A variable temperature scanning SQUID microscope

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Abstract **– We present a design of a scanning SQUID microscope (SSM) operating in a temperature range between about 5 K (2 K with pumping) and 100 K.**

I. INTRODUCTION

A number of successful Scanning SQUID Microscope (SSM) designs has been put forward in recent years. They can be divided into two groups: high-resolution microscopes with a sensor (either LTS [1-3] or HTS [4, 5]) in close contact to a sample at the same temperature; and microscopes with a sample at room temperature outside the cryostat [6]. Hereafter we shall only discuss the first group of the microscopes. Being sophisticated as they are, these systems are limited to a fixed operation temperature that is either 4.2 K or 77 K, determined by the type of the sensor. However, a number of problems such as: variation with temperature of vortex configurations in superconductors (e.g. the behavior of fractional flux quantum vortices in HTS materials), magnetic phase transitions, non-destructive evaluation of superconducting circuits and read-out of RSFQ devices, local field of antiferromagnets in the vicinity of the crystal faces, etc., require investigation of the magnetic field distribution at variable temperatures.

Below we shall discuss a design of an SSM which allows to investigate magnetic related phenomena in a wide temperature range.

II. DESIGN OF THE MICROSCOPE

A. *Design criteria.*

Variable temperature of the sample puts heavy demands on the design of all and every part of the system.

First of all since the SQUID sensor must be operated at a well-stabilized temperature, while the temperature of the sample is varying the heat exchange between them must be minimized. Naturally, this requires a cold-finger type cryostat. To maintain a high spatial resolution the SQUID must be kept close to or in direct contact with the sample surface. The former would eliminate heat conduction, but would require a complicated feedback loop. Direct contact is much easier to achieve, but the heat conduction can significantly change the SQUID operation temperature unless the contact area is made very small.

A magnetic flux generated even by a bifilar resistive heating element may cause problems in SQUID operation. Therefore, temperature regulation with a non-electric heater is preferable.

The design of a SQUID sensor for an SSQM should compromise between high magnetic field sensitivity and good spatial resolution. The former requires a bigger pick-up loop and flux focusing washer, whereas the latter dictates a small pick-up loop and a narrow linewidth, preferably of the order of 0.8-0.4 μm. Therefore the e-beam lithography must be used. Since we are aiming at investigation of magnetic phenomena in a wide temperature range, the sensor must be stable to inevitable temperature fluctuations through proximity to the sample. The obvious choice is a SQUID made of an HTS material operated at a temperature well below its T_C. Also, the design must be rather simple and flexible. This requirement is difficult to meet with SQUIDs based on bicrystal Josephson junctions. Bi-epitaxial, step-edge, ramp technology allow for arbitrary position and configuration of the junctions. However, at the present state of the HTS technology the task to make submicron devices remains a challenge.

B. *Cryostat and Scanner.*

A nonmagnetic continuous flow cryostat with a variable temperature insert (VTI) is designed and built for the microscope. Principles of operation of the VTI are the following:

A small constant overpressure of a helium gas in the bath against the recovery line is kept by a membrane pressure regulator. This pressure drives the evaporated helium to a heat-exchanger either directly (cooling) or through a copper tube placed outside the cryostat (heating). The temperature is controlled in a temperature range between about 5 K (2 K with pumping) and 300 K with an accuracy down to about 0.05 K only by the balance of flows of cold (directly from the bath) and warm (room temperature) He gas. Two valves connected to a temperature controller maintain the balance. This innovative scheme by no means affects the SQUID performance. A resistive heater mounted in the heat exchanger can be used for rapid heating in between the scans. Another one can be used to increase the pressure in the helium bath. Acoustic resonances excited by boiling helium are suppressed by using Kapitza wool threads in all gas tubes. The volume of the bath is about 2 liters of liquid helium. It is thermally insulated from the environment by a liquid nitrogen vessel with evacuated outer double walls.

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Charcoal absorbers maintain high vacuum there. Due to careful design the cryogen consumption even at extreme temperatures is very low allowing for lengthy (about 15 hours) experiments without refilling.

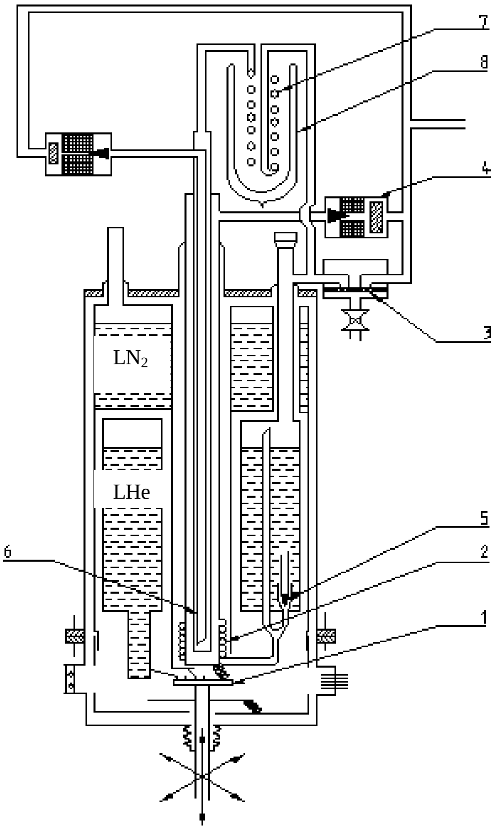

Fig. 1. The cryogenic part of the SSM: 1 – sample holder; 2 – channel for cold He gas; 3 – pressure regulator; 4 – valve; 5 – needle valve; 6 -- channel for warm He gas; 7 – warm heat exchanger; 8 – thermos with hot water (optional).

Both the sensor and the sample are placed in the vacuum chamber of the VTI. The SQUID sensor is suspended on a cantilever, which is attached to a cold finger cooled directly by LHe or LN_2. Therefore, either an LTS or an HTS SQUID sensor can in principle be used. The sample is fixed on another cold finger connected to the heat exchanger. Worth mentioning that the sample holder is fitted with 23 terminals to facilitate simultaneous electric and magnetic measurements. The sample-holder is attached to a rod fixed on a stepper motor driven XYZ translator with an attainable resolution of 0.5 micrometers. The stepper motors are equipped with incremental encoders, allowing for determination of the actual position and feedback. They are operated via an intellectual controller connected to a computer. We had considered translator schemes other than based on stepper motors (e.g., inch-worm type piezoscanners) before making the choice, and concluded that all of them loose in terms of performance/cost. The translator is based on the Vacuum Generators' Omniax base module, which has an exceptional stability, travel range, repeatability and resolution. We did not observe any interference of the properly fitted stage with the performance of the SQUID sensors.

C. *Cantilever.*

The cantilever consists of two 150 μm thick glass plates glued together at the one end through a thin insulating spacer. The inner surfaces of the glass plates are covered with gold and form a capacitor. The sensor is glued to the upper plate and bonded to the contact pads. The free ends of the plates are initially in electric contact. When the sensor touches the sample, the top plate moves upward and the contact is lost. Thereafter the capacitance between the two plates can be measured. Such a simple capacitive sensor allows for rather accurate determination of the vertical displacement of the order of 100 nm. Due to a very low elastic constant of the glass cantilever and the precise control of the vertical displacement the contact area between the sample and the SQUID can be made very small. The heat transfer through the contact is proportional to the contact area and therefore the heat load on the SQUID is negligible. The capacitor can be included in a feedback loop to maintain constant pressure on the sample surface during scanning. The capacitive sensor in principle allows for obtaining a profile of the sample surface in the same time with the magnetic imaging.

D. *SQUID sensor.*

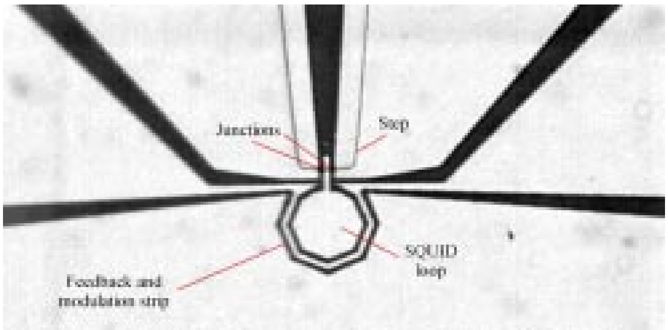

Fig. 2. A SQUID sensor with 0.5 μm wide step-edge Josephson junctions.

For the reasons given above, we chose to use $YBa_2Cu_3O_{7-\delta}$ (YBCO) step-edge Josephson junctions. The junctions are formed along the edge of a step etched in a substrate prior to deposition of the YBCO film [1]. E-beam lithography on the PMMA-Copolymer double-layer e-beam resist and ion-beam milling through an amorphous carbon mask were used both to define the steps in the substrate and to pattern the YBCO film. Importantly, the alignment marks milled in the substrate in the same run with the steps allow for accurate alignment of the SQUID patterns across the step.

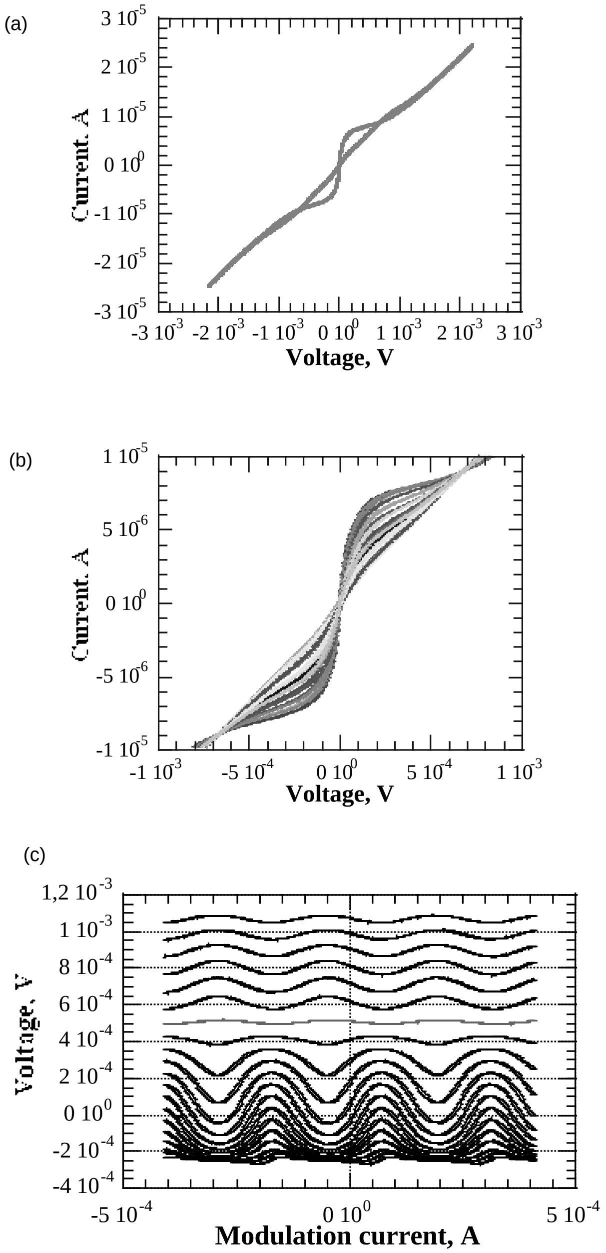

Fig. 3. IV characteristics of a SQUID with 0.5 μm wide step-edge Josephson Junctions at 4.2 K (a, b) and the SQUID response to the magnetic field produced by the modulation strip (c).

Two chips with 12 SQUIDs on each of them were fabricated to verify all important technology and design parameters. The sensor consisted of an octagonal SQUID loop 10 μm in diameter and a feedback and modulation strip 0.5 μm away (Fig.). The modulation strip had a superconducting transition at about 89 K and a critical current density close to 10^7 A/cm^2 at 4.2 K. The width of the step-edge junctions varied between 0.5 μm for 4.2 K SQUIDs and 3.5 μm for the 77 K ones giving about the same critical current at corresponding temperatures. The I-V curves were of the RSJ type, however, in a few samples we observed a second transition at bias currents about an order of magnitude higher than I_{c1}. This is due to the presence of the second weak link at the bottom of the step, as recently described in [8].

Twenty one of the twenty four fabricated SQUIDs worked at specified temperatures. The modulation amplitude was up to 40 μV and the coupling coefficient was about 250 μA/Φ_0. The SQUID is operated in a flux-locked mode. The readout electronics allows the implementation of different noise reduction techniques. It is based on a commercial programmable feedback loop PFL-100 manufactured by Conductus, Inc., modified to conform the specific type of SQUID sensor.

Individual sensors were cut from the chip and glued to the cantilever.

E. *Software.*

A program, written in LabVIEW environment, can remotely control all scanning parameters. These include scan direction, range and step, specific settings for the stepper motors (base speed, slew rate, acceleration, number of creep steps), incremental encoders (feedback mode, allowable error window, soft limits), and the SQUID (bias mode, bias current, skew, modulation amplitude and frequency, phase, offset, etc.). Readout of the SQUID feedback signal is performed by a digital voltmeter, which is also controlled by a PC computer. The distribution of the magnetic flux is automatically mapped to the color or halftone graph – SQUID feedback signal vs. the actual position (X,Y) read from the incremental encoders.

III. CONCLUSIONS

We have designed and fabricated a scanning SQUID microscope working in a temperature range 5-100 K. A novel principle was developed for temperature regulation. A simple capacitive sensor was used for accurate determination of vertical displacement of the cantilever. Submicron SQUID sensors developed in this work demonstrated good reproducibility, RSJ type IV characteristics and modulation voltage of 40 μV. However, a thorough investigation of their noise properties is required. The work is currently in progress on studying phase transitions in magnetic films, evolution of vortex configurations in HTS films, and contactless readout of RSFQ circuits.

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