Blocking capacitors are included to prevent shunting of the dc bias current away from the SQUID. The isolation transformer (MCL T1-1T) is normally included to protect the SQUID.

mably by redistributing trapped flux lines. Without feedback, this renders the SQUID useless as an amplifier. An elegant solution to this problem has been devised by Hilbert *et al.*,⁶ who connect a string of Josephson junctions in series with the input coil. The junctions act as a current clamp, in much the same way as diodes are often used to clamp the voltage at the input of a conventional NMR receiver. The performance of the limiter is largely independent of the amount of hysteresis in the current-voltage characteristic. The junctions remain in the zero voltage state once the signal amplitude has decayed below the maximum critical current, because the signals are ac. In addition to SQUID protection, a key advantage of this technique is the ability to drastically reduce pulse recovery times for high Q circuits. The dead-time of the receiver is basically determined by the ringdown of the tank from the critical current of the limiter, with the characteristic time constant Q/ω .

The limiter for the present work consists of 26 superconducting interferometers in series, with a zero field critical current of $110\ \mu\text{A}$. Running past all of the interferometers on the chip is a control line which applies a flux bias to modulate the critical current. In practice the critical current is set at $5\ \mu\text{A}$, and the normal state resistance of the line is $1\ \text{k}\Omega$. Trapped flux inhibits the tunability of the limiter, which is shielded from the earth's field by a 0.8-mm wall mumetal box. We are presently preparing smaller area, lower critical current devices more ideally suited to this application. The ease with which the interferometers can be modulated by the current in a single control line permits their fabrication on the same chip as the SQUID.

The readout circuit is shown in part (b) of Fig. 1. The dynamic resistance of the SQUID at the chosen operating point is $2\ \Omega$, measured from the dc current-voltage characteristic. A simple resonant transformer is used to step this impedance up to match the $80\ \Omega$ input of a room-temperature amplifier. Placing this transformer inside the cryostat reduces the effect of loss in the triaxial readout line, and lowers the added noise of the transformer itself. A tuned readout can be used here because of the high Q of the input circuit. Transmission line transformers can be used to obtain more bandwidth.

Figure 2 shows a noise spectrum taken with 73 dB of gain following the SQUID, which is biased to have gain $dV/d\phi = 40\ \mu\text{V}/\phi_0$. The particular device used in this case is similar to that described by Ketchen and Jaycox,⁸ having a 90 pH self inductance and a 19-turn input coil with $L_{\text{in}} = 35\ \text{nH}$. The value of $Q\rho$ is 0.64, determined by $L_p = 2.2\ \mu\text{H}$ and $R = 0.65\ \Omega$. The peak in the figure is due to the Johnson noise of the tank circuit. The off-resonance background level is the noise of the readout, confirmed by biasing the SQUID in the zero voltage state. The present readout scheme is not matched to the output noise of the SQUID at its optimal bias points, so we are unable to resolve the contribution of the SQUID to the noise level off resonance. This limits the noise temperature of the receiver to 300 mK. We are developing a cryogenically cooled FET second stage amplifier, in order to take full advantage of the low SQUID noise. The solid line is a fit to the spectrum which can be used to extract various circuit parameters, in particular the temperature of R rela-

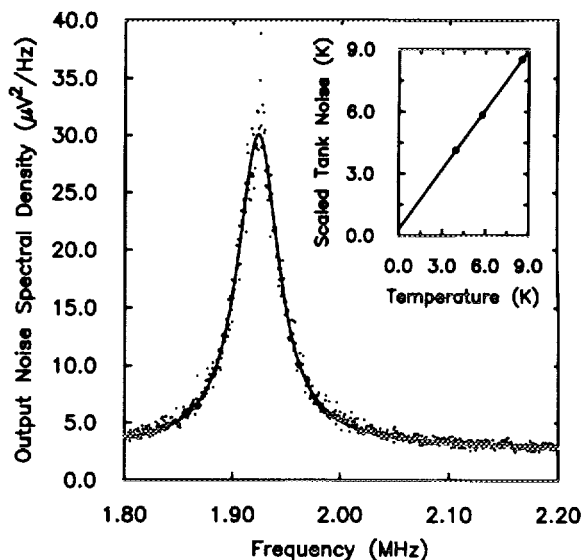


FIG. 2. Typical output noise spectrum showing the Johnson noise peak from the tank circuit at 4 K. The noise level far off resonance is the contribution of the readout amplifier. The solid line is a nonlinear least-squares fit used to extract the temperature. This noise temperature is shown in the inset as a function of the temperature of the major dissipative element in the tank, a piece of resistance wire.

tive to some fixed point. The inset of Fig. 2 shows this temperature plotted against the reading of a germanium resistance thermometer which is in good thermal contact with a heater and with a short length of Advance wire, the dominant component of R . We attribute the small offset of the vertical intercept to an extra resistance in the tank which is not coupled to the heater, most likely the capacitor dissipation. If the effective flux noise of the SQUID is $10^{-6}\ \phi_0/\text{Hz}^{1/2}$, a value we have measured at lower frequencies using an rf SQUID readout (and the dc SQUID still at 4 K), a 30 mK noise temperature at 2 MHz should be possible using a lower noise second stage.

The NMR sensitivity of this spectrometer has been tested by measuring signals from ^3He adsorbed on a surface, at 4 K and in a static field of 60 mT. The sample volume is a 0.8-cm^3 cavity, loosely packed with 2000-Å fluorocarbon spheres. ^3He can be admitted in known quantities from a room-temperature reservoir. As we have previously discussed,⁷ the minimum discernible number of spins (defined by the condition signal to noise of 1 following a 90° pulse) can be calculated to be

$$N_{\text{mids}} = \left(\frac{4k_B T^*}{\hbar\omega} \right) \left(\frac{4k_B T_{\text{sys}} v \delta f}{\mu_0 \gamma^2 \hbar^2 \omega Q} \right)^{1/2}. \quad (1)$$

Here the temperature of the spin system is T^* , and the system noise temperature, T_{sys} , is the sum of the temperature of the tank circuit and the noise temperature of the receiver. γ is the gyromagnetic ratio, δf the measurement bandwidth, and v an effective coil volume.

Two nuclear resonance signals are shown in Fig. 3. In this case the cell contained 1.5×10^{20} ^3He atoms. The free-induction decay was excited by a 16° tipping pulse lasting 40 μs and measured in a 200 kHz bandwidth. The recovery time was measured by injecting a small coherent signal, and

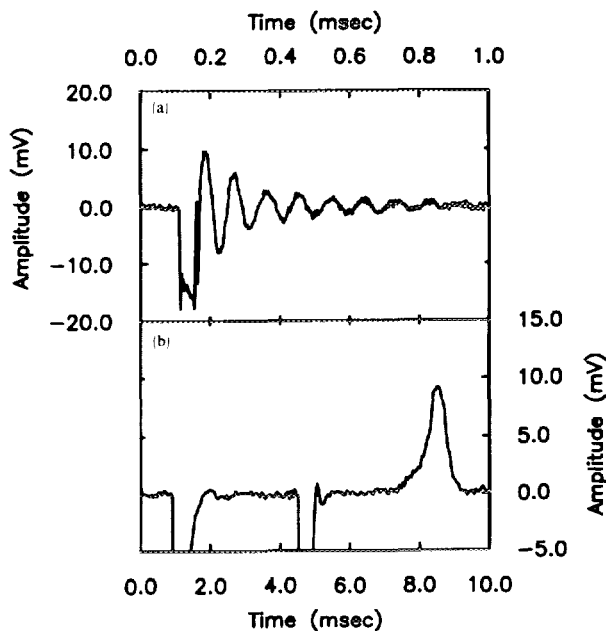


FIG. 3. (a) ^3He free-induction decay. The tipping pulse begins $100\ \mu\text{s}$ into the trace and lasts for $40\ \mu\text{s}$. The tipping angle was 16° and the total number of spins was 1.5×10^{20} . The SQUID was followed by 73 dB of gain and the signal recorded using a 200 kHz bandwidth. (b) Spin echo sequence, also seen in a 200 kHz bandwidth.

found to be $15\ \mu\text{s}$. The linear dynamic range of the amplifier was also checked in this manner, and found to be 90 dB in a 1 Hz bandwidth. The 1-dB compression point is at an input signal level of $0.25\phi_0$ peak to peak, about what one expects for a sinusoidal transfer characteristic. We have also used

the SQUID amplifier to detect spin echoes, as shown in Fig. 3(b). The observed signals correspond to $N_{\text{mids}} = 4 \times 10^{18}$ spins, measured in a 200 kHz bandwidth about 1.93 MHz, in good agreement with Eq. (1) for T^* and T_{sys} both 4 K.

We have demonstrated NMR detection at 1.9 MHz and 4 K using a dc SQUID small-signal amplifier. The SQUID is the best device presently available to match to the very low thermal noise of NMR probes at millikelvin temperatures, for signal frequencies f below about 100 MHz. The advantage held by the SQUID over other types of amplifier increases as the probe temperature is lowered towards the minimum device noise temperature, approximately $10\ \text{mK} \times f$ (MHz) for state of the art SQUID's operating at 4 K.

This work was supported by the Natural Sciences and Engineering Research Council (Canada), the National Science Foundation, and the Cornell Materials Science Center. The dc SQUID's were fabricated at the IBM Thomas J. Watson Research Center by J. Greiner and co-workers.

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