Abstract. A new probe has been developed that allows for both optical irradiation and uniaxial rotation, all in the low temperature environment of a commercial superconducting quantum interference device (SQUID) magnetometer. As part of the design process, various materials were investigated and characterized for their low temperature structural and magnetic properties, including nylon, Vespel, Delrin, Spiderwire monofilament, and PowerPro braided microfilament. Using this information, a prototype was built and operated. Characteristics of the probe will be presented along with a summary of the low temperature (T ≥ 2 K) and high magnetic field (H ≤ 7 T) properties of the construction materials.

1. Introduction

Sample rotation is a valuable tool to measure the angular dependence of magnetization [1]. Additionally, there is an ongoing research effort to study materials that show changes in magnetization with applied photonic excitation. Specifically, samples showing persistent photoinduced magnetization as well as magnetic anisotropy have been reported [2, 3]. Therefore, an experimental setup that is able to measure magnetization down to low temperatures, while allowing simultaneous in situ sample irradiation and rotation, would be beneficial.

There are a few inherent difficulties one has to consider when configuring such a system. A probe that is suitable for use with commercial superconducting quantum interference device (SQUID) magnetometers was designed because signals can be quite small, i.e. 10⁻⁶ emu and less. Although using a commercial setup is advantageous, it puts significant size and weight constraints on the probe and, most importantly, on the sample space. Also, due to the small signals involved, a minimization of the background signal from the probe is sought. Finally, care has to be taken that the system can operate at temperatures below the boiling point of ⁴He. Thermal contraction and expansion of parts must be taken into account as well the need to keep parts movable while minimizing heating. As an important part of developing the experimental setup, possible construction materials have been characterized. A probe that meets the desired specifications has been built and tested.

2. Probe Specifications and Design

The probe setup is shown in Fig. 1. Sample rotation is uniaxial about an axis perpendicular to the applied magnetic field. Rotation is done with a line connecting the low temperature sample holder to a cylinder at ambient temperatures, with an additional line for resetting. Operation
can be manual or automated by using a computer controlled stepper motor. A fiber optic cable allows for irradiation.

**Figure 1.** There are three main sections of the probe: (i) a low temperature end that sits within the SQUID coils and magnet bore and houses the rotating sample stage, (ii) a shaft that seats within an o-ring for movement of the probe through the SQUID coils that connects the high and low temperature spaces, and (iii) a head that contains the active rotation element and other probe elements. Specifically, the important design elements are: (a) the drive shaft, (b) a computer controlled stepper motor, (c) a fiber optic cable, (d) lines connecting the drive shaft to the sample stage, (e) the SQUID coils, and (f) the sample rotation stage.

The head of the probe must be light enough to accommodate the servo that translates the entire probe vertically to move the sample through the SQUID coils. The housing is aluminum because of its density, strength, and machinability. The drive shaft is aluminum with a Teflon bushing. Vacuum is achieved by the use of rubber o-rings for the drive shaft seal and for an additional access port on top. Epoxy seals the clear holes made for the fiber optics and the connection between the head and the shaft.

The shaft of the probe is constrained to be \( \approx 0.12 \) in \((0.3048 \text{ cm})\) OD and \( \approx 0.10 \) in \((0.254 \text{ cm})\) ID for use in a commercial QD-MPMS SQUID magnetometer, allowing the commercial shaft seal assembly to be used. Most of the shaft is stainless steel for strength, but the bottom portion is quantalloy, a silicon copper alloy, to minimize the background signal. The shaft is attached to the low temperature end with epoxy. For the drive lines, fishing materials were considered because they are non-metallic, thin, and strong. To decide between monofilament or braided lines, two exemplary products were studied: Spiderwire (www.spiderwire.com) 8 lb. monofilament (mono-line) and Spectra PowerPro (www.powerpro.com) 15 lb. (braided-line). The braided-line was chosen for its larger Young’s modulus, since stretching of the lines can lead to errors in sample angles. Although there is a larger magnetic signal associated with the braided-line, the amount near the SQUID coils is only \( \approx 10 \) mg. The magnetic and mechanical properties of the lines are summarized in Section 3.

For the low temperature end of the probe, the main pieces are a yoke and a rotatable sample stage. Dupont’s black acetal polymer, Delrin, was chosen for its strength and small magnetic signal. The yoke is long enough to be locally symmetric with respect to translations vertically through the detector coils, minimizing its flux contributions. The rotation stage is a hollow cylinder that has plastic on plastic bearings, with all but \( \approx 90^\circ \) open for accessibility during irradiation. The drive strings are attached to the rotation stage via nylon set screws. Our results for the magnetic properties of black Delrin, brown Vespel polyimide by Dupont, and white nylon are summarized in Section 3.
3. Probe Material Properties

During the design process, several candidate materials were investigated. Magnetic properties were measured in a Quantum Design MPMS-XL SQUID magnetometer. All samples were mounted in uniform straws using press fits, so no background signal was subtracted. Annealing of the Delrin was attempted in case additional magnetism was coming from free bonds [4], but no obvious change in the susceptibility was observed. Temperature sweeps were done at 10 mT, 100 mT, and 1 T between 2 K and 300 K. The susceptibilities fit well to a semi-empirical formula:

\[ \chi = \frac{C}{T} + L \cdot T + D, \]

and the results are given in Table 1. Field sweeps were done at 2 K, 10 K, and 100 K for fields up to 7 T, Fig. 2, and remnant magnetization after sweeping to 7 T are also reported, Table 1. Where applicable, our results can be compared with other reports [5 – 8].

| Table 1. Summary of magnetic response for candidate probe materials. Remnant magnetizations (\(M_{\text{rem}}\)) are in emuG/gram, C is emuK/gram, L emu/gramK and D is emu/gram. |
|---|---|---|---|---|---|
| \(T\) (K) | \(H\) (T) | mono-line | braided-line | Vespel | Delrin | nylon |
| C | 2 – 300 | 1e-2 | 9.1068e-7 | 1.0671e-5 | 5.3498e-7 | 7.2737e-7 | 1.5198e-7 |
| L | 2 – 300 | 1e-2 | -4.2817e-10 | -4.1188e-9 | -5.9675e-12 | 8.7623e-11 | -8.5243e-11 |
| D | 2 – 300 | 1e-2 | -1.3229e-7 | 2.5228e-6 | 8.2524e-7 | 8.2636e-8 | -3.3978e-7 |
| C | 2 – 300 | 1e-1 | 1.8156e-7 | 6.9705e-6 | 5.1352e-7 | 8.1238e-8 | 4.6009e-7 |
| L | 2 – 300 | 1e-1 | -1.0635e-10 | -2.6259e-10 | 1.1519e-10 | -6.5596e-12 | -1.1522e-11 |
| D | 2 – 300 | 1e-1 | -4.6009e-7 | 3.0526e-6 | 6.0250e-8 | -3.8064e-7 | -3.3978e-7 |
| C | 2 – 300 | 1 | 1.6334e-7 | 6.2978e-6 | 4.8105e-7 | 2.3244e-7 | 1.2383e-7 |
| L | 2 – 300 | 1 | -9.9114e-11 | -5.5029e-10 | -8.5564e-11 | -3.8625e-11 | -1.5503e-11 |
| D | 2 – 300 | 1 | -6.8050e-7 | -4.6172e-7 | -3.8356e-7 | -5.0799e-7 | -3.8064e-7 |
| \(M_{\text{rem}}\) | 2 | \(7 \rightarrow 0\) | 5.4541e-5 | 2.3989e-4 | 1.5667e-4 | 4.7316e-5 | 6.2121e-6 |
| \(M_{\text{rem}}\) | 10 | \(7 \rightarrow 0\) | 4.3157e-5 | 2.2723e-4 | 1.0041e-4 | 2.1461e-5 | 1.3669e-5 |
| \(M_{\text{rem}}\) | 100 | \(7 \rightarrow 0\) | -1.5032e-5 | 1.0743e-4 | 3.3839e-6 | 6.8093e-6 | 4.1010e-6 |

Figure 2. Magnetization as a function of field measured at 2 K (green △), 10 K (red ◆) and 100 K (black ⊔) for Vespel (a), Delrin (b), nylon (c), mono-line (d), and braided-line (e).
Additionally, for the drive lines, some mechanical properties were investigated. Force constants were examined at room temperature to test the line deformation in response to an applied force. The mono-line has a diameter of 0.010 in (0.254 mm) and a measured Young’s modulus of 1.4 Gpa, which is lower than 2.3 Gpa reported to us by Berkley Fishing. The braided-line has a diameter of 0.007 in (0.1778 mm) and a measured Young’s modulus of 68 Gpa, which is close to the 73 – 124 Gpa range reported by Honeywell for different Spectra fibers.

4. Operation

To test the probe, pieces of magnetite with the magnetic axis aligned perpendicular to the axis of rotation was measured without any applied field, Fig. 4. Sample irradiation was tested using thin films of $\text{A}_j\text{Co}_k\text{[Fe(CN)]}_6\cdot n\text{H}_2\text{O}$ oriented parallel to the applied field, Fig. 5.

Figure 3. Magnetization versus rotation angle for two different magnetite samples.

Figure 4. Magnetization versus time for thin films of $\text{A}_j\text{Co}_k\text{[Fe(CN)]}_6\cdot n\text{H}_2\text{O}$.

5. Conclusions

We have demonstrated the ability to photoirradiate and rotate samples in situ while using the convenient setup of a commercial magnetometer, a combination of features previously unreported. Probe materials and design have been presented. In the future, more careful etching of the materials may help reduce possible magnetic impurities introduced during machining.

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