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Title: Magnetic resonance force microscopy at milliKelvin temperatures
Issue Date: 2013-09-19
Chapter 3

Experimental Instrumentation

Figure 3.1: A schematic representation of the manipulation and detection circuit that is used in our experiments. The spins in the sample are excited by electromagnetic pulses generated in the RF wire. The spins are coupled to the cantilever magnet and the resulting motion causes a change in the magnetic flux through the detection coil. The detection coil is part of a closed superconducting circuit, which is coupled to a SQUID, enabling readout of the cantilever position. A piezoelectric element can be used to excite the cantilever. Superconducting radiofrequency filters are implemented in the circuit to shield the SQUID from currents due to spin excitation pulses.

In this chapter, we will describe the details of our experimental setup. The general detection scheme that is at the heart of the experiment is shown in Fig. 3.1, which includes almost all of the ingredients that are needed for an MRFM experiment. We will first discuss the fabrication and characterization of the cantilever-magnet assembly. Then, we will describe design considerations for the detection coil, show how the detection circuit transductance can be calibrated, and provide some details on the fabrication of a single-loop detection coil and RF microwire on SrTiO₃. We will then turn to the use of the SQUID
as a magnetic flux detector, discuss some background theory and its implementation in our detection system. Subsequently, the experimental chamber with the inclusion of coarse alignment motors and a fine-scanning stage will be described. Finally, we discuss the reduction of mechanical vibrations in the cryostat in which the experiment is mounted.

### 3.1 Cantilever fabrication and characterization

Figure 3.2: Optical microscope image of the IBM cantilever chip (a) and cantilever (b), aligned above a square detection coil (c) on a strontium titanate chip.

We have shown in Chapt. 2 that the ideal MRFM cantilever is ultrasoft and has a high quality factor. In our experiments, we use a single-crystalline silicon beam, fabricated by Chui et al. at IBM Almaden Research Center [31] with these design goals in mind. These cantilevers are typically 100 nm thick, 5 µm wide, and 100 µm long. They are intended for MRFM experiments in which the sample is attached to the cantilever and the readout is done with a laser interferometer [16], and hence are equipped with a thick tip and a disc-shaped mirror. We choose cantilevers that are “broken”, or deliberately break off the mirror and mass ourselves, such that only the straight part of the beam remains (see Fig. 3.2).

The cantilever magnet is a spherically shaped NdFeB based alloy\(^1\) with a diameter in the µm range. This alloy is a strong permanent magnet with a remanent field of 1.3 T [22, 23]. Using an in-house developed nanomanipulator [32] in a Scanning Electron Microscope\(^2\) (SEM), we can approach the cantilever tip to a spherical magnet of our choosing, and glue the magnet to the cantilever in an Electron Beam Induced Deposition (EBID) process with a Pt(PF\(_3\))\(_4\) precursor gas. In Fig. 3.3 we show SEM images of the nanomanipulator-

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\(^1\)MQP-S-11-9-20001-070 isotropic powder by Magnequench, Singapore.

\(^2\)FEI NanoSEM 200 by FEI, USA.
Figure 3.3: Scanning Electron Microscope images of the process of attaching the NdFeB spherical magnets to the single crystalline Si cantilever. In panel a), a magnet of suitable size is chosen from the powder on the right and attached to the cantilever that is clamped on the left. In panel b) the cantilever is shown when retracted from the powder after the gluing process.

assisted alignment (panel a) and the finished cantilever-magnet assembly after the Pt deposition (panel b).

After the magnetic particle is attached to the cantilever, it is magnetized in a 5 T field at room temperature.

The spring constant of the cantilever can be calculated according to [33]:

$$k = \frac{Ewh^3}{3l^3}$$  \hspace{1cm} (3.1)

where \(w\), \(h\), and \(l\) are the cantilever width, height, and length, respectively, and \(E\) is the Young’s modulus of the beam material. For Si-(111) a Young’s modulus of \(E = 1.85 \cdot 10^{11}\) Pa is reported [34]. A cantilever with dimensions \(w \times h \times l = 5\mu m \times 0.1\mu m \times 123\mu m\) has a calculated stiffness of \(1.23 \cdot 10^{-4}\) N/m.

The resonant frequencies of an unloaded beam can be derived from the dynamic Euler-Bernoulli beam equation [35]:

$$\frac{Ewh^3}{12} \frac{\partial^4 u(x, t)}{\partial x^4} = -\lambda_m \frac{\partial^2 u(x, t)}{\partial t^2}$$  \hspace{1cm} (3.2)

where \(u\) is the deflection as a function of position at the beam \(x\) and time \(t\), and \(\lambda_m\) is the linear mass density. The \(n^{th}\) resonance is given by

$$\omega_n = (\kappa_n l)^2 \sqrt{\frac{k}{3M_c}}$$  \hspace{1cm} (3.3)

Here, \(M_c\) is the mass of the cantilever beam. The values \(\kappa_n l\) follow from numerical solutions of the beam equation for specific boundary values. For a
beam that is clamped on one side, we find $\kappa_l = 1.875$, so that the fundamental mode frequency is

$$\Omega = \omega_1 = \sqrt{\frac{k}{0.243M_c}} \equiv \sqrt{\frac{k}{M_{\text{eff}}}} \quad (3.4)$$

Thus, we can consider the cantilever as equivalent to a massless spring with a mass $M_{\text{eff}}$ attached at its end. For an IBM cantilever with a length of 123 $\mu$m, the effective mass is 34.9 pg and the fundamental mode frequency is 9.45 kHz.

An alternative, experimental way to determine the stiffness and effective mass of a cantilever is the so-called “added-mass method” [36]. By measuring the resonant frequency of the cantilever before ($f_0$) and after ($f_1$) gluing a mass $M_m$ to its tip, the stiffness can be determined by:

$$k = (2\pi)^2 \frac{M_m}{f_1^2 - 1/f_0^2} \quad (3.5)$$

and the effective mass by

$$M_{\text{eff}} = M_m \frac{f_1^2}{f_0^2 - f_1^2} \quad (3.6)$$

We excite the cantilever in-situ in the SEM by applying an oscillating voltage to the nanomanipulator chip holder. This creates a time-varying electric field gradient, that exerts a force on charges on the cantilever surface. The force is large enough to oscillate the cantilever at its resonant frequency with an amplitude of a few microns. For an unloaded cantilever of 123 $\mu$m length, we measured a resonant frequency of $f_0 = 10350$ Hz. After gluing a NdFeB sphere with a diameter of 3.86 $\mu$m and a mass of $M_m = 0.224$ ng, the resonant frequency had shifted to $f_1 = 3483$ Hz. From Eq. (3.5) we then obtain a stiffness $k = 1.21 \cdot 10^{-4}$ N/m, which is in good agreement with the theoretical stiffness calculated above with Eq. (3.1). The effective mass, from Eq. (3.6) is 28.6 pg, which is 18 percent less than the mass calculated with Eq. (3.4). Since the measurement of the diameter of the spherical magnet is less prone to errors than the measurement of the beam dimensions, we prefer to use the effective mass obtained by the added-mass method.

At low temperatures, we characterize the cantilever resonance by studying its response to an excitation by a piezo-electric element located at the cantilever chip holder$^3$. With a lockin-amplifier$^4$ we measure the r.m.s. amplitude and phase of the SQUID voltage at the frequency at which the piezo element is actuated. In Fig. 3.4, we show the steady-state amplitude response of an IBM cantilever versus frequency (left panel), and the transient response after switching of a resonant excitation (“ringdown”) versus time (right panel).

The steady state frequency sweep is a good way to determine the resonant frequency of the cantilever very accurately. For high-Q cantilevers, the square of the amplitude is approximated well by a Lorentzian function of the frequency

$^3$Piezoceramic Tube 5A by EBL Products, Inc., USA; PICMA Chip Monolithic Multilayer Piezo Actuator PL033.30 by Physik Instrumente (PI) GmbH & Co., Germany.

$^4$SRS-830 by Stanford Research Systems, Inc., USA.
A Lorentzian fit to the data in Fig. 3.4 yields a resonant frequency of $f_0 = 3124.8$ Hz and a Full-Width-Half-Maximum (FWHM) of $\Gamma = 0.0869$ Hz, from which the quality factor $Q = f_0/\Gamma = 3.59 \cdot 10^4$ can be determined. It can be shown that the decay time of the cantilever amplitude is given by:

$$\tau_c = \frac{Q}{\pi f_0} = \frac{1}{\pi \Gamma}$$  \hspace{1cm} (3.7)

From the fit, we obtain $\tau_c = 3.66 \pm 0.03$ s. After every frequency step, we have to wait more than $3\tau_c$ for the transient cantilever response to die out sufficiently and to measure only the steady state response. For this reason, the piezo sweep is a very slow measurement. This particular frequency sweep took between 9 and 10 minutes.

In contrast, the ringdown measurement is a much more direct and fast way to measure the decay time and resonant frequency of high-Q resonators. The amplitude data in the right panel of Fig. 3.4, acquired at a different cantilever-coil alignment in the same run (hence the disagreement between parameters), can be fitted well by an exponential decay function, yielding $\tau = 3.526$ s. The measured phase lag with respect to the reference signal must be linear in time, with a slope:

$$\frac{d\phi(t)}{dt} = 2\pi(f_0 - f_{ref})$$  \hspace{1cm} (3.8)

A linear fit of our transient phase response yields $f_0 = 3125.114$ Hz. Using Eq. (3.7), the quality factor can be deduced: $Q = f_0\tau_c = 3.462 \cdot 10^4$.

Since the quality factor is a derivative quantity that depends on the resonant frequency, we rather use the cantilever damping factor $c = 2\pi \Gamma m = 2m/\tau_c$ as a characteristic property, with $m$ the effective mass of the cantilever-magnet assembly, and $\Gamma$ in unit Hz. From the piezo sweep we obtain $c = (8.93 \pm 0.07) \cdot 10^{-14}$ Ns/m and from the ringdown $c = (9.268 \pm 0.009) \cdot 10^{-14}$ Ns/m.
The typical damping factor values that we measured for IBM resonators at low temperatures range between $6.4 \cdot 10^{-14}$ Ns/m and $1.6 \cdot 10^{-13}$ Ns/m.

### 3.2 On-chip detection circuit for the MRFM cantilever

![Diagram of the on-chip detection circuit for MRFM cantilever](image)

Figure 3.5: Single stage cantilever detection circuit. A displacement of the cantilever magnet causes a flux change in the superconducting detection coil. The flux change is measured with a SQUID.

Figure 3.5 shows a diagram of our first generation SQUID-based cantilever detection circuit. The reason why the cantilever motion is not directly measured with a SQUID, but rather via an intermediate detection circuit, is that the SQUID needs to be shielded from strong static magnetic fields and radiofrequency magnetic pulses, both of which are necessary for MRFM measurements.

A displacement $x$ of the cantilever causes a flux change $\Phi$ through the superconducting detection coil with inductance $L_a$. The detection coil is connected to the SQUID input coil, which has an inductance $L_b = 1.6 \, \mu$H. The mutual inductance between the input coil and the SQUID is $M = 10 \, \text{nH}$. The voltage $U$ over the SQUID is, in Flux-Locked-Loop mode (see Sect. 3.3), proportional to the current $I$ in the detection coil. The responsivity of the detector is given by:

$$\frac{dU}{dx} = -\frac{M}{L_a + L_b} V_\Phi \frac{d\Phi}{dx}$$  \hspace{1cm} (3.9)

where $V_\Phi$ is the gain of the SQUID in V/Wb. We see that the detector becomes more sensitive as we decrease the detection coil inductance $L_a$, but is ultimately limited by the SQUID input coil inductance.

In our early experiments, we used a multiturn on-chip detection coil (see Fig. 3.6a) with an estimated inductance of $0.6 \, \mu$H. The calculated responsivity is then $dU/dx = 4.5 \cdot 10^{-3} V_\Phi \frac{d\Phi}{dx}$.  

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$^5$Quantum Design, Inc., USA.
In order to minimize the detection coil inductance, but also to implement an RF microwire close to the coil, we fabricated two generations of single-loop coils with on-chip RF wires, using electron beam lithography. The first coil, with a width of 100 \( \mu \)m and a design inductance of 0.25 nH, was patterned on a Si-(111) chip (see Fig. 3.6b). The latest design is a square coil with a width of 30 \( \mu \)m on a SrTiO\(_3\) chip and a design inductance of 0.08 nH, shown in Fig. 3.6c. To match the low detection coil inductance to the SQUID input coil, we implemented an additional transformer in the circuit. The schematic of this two-stage detection system is shown in Fig. 3.1.

The responsivity is now given by:

\[
\frac{dU}{dx} = \frac{M_{12}}{L_1L_2 - M_{12}^2} M_{2sq} V_\Phi \frac{d\Phi}{dx}
\]

\[
= \frac{\kappa_{12} \sqrt{L_{1b}L_{2a}}}{(L_{1a} + L_{1b})(L_{2a} + L_{2b}) - \kappa_{12}^2 L_{1b}L_{2a}} M_{2sq} V_\Phi \frac{d\Phi}{dx} \quad (3.10)
\]

where we have written the mutual inductance of the transformer as \( M_{12} = \kappa_{12} \sqrt{L_{1b}L_{2a}} \), with \( \kappa_{12} \in [0, 1] \) as the transformer coupling parameter. The optimal transformer parameters are \( L_{1b} = \frac{L_{1a}}{\sqrt{1 - \kappa_{12}^2}} \) and \( L_{2a} = \frac{L_{2b}}{\sqrt{1 - \kappa_{12}^2}} \). These values diverge for a perfect transformer coupling of \( \kappa_{12} = 1 \), but a more realistic estimation, like \( \kappa_{12} = 0.9 \), yields \( L_{1b} = 2.3L_{1a} \) and \( L_{2a} = 2.3L_{2b} \).

For the optimal transformer parameters, Eq. (3.10) simplifies to

\[
\frac{dU}{dx} = \frac{1}{\sqrt{L_{1a}L_{2b}} \sqrt{1 - \kappa_{12}^2}} \frac{\kappa_{12}/2}{1} M_{2sq} V_\Phi \frac{d\Phi}{dx} \quad (3.11)
\]

from which we again infer that it is advantageous to make the detection coil inductance \( L_{1a} \) as small as possible.

Figure 3.6d shows the 2-stage assembly with the \((10 \ \mu\text{m})^2\) detection coil on SrTiO\(_3\) (d1). The on-chip transformer (d2) is in this case poorly matched to the detection coil and the SQUID input coil: its design values are \( L_{1b} = 0.7 \) nH, \( L_{2a} = 0.28 \) \( \mu \)H, \( \kappa_{12} = 0.93 \), and \( M_{12} = 15 \) nH. Still, if we disregard the spurious inductance of the bonding wires, twisted pairs, and RF filters between the coils, we calculate the responsivity to be \( dU/dx = 0.12V_\Phi d\Phi/dx \), which is roughly a factor 27 better than for the single-stage detector. For the case of a coupling parameter of \( \kappa_{12} = 0.9 \) and the corresponding optimal parameter values \( L_{1b} = 2.3L_{1a} = 184 \) pH and \( L_{2a} = 2.3L_{2b} = 3.67 \) \( \mu \)H, an additional improvement of yet another factor 2.4 on this factor 27 might be expected.

### 3.2.1 Calibration procedure for the detection circuit responsivity

Since the uncertainties in estimating the spurious inductances in the detection circuit and coupling factors of the transformers are relatively large and differ per measurement run, we experimentally calibrate the detector responsivity in-situ. A calibration of \( dU/dx \) in Eq. (3.10) is possible in two independent steps.
Figure 3.6: These photographs show the three generations of detection coils that we have used. In a), a two-layer Nb detection coil with $2 \times 22$ windings is shown, which has an inner width of 230 $\mu$m, an outer width of 670 $\mu$m, and an estimated inductance of 0.6 $\mu$H. The coil is fabricated on a Si substrate. Figure b) is a SEM image of a $100\mu m \times 100\mu m$ single loop Nb coil on a Si-(111) chip. The linewidth of the loop is 4 $\mu$m, and it has an estimated inductance of 0.25 nH. At 105 $\mu$m from the coil center, we fabricated an RF microwire with a width of 10 $\mu$m. Figure c) and its inset are optical microscope images of a $30\mu m \times 30\mu m$ Nb coil with a linewidth of 1 $\mu$m on a SrTiO$_3$-(100) chip. The coil has an estimated inductance of 0.08 nH. The RF microwire has a width of 1 $\mu$m and is located at 18 $\mu$m from the coil center. Image d) shows a typical chip assembly that was used with coils b) and c): d1) is the SrTiO$_3$-(100) chip with the detection coil and the RF wire, d2) is an on-chip transformer that matches the low detection coil inductance to the high SQUID input coil inductance, and d3) is the calibration transformer.
We can write the power spectral density (PSD) function, $S_U$, of the SQUID voltage, $U$, as a function of the displacement PSD, $S_x$, of the cantilever tip:

$$S_U = \left( \frac{M_{12}M_{2sq}}{\eta L_1L_2} \right)^2 \left( \frac{d\Phi}{dx} \right)^2 V_\Phi^2 S_x \quad (3.12)$$

Here we have defined $\eta = 1 - \frac{M_{12}^2}{L_1L_2}$ (see Eq. (4.27)). In Ch. 4 we will demonstrate that the energy coupling between the cantilever and the detection coil is given by $\beta^2 \equiv \frac{(d\Phi/dx)^2}{k'\eta L_1}$ and can be determined experimentally by studying the cantilever response to a varying calibration flux. Here, $k'$ is the effective stiffness of the cantilever in the induced magnetic field of the detection coil. In terms of the energy coupling, $\beta^2$, Eq. (3.12) becomes

$$S_U = \frac{M_{12}^2M_{2sq}^2}{\eta L_1L_2^2} k' \beta^2 V_\Phi^2 S_x \equiv \xi^2 k' \beta^2 V_\Phi^2 S_x \quad (3.13)$$

the SQUID gain $V_\Phi$ can be easily measured by sending an integer number of flux quanta through the SQUID feedback coil and measuring the resulting voltage amplitude in Flux-Locked-Loop mode (see Sect. 3.3 below). The slope is typically in the order of $0.1 \text{ V/} \Phi_0$.

The only remaining unknown parameter in Eq. (3.13) is $\xi^2$. We can determine $\xi^2$ in an independent calibration step by measuring the PSD of the SQUID voltage at a temperature at which part of the primary detection circuit is in the metallic state, so that it has a finite resistance $R_1$. In our setup, this can be achieved by heating up the experiment above $T_{c,Al} = 1.2 \text{ K}$, such that the aluminium bonding wires between the detection coil and the transformer switch to the normal state. Alternatively, by implementing a tunable magnetic field, one could achieve the transition to the normal state by applying a field higher than $B_{c,Al} = 9.9 \text{ mT}$ [37].

The PSD of the voltage noise over the resistive wire is $S_{V_1} = 4k_B T R_1$ [38]. Over the primary circuits effective impedance $Z_1 = R_1 + j\omega\eta L_1$, this leads to a current noise $S_{I_1} = \frac{4k_B T R_1}{R_1^2 + \omega^2 \eta^2 L_1^2}$. The resulting PSD of the flux through the SQUID is

$$S_\Phi = \frac{M_{12}^2M_{2sq}^2}{L_2^2} \frac{4k_B T}{R_1^2} \frac{1}{1 + \omega^2 \eta^2 L_1^2 R_1^2} \equiv \frac{S_0}{1 + (f/f_0)^2} \quad (3.14)$$

We can write $\xi^2$ in terms of $S_0$ and $f_0$:

$$\xi^2 = \frac{M_{12}^2M_{2sq}^2}{\eta L_1L_2^2} \frac{\pi S_0 f_0}{2k_B T} \quad (3.15)$$

Equivalently, for a single stage detector such as in Fig. 3.5, we obtain:

$$\xi_{1\text{stage}}^2 = \frac{M^2}{L} = \frac{\pi S_0 f_0}{2k_B T} \quad (3.16)$$

In Fig. 3.7 we show the measured SQUID flux PSD above the Al transition temperature for both a single stage detector (red curve) and a double stage
detector (blue curve). The spectra are fitted with Eqs. (3.15-3.16). From the fit parameters $S_0$ and $f_0$ we obtain $\xi_{1\text{stage}}^2 = 46.7$ pH and $\xi_{2\text{stage}}^2 = 19.9$ pH.

Although the two-stage detector has roughly a two times lower measured value of $\xi^2$, its energy coupling $\beta^2$ to the cantilever magnet is three orders of magnitude larger than that of the single stage detector, because of the lower effective inductance of the primary circuit. Hence, the net linear detector responsivity of the two-stage detector $dU/dx \propto \sqrt{\xi^2 \beta^2}$ is about 20 times better.

We can now express the detector responsivity entirely as a function of experimentally verifiable quantities:

$$
\frac{dU}{dx} = \xi \beta \sqrt{k'} V_{\Phi}
$$

(3.17)

In the absence of external excitations, the motion of the cantilever is described by $\langle x^2 \rangle = \frac{k_B T}{k'}$ (see Eq. (2.8)). Hence, it follows that there exists a direct relation between the mean squared SQUID voltage and the cantilever temperature $T$, which is independent of the stiffness $k'$:

$$
\langle U^2 \rangle = \xi^2 \beta^2 V_{\Phi}^2 k_B T
$$

(3.18)
3.2.2 Microfabrication of a Nb coil and RF microwire on STO

In this section, we will discuss the fabrication and characterization of the Nb detection coil and RF wire on SrTiO$_3$ (STO) (shown in Fig. 3.6c) in more detail.

We chose a synthetic STO-(100) substrate$^6$ as an alternative to Si-(111) in order to compare the properties of paramagnetic defects between both crystals. In Fig. 3.8 we illustrate the lithography steps taken to fabricate a Nb structure on the substrate.

Figure 3.8: E-beam lithography steps for fabricating a Nb structure on an STO chip. 1) The copolymer, resist, and conducting polymer layers are spincoated. 2) The structure is written with the e-beam. 3) The conducting polymer layer is removed. 4) The resist is developed. 5) A Nb film is deposited in a sputtering chamber. 6) The resist and copolymer with the Nb film are removed, leaving only the written structure.

1. We spincoat a 300 nm layer of copolymer and a 200 nm layer of resist$^7$ on the chip. Since the substrate is non-conductive, we also have to spincoat a 50 nm layer of conducting polymer$^8$ in order to prevent local charging due to the electron beam current. The copolymer and resist layers are baked at 170 °C for 30 minutes after coating, the AquaSAVE layer is baked for 1 minute at 100 °C.

2. The chip is mounted in an electron beam lithography system$^9$ and the coil pattern is written. We used an e-beam spotsize of 52 nm for the fine structures and a charge dose of 194 µC/cm$^2$.

3. The chip is rinsed in ultrapure water, dissolving the AquaSAVE conducting polymer.

$^6$by SurfaceNet GmbH, Germany.
$^7$MMA(8.5)MMA EL 9 and 950 PMMA-A4, by MicroChem Corp., USA.
$^8$AquaSAVE 53za by Mitsubishi Rayon Co., Ltd., Japan.
$^9$eLiNE by Raith GmbH, Germany.
4. The resist is developed in MIBK:IPA=1:3 for 45 seconds, then rinsed in isopropanol. The difference in dissolution rates between the resist and the copolymer result in the overhanging resist structure shown in the figure.

5. A 147 nm thick niobium film is deposited on the chip in an in-house sputtering chamber.

6. The resist and copolymer are dissolved in acetone in an ultrasonic cleaner, “lifting off” the Nb film everywhere but at the written structure locations.

The superconductivity of the detection coil and RF wire is tested by measuring the critical current with a 4-terminal configuration in liquid helium. In Fig. 3.9 we show the resistance of a 1 µm wide RF line as a function of current through the wire. When the current is increased from $I = 0$ mA, the measured voltage drop over the wire is 0 V, until the critical current $I_{c,RF} = 11$ mA is reached. Then, the wire resistance jumps to $R = 45$ Ω. Since we generated the current by applying a voltage over a 100 Ω resistor in series with the RF wire, the current drops to 7.5 mA when the transition to the normal state occurs. Upon increasing the current further, the resistance increases slightly due to the resistive heating of the wire. When the current is subsequently decreased, the resistive heating keeps the wire above the critical temperature until 5.7 mA, at which current the wire makes a transition to the superconducting state. A similar measurement for two different detection coils yielded $I_{c, coil1} = 6.4$ mA, and $I_{c, coil2} = 4.5$ mA.

![Figure 3.9: A 4-terminal resistance measurement of a 1 µm wide, 147 nm thick Nb RF wire on an STO-(100) substrate. The measurement was conducted in a liquid 4He dewar at 4.2 K.](image)

Using the Biot-Savart law [17], we can now calculate that the maximum field we can generate at a distance of 1 µm from the RF wire center, without exceeding the critical current, is $B_{RF} = 2.2$ mT at a current of 11 mA.
3.3 SQUID theory and operation

A Superconducting QUantum Interference Device (SQUID) is a very sensitive flux-to-voltage transducer. It can be used to detect magnetic flux changes in the order of $1 \, \mu \Phi_0$. The theory of SQUIDs is quite involved and is extensively described in the literature, for example in “The SQUID Handbook” by Clarke and Braginski [39]. Here, we shortly recapitulate the basic theory and operation techniques.

The working of a SQUID is based on the phenomena of Josephson tunneling and flux quantization.

A Josephson junction consists of two weakly coupled superconducting electrodes that are connected via a point contact, a normal metal, or an insulator. The first Josephson relation describes the supercurrent flowing through the junction due to tunneling Cooper pairs:

$$I_J = I_0 \sin \Delta \phi$$

where $I_0$ is the critical current and $\Delta \phi$ is the phase difference between the macroscopic wave functions of the two electrodes. The second Josephson relation describes the time evolution of $\Delta \phi$ as a function of the voltage $U_J$ over the junction:

$$\frac{d\Delta \phi}{dt} = \frac{2\pi}{\Phi_0} U_J$$

where $\Phi_0 = \frac{\hbar}{2e} \approx 2.07 \cdot 10^{-15}$ Wb is the quantum of magnetic flux. For finite voltages, the Josephson current oscillates with a frequency $\omega_J = \frac{2\pi \langle U_J \rangle}{\Phi_0}$, or $\sim 484$ MHz/$\mu$V.

The current-voltage characteristic of a Josephson junction is highly hysteretic: on increasing the bias current $I_b$ from 0 A, the voltage changes abruptly from zero to a finite value when the critical current $I_0$ is exceeded, but when the current is again decreased, the voltage drops to zero at a value that is less than $I_0$. In order to remove this hysteresis, the junction can be shunted with a resistance $R$ connected in parallel to it. The junction hysteresis is characterized by the Stewart-McCumber parameter $\beta_c$, which is typically designed to be $\beta_c \equiv \frac{2\pi}{\Phi_0} I_0 R^2 C \leq 1$, where $C$ is the junction self-capacitance. In the limit $\beta_c << 1$ (the overdamped junction limit), the time averaged voltage over the junction is given by

$$\langle U_J \rangle = \begin{cases} 0 & , \quad I_b < I_0 \\ R \sqrt{I_b^2 - I_0^2} & , \quad I_b > I_0 \end{cases}$$

The quantization of magnetic flux through a superconducting loop stems from the fact that the phase change of the macroscopic Cooper pair wavefunction, when going around the loop once, must be $2\pi n$. Here, $n$ is the amount of flux quanta enclosed in the loop. A dc SQUID consists of two Josephson junctions connected in parallel in a superconducting loop. In Fig. 3.10a we show a schematic drawing of a SQUID with two identical junctions that are shunted with resistors $R$. The SQUID is biased with a current $I$. When a
Figure 3.10: a) Schematic drawing of a symmetric dc SQUID with shunted junctions. The bias current $I$ and the screening current $J$ determine the phase jumps at junctions 1 and 2. b) The modulation of the SQUID critical current $I_c$ versus the applied flux $\Phi_a$, calculated for a screening parameter $\beta_L = 0$ and sketched for a more realistic value of $\beta_L = 1$. We have set $\beta_c = 0$, which corresponds to perfect elimination of the junction hysteresis. c) The time averaged voltage $V$ over the SQUID is plotted versus the bias current for the extreme cases of integer and half-integer values of the normalized applied flux. The horizontal arrow indicates the modulation of critical current shown in graph b, while the vertical arrows indicate the voltage vs flux modulation shown in graph d. d) SQUID voltage versus applied flux, for three different settings of the bias current $I$. The straight line indicates the linearization of the SQUID transfer coefficient in FLL mode at the optimal working point.
magnetic field $B_a$ penetrates the loop, a screening current $J$ will flow to keep
the total flux at a multiple of $\Phi_0$. The restriction on the wavefunction phase
leads to a relation between the junction phase jumps:

$$\Delta \phi_2 - \Delta \phi_1 = 2\pi \frac{\Phi_a + L_{sq}J}{\Phi_0} = 2\pi (\phi_a + \frac{1}{2} \beta_L J/I_0)$$  (3.22)

Here, $\phi_a = \Phi_a/\Phi_0$ is the normalized applied flux, $L_{sq}$ is the SQUID inductance,
and $\beta_L \equiv \frac{2L_{sq}I_a}{\Phi_0}$ is the screening parameter, which together with the Stewart-
McCumber parameter completely characterizes the dc SQUID, in the absence
of noise.

The supercurrent through both junctions is:

$$I_1 = \frac{I}{2} + J = I_0 \sin \Delta \phi_1$$
$$I_2 = \frac{I}{2} - J = I_0 \sin \Delta \phi_1$$  (3.23)

If we assume that the SQUID inductance is negligible, so that $\beta_L \approx 0$, we
obtain for the total supercurrent:

$$I = I_0 (\sin \Delta \phi_1 + \sin \Delta \phi_2)$$
$$= 2I_0 (\sin(\Delta \phi_1 + \pi \phi_a) \cos(\pi \phi_a))$$
$$\leq 2I_0 |\cos(\pi \phi_a)|$$  (3.24)

It appears that the critical current of the SQUID, $I_c$, is different from that of
a single junction:

$$I_c = 2I_0 |\cos(\pi \phi_a)|$$  (3.25)

The periodic modulation of $I_c$ as a function of the applied flux is shown in Fig.
3.10b. It has a period $\Phi_0$. For $\beta_L=0$ we can evaluate the curve analytically.
In this case, $I_c$ oscillates between 0 and $2I_0$. For higher values of the screening
parameter, the modulation depth decreases. We have sketched the case $\beta_L = 1$,
which is in practice a common design value.

The equation for the time averaged voltage drop $V$ over the SQUID is
similar in form to Eq. (3.21):

$$V = \begin{cases} 0, & I < I_c \\ R\sqrt{I^2 - I_c^2}, & I_b > I_c \end{cases}$$  (3.26)

Since $I_c$ is periodic in $\phi_a$, the same goes for $V$. From the relation between $V$ and the bias current $I$ in Fig. 3.10c it becomes apparent that the SQUID can
be utilized as a flux sensor by applying a fixed bias current and measuring the
voltage drop $V$. In Fig. 3.10d $V$ is plotted versus $\phi_a$ for three different bias
currents, corresponding to the vertical arrows in Fig. 3.10c. In this manner
of operation, one has to set $I \geq 2I_0$ for the SQUID to be sensitive to all flux
values.

We usually operate the dc SQUID in “Flux-Locked-Loop” (FLL) mode, in
which the flux through the SQUID loop is kept at a constant value by means
of controlling the current in a feedback coil. The optimal working point for
FLL feedback is at the flux values for which \( dV/d\Phi \) is maximal. The linearized transfer function at one of the optimal working points is visualized in Fig. 3.10d. In this way, the dynamic range of the SQUID detector is increased such that not only changes in the order of \( \mu \Phi_0 \) can be detected, but many flux quanta as well.

Under optimal conditions, the spectral density of the SQUID flux noise is given by

\[
S_\Phi(\omega) = 16k_B T L_{sq}^2 / R
\]  

(3.27)

We use a commercial Quantum Design SQUID that can reach a noise level as low as 0.6 \( \mu \Phi_0 / \sqrt{\text{Hz}} \) at 0.5 K. For a typical flux-to-voltage transfer of 100 \( \mu \text{V}/\Phi_0 \), this corresponds to a voltage noise of 60 pV/\( \sqrt{\text{Hz}} \), which is more than an order of magnitude lower than the input noise of state-of-the art voltage preamplifiers. In order not to be limited by preamplifier noise, we use a two-stage detection scheme, depicted in Fig. 3.11, in which a second SQUID stage is used as a low-temperature preamplifier [40]. The sensor SQUID is coupled via a small resistor \( R_0 \) to the input coil of the amplifier SQUID. In this way, the transfer coefficient is increased by a factor 10–30 to \( \sim 1 \text{ mV}/\Phi_0 \). The two-stage SQUID is read out and controlled with commercially available electronics\(^{10}\). Both SQUIDs have to be independently biased. The voltage over the amplifier SQUID is electronically amplified at room temperature and fed back to the sensor SQUID feedback coil via a resistor \( R_f \).

The noise of the two-stage SQUID detector is determined by the flux noise of the sensor SQUID, which scales linearly with temperature. We would expect from Eq. (3.27) that the noise decreases to \( \sim 0.1 \mu \Phi_0 / \sqrt{\text{Hz}} \) at 10 mK. However, in Fig. 3.12, where we have plotted the measured white flux noise versus temperature of the experiment, we observe something different. Below

\(^{10}\)STAR Cryoelectronics, USA or Magnicon GmbH, Germany.
100 mK the noise floor is constant at $\sim 1.2 \mu \Phi_0 / \sqrt{\text{Hz}}$, while above 200 mK the noise rises significantly with temperature\[^{11}\]. The leveling off of the flux noise at lower temperatures is due to resistive heating in the shunt resistors [41]. This usually keeps the effective temperature of the SQUID at a few hundred milliKelvin.

![Flux noise spectral density versus temperature.](image)

**Figure 3.12:** Flux noise spectral density versus temperature.

### 3.4 Coarse alignment and fine scanning in the experimental chamber

Two important demands are set for the alignment of our MRFM cantilever tip: On the one hand, the cantilever tip has to be brought into close proximity of the sample in order to maximize the spin-magnet interaction. On the other hand, the cantilever magnet has to be aligned with respect to the detection coil, such that the detection sensitivity of cantilever motion is thermally limited. Because the sample has to be in the vicinity of the source of RF radiation, in our case a superconducting microwire, the in-situ alignment amounts to finding the optimal position with respect to the detection coil and the RF wire.

We manually align the cantilever to within a few micrometers of the ideal position at room temperature. As we mount the experiment in our cryostat and start cooling down to ultralow temperatures, the thermal contraction of the device causes a misalignment that is optically unverifiable. This necessitates

\[^{11}\]During these particular measurements, we did not reach the usual lowest noise floor of $0.6 \mu \Phi_0 / \sqrt{\text{Hz}}$, nor did the noise at higher temperatures seem to follow a $\sqrt{T}$ behavior. The former we attribute to a sub-optimal FLL working point, the latter to “jumps” in the voltage signal due to electronic shielding problems.
the implementation of an automatic positioning system with a sufficiently large range in all three dimensions that is compatible with cryogenic temperatures.

For the coarse alignment in our experiment, we have implemented a commercial solution: a “PiezoKnob”\textsuperscript{12} is a threaded spindle, contained in a nut, that can be rotated by applying torque pulses via piezo-ceramic elements in the head of the spindle. Thus, the translational range of the spindle can be in the cm range, while the stepping resolution is tens of nm. In order to reduce the friction and to improve the thermal and mechanical properties of the PiezoKnobs, we have developed new threaded spindles and nuts in-house, made out of bronze with a WS\textsubscript{2} coating, keeping only the PiezoKnob head from the original design.

Our experimental chamber is shown in Figs. 3.13a and b. In our implementation, three PiezoKnobs (1) are mounted in a brass platform (2), to which we have rigidly clamped a bronze rod with a cantilever holder (3) at its end. With strong tension springs (4) the platform is pulled towards the top of an Al chamber (5) that acts as a superconducting shield for the experiment. The chamber is coated with Au to improve thermalization at low temperatures. On the inside, it is covered with Nb foil in order to retain superconducting shielding above the critical temperature of Al. By independently driving the three PiezoKnobs, we can explore one translational and two rotational degrees of freedom of the platform, enabling us to move the cantilever tip with respect to the sample chip (6) in three dimensions, with a lateral range of approximately (400 $\mu$m)$^2$ and a vertical range in the order of a few mm. Close to each PiezoKnob, we have mounted a capacitive sensor\textsuperscript{13} (7) between the platform and the shielding chamber. The three sensors are read out with a capacitance bridge\textsuperscript{14}, which enables us to infer the vertical displacement of each knob with an accuracy of $\sim 1 \mu$m. It is then straightforward, using the dimensions of the platform, to calculate the position of the cantilever tip. The Printed Circuit Board (PCB) with the cantilever detection circuitry is mounted, via thin brass rods that penetrate the Al shielding, on a home-built 3D fine-scanning stage (8).

The fine stage, machined out of Al, is shown separately in Fig. 3.13c. Its working is based on a flexure hinge design \textsuperscript{[32]}; a movable central part of an Al block is connected to the outer body of the block by thin ridges and can be moved by actuating laminated piezoelectric extension stacks\textsuperscript{15}. Two $z$-stages are mounted on the moving part of an $xy$-stage. The scan range at room temperature was measured to be ($\Delta x, \Delta y, \Delta z$)$_{300K}$ = (18 $\mu$m, 20 $\mu$m, 16 $\mu$m). The actuation constant of this type of piezo stack is reported to decrease more than a factor of 4 when cooling from room temperature to 40 K \textsuperscript{[42]}, with a trend that suggests, upon linear extrapolation, a factor 6.7 decrease when cooling to $\sim 0$ K. From this extrapolation we estimate that the fine stage range at the base temperature of the cryostat is ($\Delta x, \Delta y, \Delta z$)$_{10mK}$ = (2.7

\textsuperscript{12}Janssen Precision Engineering B.V., The Netherlands.
\textsuperscript{13}Eurocircuits N.V., Belgium.
\textsuperscript{14}2500A by Andeen-Hagerling, Inc., USA.
\textsuperscript{15}P-883.51 by Physik Instrumente (PI) GmbH & Co. KG, Germany.
Figure 3.13:  

a) Schematic drawing of the MRFM experimental chamber. The numbers indicate the visible parts that are shown in the schematic drawing, with the addition of the SMA connectors for connecting semirigid coaxial lines in the cryostat to the RF microwave on the chip inside the chamber (9), and Cu rods for mounting the chamber inside the cryostat (10). 

b) The fully assembled experimental chamber. 

c) The fine-scanning stage with the PCB mounting rods on top (part 8 in drawing a).
There is a significant difference in the thermal expansion coefficients for Al (23 ppm/K at r.t. [43]) and for our piezo stacks (7 ppm/K at r.t. [42]), which causes a drift of the detection coil with respect to the cantilever tip when the experiment is cooled down. The length of the stacks is 18 mm. Taking into account the variation of the thermal expansion coefficients with temperature, we calculate that the mismatch in thermal contraction is in total 49 µm. Since the z-stages have a designed leverage of 2, the calculated finestage drift is $(\delta x, \delta y, \delta z)_{\text{finestage,calculated}} = (49 \ \mu m, 49 \ \mu m, 97 \ \mu m)$.

The total thermal drift between cantilever and detection coil is also due to the contraction variations between the Al shielding box, the brass platform, and the bronze PiezoKnob spindles and cantilever rod. Typically, we measure a drift of $(\delta x, \delta y, \delta z)_{\text{total,measured}} = (21 \ \mu m, 65 \ \mu m, 133 \ \mu m)$.

### 3.5 Vibration isolation in a cryogen-free dilution refrigerator

In order to cool our experiments to ultralow temperatures, we acquired a commercial cryogen-free dilution refrigerator\(^{16}\). This cryostat has a base temperature of less than 10 mK and a specified cooling power of 650 µW at 120 mK, which translates to 5 µW at 10 mK [43]. It is cooled to liquid $^4$He temperatures by a 2-stage pulse-tube refrigerator\(^{17}\), which has a first stage cooling power of 36 W at 45 K and a second stage cooling power of 1.35 W at 4.2 K. The implementation of a pulse-tube cooler greatly reduces the labour intensity of precooling the dilution refrigerator as compared to cryogen-cooled machines. More importantly, in light of the steep global increase of helium scarcity [44], running the cryostat is much less costly and does not depend on the quality or quantity of the helium supply.

However, the cooling principle of our pulse-tube refrigerator relies on a varying pressure between 0 and 17 bars [45], which is provided by a compressor. This results on the one hand in square wave-like, low frequency, kilonewton forces acting on the top parts of the cryostat, and on the other hand in kilohertz range acoustical vibrations due to the gas flow through the pulse-tube regenerator and the flexible hoses connected to the rotary valve and expansion vessels. Since we aim to image our samples with atomic resolution using an ultrasoft cantilever, we have implemented several modifications in order to reduce the vibrations that couple into the mechanical loop between tip and sample.

The various cryostat modifications are shown in Fig. 3.14. We can distinguish between measures that reduce the forces acting upon the cryostat, and measures that isolate the experiment from environmental vibrations.

We will first discuss the force reducing measures. The pressure variation in the pulse-tube (PT) is realized though a rotary valve that switches the PT

\(^{16}\)CF-650 by Leiden Cryogenics, The Netherlands.

\(^{17}\)PT415-RM by Cryomech, USA.
Figure 3.14: Schematic representation of the commercial cryogen-free dilution refrigerator with the implementation of vibration-reducing modifications.
inlet between the 0 and 17 bar outputs of a compressor, with a frequency of 1.4 Hz. In order to reduce the horizontal forces acting on the cryostat we have lengthened the hose between the PT and the rotary valve, implementing a flexible “swan-neck” shape, and placed the rotary valve on a swinging platform inside an acoustic isolation box. The hoses between the rotary valve and the compressor are loosely suspended with ropes from the ceiling of an adjacent hallway. In this way, the expansion of the hose between PT and rotary valve results in an acceleration of the rotary valve and the hoses to the compressor, rather than that of the PT head.

In the default configuration, the PT is rigidly connected to the cryostat at the room temperature, 50 K, and 3 K stages. The periodic expansion of the PT due to the pressure variations is reported to be 25 µm [46]. In order to reduce the forces acting between the top three cryostat stages, caused by this expansion, we have lifted the PT a few cm, so that it is resting on support rods and a rubber ring on the room temperature plate. The PT is thermalized to the 50 K and 3 K stages with soft copper braids. In Fig. 3.16a, two photographs show the PT when it is lifted completely out of the cryostat (left) and when it is reinstalled at a higher position (right), thermalized by the Cu braids.

Now we turn to the vibration isolation of the experimental chamber. The three support legs of the cryostat are placed on a concrete block that is separated from the foundation of the surrounding building. We have stiffened the connection between the legs and the outer vacuum chamber (OVC) by making the leg connections to the room temperature plate more bulky as well as by adding rods between the bottom of the OVC and the legs, thus creating triangular support structures.

The default connection between the 3 K plate and the still plate is provided by rigid poles. We have removed these poles and suspended the lower three stages of the cryostat from the 3 K plate with tension springs\(^\text{18}\) (see left panel of Fig. 3.15a). The total mass of the suspended part is ~ 55 kg. We use 5 pairs of springs with a stiffness of 1.31 N/m, which leads to an estimated vertical resonant frequency of 3 Hz. In order to reduce the vibration amplitude at this frequency, which is uncomfortably close to the second harmonic of the PT excitation, we implemented an eddy current damper that is thermalized at the 3 K plate.

Furthermore, we added a mass-spring vibration isolation system below the mixing chamber plate, consisting of three 5 kg brass masses, suspended from tool steel ring springs, which was designed to provide a 100 dB vibration isolation in a frequency range between 1 kHz and 5 kHz [47] (see right panel of Fig. 3.15a). The masses are thermalized to the mixing chamber with commercial soft Cu tape, clamped with brass bolts. The experiments were always mounted on the lowest or second-to-lowest mass of this mass-spring system.

Figure 3.15 shows the improvement in SQUID noise due to the suspension of the still plate and the implementation of the mass-spring vibration isolation. This was an early modification, before lifting the PT and adding the leg stiffening rods. The red curve in graph b shows the PSD of the SQUID voltage noise

with the SQUID on the mixing chamber plate in the cryostat as delivered (but
with the rotary valve suspension and the cryostat placement already as drawn
in Fig. 3.14). The black curve was measured with the SQUID on the second
suspension mass, after implementation of the still suspension. The signals are
resulting from the motion of the detection coil in the ambient magnetic field
gradient that exists inside the cryostat. Although we did not calibrate for a
displacement measurement, it becomes clear from the spectra that the suspen-
sion systems lead to a tremendous reduction of the vibrations over the whole
measurement bandwidth. Above 200 Hz, the noise floor is now determined
by the intrinsic detection SQUID flux noise instead of experimental vibrations.
Below 10 Hz, the harmonics of the PT modulation \((n \cdot 1.4 \text{ Hz})\) emerge that were
first obscured by low frequency background noise. The total noise reduction is
quantified in graph c, where we have plotted in black the absolute difference
in r.m.s. voltage noise \(\Delta_{r.m.s.,\text{abs}}\) before and after the modification versus the
integration bandwidth \(\Delta f\):

\[
\Delta_{r.m.s.,\text{abs}}(\Delta f) = \sqrt{\int_0^{\Delta f} S_{V,\text{before}} df} - \sqrt{\int_0^{\Delta f} S_{V,\text{after}} df}
\]  (3.28)

In red, we have plotted the the ratio \(\Delta_{r.m.s.,\text{rel}}\) between r.m.s. noise before and
after the modifications:

\[
\Delta_{r.m.s.,\text{rel}}(\Delta f) = \frac{\sqrt{\int_0^{\Delta f} S_{V,\text{before}} df}}{\sqrt{\int_0^{\Delta f} S_{V,\text{after}} df}}
\]  (3.29)

The absolute reduction over the whole bandwidth is 0.25 V, the bulk of which
is gained at frequencies below 1 Hz. The full-bandwidth relative improvement
is a factor 5.1.

The effect of lifting the PT cooler was quantified by measuring the vi-
brations at the 3 K plate with rotating-coil geophones\(^\text{19}\). The geophones are
calibrated for vertical displacements in a frequency range between 1 Hz and
100 Hz. In Fig. 3.16b we show the PSD of the displacement noise before
and after lifting the PT. The vibration peaks at multiples of the PT modula-
tion frequency are clearly reduced up to the 8th harmonic. Integration of the
spectra leads to the curves for the absolute (black) and relative (red) r.m.s.
displacement reduction, shown in graph c. The total reduction in vertical dis-
placement over the calibrated bandwidth is 0.94 \(\mu\text{m}\), which is dominated by
the reduction of the PT fundamental frequency at 1.4 Hz. The most significant
steps in the vibration reduction can all be clearly related to higher harmonics
of the PT vibration. The relative improvement below 4 Hz is a factor 2.9, but
since the displacement at 4.2 Hz remains relatively large, the full-bandwidth
improvement reduces to a factor 2.3.

The latest modification was to add rods between the OVC and the cryostat
support legs in order to make the construction stiffer. In the original design,\(^\text{19}\)GS11D, by Geospace Technologies, USA.
Figure 3.15: Vibration reduction after suspension of the still plate from the 3 K plate and adding a mass-spring system below the mixing chamber. a) Photographs of the modifications. b) PSD spectra of the SQUID noise. c) Absolute (black) and relative (red) vibration reduction vs bandwidth.
Figure 3.16: Vibration reduction after lifting the pulse-tube cooler. a) Photographs of the modifications. b) PSD spectra of geophone signals. c) Absolute (black) and relative (red) vibration reduction vs bandwidth.
the connection between the support legs and the room temperature plate was quite floppy. The stiff cylindrical OVC connected to this plate acted like a vibration antenna that seemed to resonate with the 3rd harmonic of the PT modulation at 4.2 Hz. Indeed, if we compare the PSDs of the displacement of a detection coil at the bottom suspension mass, with and without the extra rods (see Fig. 3.17a), we observe that the peak at 4.2 Hz is reduced by a factor 2.5. In graph b we can see that the modification increases the measured vibrations at some other frequencies, most clearly at 1 Hz and at 342 Hz, but that it is still an improvement of a factor 1.2 when integrated over the whole measurement bandwidth. In Fig 3.17c, we have plotted the horizontal r.m.s. displacement of the detection coil with respect to the Al shielding box versus the integration bandwidth after the implementation of all modifications. The total r.m.s. displacement noise is only 61 pm, which is more than sufficient to enable atomic-scale imaging.
Figure 3.17: Vibration reduction after adding leg-stiffening rods between the OVC and the support legs. 

- **a)** PSD spectra of the SQUID detection coil displacement.
- **b)** Absolute (black) and relative (red) vibration reduction vs bandwidth.
- **c)** Net vibration after all modifications.