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High resolution magnetic imaging : MicroSQUID Force Microscopy

K. Hasselbach¹, C. Ladam^{1,4} V.O. Dolocan^{1, 5} D. Hykel¹, T. Crozes¹, K. Schuster², D. Mailly³

 1 Institut Néel, CNRS et Université Joseph Fourier, BP 166, F-38042 Grenoble Cedex 9, France

 2 Institut de Radio Astronomie Millimétrique 300 rue de la Piscine, Domaine Universitaire F-38406 Saint Martin d'Hères, France

 3 Laboratoire de Photonique et de Nanostructures, CNRS, Site Alcatel de Marcoussis Route de Nozay F-91460 Marcoussis France

E-mail: klaus.hasselbach@grenoble.cnrs.fr

Résumé. Magnetic imaging at the micrometer scale with high sensitivity is a challenge difficult to be met. Magnetic force microscopy has a very high spatial resolution but is limited in magnetic resolution. Hall probe microscopy is very powerful but sensor fabrication at the one micron scale is difficult and effects due to discreteness of charge appear in the form of significant 1/f noise. SQUID microscopy is very powerful, having high magnetic resolution, but spatial resolution is usually of the order of 10 μ m.

The difficulties lay mostly in an efficient way to couple flux to the sensor. The only way to improve spatial resolution is to place the probe close to the very edge of the support, thus maximising coupling and spatial resolution. If there has been found a way to bring close the tip, there must be also found a reliable a way to maintain distance during scanning.

We want to present recent improvements on scanning microsquid microscopy : Namely the improved fabrication of microSQUID tips using silicon micro machining and the precise positioning of the micrometer diameter microSQUID loop by electron beam lithography. The microSQUID is a microbridge DC SQUID, with two opposite microbridges. The constrictions are patterned by high-resolution e-beam lithography and have a width of 20 nm and a length of about 100 nm.

The distance control during scanning is obtained by integrating the microSQUID sensor with a piezoelectric tuning fork acting as a force sensor allowing to control height and even topographic imaging. The detector is placed in a custom built near field microscope and the sample temperature can be varied between 0.1 Kelvin and 10 K.

The microscope is used to study magnetic flux structures in unconventional superconductors and will be used to observe thermal domains in superconducting detectors in the voltage state.

Introduction

Magnetic imaging at room temperature is achieved using commercial magnetic force microscopes (MFM) or Kerr effect microscopy to name only a few techniques. Magnetic

⁴ Present address : SINTEF Materials and Chemistry Høgskoleringen 5, Trondheim, Norway

 $^{^5\,}$ Present address : LMCQ/Laboratoire de Matière Condensée Quantique CEA/Saclay F- 91191 Gif sur Yvette Cedex

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imaging at cryogenic temperatures allows for high resolution magnetic imaging of ferro magnetic structures and the study of vortex physics in superconductors.

A recent review on local magnetic probes of superconductors [1] describes different techniques employed. Among the scanning probe approach MFM microscopy is used for imaging vortices in superconductors at temperatures as low as 4.2 K using either optical [2] or piezoresistive [3] detection schemes. Scanning Hall probe microscopy [4, 5] is successfully employed for imaging vortices. Very successful SQUID imaging has been done using mechanical translation without an approach control [6, 7, 8, 9] though the spatial resolution is limited by the size of the SQUID's pick-up loop and the distance between tip and sample. The size of the SQUID loop has recently been [10] reduced down to a size of about 190 nm by focused ion beam patterning of Nb circuits. The question remains how to approach the nanoSQUID close to the surface. We present here the Grenoble scanning μ -SQUID -microscope [11] with high spatial resolution, functioning at temperatures below 10K capable of acquiring simultaneously topographic and magnetic images using as force sensor a quartz tuning fork. These force sensors have been used in the past to build near field scanning optical microscopes [12]. This approach has been extended by atttaching small STM tips to the tuning fork in order to combine AFM and STM techniques. It has been shown that this can be used at low temeratures as low as 60 mK [13] and in high magnetic fields.

Instrument

The microscope is placed in a inverted dilution refrigerator (Sionludi) built at the Néel Institute. The refrigerator is inside a cylindrical vacuum chamber of 400 mm height and 200 mm diameter. The main body of microscope, figure 1, is situated on the still stage of the refrigerator. The microscope consists of a large range scanner [14] displacing the sample. The scanner is carried by a linear piezoelectric motor for the coarse approach to the SQUID-AFM sensor. Sample and sensor are connected with weak thermal links to the mixing chamber allowing to heat sample and SQUID independently. Sample and SQUID reach a base temperature of 0.45 K while the still is at 0.9 K.



FIG. 1. Schematic drawing of the disposition of the SQUID-AFM microscope inside the reversed dilution refrigerator, sample and SQUID are thermally weakly anchored to the mixing chamber, heaters and thermometers on the sample (not shown) allow for temperature control.

The purpose of the microscope is to observe vortices in bulk superconductors [15, 16], nanoengineered superconducting circuits [17] and magnetic domains [18] in ferromagnets at low temperatures. The SQUID is sensitive to the component of magnetic flux perpendicular to its area. The measured signal depends on the mutual inductance between the current distribution

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in the sample and the SQUID loop. The magnetic vortex size is given by the penetration depth of the magnetic induction, B, in the superconductor, setting the scale over which the screening currents circulate around the vortex core. The typical penetration depth is of the order of 0.1 μ m, diverging at T_c .

In order to obtain high spatial resolution we chose to scan a 1μ m diameter SQUID loop figure 2. The Josephson Junctions of these SQUIDs are constituted of Dayem Bridges of 300 nm length, 20 nm width and 30 nm thickness. The width of a SQUID branch is 200 nm. The critical current is measured by a direct current biasing scheme [19, 20] : A dc current is ramped up, when the critical current is reached the applied current is reset to zero and the SQUID becomes superconducting again.

Advantages of these μ -SQUIDs are multiple : simplicity of fabrication : one single level of e-beam lithoraphy and week flux pinning as the line width is very narrow. We used SQUIDs made of two different materials Nb and Al. The critical current of the Al SQUIDs is of the order of 150 μ A at low temperatures. 150 μ -SQUID probes are fabricated on a 2 inches diameter Si wafer. Each SQUID chip is precision cut with a dicing machine. A precision of 5 μ m is obtained, limited only by chipping of the Si, figure 3.



FIG. 2. Scanning Electron Microscope image (SEM) of an aluminium μ SQUID. The two constrictions of nominally 20 nm width are Dayem bridges and act as Josephson Junctions.



FIG. 3. SEM picture of the Si chip carrying the μ SQUID detector after dicing with a diamond saw.

We are exploring new ways of controlling precisely the placement of the SQUID relative to the apex of the Si chip, using Si deep-etching by means of an Alcatel 601 E reactor employing a Bosch process. Here a resist mask, defining the shape of the cantilever 4 top, is aligned with the SQUID pattern. The resist mask serves then as etch mask for the deepetching (100 μ m in about 15 minutes, for 100 μ m vertical etching the lateral etch is about 1 μ m). By this means it becomes possible to control precisely the distance between apex and SQUID, figure 4 bottom, and ultimately to reduce this distance to the order of 1μ m.

It is this apex of the Si chip that serves as tip for distance control and topographic imaging. The sample is scanned at an angle of less than 5 degrees relative to the Si chip. For a 5μ m distance between tip and SQUID and an approach angle of 5 degrees the SQUID sample distance is 0.43 μ m.

The closeness of the SQUID to the sample demands a good control of the distance between tip and sample. Three choices are possible : either the sample and tip are continuously in contact or a distance sensor is incorporated and a feedback loop is used, or scanning takes place at constant height above the sample's surface. The SQUID microscope has a tuning fork for distance control.

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FIG. 4. SEM pictures of the μ -SQUID sensor. At the top an overview is presented of the Si deeply etched μ SQUID sensor, the small SQUID loop is patterned at the end of the narrow ridge.

Bottom : The μ -SQUID sensor of 1 μ m diameter is closely aligned with the Si tip.



FIG. 5. Microscope image of the 2 mm long SQUID Si chip epoxied on the Quartz tuning fork and connected by Al wires to the holder.



FIG. 6. Resonance curve, phase and amplitude, of a tuning fork carrying a SQUID at 400 mK, under cryogenic vacuum. The Q factor is 17000, the full-lines follow the behavior of a harmonic oscillator.

The tuning fork is a 6 mm long, 0.5 mm wide Quartz watch crystal. It is designed for a resonance frequency of 32765 Hz at room temperature. Scanning probe schemes were developed using this type of tuning forks to which small and light tips were attached [12, 21, 22].

In our case the SQUID chip wheighs about 30 % of the mass of one prong thus the resonance frequency of the tuning fork is shifted from 32,7 kHz to about 25 kHz. Knowing the Young modulus of the piezocrystal and the expression of the spring constant of a beam of given dimensions a spring constant of the order of 40000 N/m is obtained for the bare tine. We can estimate the amplitude of oscillation from the amplitude of the tuning fork current

measured[12, 22, 23]. Amplitudes used are in the order of 5 to 10 nm. The quality factor of the resonator, Q, is of the order of 100 to 200 at room temperature and 5000 to 10000 at 4.2 K and up to 17000 at 0.4K. For regulation of the high quality resonators the PLL scheme [21, 24] is now well established.

Penetration depth : example of Sr_2RuO_4

The penetration depth (λ) is a measure of the superfluid density and its temperature variation gives an indication on the amount of available quasiparticle excitations, or in other words on the symmetry of the order parameter (fully gapped, lines, nodes etc...). Very sensitive measurements [25] detect the temperature dependence of $\lambda(T)$ but the absolute value is difficult to obtain directly. Direct magnetic imaging may be a way to obtain values for the penetration depth. It was shown that the spreading of the magnetic field of a vortex at the surface of a superconductor can be modeled in coupling the London equations in the superconductor to the Maxwell equations in vacuum. The magnetic field at a height z above the surface of a superconductor of thickness d can then be analytically calculated [6, 26].

$$h_z = \frac{\Phi_0}{(2\pi\lambda_{ab})^2} \int d^2 \vec{k} e^{i\vec{k}\cdot\vec{r}} \frac{e^{k(d/2-z)}}{\alpha \left[\alpha + k \cdot \coth(\alpha d/2)\right]}$$

where

$$\vec{r} = \{x, y\}, \vec{k} = \{k_x, k_y\}, k = \sqrt{k_x^2 + k_y^2}, \text{ and } \alpha = \sqrt{k^2 + \lambda_{ab}^{-2}}.$$

It is necessary to integrate the vortex stray field over the geometric area of the SQUID in order to compare with an experimental flux profile. The typical shape of such a flux profile can be seen in figure 7. The aim is to obtain error bars for the penetration depth and the height of the SQUID sensor, thus improving the confidence in the measurement of the penetration depth.



FIG. 7. A vortex profile and two fits for either h=1.15 μ m and λ = 0.175 μ m (dotted), or h=1.05 and λ = 0.26 μ m (solid), data (crosses) of Sr₂RuO₄ at 0.4K. The fits are very similar.



FIG. 8. Regions of confidence for the fit with two free parameters height of SQUID above the sample surface and penetration depth. The color scale indicates the quadratic error between model and data (same as figure 7). $\lambda = 0.26 \mu \text{m}$ $\pm 0.05 \mu \text{m}$ and height $1.05 \ \mu \text{m} \pm 0.05$ μm .

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From the charting of the confidence region of the fit with two free parameters, height of SQUID above the sample surface and penetration depth it becomes easily to give error bars : $\lambda = 0.26 \mu \text{m} \pm 0.05 \mu \text{m}$ and for the height $1.05 \mu \text{m} \pm 0.05 \mu \text{m}$. The strong dependence of the penetration depth on the height becomes weaker when the SQUID gets closer, The amount of flux captured increases on approaching. The second point is that the height should be well known from the beginning so that it would not be necessary to treat it as free parameter. Well defined tip geometries figure 4 and a calibrated approach angle between SQUID and sample surface will allow in the future to approach the SQUID closer to the surface and to define precisely its distance from the sample surface.

Conclusion and outlook

We have presented a μ SQUID microscope and have indicated the principal direction of further improvements that will transform SQUID microscopy in even a more powerful and quantitative tool. Namely closer scanning (0.5 μ m range) will inhance the flux capture of the SQUID, and the integration of large displacement slip stick based motors will allow in the future to study magnetic fingerprints of superconductors and superconducting devices with sub μ m resolution on the millimeter scale at very low temperatures.

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