Measuring Sr_2RuO_4 down to 0.5 K with a Commercial SQUID Magnetometer Combined with ^{3}He Refrigeration

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Abstract

To perform high-sensitivity, absolute magnetization measurements below 2 K, we developed a very slim ^{3}He cryostat that can be inserted into the sample space of a de facto standard, commercial SQUID magnetometer. We were able to measure the magnetic moment of the order of 10^{-6} emu down to 0.5 K. It features an easy operation and fully-automated data acquisition. We successfully observed the 1.5-K superconducting transition of a Sr_2RuO_4 single crystal.

Key words: Sr_2RuO_4, magnetization, SQUID, ^{3}He
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In recent years the discoveries of some interesting low-T_c oxide superconductors (Sr_2RuO_4 [1] and $Cd_2Re_2O_7$ [2]), heavy-fermion superconductors [3], and organic superconductors (ET salts [4], etc.) and ferromagnets (*p*-NPNN [5]) have increased demands for low-temperature magnetization measurements.

Magnetometers utilizing SQUID technology are widely used as a powerful tool to investigate superconductivity, ferromagnetism, antiferromagnetism, paramagnetism, and other magnetic phenomena. In most cases commercial magnetometers have been used with only a very few exceptions [6], and relying on those commercial products, we have not been able to measure magnetization below 1.8 K. This has required us to build a homemade apparatus for revealing the superconducting transition of, for example, Sr_2RuO_4 by DC magnetization measurements. If we could develop a ^{3}He refrigeration system that can be easily installed into commercial SQUID magnetometers, it would be quite useful for measuring not only Sr_2RuO_4 but also other unconventional superconductors and exotic magnets.

We have recently succeeded in measuring magnetization down to 0.5 K by integrating ^{3}He-refrigeration technique into the MPMS from Quantum Design, Inc. We were able to observe the 1.5-K superconducting transition of Sr_2RuO_4 easily by an MPMS, using a millimeter-sized crystal.

The ^{3}He cryostat consists of a sample rod, a main pipe, bellows, and a box. A sample and a thermometer plus a heater for temperature control are attached to the lower end of the sample rod.

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The main pipe is a liquid-^{3}He container with a vacuum jacket at the bottom end so that the liquefied ^{3}He is thermally insulated from the outside and endures sufficiently long. The main pipe is slim enough to fit into the sample space (the inner diameter = 9 mm) of an MPMS.

After loading the sample rod from the top, the main pipe is suspended at its neck in the sample space of the MPMS. The pipe has to be scanned, *i.e.* moved up and down, by an RSO (Reciprocating Sample Oscillation) servo motor unit. Therefore, the bellows is used to interconnect between the pipe and the ^{3}He gas handling system.

The box covers all the three parts above, in order to keep the MPMS sample space sealed and to maintain the pressure difference between inside and outside of the bellows minimal. The latter ensures a light movement of the bellows.

The usage is quite simple. After warming up the MPMS sample space to 300 K, it is vented by helium gas and the box, through its bottom pipe, is connected to the top opening of the RSO unit. Then the main pipe with the sample rod inside is inserted into the MPMS and the bellows is connected to the main pipe. Connectors of electrical wiring are also mated. After the lids of the box have been secured, the ^{3}He cryostat is evacuated via the bellows and now the system is ready to be cooled down.

When the MPMS temperature has gone down to around 4.2 K, the auxiliary rotary pump is started to maintain 1.6 K, at which temperature ^{3}He gas is condensed. When the condensation is complete, the liquefied ^{3}He is pumped and the base temperature of 0.5 K is reached finally.

The base temperature lasts for ten hours as long as no heater power is applied. Just before the liquid ^{3}He has dried up, the very minimum of 0.43 K is achieved. The temperature rise during the scan is not higher than a few millikelvin.

^{3}He pumping speed is continuously controlled by a motor-driven, metering needle valve. This, together with GUI-based measurement software, enables a fully-automated data acquisition.

Sensitivity of 10^{-6} emu in magnetic moment is readily achieved after background subtraction.

The measurement result of a Sr_2RuO_4 single crystal is shown in Fig. 1. The planar sample, grown by a floating-zone method, weighed 3.23 mg and was $2 \times 1.2\,\mathrm{mm}^2$ in area, 0.25 mm in thickness. Magnetic field of 2 Oe was applied perpendicular to the plane. We see a large diamagnetic signal due to superconducting transition and the critical temperature is near the optimum (1.5 K). If we crudely approximate the shape of the sample by an ellipsoid of the same spans, the perfect diamagnetism corresponds to $\chi = -0.36\,\mathrm{emu/cm}^3$. Therefore, we may conclude that our sample is still not perfectly diamagnetic. This may be consistent with the fact that the diamagnetic susceptibility under field-cooling is greatly suppressed, indicating a strong pinning effect. We speculate that a secondary phase (*e.g.* Ru metal precipitation) may have acted as pinning centers.

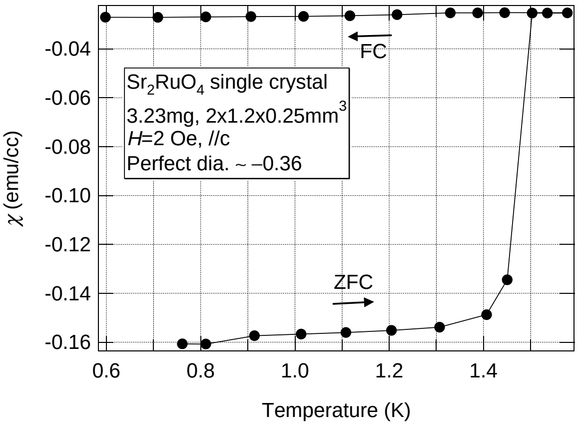

Fig. 1. The temperature dependence of dimensionless magnetic susceptibility of Sr_2RuO_4.

In the present, measurement sensitivity is limited by the background signal due to the moving cryostat. We are working on decreasing the background by making it out of a novel material.

References

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