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Design and Construction of a top loading dilution refrigerator probe for a superconducting quantum interference device DC magnetometer

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Abstract. A commercially available SQUID (Superconducting QUantum Interference Device) DC magnetometer is often limited by its relatively high temperature (≥ 1.9 K) and low magnetic field (≤ 7 T) operating environment. The need for the lower temperature and higher field DC magnetization measurements keeps growing as more materials show interesting physical phenomena with relevant energy scales that require millikelvin temperatures. To meet these needs we have developed a SQUID DC magnetometer which operates in the top loading dilution refrigerator of a 16 T superconducting magnet. An essential part of this low temperature and high field SQUID magnetometer is a specialized probe which can adapt the SQUID electronics and low friction mechanical sample shaft. The details of magnetometer probe and preliminary testing results are described in this paper.

1. Probe Design Consideration

The main parts of a SQUID DC magnetometer consist of SQUID electronics [1], superconducting detection coil and a mechanical shaft that moves a magnetic sample to induce detectable flux change. In constructing the magnetometer, each part presents its own challenge. For example, the nature of superconductor in SQUID circuit requires good magnetic shielding and low temperatures to keep the entire circuit in the superconducting state. The superconducting detection coil, which is usually wound as a second-order gradiometer [2] to exclude external magnetic field changes, has similar field and temperature limitations. For low temperature applications, the mechanical shaft, which allows the sample to be driven up and down through the pickup coil, must have very low friction to avoid heating. For effective magnetic shielding and temperature stability of the SQUID circuit, both passive and active shielding methods using a niobium can and a compensation coil were considered at the location of 1 K pot. In addition, providing effective heat exchange to the sample is much easier in a sample in liquid environment than sample in vacuum. To reflect all the design considerations above and obtain the practical turn-around time for sample change, a top loading dilution refrigerator with a liquid ^3He-^4He mixture in sample space was chosen as a platform cryostat and all the parts for magnetometer including SQUID circuits were mounted on a removable probe as schematically shown in Figure 1.

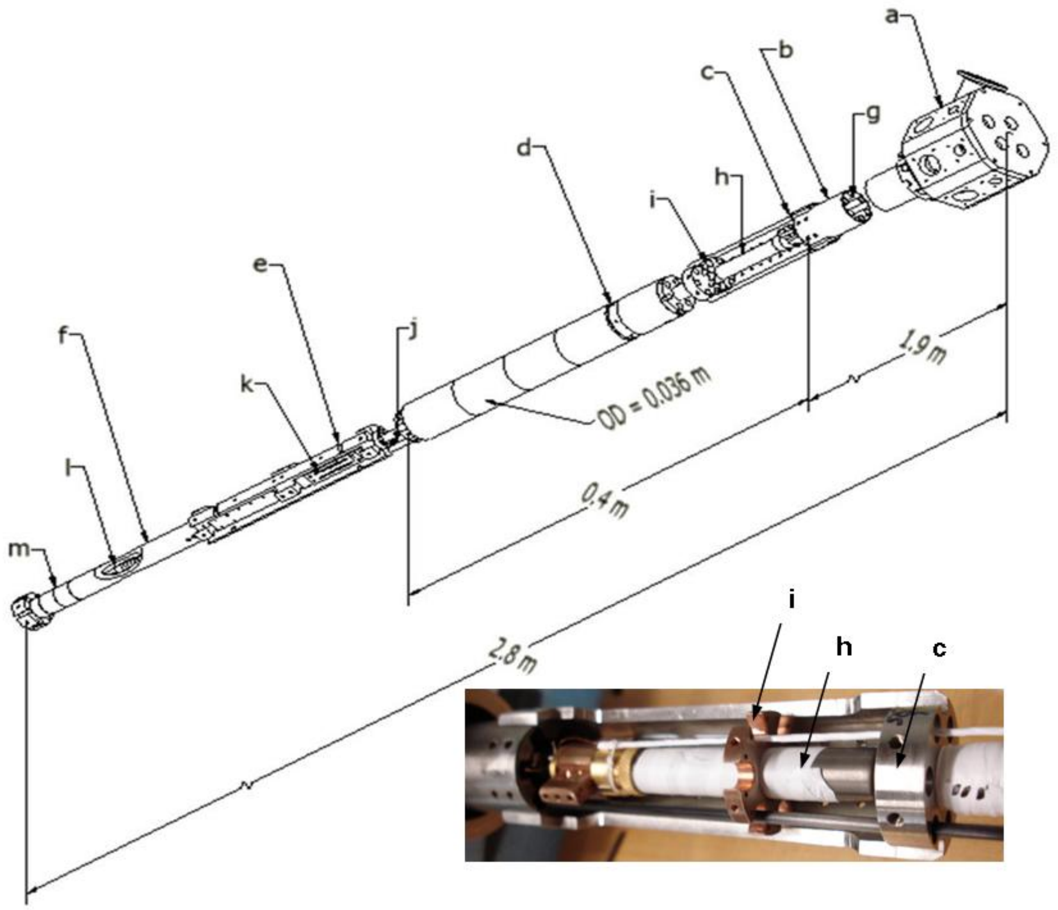

Figure 1. Schematics and picture of top loading dilution refrigerator probe for SQUID magnetometer. The parts include **a**. probe head, **b**. body tube, **c**. 1 K pot cavity, **d**. fixed displacer, **e**. adjustable displacer, **f**. detection coil former, **g**. wire guiding tube, **h**. SQUID electronics, **i**. copper flange, **j**. mechanical shaft, **k**. SIP socket, **l**. sample rod, and **m**. detection coil.

2. Probe Construction and Test Result

The probe can be divided into 6 main parts; probe head, body tube, 1K pot cavity, fixed displacer, adjustable displacer, and detection coil former. The probe head (Figure 1**a**) has hermetically sealed electrical connectors and a mechanical linear feed through (LFT) to move sample shaft and is made of 6061 aluminum to reduce overall weight. Since the total length of the probe is longer than 2.5 m whereas the diameter of the body tube is only about 36 mm, the weight of probe becomes an issue when handled by one person. A long body tube (Figure 1**b**) was made out of titanium and a titanium O-ring flange was welded into the body tube to provide the vacuum connection between the body tube and the probe head. The interior of the tube also contains 6 wire guide tubes and baffles. A stainless steel (SS) 1 K pot cavity is made in two pieces; base and cap. The base part of this SS cavity, shaped as a half open cylinder, plays a role of connecting the body tube to the fixed displacer in addition to providing housing for SQUID, compensation coil and other thermometry. The open cylinder geometry was adapted to allow an easy access to the SQUID and other electronics. When the probe is fully loaded to the cryostat, this cavity sits on the temperature controlled 1 K pot, and provides thermal link between the 1 K pot and SQUID with help of a copper flange (Figure 1**i**) that also secures SQUID electronics body to the cavity.

When the probe is not loaded, the top loading dilution refrigerator is equivalent to the dilution refrigerator with a hole in the mixing chamber top and thus cannot function as a dilution refrigerator. It makes a complete dilution unit only when the fixed displacer in the probe plugs the mixing chamber top and displaces the mixture up into the heat exchangers and still. The fixed displacer (L = 280 mm, OD = 36 mm) is made out of G-10 garolite rod (Figure 1**d**) and has radially situated six feed holes for electric wires and the mechanical shaft. Since this fixed displacer acts as a long plug to isolate the mixing chamber from other part of the dilution refrigerator, sealing the six feed holes from ^{3}He-^{4}He mixture are somewhat critical. The holes for electric wires are sealed by filling Apiezon-N grease into the holes around the electric wires. However, the grease filling method could not be used to seal the feed hole for the mechanical shaft because the shaft has to move up and down to induce the magnetic signal from the sample while the grease would act as glue at low temperatures. For this problem the shaft was made with a diameter which snugly fits into the feed hole to provide a reasonable sealing and minimize the thermal heating due to shaft movement. The mechanical shaft (Figure 1**j**) was made out of two pieces of carbon fiber tube (OD = 0.32 mm). The two tubes are connected using G-10 tube to allow the length adjustment. The section of the carbon fiber tube where it passes one of the holes in the fixed displacer was covered by a G-10 tube, whose diameter fits snugly on the displacer's hole. To reduce the friction in the inner surface of the displacer's hole, a thin wall SS tube was inserted inside the hole.

The adjustable displacer (Figure 1**e**) is made out of G-10 garolite and electric SIP connectors (Figure 1**k**) are mounted to use for electrical measurements such as thermometers. One of the main purposes of this displacer is to adjust the amount of ^{3}He-^{4}He mixture at the mixing chamber by replacing the mixture with adjustable volume of the displacer. In our probe, the adjustable displacer also provides a straight-motion guide to the mechanical shaft before the shaft enters into the detection coil area (Figure 1**f**). For test purposes a NbTi superconducting wire was wound on a G-10 tube (OD = 1.9 cm) to form a gradiometer (Figure 2) and the superconducting wires coming out of the coil are tightly twisted and secured to the G-10 rod, which extends all the way to the 1 K pot cavity where the wires enter into the SQUID electronics.

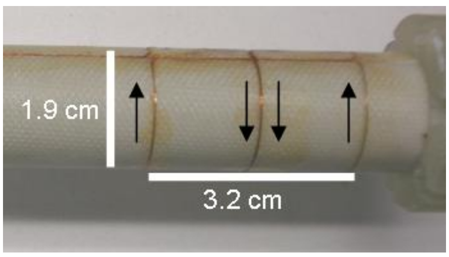

Figure 2. Second-order gradiometer detection coil. NbTi superconducting wire was used to form a total of four turns of detection loops.

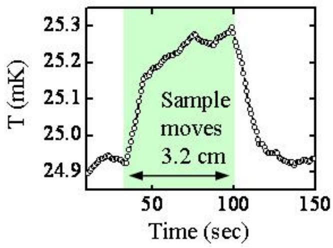

Figure 3. Heating effect due to sample moving. The sample was continuously moved for 3.2 cm with speed of 3 cm/min.

A complete probe without any electric components and mechanical shaft was cooled down in a commercial top loading dilution refrigerator. An activated ^{60}Co single crystal was mounted at the end of probe for the base temperature measurement [3]. The base temperature of the bare probe was ~ 7.5 mK. When all the electric components and necessary parts including mechanical shaft were equipped into the bare probe, the base temperature became ~ 15 mK. Near this base temperature at 25 mK, the heating effect due to the mechanical shaft moving was tested by continuously moving the shaft for ~ 3.2 cm with speed of 3 cm/min. The moving distance, 3.2 cm is the similar the height of detection coil. Figure 3 shows the result of temperature changing when the sample is moving at 24.9 mK. The temperature increased to 25.3 mK during the movement of mechanical shaft and shows plausible use of this probe for low temperature application. As a test sample 1.9 mg of the paramagnetic salt Gadolinium Sulfate ($Gd_2(SO_4)_3{\cdot}8H_2O$) in powder form was mounted using a commercial straw. Figure 4 shows raw SQUID signals as the test sample moves through the detection coil at roughly 500 mK as measured at the top of the mixing chamber. At this temperature, the moment of the test sample is expected to be in the order of ~ 10^{-4} emu·G at 10 G and increases as the external magnetic field increases. As shown in Figure 4, the overall SQUID amplitude increases as expected as the magnetic field increases and the noise level was less than the size of the data point. The shape of the signal however, did not turn out to be symmetric around the center of the detection coil. This imbalance of signal is most likely due to the imbalance of coil winding or non homogenous temperature profile along the 3.9 cm of coil axis and requires future improvement. The SQUID signal was able to detect the test sample moment down to 50 mK, see Figure 5.

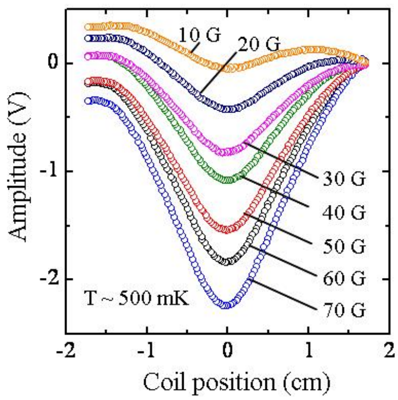

Figure 4. Raw SQUID signals at different magnetic field. The signals were detected as the sample moves through the second-order gradiometer NbTi detection coil.

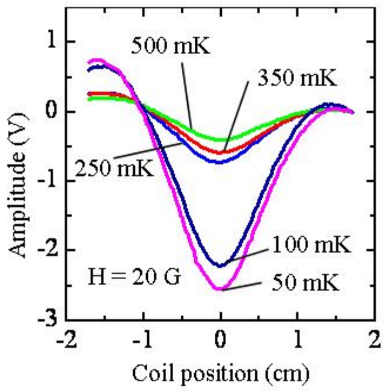

Figure 5. Raw SQUID signals at different temperatures. The signals were detected as the test sample moves through the second-order gradiometer NbTi detection coil.

3. Conclusion

As a part of low temperature and high magnetic field SQUID DC magnetometer project, a specialized top loading probe was designed and constructed. The probe was designed such that all the magnetometer components including SQUID electronics, detection coil, and sample moving shaft are placed within the probe. The probe was tested in a commercial top loading dilution refrigerator and the result shows that the base temperature at 25 mK increased ~ 1.6 % when the sample was moving about 3.2 cm with the speed of 3 cm/min. The moment of the test sample was successfully detected down to 50 mK but the imbalance of coil winding needs future improvement.

Acknowledgements

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