

Chapter 2

Methods

The experimental setup used in this work consists of three major subsystems. The first and foremost in importance is the SQUID-on-tip. In Sect. 2.1 we describe the procedure used to fabricate it. The second subsystem is the tuning-fork assembly, which is described in detail in Sect. 2.2. These two subsystems comprise the sensor assembly of the scanning SQUID microscope. Their integration into one assembly is described in Sect. 2.3. The third and last subsystem is the fabrication of samples. I will give a brief yet thorough description of this procedure in Sect. 2.4.

2.1 SQUID-on-Tip Fabrication

We pull a quartz tube with an outer diameter of 1 mm and inner diameter of 0.5 mm to form a pair of sharp pipettes with a tip diameter that can be controllably varied between 100 and 400 nm using a commercial pipette puller.¹ Then, we either apply a thin layer of indium or deposit a thin (200 nm) film of gold on two sides of the cylindrical base of the pipette. Afterwards, the pipette is mounted on a rotator (see Fig. 2.1) and put into a vacuum chamber for three steps of thermal evaporation of aluminum. The rotator has an electrical feedthrough for two operations. First, a 2 mW red laser diode is connected, which allows for the alignment of the tip main axis with respect to the source. This defines our zero angle. The second electrical connection is for an in-situ measurement of the tip's resistance during deposition. A finite resistance typically appears after depositing approximately 70 Å in the third stage (see below). In the first step (see Fig. 2.2), 25 nm of aluminum are deposited on the tip tilted at an angle of -100° with respect to the line to the source. Then the tip is rotated to an angle of 100° , for a second deposition of 25 nm. As a result, two leads on opposite sides of the quartz tube, reaching all the way to the apex, are formed. In the last step 20–22 nm of aluminum are deposited at an angle of 0° , coating the apex ring of the tip. The resulting structure has two leads connected to a ring. “Strong” superconducting regions are formed in the areas where the leads make contact with

¹ Sutter Instruments P-2000.

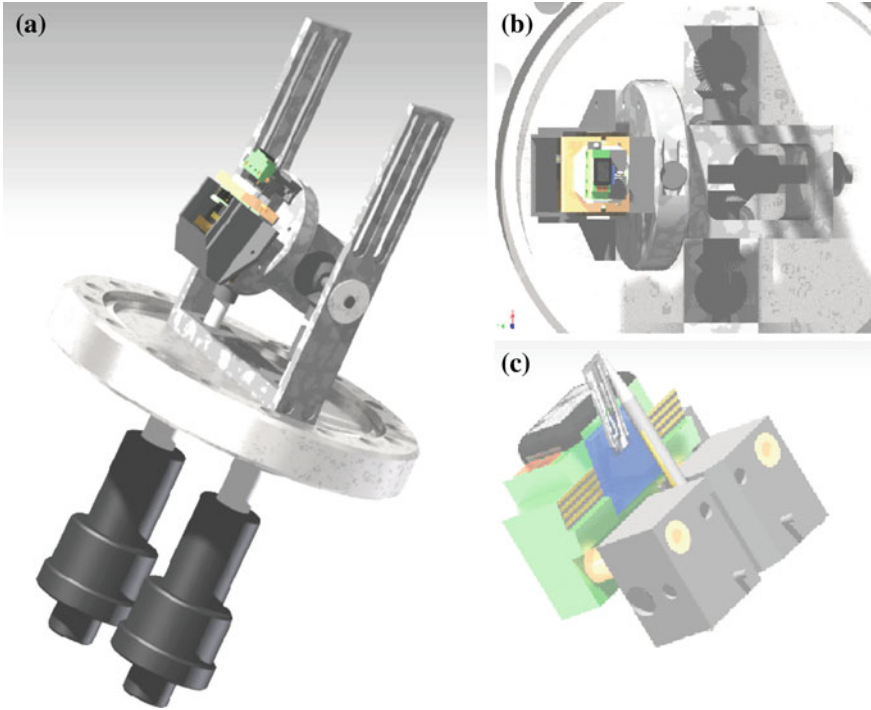


Fig. 2.1 **a** A CAD image of the rotator used to rotate tips in the vacuum chamber. The original design of this rotator was based on a similar device from Yoo et al. [1]. **b** A close-up view of the *gear-box* holding the rotating stage with the tip holder. **c** The tip holder, co-linear with the laser diode on the rotator. The tuning-fork assembly is not present during deposition for practical reasons

the ring, while the two parts of the ring in the region of the gap between the leads constitute two weak links, thus forming the SQUID. Its typical room-temperature resistance is $1.5\text{ k}\Omega$. Since the device is highly sensitive to electrostatic discharge, it is shorted when being transferred from the evaporator to the microscope.

2.2 Tuning Fork Assembly

The idea to use a quartz tuning-fork as a topography sensor was suggested to us by Dr. Michael Rappaport. He had previously built a tuning-fork based NSOM [2], and the basic principles of operation were taken from his fiber-optic design and adapted to our much larger SQUID-on-tip.

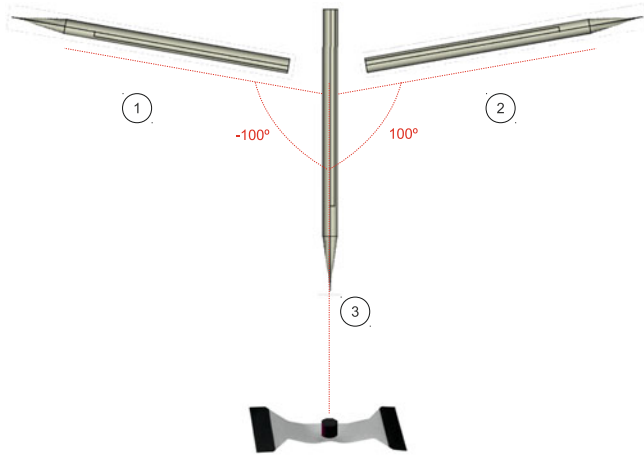


Fig. 2.2 A schematic of the three-stage thermal deposition of a SOT

2.2.1 Preparation

We use commercial² quartz tuning forks, laser-trimmed to have a resonance at 2^{15} Hz, which are normally used as time bases in digital watches. Preparing one for microscopy entails the removal of the vacuum can and the two electrodes soldered to it (see Fig. 2.3). We then glue it to a $5 \times 5 \times 0.5 \text{ mm}^3$ quartz piece which was pre-coated with two gold electrodes ($70 \text{ \AA}$ Cr/ $2000 \text{ \AA}$ Au). The two tuning-fork electrodes are bonded to those quartz electrodes, which are in turn connected to external wires via two additional bonds. This allows for the electrical readout of the voltage, which develops on the tuning-fork due to the piezoelectric effect. In principle this would have been enough, since it is possible to electrically excite the tuning-fork with a voltage source and read the current through it using a current-to-voltage converter [3]. However, it is more advantageous to decouple the excitation from the readout by driving voltage to a dither piezo, which mechanically excites the tuning-fork (Fig. 2.3d). We used two types of dither piezos for this purpose, one of which³ had the same dimensions as the quartz plate and was therefore used both as the plate to which we glued the tuning-fork and as its mechanical excitation medium. The second⁴ was slightly bulkier and was therefore placed on the side of the tuning-fork holder.

² Polaros Electronics, TB38-20-12.5-32.768 KHz.

³ EBL#4.

⁴ PI PICMA PL033.30.

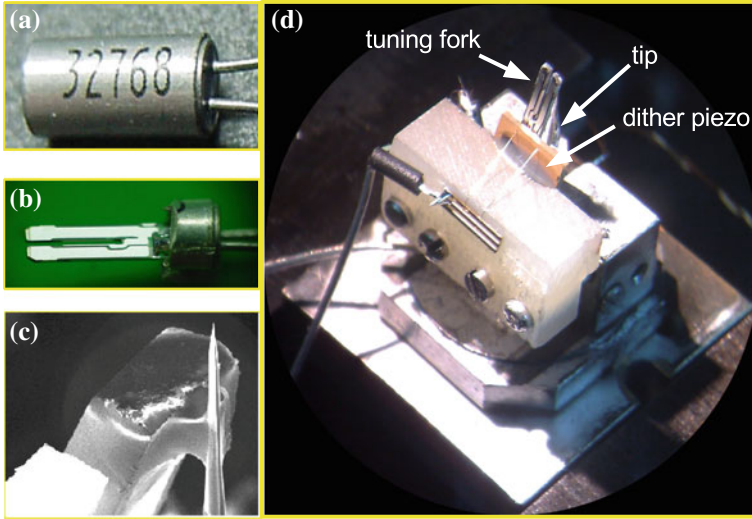


Fig. 2.3 **a** A commercial tuning fork encased in a vacuum can. **b** The same tuning fork after the vacuum can had been removed. **c** A SEM micrograph of a tip glued to a prong of a tuning fork. **d** The tip/tuning fork assembly. The tuning fork itself is glued onto the dither piezo with E32 and then glued to the tip with Araldite 2020

2.2.2 Gluing a Tip and Its Effect on Resonance

In our microscope setup, we glue the SOT to one of the prongs of the TF (see Fig. 2.3c). This can change the resonant frequency [4] dramatically. Therefore, we try to use as small amount of glue as possible. This is made possible by using a two-part epoxy with a rather low (150cP) viscosity, which enables us to place a very small drop of glue on the part joining the two. The longer the SOT protrudes above the edge of the prong, the softer its effective spring constant [5] becomes (for a cylindrical tip)

$$k_{\text{eff}} = \frac{3\pi E r^4}{4l^3}$$

Here r , l and E are the radius, length and Young modulus of the tip, respectively. Thus, if it protrudes too much, the vdW forces of interaction with the sample will dampen its motion before the tuning fork's prong actually “feels” anything. Consequently, we always try to position the tip with respect to the tuning fork so that the SOT itself protrudes only slightly above the edge of the prong (typically a few tens of microns). We then excite the tuning fork and measure its resonance, specifically noting the location of the peak, its amplitude and its width. This measurement is performed at room temperature and atmospheric pressure and repeated at low temperature (300mK) in vacuum for comparison. More often than not, the low-

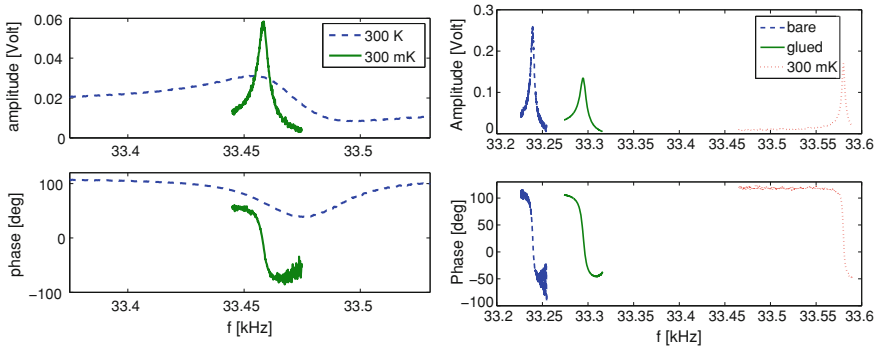


Fig. 2.4 The resonance of our quartz tuning fork. *Left* comparison between the Q-factor at room temperature and at low temperature. For ease of comparison, the latter was shifted by 80 Hz so that its peak overlaps with that of the former; *Right* the “bare” and “glued” curves are at room temperature, with “bare” meaning that the tuning-fork is not glued to the tip. The 300 mK curve is that of the “glued” tip cooled down to 300 mK

temperature, vacuum resonance is much sharper than the room-temperature, ambient pressure one (see Fig. 2.4).

2.2.3 Mechanical Versus Electrical Excitation and Readout

In the equivalent electrical model for a resonating tuning-fork [6, 7], there are four parameters which should be considered: the stray capacitance, $C_0 \sim pF$ and the RLC-equivalent parameters, which in our case are $R = 30 k\Omega$, $C = 1 fF$, $L = 23.6 kH$. The stray capacitance is in fact that of the contacts. This means that the tuning-fork is in effect a reactive impedance, with an effective impedance of $\approx 1 M\Omega$. Any additional stray capacitance, mostly wires, will attenuate the signal considerably.

There are two methods of exciting and measuring the resonance of the tuning-fork:

1. Apply a voltage between the two prongs of the tuning-fork and measure the current through it. This is a compact method since it requires as little as two wires. The disadvantage with such a method is that there is coupling between the electrical excitation (piezoelectric effect) and the current readout, giving rise to a large background signal. In order to overcome this, one can insert a variable capacitor and a center-tapped transformer [3], which can be varied so as to exactly cancel the stray capacitance.
2. Mechanically excite the tuning-fork using an external dither piezo and reading the voltage between the two prongs. This way, the excitation is electrically decoupled from the signal readout. There still remains, however, the problem of signal attenuation due to cables. Once again one may employ the variable capacitor method. Another, more complicated but by far superior solution, is to use a

trans-impedance converter right next to the tuning-fork before the signal is attenuated by the experimental setup's cables, so that the effective impedance is converted from a few $M\Omega$ to a few $k\Omega$.

We used both techniques in our setup. The trans-impedance converter we used was made by attocube, and comprised of a commercial GaAs MESFET, which can work at cryogenic temperatures (charge carriers in GaAs do not freeze as those in silicon do).

2.3 Scanning SQUID Microscopy

The microscope's design is based on commercial coarse positioners and piezoelectric scanners from attocube. It consists of two parts: the bottom flange, which holds the coarse- z positioner, the xyz scanner and the sample; and the top flange, which holds the coarse- x and y positioners and the tip holder assembly (see Fig. 2.5). Our microscope's feedback mechanism relies on the behavior of a quartz tuning fork's (TF) resonance as it moves closer and closer to the surface of a sample. Both the quartz TF's modes and the above-mentioned behavior have been studied extensively and are widely used today in near-field scanning optical microscopes (NSOM) and atomic force microscopes (AFM) [8, 9]. Our tuning-forks are laser-trimmed so that they peak at $2^{15} = 32,768$ Hz.

2.3.1 Control Electronics

We divide the electronics section into three parts: (a) the SQUID on tip; (b) the tuning fork; and (c) coarse and fine motion.

2.3.1.1 SQUID on Tip Electronics

A SQUID is by definition a low impedance device. Typically, room-temperature electronics are optimized for high impedances. Consequently, this impedance mismatch results in a decrease in the signal-to-noise ratio. There are many schemes which tackle this problem successfully, e.g. non-dispersive read-out [10] and transformer impedance matching [11]. We chose to use a SQUID series array amplifier [12], which is in essence an array of 100 SQUIDs connected in series. The current through the SOT passes through a line which is inductively coupled to this array. This current induces a change in the flux through them, and as they are phase-coherent (if properly cooled down) this change in flux changes their critical current and accordingly the voltage on them. The SSAA has an additional FLL to keep the working point of the SQUIDs at their most sensitive (and most linear) region. Effectively, the

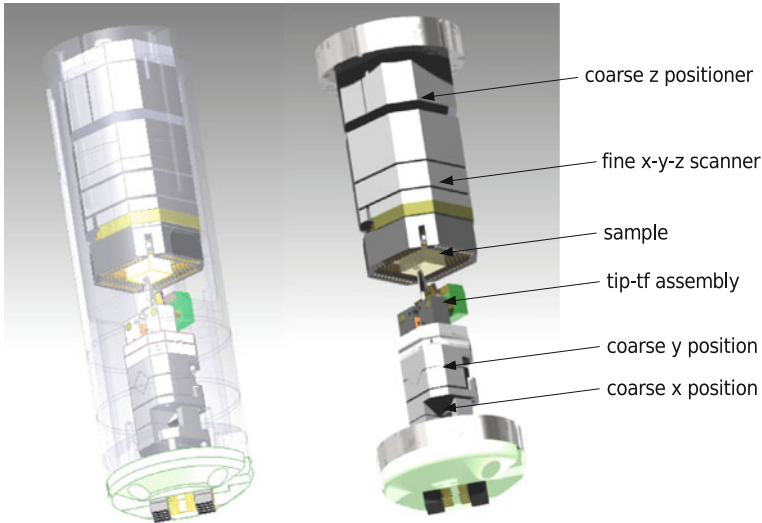


Fig. 2.5 Schematics of the microscope assembly. *Left* the microscope with the outer shell (made transparent for clarity); *Right* the microscope without outer shell. The lower part includes the coarse x and y positioners and the tip holder while the upper part includes the z positioner, the xyz scanners and the sample holder

SSAA functions as a flux-to-voltage converter, and with the SOT's current inductively coupled to a cryogenic, low-impedance current-to-voltage converter. A scheme of the measurement circuit is given in Fig. 2.6. We voltage bias the SOT using the small parallel resistor (R_b) and measure the current through the SOT using the SSAA. Currently, the room-temperature feedback box is bandwidth limited to 1 MHz, which means that we cannot measure signals at frequencies higher than 500 kHz. Moreover, the SSAA current noise density is $\approx 2.5 \text{ pA/Hz}^{1/2}$, which sets our system noise limit (see Sect. 3.1.1)

2.3.1.2 The Tuning Fork and Its Feedback Loop

One can monitor the height of the resonance peak and set the feedback threshold to, for example, 50% of the maximum. This method, also known as slope detection [13], is not fast enough, since quartz tuning-forks have relatively high Q -factors, especially at low temperatures (typically 100,000) [13, 14]. As explained in Refs. [13, 15], a phase-locked loop (PLL) increases the bandwidth and makes scanning probe microscopy with tuning-forks possible at reasonable scan speeds. A scheme of the entire measurement system with emphasis on the PLL block-diagram is shown in Fig. 2.7. As already mentioned in Sect. 1.1.3.2, interaction forces between the tip and the sample result in a frequency shift of the tuning-fork's resonance. The basic idea behind the PLL is to track this resonance in a closed loop circuit and compare

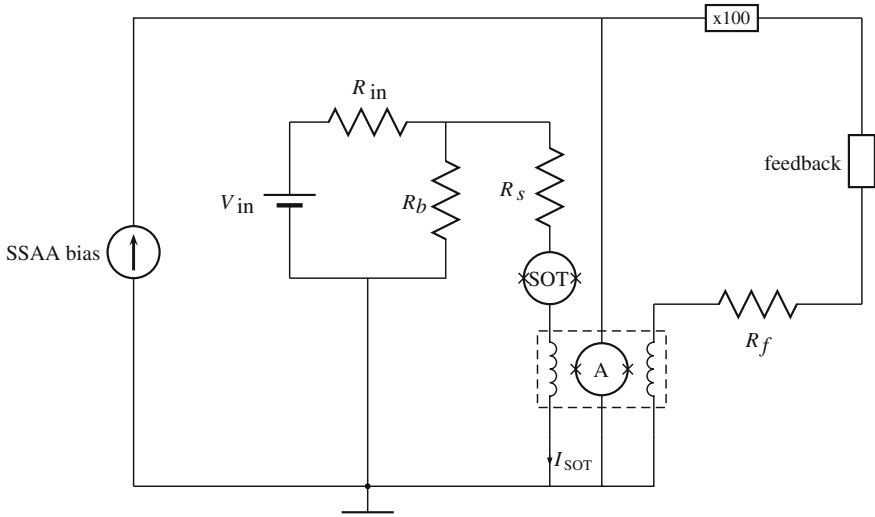


Fig. 2.6 The SOT circuit. Here “A” represents the SQUID series array amplifier (SSAA). The $5\text{ k}\Omega$ series resistor, R_b and the SSAA are thermally coupled to the fridge’s 1 K tube coil. The preamp ($\times 100$) and the feedback circuit are battery-operated room-temperature electronics. R_s is a parasitic series resistance to the SOT

its current location with the fundamental one to give the frequency shift, Δf . This frequency shift serves as the error function of the height, or z , feedback loop.

2.3.1.3 Coarse and Fine Motion of the Microscope

The coarse motion is performed by three attocube positioners ($1 \times \text{ANPz101RES}$, $2 \times \text{ANPx50}$), one for each axis, based on the slip-stick [16] mechanism. The positioners themselves are driven by a saw-tooth pulse from a high-voltage driver (attocube ANC-150). The z -axis positioner incorporates a resistive encoder, which allows for a resistive readout of the current displacement of the positioner. This resistor is calibrated so we can translate the value in Ohms to displacement in μm . Scanning is performed by an attocube integrated xyz scanner (ANSxyz100), with an $X - Y$ scan range of $30 \times 30 \mu\text{m}^2$ and a Z -scan range of $15 \mu\text{m}$ (all at low temperatures). We drive these piezoelectric scanners with high voltage from an SPM controller (either RHK SPM-100 or attocube ASC500).

The output of the PLL (in the form of a frequency shift, Δf) serves as the input of the z -height feedback loop (point no. 6 in Fig. 2.7). This second feedback loop (the first being the phased-locked one) controls the height of the tip above the sample by changing the voltage going to the z -scanner piezo.

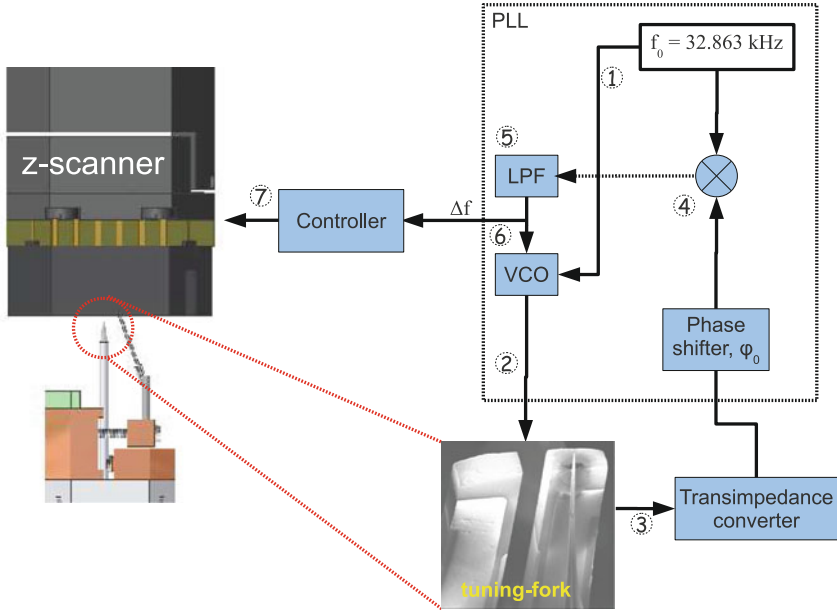
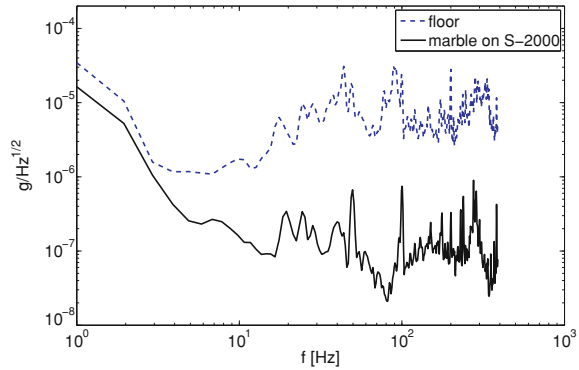


Fig. 2.7 A scheme of the height control loops. (1) The PLL sends an excitation voltage at the base resonant frequency, $\cos(f_0 t)$, to the VCO; (2) the VCO adds the base excitation phase together with any error corrections from the PLL feedback loop, and sends it, i.e. $\cos(f_0 t + \varphi_0)$, to the tuning-fork; (3) the current from the tuning-fork may experience a phase shift, φ , due to change in height and is then converted at room-temperature by a transimpedance converter; (4) the voltage from the converter is phase shifted by φ_0 and mixed with the base resonance frequency; (5) the mixed signal $\cos(2f_0 t + \varphi_0 + \varphi) + \cos(\varphi_0 + \varphi)$ is passed through a low-pass filter to produce the error signal, φ ; (6) this error signal is fed back to the VCO and; (7) knowing the original resonance curve, e.g. Fig. 2.4, one can convert the phase shift into frequency shift, Δf , which serves as the feedback parameter for the height control loop

2.3.2 Approach Procedure

The microscope assembly's heart revolves, then, around the SOT and the TF, designed as a rigid structure holding the two, one against the other, each resting on either coarse motors or piezoelectric scanners. At room temperature the distance between the SOT/TF assembly and the sample is a few hundred of microns, and once they are cooled to low temperature, we initiate what is known as the approach procedure. This involves fully extending the z-piezo while simultaneously trying to close a feedback loop with the frequency shift as the set-point parameter (typically 100 mHz in our case). If the set-point is not reached, the z-piezo is fully retracted and the z-coarse motor moves by a few steps forward, repeating the entire procedure all over again. As with every feedback loop, there is the subtle interplay between the three (or two) parameters of the PID (PI) loop, known as “proportional”, “integral” (and “derivative”). Usually, if one wants the loop to close in a reasonable time, an

Fig. 2.8 Comparison of vibrations' level. The measurement was performed using a Wilcoxon 731A accelerometer, which measures acceleration in units of Earth's gravity field, g . The signal is then fed to a spectrum analyzer



overshoot above the desired value is obligatory. In the case of our SOT, however, an overshoot is highly dangerous, as the device itself, the SQUID, is located at the very edge of the tip, making it the first to be in potentially hazardous contact with the sample. This is also in practice what we had encountered in the first few approach sequences, all resulting in the tip crashing into the sample. In order to overcome this obstacle, we patterned our samples in a meander, or serpentine-like, shape. The original idea was to run current through the meander, so that the magnetic field resulting from this current (according to Biot-Savart law) would be observable tens of microns away from the sample. I will discuss this in more detail in Sect. 3.2. With the feedback loop closed we could then maintain a constant height above the sample and acquire images.

2.3.3 *Vibration Isolation*

If one wants to approach the surface of a sample within a few nanometers and maintain a constant height of a few nanometers above it, one needs to properly isolate the system from its surroundings, namely from vibrational, acoustical and electric noises. For structural vibration isolation, we placed the dewar on a massive marble block, weighing 920 kg. This block, in turn, was placed on four commercial isolators (Newport S-2000). These isolators attenuate vibrations from the environment at low frequencies, having already a 20 dB attenuation at 10 Hz. The disadvantage of these isolators is that they typically have a resonance, i.e., amplification, at around 1 Hz. To reduce acoustical noise, we first coated the entire dewar with an acoustipipe, thereby damping its self-resonating mode. In addition, we enclosed the entire upper part of the system (from the marble block and upwards) with a 45 dB attenuation factor of an acoustic enclosure. The combination of the isolators and the enclosure created a sufficiently quiet environment in which we were able to safely scan samples with a tip-sample separation of a few nanometers (Fig. 2.8).

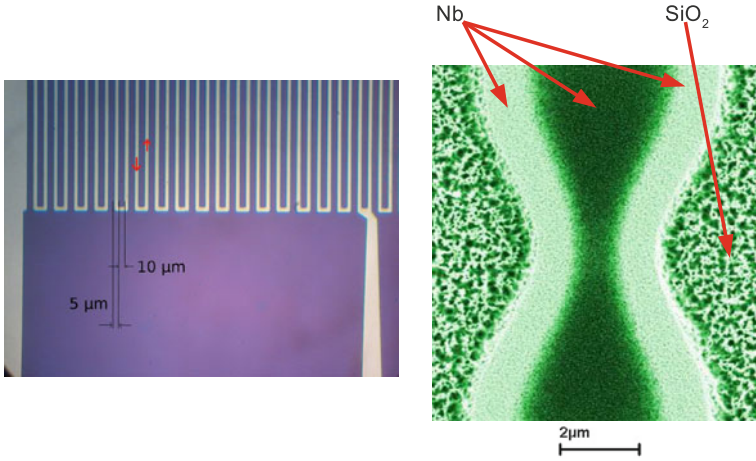


Fig. 2.9 *Left* an optical image of the Al serpentine deposited on a Si/SiO₂ substrate. The *arrows* indicate the direction of current running through it, in a serpentine trajectory; *Right* a SEM image of a Nb serpentine. It is very similar in shape and pattern, except for a constriction (shown) which repeats every 100 μm

2.4 Fabrication of Samples

The samples in this work were all prepared by our group in the clean-room facilities offered by our department. The mask itself is rather simple, having a serpentine structure going from one end to another. The main challenge, however, is to successfully fabricate such a structure with a minimum amount of defects so that current can flow unhindered from one side to the other. With a sample size of approximately 5×5 mm and a pitch of 15 μm (the width of the film is 5 μm and each two neighboring films are 10 μm apart), this amounts to a continuous line of the order of one meter!

The aluminum samples were deposited on a Si/SiO₂ substrate by e-gun evaporation in a background pressure of 1×10^{-6} Torr, and the photo resist (Shipley Microposit 1805) was removed using the lift-off technique. An optical image of the aluminum serpentine is given in Fig. 2.9. The niobium samples were deposited on a Si/SiO₂ substrate in the same vacuum chamber, with a substrate temperature of 300°C during deposition [17] and a rate of 0.3 nm/s. Then the Nb was capped with 3 nm of gold and the photolithography was performed using the previously mentioned 1805 photoresist. The gold layer was removed using argon ion-milling and finally the Nb was removed using reactive ion etching with SF₆ plasma. The complete recipe was taken from Ref. [18]. The mask used was slightly different, having a constriction (shown in Fig. 2.9) repeating itself approximately every 100 μm.

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