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Observation of NMR signals from a thin ^{3}He slab

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NMR free precession signals from a slab of normal ^{3}He of thickness comparable to the superfluid coherence length have been observed using a SQUID NMR spectrometer at a frequency of 880 kHz. This spectrometer is based on a SQUID with Additional Positive Feedback, directly coupled to a low noise room temperature amplifier, and operated in flux locked loop mode using the direct offset integration technique, with a bandwidth of several MHz. The sensitivity is such that the signal from a 100 nm slab on a 1 cm^2 surface, corresponding to 2×10^{17} spins, is clearly resolved.
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1. INTRODUCTION

This paper describes preliminary NMR studies of a thin ^{3}He slab resting on a silver surface. Slabs for which the size of the Cooper pair, given by the superfluid coherence length $\xi(T)$, is comparable to the thickness of the slab d are expected to exhibit a phase diagram very different from bulk liquid.[1,2] Suppression of the order parameter is expected to be strongly influenced by quasiparticle boundary scattering,[3] with diffuse scattering leading to a suppression of T_c.[4] The simplicity of this slab geometry and the possibility to vary the film thickness over a wide range and at constant pressure, eliminating pressure dependent strong coupling effects, make it particularly attractive. NMR should play an important role in determining the phase diagram as a function of $d/\xi(T)$, in studying the influence of surface scattering on the superfluid phases, and in revealing possible new physics of "two dimensional superfluidity" when $d < \xi(T)$. However to measure a single slab of stable and well characterised thickness is technically demanding. The zero temperature coherence length at zero pressure is $\xi(0) = 70$ nm, and a slab of this thickness on a surface of area 1 cm^3 contains $\sim 10^{17}$ spins. In order to measure the weak NMR signal from such a sample, we have developed a sensitive NMR spectrometer based on a DC SQUID preamplifier. This paper reports the performance of this spectrometer in measurements on normal ^{3}He films.

So far several approaches have been adopted in the study of superfluid ^{3}He films. Measurement of ^{3}He film flow over the polished rim of a beaker[5] found a suppression of the superfluid transition temperature T_c, consistent with theoretical predictions for diffuse boundary conditions. This suppression could be eliminated by coating the substrate with a thin ^{4}He film, presumably giving rise to specular boundary conditions. A disadvantage in this method is the continuous thinning of the film arising from emptying of the beaker. Metastable films have been grown and their superfluid density measured by a torsional pendulum technique.[6] In both these flow experiments evidence for a transition from a B-phase to an A-phase like state has been reported as the thickness of the film is decreased. Our own progress in developing a torsional pendulum for measuring the superfluid density is reported in these proceedings. Recently third sound resonances, driven and detected capacitatively, have been observed in the Van der Waals film on a horizontal copper surface, also allowing a measurement of the superfluid density.[7] Again a change in behaviour below 195 nm is attributed to a transition to a new equilibrium state. An alternative approach to the study of the influence of finite size on the superfluid phase diagram is to confine liquid ^{3}He in a restricted geometry. For example NMR on ^{3}He in a stack of 3000 Mylar sheets spaced by 300 nm identify the equilibrium phase to be the A-phase, even at low pressures.[8]

2. SQUID NMR SPECTROMETER

Our NMR spectrometer uses a DC SQUID, whose voltage-flux transfer characteristic is enhanced by Additional Positive Feedback. This allows the output of the SQUID to be directly coupled to a low noise room temperature amplifier, achieving an overall noise of typically $1.5\mu\phi_0/\sqrt{\text{Hz}}$. This amplifier is linearised and the working point stabilised by operation in flux locked loop mode, using the direct offset integration technique. Using this method amplifier bandwidths up to 7.5 MHz have been achieved.[9]

The NMR receiver coil, of inductance 8.37 μH, forms part of a series tuned circuit with the input coil of the SQUID, resonating at 880 kHz. The circuit has a Q of 17, limited by a series resistor mounted on the mixing chamber, and includes a Q-spoiler to reduce its recovery time following the NMR transmitter pulse. The particular SQUID used for the present experiment has an integrated input coil of inductance 470 nH, with a coupled energy sensitivity of $330h$. As reported previously[10] the gain stability of this arrangement is better than 1%, a significant improvement over performance open-loop,[11] of crucial importance in the subtraction of coherent background signals.

3. EXPERIMENTAL DETAILS

This experiment is mounted on a new ultra-low temperature facility at Royal Holloway, which provides a platform for experiments down to $100\mu K$, with extensive space for mounting experiments and a high level of access for experimental services. Extended space between the 4 K flange and 1 K pot plate and between the 1 K pot plate and still plate provides ample space for mounting DC SQUIDs. The DC SQUID preamplifier stage described above is mounted on a probe, tested at 4 K in a transport dewar, and then inserted without disassembly through a 40 mm diameter tube into the inner vacuum can. Thus the SQUID is operated in vacuum and in this work it is thermally anchored at 4 K.

The ^{3}He sample cell is situated at the centre of a solenoidal NMR magnet, which is located in the ^{4}He bath. The lower inner surface of the cell consists of a highly polished silver foil, sealed to a Stycast body. The open volume of the cell body is a cylindrical cavity 13 mm in diameter and 1 mm high. The silver foil is cut with a tab which passes horizontally through the wall of the cell, and through which the film is thermalised. The rectangular receiver coil of width 15 mm, height 3.3 mm and overall length 13 mm is wound in two sections 5 mm in length from 0.19 mm diameter copper wire. The estimated field constant for this coil at its centre, the relevant parameter for calculating the expected signal size, is 3.87 mT/A. The transmitter coil has a saddle geometry and is wound on a MACOR former. This is fixed rigidly relative to the sample cell, and the assembly is mounted on a silver plate which in turn is thermally linked the top flange of the nuclear stage via a silver rod 10 mm in diameter and 20 cm long, coupled through two cone joints. A platinum wire NMR thermometer is also mounted from the silver plate.

The nuclear stage is fabricated from 1 mm copper sheets, spaced by teflon and diffusion welded into a copper flange, closely following the design of Dmitriev et al.[12] It has an effective mass of 42 moles in the maximum field of the demagnetization solenoid. A ^{3}He melting curve thermometer and

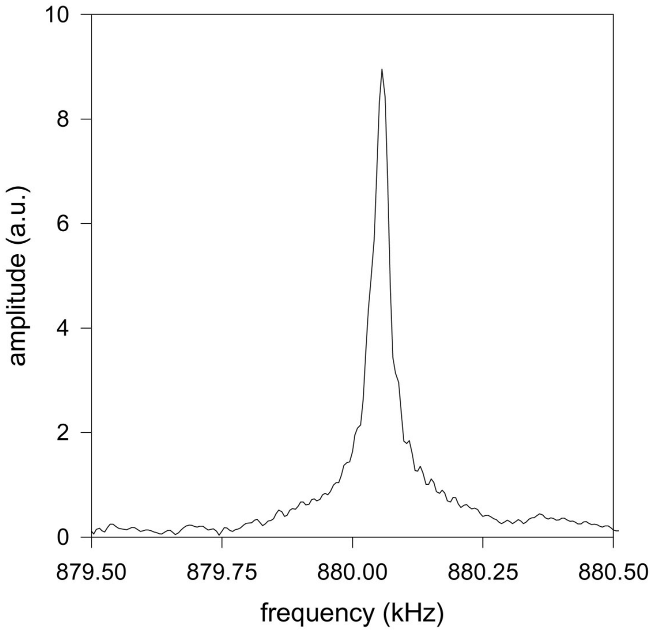

Fig. 1. NMR signal at 14 mK from 2×10^{17} ^{3}He spins

a current sensing noise thermometer are also mounted on the flange. The stage is pre-cooled by a custom designed Oxford Instruments Kelvinox 400 dilution refrigerator.

4. RESULTS

The ^{3}He sample is admitted to the sample chamber in doses from a room temperature standard volume, the pressure in which is monitored by a Paroscientific Digiquartz pressure gauge. The fill line, which is relatively weakly thermally anchored to the 50 mK plate and mixing chamber, is heated along its length to 1 K during this process, while the cell is held below 100 mK in order to ensure condensation of the sample in the cell.

The figure shows the NMR signal at 14 mK from 2×10^{17} ^{3}He spins, equivalent to 7×10^{-3} STPcm^3, corresponding to a film of thickness 100 nm on one surface. The signal is captured by a Tektronix TDS430A oscilloscope, downloaded to a PC where it is Fourier transformed after zero filling. Due to the relative narrowness of the NMR line no background subtraction of magnetoacoustic resonances was necessary, since these could be avoided by suitable tuning of the magnetic field. This result demonstrates that signals of reasonable quality are measurable from such small samples, with a signal to noise ratio in a single shot ~ 4. Quadrupling the number of ^{3}He spins in the sample produced a corresponding increase in magnetization, as inferrred from S_p/T_2^* (where the peak height S_p and T_2^* are inferred from Lorentzian fits). In principle eddy current heating of 1 nJ per $\pi/2$ transmitter pulse is achievable, permitting experiments well below 1 mK.

The noise temperature for a similar SQUID, determined by measurements of the Johnson noise in the input tuned circuit from 4.2 K to 1.5 K, was estimated at 100 mK. However the present SQUID performed rather worse, which we attribute to a short which arose between the integrated input coil and the SQUID. Eliminating this problem should result in significant improvements in signal to noise with the current set-up, with an acchievable minimum number of spins detectable in a single shot $\sim 10^{16}$. Improvements on this sensitivity will result from further progress towards an NMR spectrometer with quantum limited energy resolution.

The bottleneck for thermalisation of the sample is expected to be the boundary resistance between the ^{3}He film and silver surface. Actually the surface to sample volume ratio is comparable to that achieved in bulk samples cooled by silver heat exchangers. Extrapolating a high temperature value for the Kapitza resistance R_k for a clean polished metal of area A, $AR_kT^3 = 0.005\ \mathrm{m^2K^4W^{-1}}$, we find a thermal time constant of 300 s at 1mK, which is acceptable. Since the magnetization of a normal Fermi fluid film is temperature independent in the region of interest we have no empirical information thus far on the cooling of the film.

We did find that measurements were complicated by an apparent physical instability of the film, which led to a time dependence of the NMR signal. For example, with the cell controlled at a temperature of 7.2 mK we found the NMR signal from a 400 nm film to decrease approximately linearly with time by a factor of three over 90,000 s. This film was more stable at 60 mK. Operations such as applying a sequence of large NMR pulses or heating the fill line tended to restore the signal. Although the experimental systematics were not fully established, we believe that the problem arises from temperature gradients within the cell, since the lower surface is silver, while the upper is Stycast. A flow of the normal ^{3}He film in reponse to a temperature gradient was observed in the first flow measurements on ^{3}He films.[5] This is believed to arise from a giant thermomechanical effect in confined liquid ^{3}He, analogous to thermopower in electronic systems, and described by irreversible thermodynamics.[13,14] We note that in our the torsional oscillator experiments, reported elsewhere in these proceedings, the film appeared to be stable with time, probably because in this case the design of the sample chamber ensures isothermal conditions.

In this work we have demonstrated the sensitivity of the spectrometer in measurements on normal ^{3}He films. Future work will involve improvements in spectrometer performance and cell design in order to investigate the two dimensional superfluid phase diagram.

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