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Fully Automated AC Susceptometer for milli-Kelvin Temperatures in a DynaCool PPMS

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***Abstract*—We have developed a commercial apparatus to measure the AC-susceptibility of small samples (1 mg to 500 mg) from 50 mK to 4 K using a versatile top loading dilution refrigerator. This susceptometer is readily available to perform fully automated AC measurements in both, cryogen free and liquid cooled Physical Property Measuring Systems (PPMS). AC susceptibility measurements can be performed with AC excitation fields in the range of 0.01 Oe–4 Oe (peak) for frequencies from 10 Hz to 10 kHz and in the presence of a static DC field of up to 12 T. The design of the susceptometer employs a novel approach which virtually eliminates heating of the sample stage by thermally anchoring the coil set at 1.8 K, using superconducting wire for the excitation coil, and using a coil design which limits induced eddy currents on the dilution unit. The sample is mounted to a sapphire sample holder attached to the dilution stage and positioned in the center of one of the pickup coils. An additional trim coil on the coil set allows for dynamic removal of any background signals during the measurement, facilitating sample measurements for moments as small as 2×10^{-7} emu. We present measurements for various samples to demonstrate the capabilities and performance of this new instrument. In the current design, AC susceptibility measurements down to 50 mK can be performed in less than 8 hours after mounting the sample.**

***Index Terms*—Dilution refrigerators, Cryogenics, Magnetic susceptibility, Magnetometers, Materials testing.**

I. Introduction

Magnetic Susceptiblity measurements at sub-Kelvin temperatures are a valuable tool to investigate fundamental material characteristics. However, due to the challenges imposed by performing these measurements at extremely low temperatures, availability of such measurements historically has been extremely limited. Measurements typically have only been possible on large dilution refrigerator (DR) systems with custom experimental setups [1], [2], requiring complicated sample mounting procedures and resulting in long cool down times. Additionally, in order to limit heating of the DR sample stage, AC drive and frequency typically had to be limited, imposing a minimum feasible sample size to overcome the small signals. Most systems traditionally would also report relative data and require manual post-processing to remove background signals.

We present a novel, compact design for a fully automated AC susceptometer that overcomes these issues and allows sample measurements for moments as small as 2×10^{-7}emu with a phase accuracy of ±2°.

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II. Design

A. Coil Set

Since we designed our susceptometer for the Quantum Design DR for the PPMS DynaCool [3], [4], we decided to thermally and mechanically anchor the susceptometer's coils to the puck interface at the bottom of the sample chamber rather than the DR sample stage. This allows us to use the existing wiring and provide cooling at a temperature of about 1.8 K, thus not contributing any additional heat load to the DR.

The drive coils are wound onto a G-10 former using #40 (0.003" diameter) Cu-clad single-filament superconducting NbTi wire in order to reduce local heating. To minimize field inaccuracies due to sample location and size, the drive coil is designed in such a way as to yield maximum field homogeneity at the sample location. Furthermore, to prevent eddy current heating and spurious signals, the drive coil is actively shielded such that virtually no stray field is present at the location of the DR sample stage or at the sample chamber walls. The drive coil produces a field of about 0.095 Oe/mA.

The pickup coils are wound from #38 (0.004" diameter) Cu wire arranged as a first order gradiometer with each side having a total of 442 turns. The sample location is at the center of the upper part of the pickup coils.

In addition to the primary drive and the pickup coils, the coil set features two additional coils: a calibration coil and a trim coil. The calibration coil comprises a single turn made from the same type of wire as the pickup coil, centered at the lower part of the pickup coils. This additional coil allows on-the-fly measurement of attenuation and phase shifts introduced by the system in order to correct for it which enables the system to report absolute phase angles to better than ±2°. The trim coil is made of multiple turns wound in such a way that it has a large mutual inductance with the gradiometer without contributing any field at the sample location. This allows for removal of frequency dependent background signals in order for the system to be able to report absolute susceptibility values. The trim coil is made from the same type of wire as the primary drive coil.

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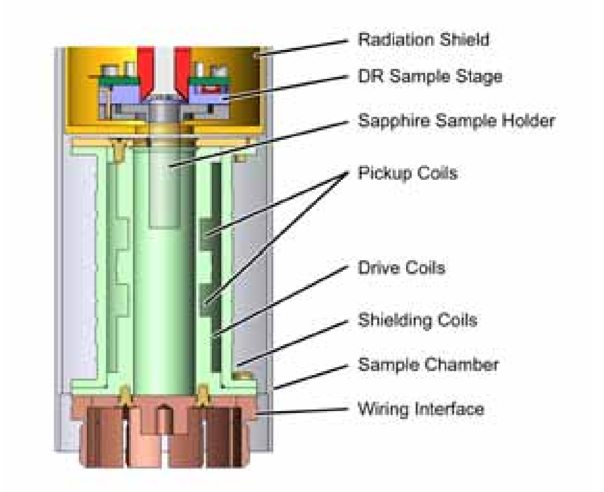

Fig. 1. Cross-sectional view of the coil set inside the sample chamber and the bottom end of the DR with one of the AC sample holders attached. The sample is mounted to the bottom end of the sapphire sample holder in the center of the upper part of the pickup coils. The radiation shield, sample chamber walls, and wiring interface are all at the same temperature of 1.8 K or 2.5 K when sample is above 1 K. (Some details omitted for clarity)

When the DR sample stage temperature is below 1 K, the coil set is held at a constant temperature of 1.8 K. In that configuration, we were able to source up to the maximum current of our AC electronics (50 mA) in background fields up to 13.7 T without driving the coil normal. For sample temperatures between 1 K and 4 K, the coil is held at a higher temperature of 2.5 K, which slightly reduces the maximum current possible. We are however able to use our maximum AC drive of 4 Oe at all temperatures up to a DC background field of 12 T.

B. Sample Holder and Mounting

Fig. 1 shows a cross-sectional view of the coil set and the sample holder inside the sample chamber of the host system.

The sample holder consists of a single crystal sapphire rod with a diameter of 4 mm (0.157"), which attaches to the DR sample stage and extends through a hole in the protective cover/thermal shield into the center of the upper part of the pickup coils. We tested two different sample holder designs, allowing to easily mount thin film samples in orientations parallel as well as perpendicular to the AC field.

In order to characterize the thermal lag between the sample mounted to the bottom end of the sapphire sample holder and the DR sample stage which houses the sample thermometer, we mounted a second thermometer to the bottom of the sample holder and monitored both temperatures as function of time while stepping through various temperatures. Down to the lowest temperature of the DR (50 mK), the second thermometer reached the temperature of the DR sample stage, indicating that the radiation from the coil set at 1.8 K does not contribute additional heating. Keeping in mind that the standard thermal shield for the DR is at the same temperature, this is not all that surprising. The time constant to reach equilibrium between the sample and stage thermometers becomes appreciable only below about 100 mK, eventually reaching values of the order of ten minutes at 50 mK.

C. Measurement Electronics and Data Analysis

Signal generation and analysis is performed using custom, specialized electronics developed for this task. With a Texas Instruments TMS320VC5510A Digital Signal Processor (DSP) [5] at its core, the electronics are capable of generating two independent sinusoidal currents with amplitudes of up to 50 mA while simultaneously performing lock-in analysis on two voltage signals returned from the coil set. The primary current drive is used to generate the AC field at the sample's location while the secondary channel drives the trim coils to remove any background signals caused by coil imbalance and other non-sample contributions. The current of the secondary channel consists of a sine wave with the same frequency as the primary channel with a fixed amplitude and phase relationship. The amount of current required and relative phase to the main AC signal are calibrated once with no sample mounted. This frequency-dependent calibration is then applied for each subsequent measurement with a sample in place.

The primary voltage read-back connects via a selectable variable gain to the signal of the pickup coils and the DSP performs a lock-in analysis on the signal. The secondary channel reads the signal of the calibration coil centered in the lower part of the pickup coils. As the sample signal at its location is negligible, the phase and amplitude information from this second channel can be used to correct for system-related corrections, allowing to report both phase and amplitude of the signal with greater accuracy.

For small signal samples that don't exhibit any AC losses (e.g., paramagnetic or superconducting samples), the electronics is able to further reduce the background signal even with the sample in place. Due to the fact that the background and the sample signals are out-of-phase from each other, the background signal can be further reduced by performing a constrained minimization of the overall signal amplitude. The electronics can achieve this in an automated way by making small adjustments to both, the amplitude and phase of the secondary signal driving the trim coil starting from the values obtained by the calibration without a sample.

III. EXAMPLE DATA

In order to demonstrate the capabilities and performance of our susceptometer we measured the superconducting transitions for two samples, a Titanium single crystal and a small sample of $Ir_{1-x}Ru_x$.

A. Ti Single Crystal

We measured a sample weighing 23.0 mg cut from a single crystal Ti rod, similar to what has been reported in [6]. Fig 2 shows the superconducting transition measured in zero DC background field for relatively fast sweeps, both with increasing and decreasing temperature. This data shows the quality of the thermal coupling between the DR sample stage and the sample even when sweeping temperature, as there is almost no discernable difference between the cooling and

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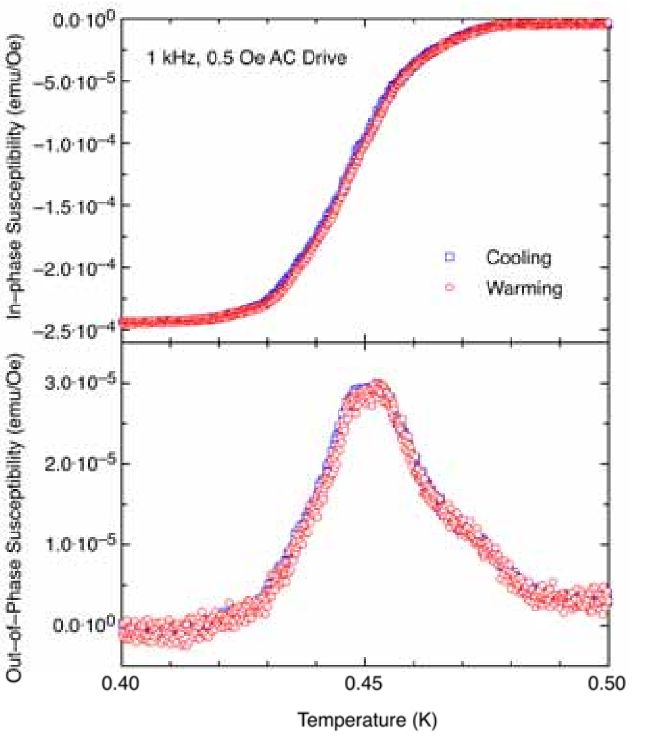

Fig. 2. In-phase (top) and out-of-phase susceptibility (bottom) for the superconducting transition of the Ti single crystal measured while sweeping temperature at a rate of 10 mK/min. This data shows the exceptional speed and reproducibility of the instrument: each data set took only 10 minutes to collect and there is no discernable temperature difference between the data acquired while cooling and warming.

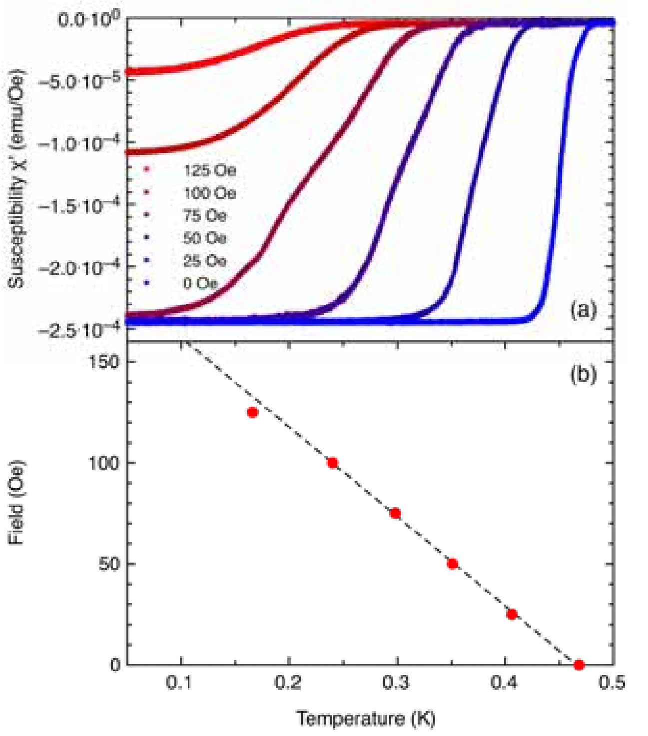

Fig. 3. (a) Superconducting transition for the Ti sample measured with an AC amplitude of 0.5 Oe at a frequency of 1 kHz in varying DC background fields. Each data series is measured while continuously cooling the sample. (b) Critical field as function of temperature extracted from the above measurements. The dashed line has a slope of −443 Oe/K.

warming curves. The fact that the out-of-phase component is zero in the superconducting phase also shows how well the background removal using the trim coil and phase calibration using the calibration coil work. It is important to note that the data presented is the data as reported by the instrument, without any additional post processing applied. The small non-zero out-of-phase signal in the normal phase is due to eddy currents in the sample itself.

Defining the critical temperature T_c as the temperature at which the susceptibility changed by 10% of the full transition, we measure a critical temperature $T_c = 468$ mK, in good agreement with [6] which reports a slightly higher value but used the onset of the transition to define T_c.

Repeating the same measurement with varying DC background fields, we can then trace the critical field as function of temperature — we once again use the 10% criteria to define the transition temperature. The results of those measurements are shown in Fig. 3. We calculate a slope of $(dH/dT)_{T=T_c}$ of −443 Oe/K, again in good agreement with [6].

In Fig. 4 we show the same transition measured with different frequencies. This data was collected while stabilizing the temperature at steps of 2.5 mK. While lower frequencies obviously result in noisier data, the data clearly shows that we don't get any sample or DR stage heating even at the higher frequencies. The fact that the out-of-phase signal in the sample's normal state scales with frequency proves that this is indeed a result of the eddy current losses in the sample itself.

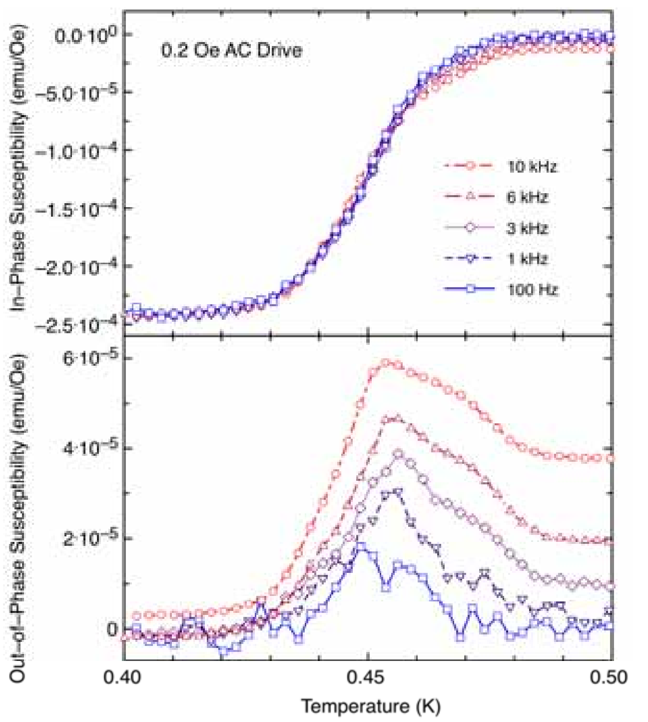

Fig. 4. Frequency dependence of the in-phase and out-of-phase susceptibility for the Ti sample.

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B. $Ir_{1-x}Ru_x$

In order to demonstrate the capabilities of the susceptometer for low signals as well as at the lowest temperatures of the DR, we measured the superconducting transition of a $Ir_{1-x}Ru_x$ sample. Varying the amount of Ru and/or changing the DC background field allow tuning the transition temperature of this material. For the sample measured here we verified the transition temperature in various small DC background fields using both, electrical resistivity as well as specific heat [3].

Fig. 5 shows the in-phase susceptibility for the sample's superconducting transition for various DC background fields. All data has been measured using an AC amplitude of 10 mOe and frequency of 10 kHz with 1 s averaging while continuously sweeping the temperature. The time required to acquire each data set was 20 minutes. The lower graph in Fig. 5 shows the zero field data in more detail in order to highlight the susceptometer's performance. The peak-to-peak scatter of the data is about 5×10^{-6} emu/Oe, corresponding to 5×10^{-8} emu in absolute signal.

IV. Conclusion

We presented a novel design for a fully automated AC susceptometer compatible with the PPMS DynaCool dilution refrigerator option. Due to choices made in the design of the coil set, overall system thermal and electronics design, we are able to directly measure absolute values of the AC susceptibility for samples down to 50 mK without the need for data post-processing to remove background signals or report accurate phase information. The ease of sample mounting which does not require interfering with the coil set or other parts or the susceptometer combined with the fast thermal performance of the system allows for data to be acquired typically within 8 hours after mounting a sample. The example data we presented clearly show the capabilities and accuracy of the susceptometer.

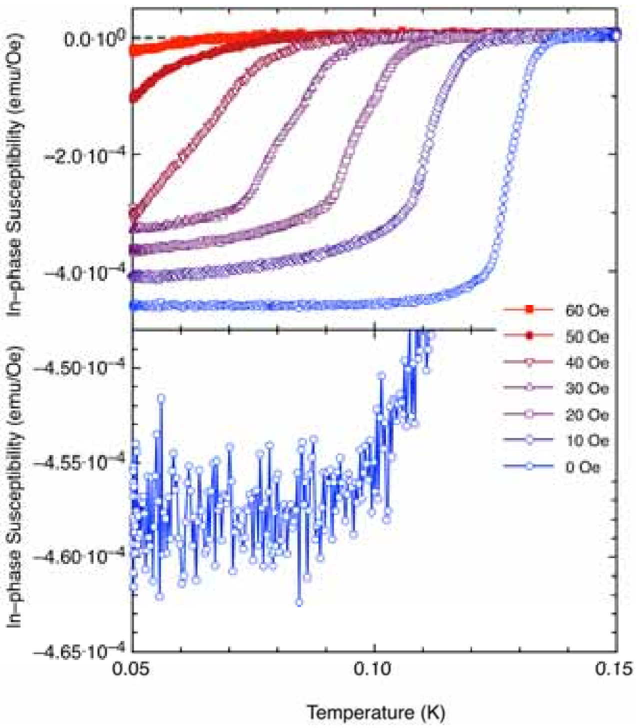

Fig. 5. In-phase susceptibility for the $Ir_{1-x}Ru_x$ sample measured using an AC excitation of 10 mOe and a frequency of 10 kHz for various DC background fields. The lower graph highlights the noise level for the zero field data.

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References

[1] L. Yin, J. S. Xia, N. S. Sullivan, V. S. Zapf, and A. Paduan-Filho, "Magnetic Susceptibility Measurements at Ultra-low Temperatures," *J. Low Temp. Phys.*, vol 158, no. 3, pp. 710–715, 2009.
[2] M. A. Schmidt, D. M. Silevitch, N. Woo, and T. F. Rosenbaum, "Sub-Kelvin ac magnetic susceptometry," *Rev. Sci. Instr.*, vol 84, no. 1, p. 013901, 2013.
[3] http://qdusa.com/sitedocs/productBrochures/dr12.pdf, Quantum Design, Inc.
[4] http://www.qdusa.com/products/dynacool.html, Quantum Design, Inc.
[5] http://www.ti.com/product/TMS320VC5510A, Texas Instruments, Inc.
[6] M. C. Steele and R. A. Hein, "Superconductivity of Titanium," *Phys. Rev.*, vol 92, no. 2, pp. 243–247, 1953.