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Low Cost High Sensitivity Superconducting Magnetometers and Gradiometers - AF171-017

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Final Technical Report

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Final Technical Report

Introduction

Report Developed under SBIR purchase order FA9550-19-C-0001. This final report describes research and development activities undertaken during the period between March 15th, 2019 and March 14th, 2022.

This report summarizes following sections of the project.

- Project objectives
- Hardware development
- Software integration
- Demonstration
- Future development

1. Project Objectives

It was our intent to develop a new magnetic sensing capability using the high T_c SQUID sensors developed at UC Riverside and to non-destructively evaluate samples and devices in-situ in a controlled cryogenic environment of the Physical Property Measurement (PPMS) family of instruments [1,2,3].

Figure 1 shows a complete set-up of the high T_c SQUID system in Quantum Design's Dynacool system, which is a cryogen free PPMS. Its main components include a cryogenic probe (Optical Multifunction Probe; OMFP [4]) inside the dewar, an optical sample imaging system (Camera), a cartesian positioning system and new SQUID electronics. The motorized X-Y-Z cryogenically compatible piezo positioning system allows the magnetic sample under test to be moved near the SQUID while being visualized under the camera.

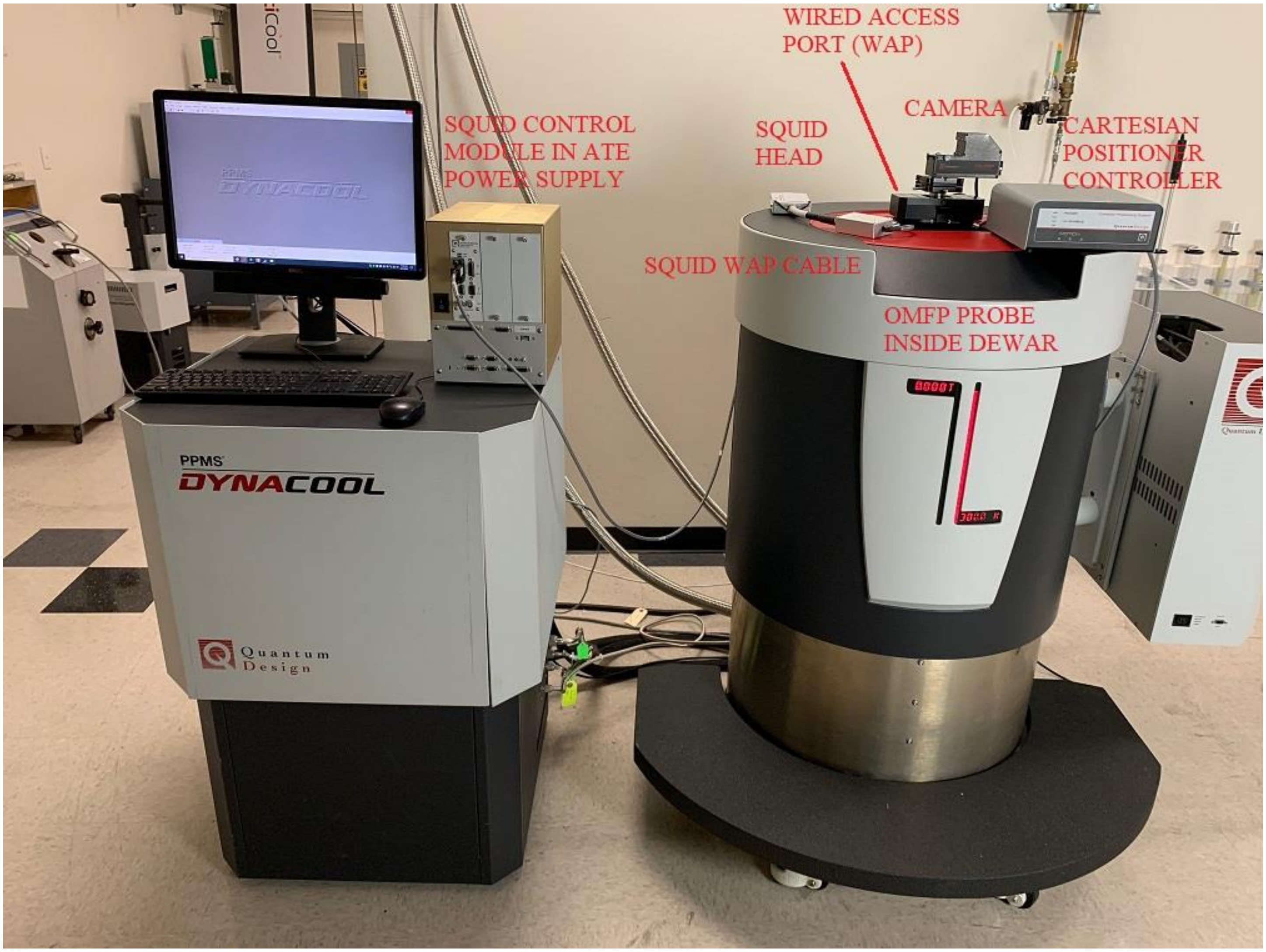


Figure 1. High T_c SQUID System Set-up in QD’s Dynacool System, where a cryogenic probe (OMFP)is inside the sample space in the dewar.

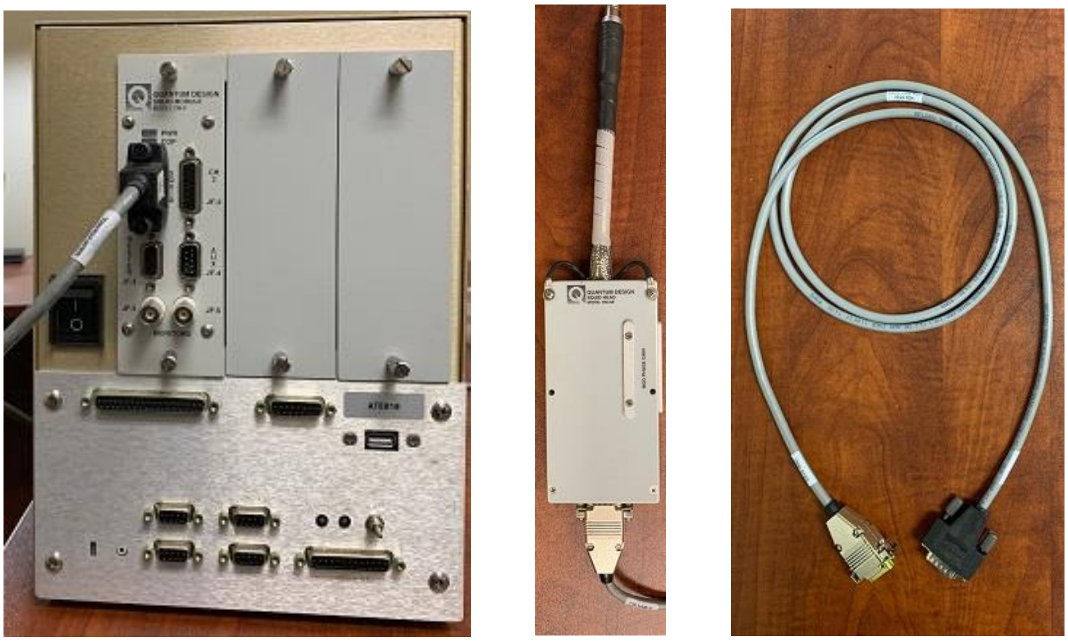
The cryogenic portion of the OMFP probe is shown in Figure 2, where an optical window is located on the left and a probe capsule on the right end. In the middle section 4 carbon fiber tubes support the probe and carry electrical wires inside the tube with 6 relay lenses precisely positioned for an image transmission. Inside the mu-metal shielded capsule there are important components such as an objective lens, a SQUID sensor, a sample holder standoff, and a 3-axis piezo stack for controlling the distance between the SQUID and the sample.

Figure 2. Cryogenic OMFP probe for high T_c SQUID detection.

2. Hardware development

SQUID Electronics

The SQUID electronics consists of a SQUID control module, a SQUID Head, and a Power supply (ATE Chassis Assembly). Originally developed for Quantum Design's low temperature Nb SQUID, these components comprised the starting point for the final electronics developed for this contract. The SQUID Head, containing the preamplifier circuitry for the SQUID signal, was extensively modified to operate the high T_c SQUID due to the differences in inductances of modulation and feedback coils of these sensors when compared to traditional Nb SQUIDs.



(a) (b) (c)

Figure 2. SQUID Electronics consisting of (a) SQUID control module mounted in ATE Power Supply, (b) SQUID Head, and (c) SQUID control cable.

The SQUID detection system consists of the followings.

1. A set of superconducting detection coils inductively coupled or directly coupled to a magnetically shielded DC SQUID.
2. A SQUID head that biases the SQUID provides feedback control and signal amplification.
3. A Control Area Network (CAN)-based SQUID control module with a digital signal processor (DSP) performing synchronous AC detection.

The magnetic signal from the sample is detected by a planar pick-up coil and connected either via a flux transformer or directly coupled to the SQUID. In our case we have chosen to use the directly coupled magnetometer design since it has been demonstrated to be the most successful configurations in practical applications [6, 7, 8, 9]. The SQUID signal is amplified in the SQUID Head and processed by Digital Signal Processor (DSP) in the module.

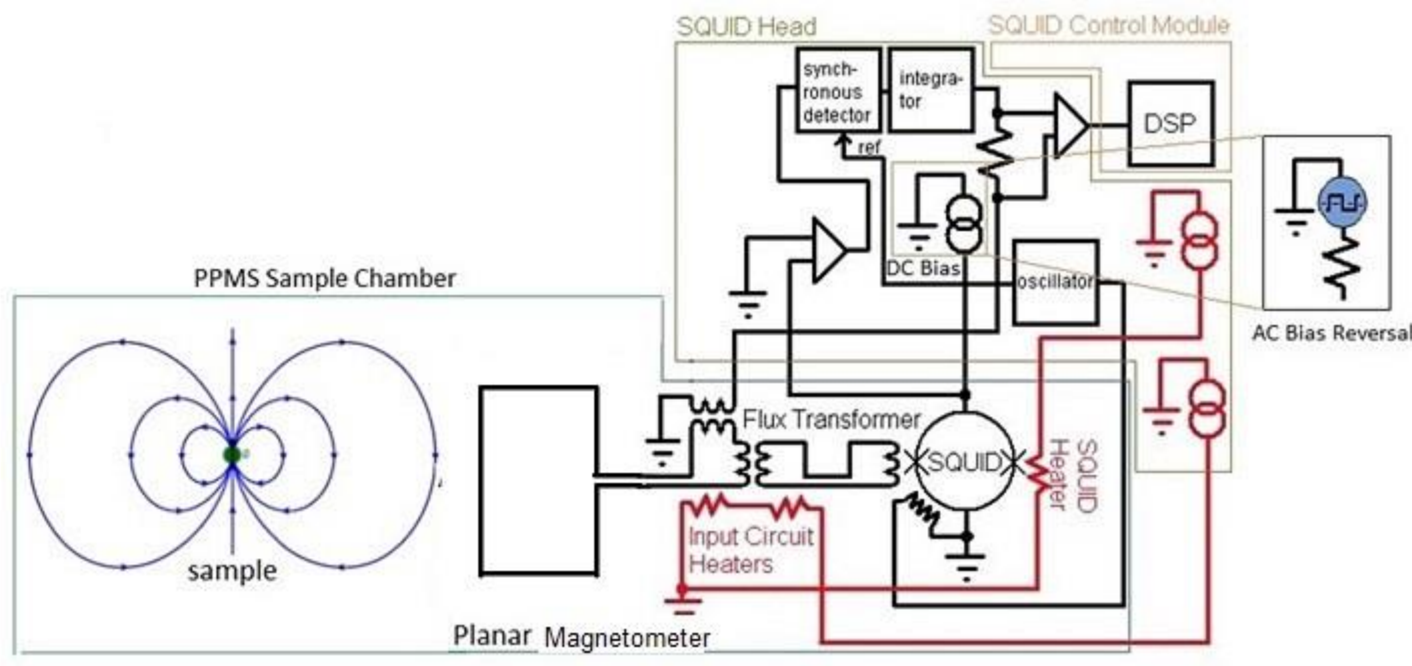


Figure 3. Detection system diagram of the planar SQUID magnetometer in the PPMS sample chamber.

Optical Multifunction Probe (OMFP)

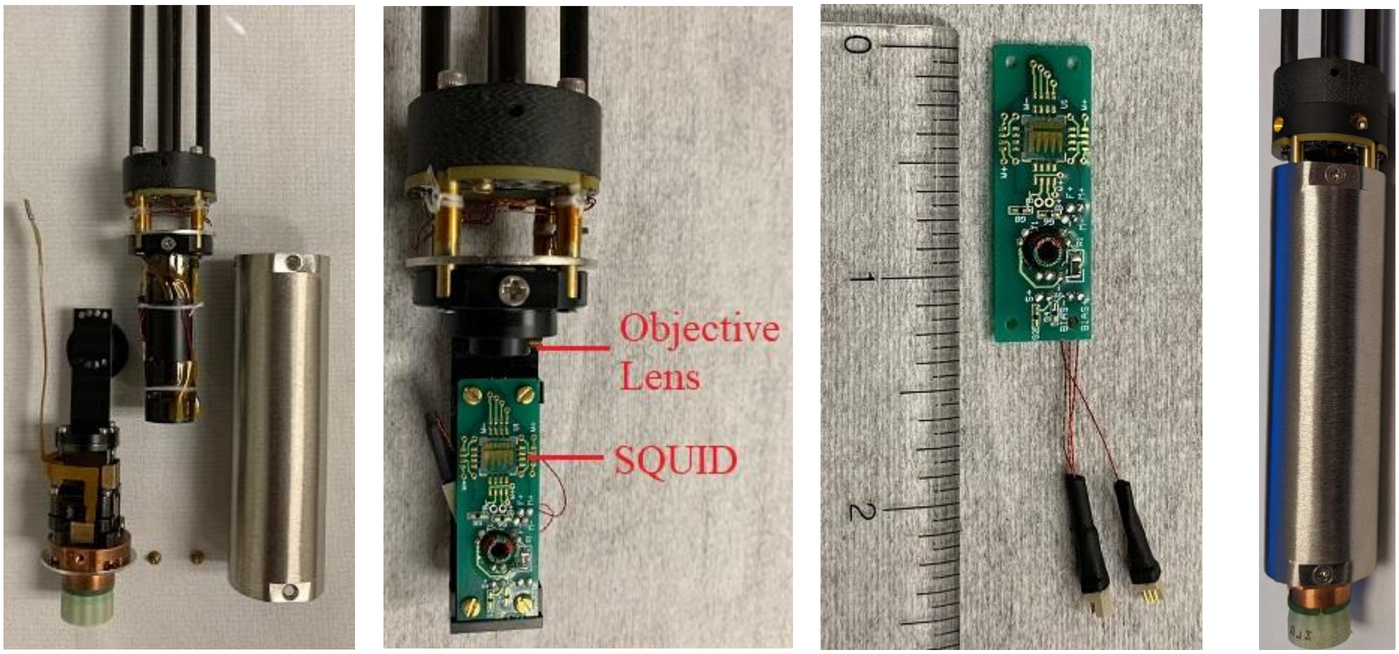
In this program QD's Optical Multifunction Probe (OMFP) was modified to incorporate a high T_c SQUID sensor chip [4]. The OMFP, was originally designed to allow the user gain optical access to experiments within the variable temperature and magnetic field environment of the PPMS family of instruments [1,2]. At the top of the OMFP, a wired access port (WAP) features an axial optical port window. The WAP also provides modular feedthrough connectors that can be configured to allow electrical signals to the sample space. Adjustable optical mounts can be placed along the length of the probe to position relay lenses and objective lens. The capsule at the bottom of the OMFP contains a three-axis piezo stack enabling the sample to be moved in proximity of the stationary SQUID sensor. The capsule was redesigned with new components needed to integrate the high T_c SQUID chip into the OMFP. The SQUID printed circuit board (PCB) was redesigned to accommodate the high T_c SQUID sensor at the right position opposite to a sample. A sample holder made of black Delrin is mounted on top of the three-axis piezo stack.

During the measurements the sample chamber is purged, backfilled with He gas, purged again, and held in a medium vacuum, or to about just below 10 torr at room temperature. During experiments, the sample chamber pressure may drop to 1-2 torr at PPMS's base temperature. Retaining a low pressure of He gas in the chamber enables the SQUID, the sample, and chamber walls to reach a uniform temperature.

Details of the OMFP Probe construction are described below.

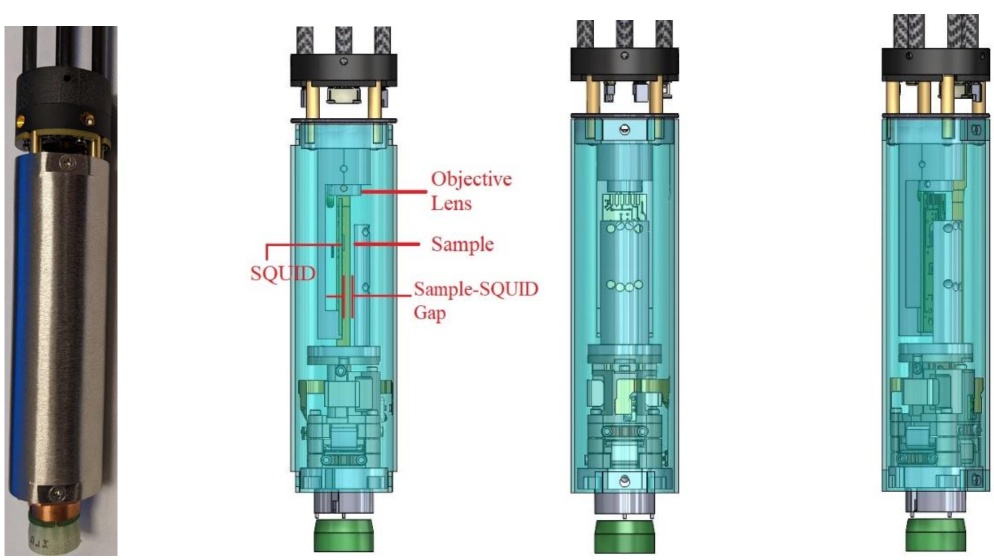
Main hardware design includes:

- Mu-metal shielding top, bottom, and tube
- Mu-metal shield works as a structure
- New top end capsule connecting ring and SQUID holder
- Optical access hole = 0.25"
- Objective lens with 10 mm focal length inside the Mu-metal shield
- New non-metallic sample stage standoff (made of Delrin)
- SQUID is mounted on PCB
- SQUID is 1" away from piezo stack (stainless steel hardware)
- Extending flex circuit cable



(a) (b) (c) (d)

Figure 4. (a) From the left to the right: motorized sample stage including sample standoff made of non-magnetic Delrin, stationary SQUID sensor with transformer carrier and, mu-metal shielding acting as the structure for assembly. (b) SQUID mount on the end of the OMFP with a SQUID and a transformer mounted on a PCB under the objective lens. (c) Detail of SQUID PCB with the SQUID sensor and toroidal transformer. (d) Fully assembled view, where the mu-metal shielding provides the enclosing structure for the assembly.



(a) (b)

Figure 5. (a) Views of the assembled probe capsule. (b) The drawing shows the interior of the mu-metal shield, with relative positions of SQUID PCB and sample stand-off on top of the piezo stack. Note imaging of the SQUID-Sample gap is possible with the camera at top of the probe and controlled by the PPMS software.

Key features of the OMFP-SQUID system:

- Customizable 1″ free-beam access port and internal ½″ mounts along the optical path.
- Modular feedthroughs available for electrical signals into the sample space.
- Optical camera allows for in-situ fine alignment and focus.
- 3-axis piezo-positioning system enables multiple samples or regions of interest to be investigated at one time.
- Complete integration of imaging and positioning with the PPMS MultiVu console software including availability of sequence commands to pre-program experiment scripts.
- Includes a test station for ex situ alignment of optical elements and test of proper electrical connections the sample.

Figure 6. OMFP specifically designed for High T_c SQUID application.

(a) (b)

Figure 7. (a) Securing the OMFP in the alignment station on the left to show the image of the USAF Resolution Target in the PPMS MultiVu application software. (b) USAF Resolution Target imaged on the alignment station, where lines and spaces (1.55 um) of Group 8 Element 3 are visible (enclosed by red oval).

High T_c SQUID

High T_c SQUID sensors were fabricated by helium ion beam direct-write technology developed by Prof. Cybart at University of California, Riverside [5, 6]. Large circuit features such as electrical contacts and micron wide strips of YBCO film were patterned with conventional photolithography. Then samples were transferred to a Zeiss Orion helium ion microscope (Figure 8) and helium ion beam was directly scanned across the YBCO line to make tunnel junctions. Furthermore, helium ion beam with different dosage was utilized to pattern nano-meter scale patterning of YBCO film to define small scale geometry in-situ. Low dose He ion beam was used to fabricate the tunnel junctions, while high dose He ion beam was used to convert superconductors to insulators in a nm scale.

Figure 8. Zeiss Orion Helium Ion Microscope, where Helium ion beam with different dosage was utilized to pattern YBCO film in a nano-meter scale.

Figure 9 shows temperature-dependent resistivity of epitaxial $YBa_2Cu_3O_{7-\delta}$ films as a function of ion-beam-induced damage [10]. At low Ne ion dose the film behaved like a typical metal above the superconducting transition temperature. At higher Ne ion dose the material went through metal-insulator transition.

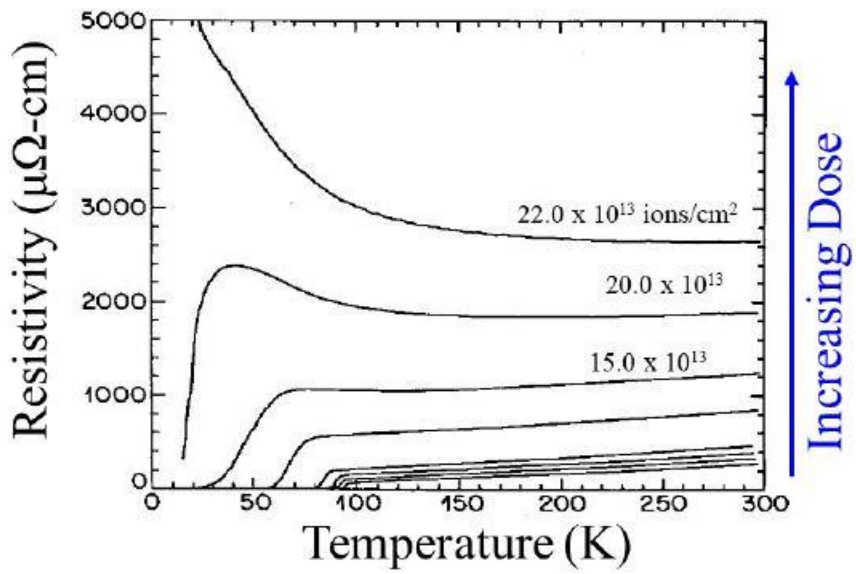


Figure 9. Resistivity as a function of temperature for a single $YBa_2Cu_3O_{7-\delta}$ film. Increasing low Ne ion dose yields weakening superconductivity. The higher Ne ion dose results in metal-insulator transition.

Figure 10 shows the design of 5 mm x 5 mm SQUID chip with 8 directly coupled SQUID magnetometers, where a hexagonal pick-up loop size is 300 μm. White regions are bilayers of YBCO (40 nm) and Au (250 nm), whereas light green areas are a substrate with no films. In dark green areas Au is etched to leave YBCO film, where Josephson junctions and SQUID washer are patterned by helium ion beam direct write technology.

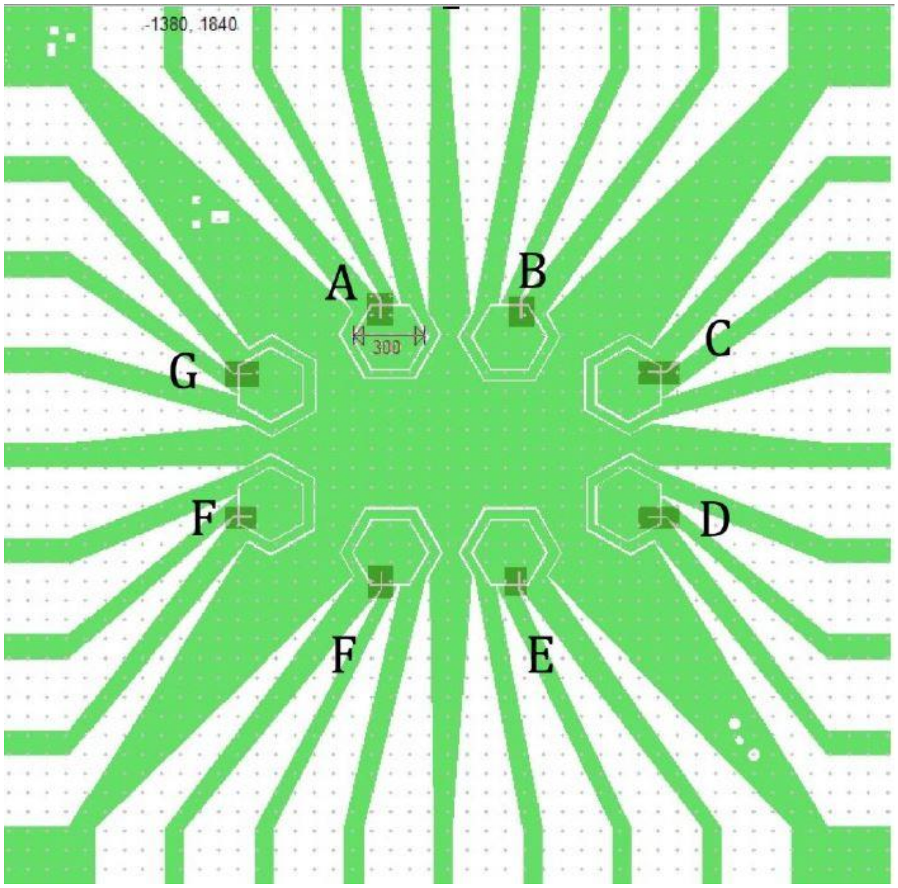
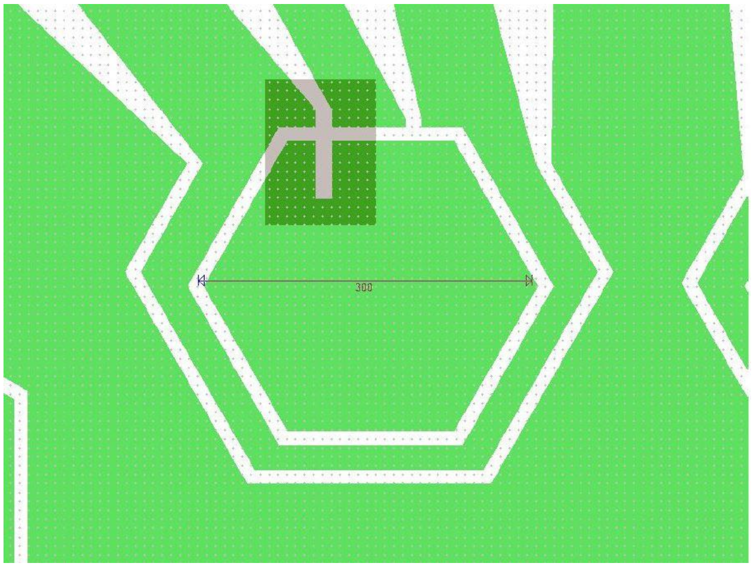
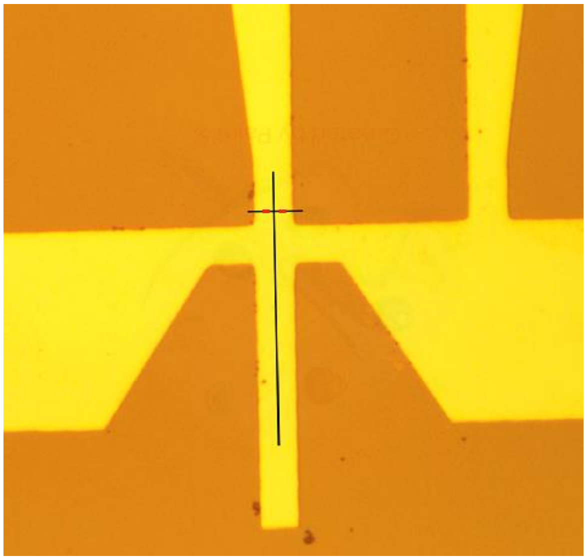


Figure 10. The SQUID chip consists of 8 directly coupled SQUID magnetometers, where white regions are bilayers of YBCO and Au film. Dark green areas are YBCO film only, where SQUID junctions are fabricated. Light green areas are substrate with no films.

One of the directly coupled magnetometer is shown in Figure 11 (a), where a hexagonal pick-up loop is 300 μm in diameter. A loop surrounding the pick-up loop is modulation/feedback loop. A dark green area was etched by KI to remove Au and leave YBCO film. In Figure 11 (b) thin black lines become insulator after the exposure of high dose of He ion beam. Short red lines are two junctions, where the low He ion beam dose is optimized to obtain desirable critical current of the Josephson junctions. The SQUID junctions are 400 nm wide with a tunneling barrier of only 2 nm. The slit width and length of the SQUID washer are 500 nm and 100 μm respectively. Even though the SQUID loop was 500 nm by 100 μm, line flux could penetrate up to twice of the penetration depth.



(a)

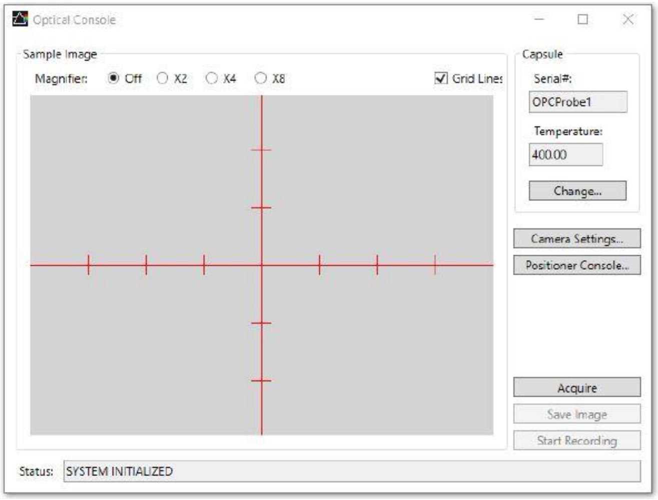


(b)

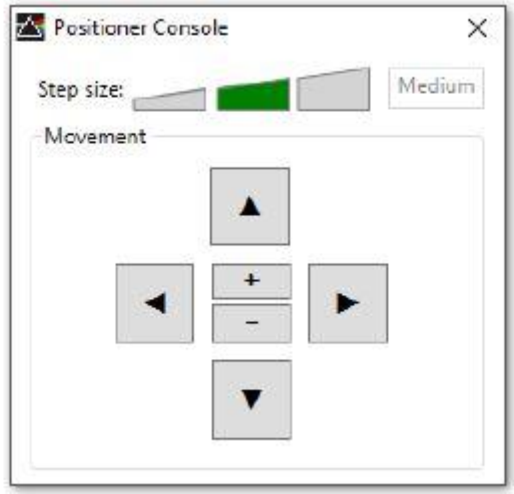
Figure 11. (a) One directly coupled magnetometer is shown. A dark green area, where Au film is etched away by KI solution, is YBCO film. (b) SQUID is patterned, where black areas (SQUID slit) become insulator after the exposure of high dose of He ion beam. Red lines are two junctions, where the low He ion beam dose is optimized to obtain desirable critical current of the junctions.

3. Software integration

The OMFP option Software (SW) was integrated into Quantum Design's MultiVu graphical user interface for the PPMS system. In general, the MultiVu SW can be used by the user to set experimental parameters such as sample chamber Temperature, applied external magnetic field, and background pressure. In addition, the software allows for simultaneously logging various parameters defined by the user and plot them in real time. When the OMFP option is first activated, both Optical Console and the Positioner console can be accessed at any time via the instrument menu. The former window [Figure 12(a)] displays the large sample image reticle where the image is shown once the camera is actively acquiring data. The camera settings can be controlled by setting dialog window [Figure 12(c)]. The latter window displays a simple interface for adjusting the sample position [Figure 12(b)]. The horizontal and vertical position of sample can be adjusted by the arrows pointing in the corresponding directions. The [+] and [-] buttons control movement along the axial direction (which we will call z-direction) which allows the sample to be positioned in focus at the proper focal distance from the objective lens. Once this z-axial position is set it will remain fixed, and the sample moved in the x-y plane to approach the SQUID for magnetic detection. To facilitate careful movement of the sample near the SQUID without collisions, the step size of the motorized sample stage is selected accordingly to aid the movement of the sample: larger steps when the sample is far-away from the SQUID and smaller steps when the sample is closer to the SQUID sensor.



(a)



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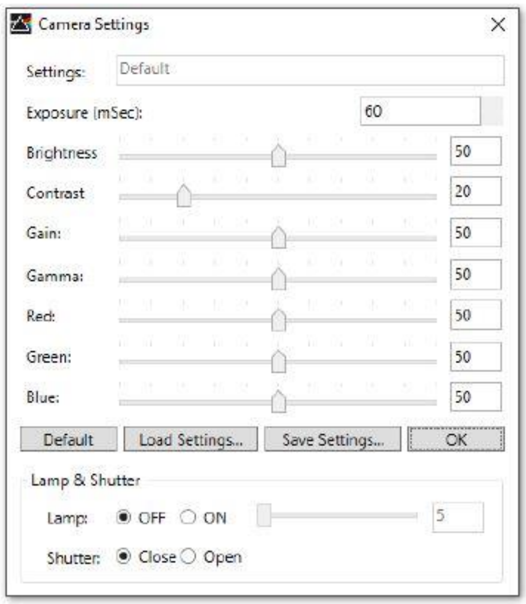


(c)

Figure 12. (a)Optical Console window, (b) Positioner Console window, and (c) Camera Settings dialog window after activating the OMFP Option.

The SQUID control application software is currently a stand-alone and separately activated. The SQUID monitor dialog shows:

- The tune voltage (labelled: "Cyclonic SQUID Eye" for fun) when used in an open loop operation, or the SQUID signal when in a feedback loop operation,
- Selection of Channel 1 or 2,
- A bias display with user defined inputs of Channel 1 or 2,
- Quench commands of either: Transformer only of Transformer and SQUID,
- A SQUID range control (with x1 most sensitive to x1000 least sensitive),
- Operational mode selection (user chooses from normal, reset, and tune),
- DC offset voltage: both coarse and fine adjust, in the normal mode.

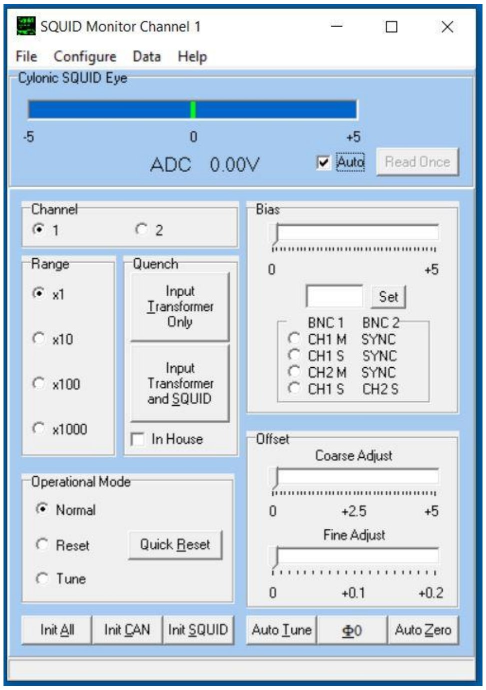
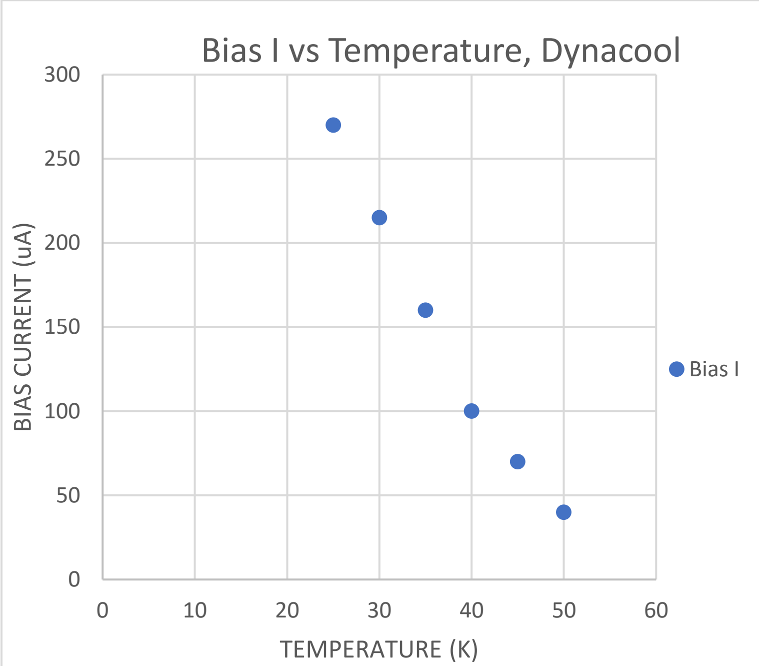


Figure 13. SQUID monitor window to control the SQUID operation.

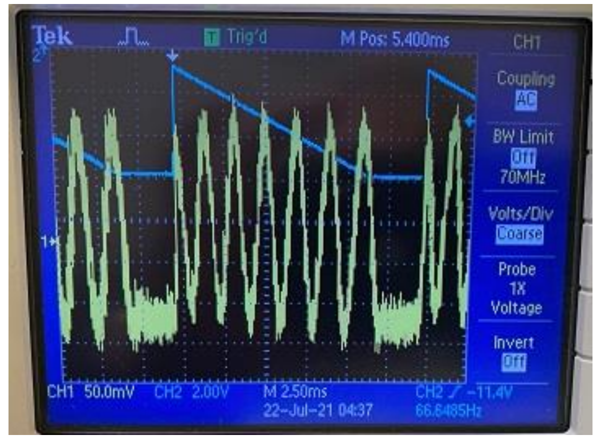
Although not necessary for these experiments, SQUID control application will be integrated with MultiVu SW in the future.

4. Results and Demonstration

Directly coupled SQUID with 300 um hexagonal pick-up loop was successfully operated in Dynacool at various temperatures between 25 and 50 K. This was an encouraging result because it was anticipated that vibrations and electrical noises from the compressor and other components in the Dynacool could affect the SQUID performance adversely. Also, there was a question if the designed single layer mu-metal shield being effective enough. However, this experiment demonstrated a good performance of high T_c SQUID magnetometer in a wide temperature range. Figure 14 showed (a) bias current vs. temperature and (b) SQUID triangle at 25 K, where the tune voltage was 280 mV at bias current of 270 μA.



(a)



(b)

Figure 14. (a) Bias Current as a function of temperature in Dynacool, and (b) SQUID triangle (tune voltage) (yellow signal, 280 mV) at 25 K.

The SQUID was DC-biased so that 1/f noise was observed at low frequencies as shown in Figure 14. Although it was anticipated that the flux noise would be lower at lower temperatures, the data did not show this trend clearly. There was a noise peak near 1 kHz, whose origin was not identified but suspected to be due to the electronic components in the SQUID circuit. The lowest white noise reached about 100 $\mu\Phi_0/\sqrt{Hz}$.

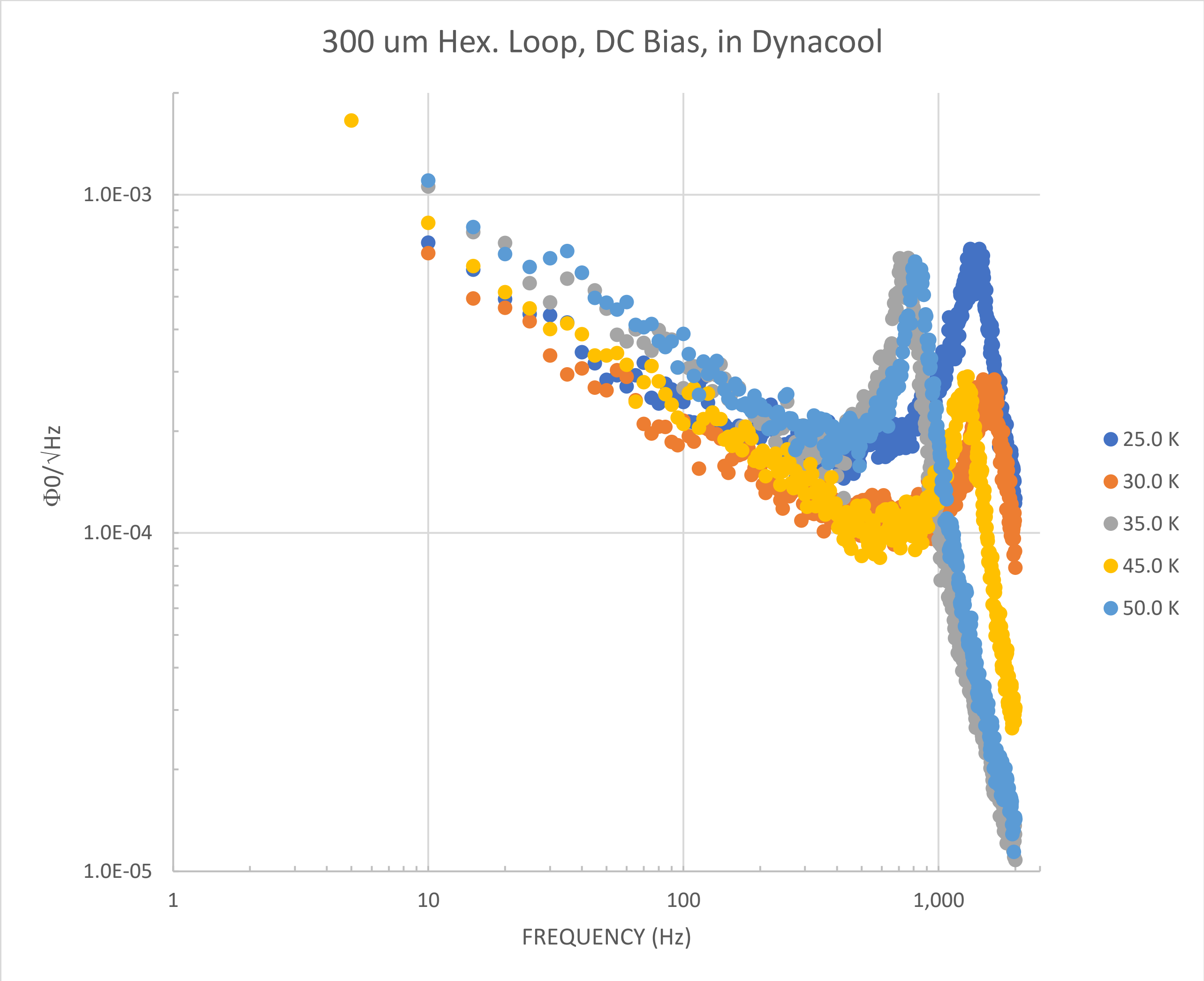


Figure 14. Flux noise of 300 um hexagonal loop SQUID operated in Dynacool at between 25 K and 50 K.

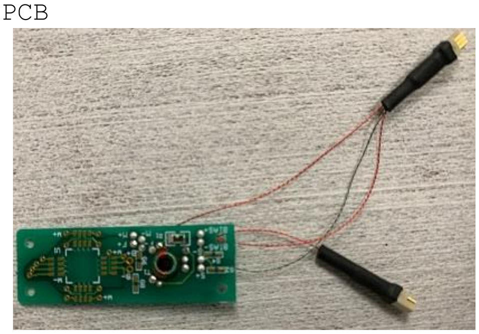
5. Future development

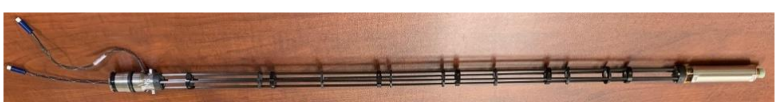
Hardware and software components are integrated for PPMS family of instruments such as PPMS and Dynacool at Quantum Design. All the components are delivered to UC Riverside for further development. The most interesting and challenging aspects of the project are to try to measure a signal from a sample, where all components are assembled and being operated. Questions remain how other components might affect the SQUID when they are being powered since the SQUID is the most sensitive detector of the magnetic flux in existence. Furthermore, software integration might be desired to operate the system in an automated way.

Since YBCO films are known to be sensitive and degrade in air over time, it is imperative to protect the surface of nano-scale device such as these SQUIDs in order to be able to use them in a commercial environment. Prof. Cybart group examined a 1% bromine-ethanol etchant solution to conclude that bromination of YBCO had minimal electrical consequences [11]. It is important to research further on the performance of the brominated SQUID devices.

Key components, which are going to be delivered to UC Riverside, are listed below.

Items delivered to UC Riverside

Item	Description	Qty
SQUID Control Module in ATE Power Supply 	A CAN-based module, powered by the ATE power supply, employing a digital signal processor (DSP) which performs synchronous AC detection.	1
SQUID Head 	Biases the SQUID and provides feedback control and signal amplification.	1
SQUID Control Cable 	Connects SQUID module and Head.	1
SQUID PCB 	Accommodates 5 mm x 5 mm SQUID chip on the PCB with Molex connectors.	2
SQUID WAP Cable 	A cable connecting WAP feedthrough to SQUID Head	1

Cartesian Positioner Controller 	Sends the high voltage signals required generate stepping action in the motorized sample positioner stage inside the mu metal capsule.	1
Wired Access Port (WAP) 	Includes multiple vacuum feedthroughs to pass electrical and signals into the sample space.	1
Cartesian Positioner 	Contains piezo-driven positioner stack with Delrin standoff.	1
Probe 	Carries electrical signals to and from the positioner piezo-drivers and SQUID; houses optical elements for imaging the sample. SQUID is enclosed by mu-metal shield.	1
Camera 	Allows for imaging of the sample while installed on the OMFP installed inside the PPMS sample chamber.	1

6. References

[1] Physical Property Measurement System (PPMS) manufactured by Quantum Design (www.qdusa.com).

[2] Dynacool manufactured by Quantum Design (www.qdusa.com).
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[4] Optical Multi-Function Probe (OMFP) manufactured by Quantum Design (www.qdusa.com).

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Appendix

OMFP Specifications

Temperature (T)	
Range*	350 to 5 K (Dynacool, PPMS)

Axial Optical Window	
Coupling Type	SM1 (1" diameter)
Included Coating	350 to 700 nm and 650 to 1050 nm

Camera Resolution	< 2 µm**

Piezo-Positioner Stack	
Maximum Travel	3 mm (all axes)
Minimum Step Size***	1 µm to 1 mm (approx.; user controlled)
Control Mode***	Open Loop

Operational Range	0 to 16 T

*Base temperature of 5 K represents the minimum achievable under 10 mW of radiant flux.

**Based on resolving individual lines within group 8, element 3 of the 1951 USAF resolution test chart.
***Due to hysteretic effects intrinsic to the piezo-resistive drive elements and open loop operation, precise step sizes may not be repeatable between different temperatures, upon changing drive direction, or at the extreme points on the available range.