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Advances in SQUID-detected magnetic resonance force microscopy

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A

FEEDBACK COOLING OF THE CANTILEVER'S FUNDAMENTAL MODE

The fundamental sensitivity of MRFM is limited by the thermal force noise. This noise can be reduced by lowering the temperature of the cantilever. However, this is complicated by the limited thermal conductance of all the different components combined with the constant influx of power from various sources. This can create large temperature gradients between the experiment's vital components, such as the sample and the cantilever, and the source of cooling (e.g. the mixing chamber). To give an example from Poggio et al.: in a cryostat with a base temperature of 250 mK, the cantilever temperature saturated at 2.2 K due to the laser used for the detection of the cantilever [44].

In this appendix, we discuss our efforts to cool the cantilever, focusing on the achieved cantilever temperature and thermal force noise, and the feedback cooling of our resonator to an effective mode temperature below 150 μ K. Note that the experiments in this chapter were performed in one of the older versions of the setup than that explained in Ch. 2. All relevant experimental details will be discussed.

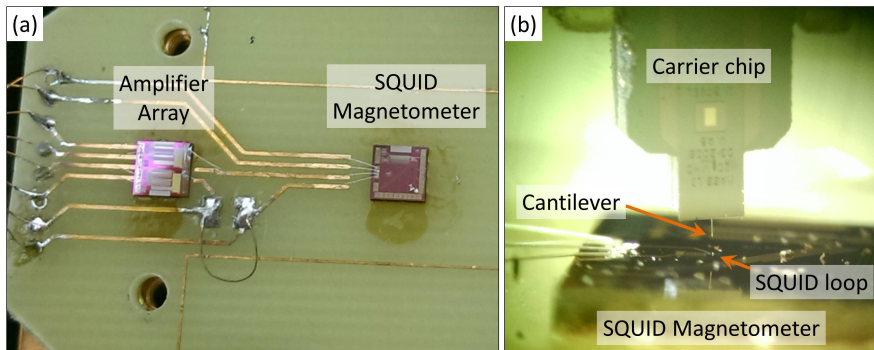


Figure A.1: Setup used for the experiments described in this section. (a) Photograph of the improvised two-stage SQUID, consisting of a magnetometer coupled to a SQUID array. (b) Optical microscope image of the cantilever placed directly above the SQUID loop of the magnetometer.

A.1 CANTILEVER TEMPERATURE AND THERMAL NOISE FORCE

In order to achieve the lowest possible cantilever temperature, several measures were taken to ensure a good thermalization to the mixing chamber of the dilution refrigerator. First of all, the silicon chip which supports the cantilever is attached to a copper block using a brass spring, and further thermalized using some silver epoxy at the base of the chip. This copper block is directly connected to the mixing chamber via a silver wire with a diameter of 1 mm and a length of about 30 cm. When we assume the silver is the limiting factor in the thermal conductance, this configuration leads to an optimal power tolerance of 3 nW/mK at 100 mK. However, since this estimate ignores the thermal resistance at the interfaces of different components and materials, the actual thermal conductance should be much lower.

To determine the lowest temperature at which the cantilever saturates, we place the cantilever directly above a SQUID¹ (see Fig. A.1) in order to maximize the coupling and thereby the SNR. We measure the thermal motion of the cantilever by looking at the power spectral density of the SQUID signal at different temperatures. Two representative curves measured at 12 mK and 500 mK can be seen in the upper image of Fig. A.2. The thermal motion of the cantilever's fundamental mode at 2727 Hz is clearly visible above the noise. We use a cantilever with a magnet with a radius of 1.5 μm , and a stiffness of 50 $\mu\text{N/m}$. The smaller bumps visible in the bottom

¹PTB GX1GFM Magnetometer, connected to a PTB 5X1GF4 SQUID series Array used as amplifier

curve are mechanical vibrations of the setup amplified by the transfer function of the cantilever.

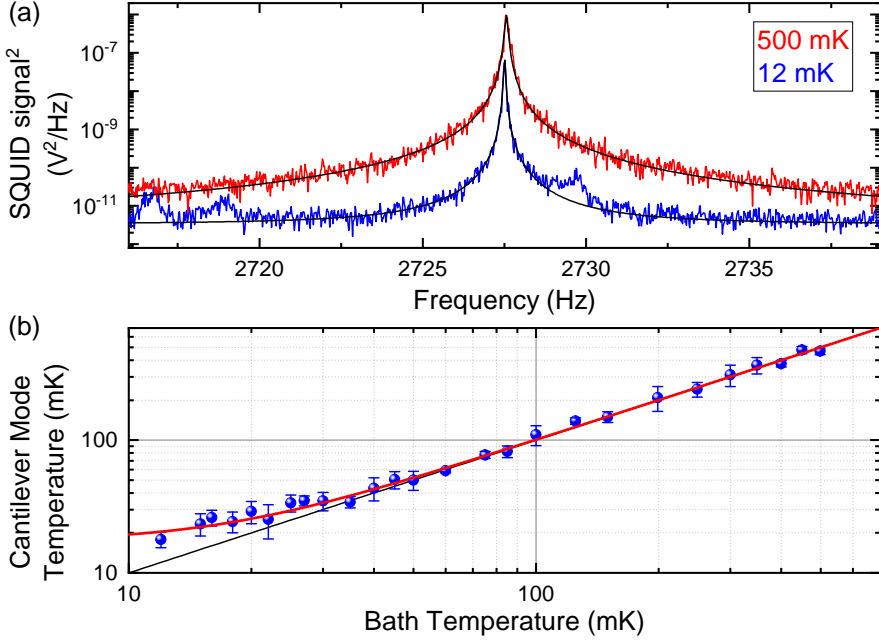


Figure A.2: (a) SQUID signal for two bath temperatures. The black lines are Lorentzian fits to the data. (b) Cantilever noise temperature calculated from the area beneath the peaks obtained from the fit. The red solid line is a fit to the data using Eq. A.2, with $T_0 = 17.3 \pm 3.3$ mK, and $n = 2.2 \pm 0.6$.

Once the coupling between the magnetic particle and the SQUID is known, the area obtained from a fit of the data to a Lorentzian peak can be used to calculate the effective mode temperature, and via the equipartition theorem² this can be translated to the mean square displacement of the resonator. The coupling, which depends on the position of the cantilever with respect to the SQUID, can be experimentally determined by using a calibration coil in the SQUID input coil circuit to drive the cantilever [53]. Alternatively, the coupling can be determined by assuming that at high temperatures the cantilever temperature is equal to the bath temperature. This gives us

$$\frac{1}{2}k\langle x \rangle^2 = \frac{1}{2}k_B T = \frac{1}{2}k_B \beta A \quad (\text{A.1})$$

²The equipartition theorem relates the thermal energy of a system to its average potential energy. In the case of an harmonic oscillator, it simply states that $\frac{1}{2}k_B T = \frac{1}{2}k\langle x \rangle^2$.

with β the conversion factor between the area and the temperature in units K/V^2 , and A the area under the curve of the power spectral density.

The measured cantilever noise temperature is shown in Fig. A.2(b). Our assumption that at high temperatures the cantilever temperature is equal to the bath temperature is justified by the linear behavior observed above approximately 50 mK, as indicated by the black diagonal line. Below this temperature, a saturation of the cantilever noise temperature can be observed. The red solid line is a fit to the data using a saturation curve [47]:

$$T_N = (T^n + T_0^n)^{1/n} \quad (\text{A.2})$$

The best fit to the data was obtained when using a saturation temperature $T_0 = 17.3 \pm 3.3$ mK, and an exponent $n = 2.2 \pm 0.6$, where the value of the exponent n is determined by the temperature dependence of the limiting thermal resistance. The acquired value for n indicates that the thermal conductance is due to conduction electrons [109], whereas the first Oosterkamp MRFM setup, which was used in the work of Usenko et al. [47], appeared to be limited by phonon processes or boundary effects. This difference could be due to the improved direct electrical connection of the cantilever to the mixing chamber via the silver wire and silver epoxy. Since the thermal conductance via electrons is much better than via phonons at low temperatures, this might also explain the reduction in the saturation temperature of the cantilever by nearly a factor of 2 when compared to the work by Usenko et al..

By using the conversion factor defined in Eq. A.1, we can now use the SQUID signal power spectral density to calculate the displacement noise, as shown in Fig. A.3.

With the data we have gathered so far, we can calculate some numbers which indicate the final measurement sensitivity of our setup, such as the thermal force noise, which is given by

$$\sqrt{S_F} = \sqrt{4k_B T \frac{k}{2\pi f_0 Q}} \quad (\text{A.3})$$

In recent years, several groups have reached astonishingly low values for the thermal force noise, reaching well within the zeptoNewton range, using a variety of resonator geometries [228–231]. However, most of these geometries are unsuited for MRFM. For our cantilever, assuming $k = 50 \mu\text{N}/\text{m}$, $f_0 = 2727$ Hz and $Q = 5 \cdot 10^4$ at low temperatures, we calculate a force noise $\sqrt{S_F} = 0.23 \text{ aN}/\sqrt{\text{Hz}}$. This value is very close to the lowest values ever reported for the soft cantilever geometries necessary for magnetic resonance force microscopy [45, 232, 233].

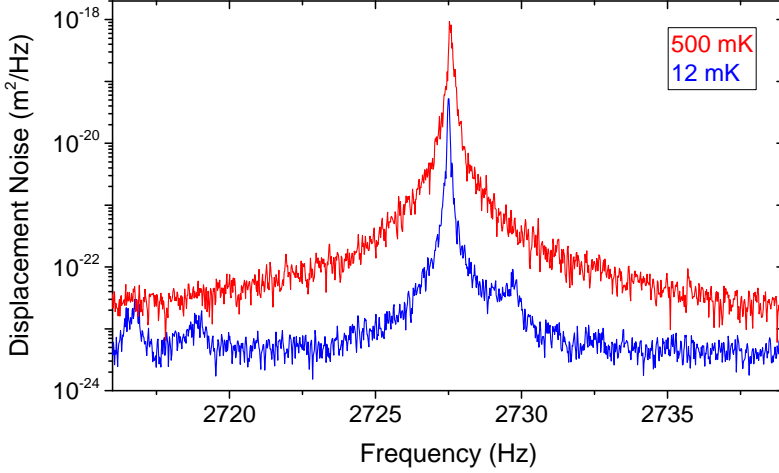


Figure A.3: Displacement spectra showing the thermal motion of the cantilever for temperatures of 500 mK (red) and 12 mK (blue).

A.2 FEEDBACK COOLING OF THE CANTILEVER'S FUNDAMENTAL MODE

Since the effective temperature of our resonator appears to be limited by the performance of the dilution refrigerator, the next step to reduce the motion of the resonator is to implement a technique called feedback cooling. In this technique, a high precision measurement of the motion of a resonator is used to perform active feedback on said motion, thus introducing an additional damping of the resonator. Feedback cooling is widely used for a variety of reasons:

- One of the ultimate goals is to use MRFM to detect the magnetic moments of individual nuclei with Ångström-scale spatial resolution. Detecting such small forces requires the smallest possible spring constant. However, since the mean-square amplitude of a cantilever's Brownian motion is given by $\langle x^2 \rangle = k_B T / k$, reducing the spring constant might lead to a thermal motion exceeding the desired imaging resolution [234]. Furthermore, the Brownian motion of the resonator also introduces a fluctuation of the polarizing field felt by the spins with an amplitude of $B_x = x_{\text{rms}} \partial B / \partial x$ [235]. To solve these issues feedback cooling must be used to reduce the cantilever's motion to within an acceptable range.
- As the quality factor of a resonator increases, its bandwidth is reduced. Since

the amplitude of a resonator decays with a typical time $\tau = Q/\pi f_0$, the time between independent measurements becomes very large. By reducing the quality factor using feedback, the decay time can be kept short, which increases the bandwidth of the experiment without sacrificing the force sensitivity [57].

- A cold resonator with a low number of phonons is useful for a variety of experiments exploring the limits of quantum mechanics [236–238]. This topic is extensively covered in the thesis of de Voogd [59].

The setup used for the feedback cooling experiment was the same as the one used in the previous section on the effective cantilever temperature, but at a position with a slightly better coupling to the pickup loop, which affects the detection noise floor.

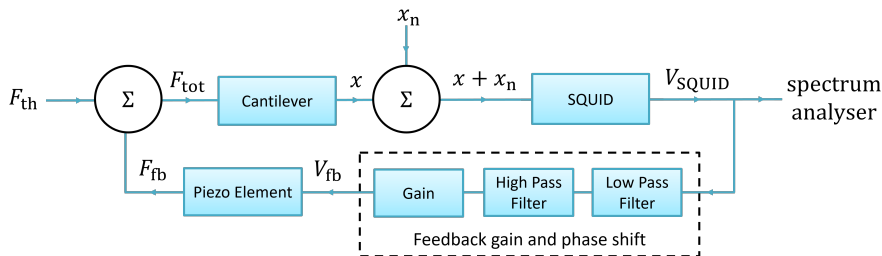


Figure A.4: Diagram of the experimental setup used for the feedback cooling

In order to perform the feedback, the SQUID signal containing the information about the cantilever motion is sent through a low-pass filter followed by a high-pass filter³ to add a gain and phase shift. The bandwidth of both filters is adjusted to obtain the desired phase shift with a random attenuation. This signal is then sent to an amplifier with a tunable gain⁴. This altered signal is then sent to a piezoelectric element which is mechanically coupled to the cantilever. A diagram of this setup can be seen in Fig. A.4. When the phase shift is set in such a way that the feedback is negative, this scheme results in a damping of the cantilever motion, proportional to the velocity of the cantilever. Simultaneously, the SQUID signal is sent to a spectrum analyzer to measure the resulting cantilever motion.

The response of the cantilever to this feedback signal can be calculated from an equation of motion very similar to the one defined in Eq. 2.10, but with an additional force term [44]:

$$m\ddot{x} + \Gamma\dot{x} + kx = F_{\text{tot}} = F_{\text{th}} - g\gamma(\dot{x} + \dot{x}_n), \quad (\text{A.4})$$

³SRS SIM965 Analog Filter

⁴SRS SIM911 BJT Preamplifier

where F_{th} is the random thermal force, g the gain of the feedback, and x_n is the detector displacement noise. The reason this last term is present here and not in Eq. 2.10, is that now also the detector displacement noise is coupled back to the cantilever motion via the feedback mechanism.

Considering this equation of motion, the spectral density of both the actual displacement and the measured displacement of the cantilever can be calculated, following Poggio et al. [44]. The actual displacement spectral density is given by

$$S_x(\omega) = \left[\frac{1/m^2}{(\omega_0^2 - \omega^2)^2 + (1+g)^2 \frac{\omega_0^2 \omega^2}{Q_0^2}} \right] S_F + \left[\frac{g^2 \omega_0^2 \omega^2 / Q_0^2}{(\omega_0^2 - \omega^2)^2 + (1+g)^2 \frac{\omega_0^2 \omega^2}{Q_0^2}} \right] S_{x_n}, \quad (\text{A.5})$$

and the measured displacement spectral density by

$$S_{x+x_n}(\omega) = \left[\frac{1/m^2}{(\omega_0^2 - \omega^2)^2 + (1+g)^2 \frac{\omega_0^2 \omega^2}{Q_0^2}} \right] S_F + \left[\frac{[(\omega_0^2 - \omega^2)^2 + \omega_0^2 \omega^2] / Q_0^2}{(\omega_0^2 - \omega^2)^2 + (1+g)^2 \frac{\omega_0^2 \omega^2}{Q_0^2}} \right] S_{x_n}, \quad (\text{A.6})$$

with S_{x_n} the spectral density of the detection noise, and $S_F = 4k_B T \Gamma$ the standard thermal force spectral density with T the cantilever temperature at zero gain.

The result of the feedback cooling of the cantilever, starting from a Q-factor of $5.2 \cdot 10^4$ at a temperature close to 20 mK is shown in Fig. A.5. The solid lines are fits to the data according to Eq. A.6. Fitting the data can be challenging due to the high number of parameters and, especially in the case of the purple curve, the low SNR. In the procedure we followed, we fix the parameters for the mass and spring constant by calculating them based on the cantilever geometry. For T , we take the calibrated cantilever temperature based on the procedure described in section A.2. We fix Q_0 at the value obtained from a Lorentzian fit to the data at zero gain. This only leaves three parameters free: the resonance frequency f_0 , the gain g , and the detection noise S_{x_n} .

This procedure works well for all but the highest gain data. To get a reliable value for the gain for this data, we also fix f_0 and S_{x_n} to the values found for the second highest gain. The gain we obtain in this way can then be used to calculate

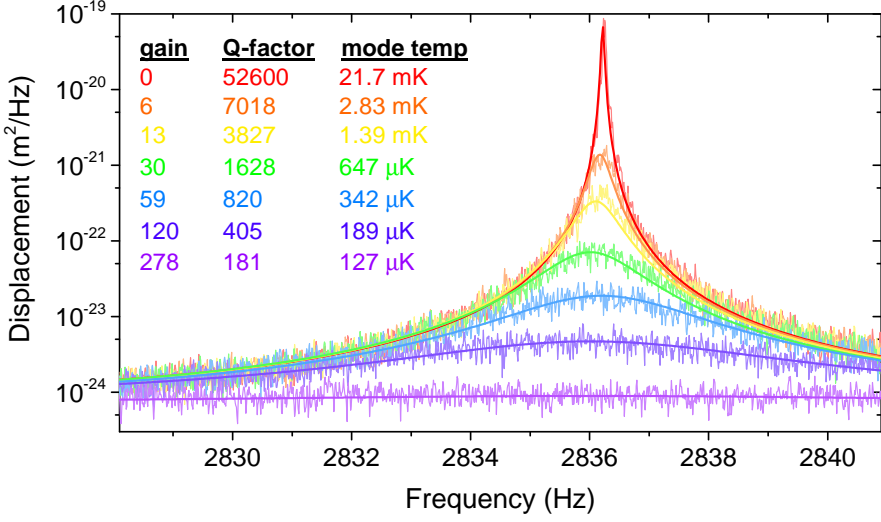


Figure A.5: The main result of the feedback cooling, indicating a final mode temperature of 127 μK . The solid curves are fits to equation A.6. The Q factors of all curves (apart from the bottom one) are obtained from a Lorentzian fit to the data.

the Q using $Q = Q_0/(1 + g)$ [48, 239].

The fit values obtained from Fig. A.5 can be used to calculate the final mode temperature achieved by the feedback cooling, using [44]

$$T_{\text{mode}} = \frac{T}{1 + g} + \frac{k\omega_0}{4k_{\text{B}}Q_0} \left(\frac{g^2}{1 + g} \right) S_{x_n}, \quad (\text{A.7})$$

from which we find that at our maximum gain (limited by the detection noise) we have achieved a mode temperature of 127 μK . The minimal achievable temperature is given by

$$T_{\text{min}} = \sqrt{\frac{k\omega_0 T}{k_{\text{B}}Q_0}} S_{x_n} = 122 \mu\text{K}, \quad (\text{A.8})$$

given our parameters and measured force noise.

This mode temperature corresponds to a phonon occupation number

$$N_{\text{phonons}} = \frac{k_{\text{B}}T_{\text{mode}}}{\hbar\omega_0} = 937, \quad (\text{A.9})$$

at a thermal force noise of 0.23 $\text{aN}/\sqrt{\text{Hz}}$ and a displacement noise floor of 840 $\text{fm}/\sqrt{\text{Hz}}$.

B

LIMITATIONS OF THE MECHANICAL GENERATION OF RADIO-FREQUENCY FIELDS

While the higher modes have the potential for generating very large B_1 fields with very little dissipation, we have also encountered some serious drawbacks of the presence and use of the higher modes. In this appendix, we will demonstrate some of these limitations based on saturation experiments performed on copper, as described in Ch. 4.

B.1 OFF-RESONANT COUPLING

The first issue we discuss is the unintended driving of one or more of the higher modes, even when the applied RF pulse is far off-resonance with the higher mode. During the pulse, the cantilever higher mode acts like a forced damped harmonic oscillator, with equation of motion

$$\ddot{x}(t) + \Gamma\dot{x}(t) + \omega_0^2 x(t) = \frac{F_0}{m} \cos(\omega t), \quad (\text{B.1})$$

with m the mass of the oscillator, $\omega_0^2 = k/m$ the natural frequency, Γ the damping, and F_0 the amplitude of the external force. For $\omega \neq \omega_0$, no damping, and the initial conditions where the cantilever is stationary at $t = 0$, the general solution is given by

$$x(t) = 2A_0 \sin\left[\frac{(\omega_0 + \omega)t}{2}\right] \sin\left[\frac{(\omega_0 - \omega)t}{2}\right], \quad (\text{B.2})$$

with

$$A_0 = \frac{F_0/m}{\omega_0^2 - \omega^2}. \quad (\text{B.3})$$

So, if $\omega \approx \omega_0$, we can look at $x(t)$ as the product of a slow modulation with an amplitude $2A_0 \sin((\omega_0 - \omega)t/2)$, and a rapid oscillation with amplitude $\sin((\omega_0 + \omega)t/2)$. The amplitude of the modulation increases as ω approaches ω_0 . When $\Gamma > 0$, the motion decays to zero as time progresses, resulting in a steady oscillation with amplitude

$$A(\omega) = F_0 / [m^2(\omega_0^2 - \omega^2)^2 + \Gamma^2\omega^2]^{(-1/2)} \quad (\text{B.4})$$

When the pulse is switched off, the cantilever motion starts to decay following

$$x(t) = A_1 \exp(-\omega_0 t/2Q) \cos(\omega_0 t), \quad (\text{B.5})$$

where A_1 is the amplitude of the cantilever at the end of the pulse. We assume weak damping.

We demonstrate the effect of this behaviour in Fig. B.1(a), where we show the direct frequency shift after an RF pulse with a frequency of 950 kHz, which is in between the higher modes at 756 kHz and 1009 kHz, and very short pulse durations. We observe an oscillation of the direct frequency shift, which in the past was wrongfully interpreted as a potential Rabi oscillation [205]. The period of the oscillation is 18 μ s, which indicates that it originates for the beating with the higher mode at 1.009

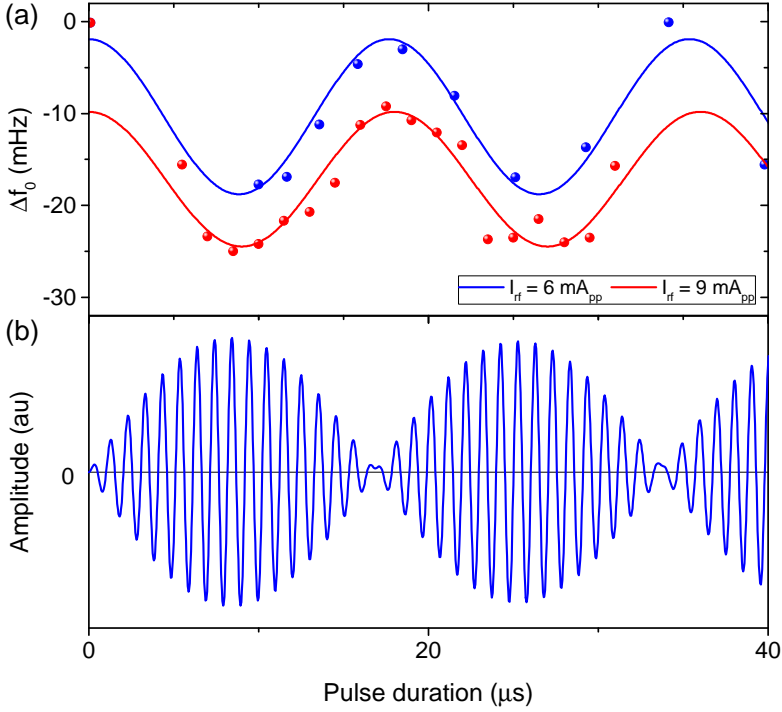


Figure B.1: (a) Direct frequency shift Δf_0 versus pulse duration at RF frequency $f_{RF} = 950$ kHz, $T = 30$ mK, and $h = 0.95$ μm , for 2 different B_1 fields. The solid curves are fits to a simple cosine, from which we extract oscillation periods of 17.7 ± 0.3 and 18.0 ± 0.2 μs for the 6 and 9 mA_{pp} data sets, respectively. (b) Simulation of the motion $x(t)$ of the 1.009 MHz higher mode when excited by a 950 kHz drive force.

MHz, 59 kHz off-resonance. Fig. B.1(b) shows the simulated amplitude of the 1.009 MHz higher mode when excited with a periodic driving force at 950 kHz.

Thus, the ‘‘Rabi’’ oscillations observed for very short pulse times are in reality caused by the motion of one or more of the higher modes, generating a B_{RF} field with an amplitude which varies with the slow beating frequency, and with a duration determined by the exponential decay with characteristic time $\tau = Q/\pi f_n$.

B.2 NON-LINEARITIES

The second issue is the inherent non-linearity of the higher modes of the cantilever. In Fig. B.2(a) we vary the frequency of the drive field we use to excite the higher mode at 756 kHz. We drive the higher mode using RF currents of about $3 \mu\text{A}_{\text{pp}}$ (black), $10 \mu\text{A}_{\text{pp}}$ (red), and $30 \mu\text{A}_{\text{pp}}$ (green). The solid lines in the figure are guides to the eye. The asymmetry of the curves shows that even at extremely small driving amplitudes the non-linearities of the cantilever dominate the total B_{RF} field.

In Fig. B.2(b) we see a measurement where we drive the same higher mode, but now far away from the sample. In this case, we drive the mode using the cantilever piezo, and we measure the response using the induced magnetic field in the pickup loop. Also far from the surface we observe a clear non-linearity, indicating that at least parts of the non-linearity are an inherent property of the cantilever. We assume that the non-linearities are caused by stress at the surface of the cantilever, which would mean this issue intensifies for higher mode numbers. The slight mismatch between the mode frequency in both figures is attributed to a small sample-induced frequency shift in the top figure.

B.3 TEMPERATURE DEPENDENCE OF QUALITY FACTOR

Finally, we report a large temperature-dependence of the quality factor of the higher modes on the cantilever temperature. Measurements of the temperature-dependence of the Q-factor for the 756 kHz mode is shown in Fig. B.3(b). The solid red line in this curve indicates a $1/T$ dependence. The precise origin of the dissipation responsible for this Q-factor is unknown. For low temperatures, the Q-factor increases to over $5 \cdot 10^5$, with a characteristic time $\tau > 0.2$ s. For higher frequency modes, Q-factors in excess of 1 million have been observed.

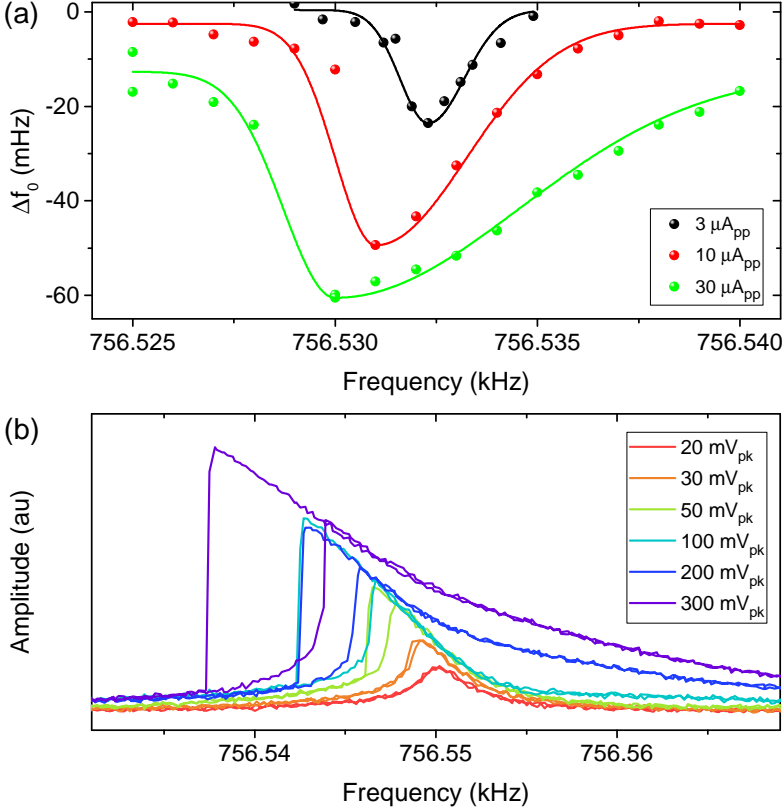


Figure B.2: (a) Direct frequency shift Δf_0 for various drive frequencies around the higher mode frequency. The amplitude of the RF currents are 3 μA_{pp} (black), 10 μA_{pp} (red), en 30 μA_{pp} (green). The solid lines are guides to the eye. An asymmetry of the signal and thus B_{RF} field generated by the higher mode indicates a strong non-linearity of the mode. (b) Response of the higher mode when driven by the cantilever piezo at various drive amplitudes far away from the sample. The drive frequency is swept from frequencies below the resonance to frequencies above the resonance and back. The signal is obtained from the magnetic field measured by the SQUID.

It is possible to convert the Q -factor to a dissipation constant. The shape of the higher vibrational modes of the cantilever induces a rotation of the magnet at the tip of the cantilever, which means we can calculate the dissipation constant using

$$\Gamma_n = \frac{\kappa_n}{Q\omega_n}, \quad (\text{B.6})$$

where κ_n is the torsional spring constant, given by

$$\kappa_n = \omega_n^2 I, \quad (\text{B.7})$$

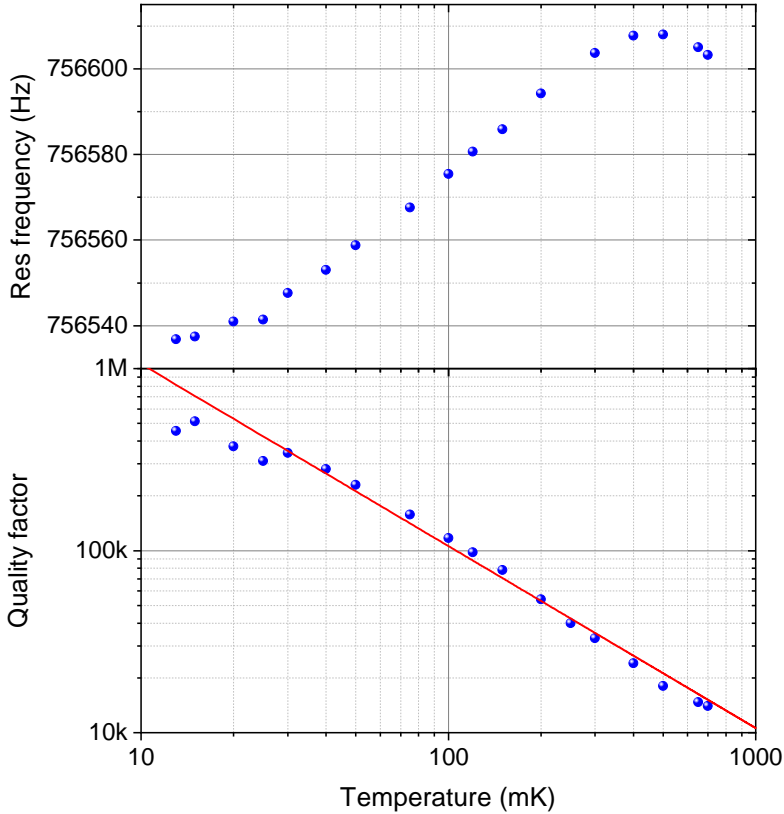


Figure B.3: (a) Resonance Frequency and (b) quality factor of the higher mode at 756 kHz measured for different temperatures of mass 3, when the magnetic particle is far away ($> 5 \mu\text{m}$) from the surface. The red solid line in (b) indicates a $1/T$ dependence.

with I the moment of inertia. When we assume the moment of inertia is dominated by the rotation of the heavy spherical magnet, it is given by $I = (2/5)mR_0^2$ with $m = 2 \cdot 10^{-13}$ kg the mass of the magnet and with $R_0 = 1.7 \mu\text{m}$ the radius. If we take the 6th higher mode at 756 kHz as an example, we find $\kappa_6 = 5 \cdot 10^{-12}$ Nm/rad, which at a temperature of 20 mK leads to a dissipation constant $\Gamma_6 = 3 \cdot 10^{-24}$ kg m²/s and a thermal torsional noise of about $2 \text{ yNm}/\sqrt{\text{Hz}}$. However, our detection sensitivity is not sufficient to detect the thermal motion for most of the higher modes.

C

QUENCHING OF SQUID MODULATION UNDER RADIO-FREQUENCY INTERFERENCE

In this appendix, we briefly expand upon the results presented in Ch. 6, in particular Fig. 6.5, to show the influence of the amplitude of the RF interference or crosstalk on the depth of the SQUID modulation, and the corresponding SQUID noise.

C.1 QUENCHED SQUID MODULATION

As we have seen in Ch. 6, the peak-to-peak amplitude of the SQUID modulation (ΔV_m) is reduced when the SQUID is exposed to a large RF flux. When we apply a test flux Φ_a to the SQUID in combination with RF interference Φ_{rf} , the time-dependent SQUID voltage response to Φ_a is given by [152]:

$$V(t) = \Delta V_m \cdot \cos \frac{2\pi\Phi_a}{\Phi_0} \cdot J_0 \left(\frac{2\pi\Phi_{\text{rf}}}{\Phi_0} \right), \quad (\text{C.1})$$

with $\Phi_0 = 2.068 \cdot 10^{-15}$ Wb the magnetic flux quantum, and J_0 the zeroth-order Bessel function. In Ch. 6 we only looked at the response of the SQUID voltage under a constant RF amplitude, but we can also reconstruct the entire response by varying the RF amplitude. The result of this measurement can be seen in Fig. C.1 for a constant frequency RF interference at 1908 kHz. We applied a test flux using the generator of the SQUID electronics with an amplitude of a little over $2\Phi_0$. The inset shows an example of the SQUID modulation for this applied test flux combined with RF flux at constant amplitude and constant frequency.

Since we have only measured the absolute amplitude, the data follows the absolute value of the zeroth-order Bessel function, shown as the red solid line. For a peak RF flux of $0.38\Phi_0$ the amplitude of the SQUID modulation is reduced to zero,

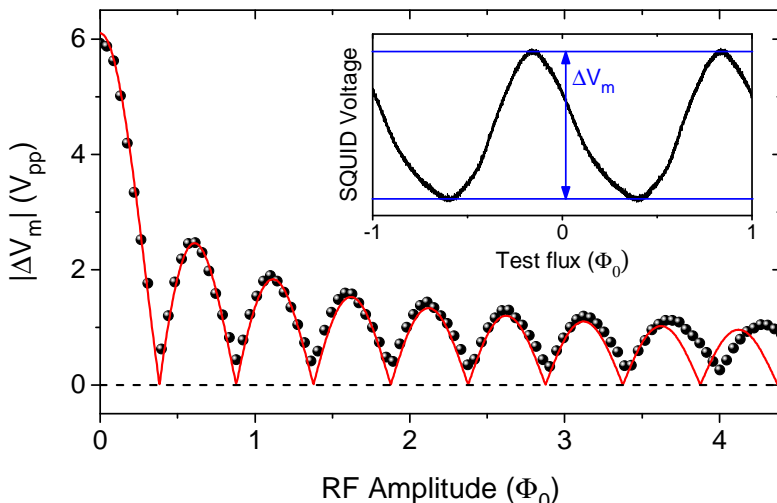


Figure C.1: Measured SQUID modulation depth as a function of the amplitude of the RF interference. This inset shows the way the modulation depth is extracted from the raw SQUID-flux response. The red solid line is a fit to Eq. C.1.

independent of the test flux Φ_a . For large RF amplitude we see that the period of the oscillation increases. An increasing period means that the amplitude of the RF interference reaching the SQUID reduces. We speculate that this is linked to the observed dissipation of power for high frequency, high amplitude RF fields, as described in Ch. 7. The idea behind this hypothesis is that we can make the data and fit match by stretching the horizontal axis by a value proportional to the RF amplitude squared. This scaling is consistent with the amplitude-dependent heating observed. An experimental check for this hypothesis would be to repeat the experiment at a higher RF frequency, as the dissipation of the RF wire scales as $f^{1.5}$ for high f . In that case, the deviation of the data from the fit should appear at lower RF amplitudes. This check has not been done yet.

C.2 POSSIBILITIES

The measurement presented in Fig. C.1 might have important consequences for SQUID-based MRFM experiments, if for some reason the compensation scheme presented in Ch. 6 cannot be used. For example in experiments on electrons, where the required GHz-range frequencies pose a challenge for the compensation. In those cases, the negative effects that the pulse has on the SQUID modulation can be reduced by selecting a suitable amplitude for the pulse.

Furthermore, one could use a measurement such as that presented in Fig. C.1 to check the amplitude of the magnetic field that we create at the location of the sample because the geometry of the pickup loop is known. Especially in the case of GHz pulses this cannot be done directly using the SQUID because of the limited bandwidth, but the amplitude of the pulse might show up in the amplitude of the SQUID modulation.

D

FABRICATION RECIPES

This appendix discusses some of the basic fabrication recipes used for the work presented in this thesis, as we believe this might be useful for future students.

D.1 DETECTION CHIP

We start the fabrication of the MRFM detection chips from 350 nm thick NbTiN films grown on top of a silicon wafer. All details about this film are given by Thoen et al. (our films are grown using the Nordiko 2000 Static) [55]. The pickup loop and RF wire are fabricated with a top-down approach using reactive-ion-etching (RIE). We use the following fabrication recipe:

Resist and spincoating:

- Resist: Negative E-beam resist AR-N 7700.18.
- Spincoat at 1500 rpm for a thickness of 0.65 μm .
- Bake at 80°C for 90 seconds on a closed hotplate.

Exposure using Raith EBPG 5000+ at the Kavli Nanolab Delft:

- E-beam dose 150 $\mu\text{C}/\text{cm}^2$.
- Spotsize 66 nm. With proximity effect correction (PEC).

Development after exposure:

- Postbake at 110°C for 120 seconds on a closed hotplate.
- MF321 developer, 180 seconds.
- H₂O, 30 seconds.
- Rinse with H₂O, dry with nitrogen.

Reactive-ion-etching using Leybold F2 at the Kavli Nanolab Delft:

- 13.5 sccm SF₆, 5.0 sccm O₂.
- 50 W forward RF power, 8 W backward RF power. 320 V_{DC} bias.
- Etching time: 335 seconds + 10 second overetch.
- O₂ plasma descum to help in resist removal.
- 20 sccm O₂, 30 W forward RF power, 100 seconds.

Stripping of the resist before dicing:

- PRS-3000 (positive-resist stripper), 40°C, sonicate for 15 minutes.
- Acetone, 40°C, sonicate for 5 minutes.
- IPA, 40°C, sonicate for 5 minutes.
- Rinse with IPA, dry with nitrogen.

Dicing of the detection chips at the Kavli Nanolab Delft:

- Apply positive photoresist S1805 to prevent surface damage during dicing.
- Spincoat at 4000 rpm.
- Bake at 110°C for 120 seconds on a closed hotplate.
- Dice using DAD 3220 wafer dicer.

Resist stripping after dicing:

- PRS-3000, heated to 85°C au bain-marie, 15 minutes.
- Move beaker to sonicator, sonicate for 5 minutes.
- Reheat to 85°C au bain-marie, 10 minutes.
- Move beaker to sonicator, sonicate for 5 minutes.
- Acetone, room temperature, sonicate for 5 minutes.
- IPA, room temperature, sonicate for 5 minutes.
- Rinse with IPA, dry with nitrogen.

Final measured thickness of the structures on the chip using DektakXT in Leiden: 400 nm, approximately 350 nm NbTiN and 50 nm overetch into the silicon substrate.

D.2 DOUBLE LAYER RESISTS FOR SPUTTERING

For sample fabrication using sputtering, a double resist layer with an undercut is required for proper lift-off and to prevent dog-ears. We identified 2 recipes which seem to work well with the available sputtering machines.

Recipe 1: thin samples.

Resist and spincoating:

- Spincoat PMMA 200k, AR-P 642.06, 4000 rpm. Thickness 0.2 μm .

- Bake at 180°C for 180 seconds on open hotplate.
- Spincoat PMMA 950k, AR-P 672.042, 4000 rpm. Thickness 0.25 μm .
- Bake at 180°C for 180 seconds on open hotplate.

Optimal E-beam dose: 280 $\mu\text{C}/\text{cm}^2$. Suitable for samples with a thickness of up to about 150 nm. Very large undercut due to large difference in polymer length.

Recipe 2: thick samples.

Resist and spincoating:

- Spincoat PMMA 600k, AR-P 662.06, 4000 rpm. Thickness 0.4 μm .
- Bake at 180°C for 180 seconds on open hotplate.
- Spincoat PMMA 950k, AR-P 672.042, 4000 rpm. Thickness 0.25 μm .
- Bake at 180°C for 180 seconds on open hotplate.

Optimal E-beam dose: 300 $\mu\text{C}/\text{cm}^2$. Suitable for samples with a thickness of up to about 350 nm, at the expense of a smaller undercut.

Development and lift-off.

Both options are developed following the same recipe:

- MIBK:IPA (1:3), 60 seconds.
- IPA (stopper), 30 seconds.
- Rinse with IPA, dry with nitrogen.

Liftoff after sample deposition:

- Acetone, 52°C, 20 minutes.
- Spray with acetone while keeping the chip submerged.
- Transfer to clean acetone, sonicate for 2 minutes.
- Inspection of the chip using an optical microscope, still submerged in acetone.
- When lift-off successful, sonicate in ethanol for 2 minutes.
- Sonicate in IPA for 2 minutes.
- Rinse with IPA, dry with nitrogen.

D.3 SPECIFIC SAMPLES

In this section, we will briefly discuss the recipes used for the specific samples used in the experiments presented in this thesis.

D.3.1 Copper

We aim for a sample consisting of about 100 nm of copper, capped with a 10-20 nm layer of gold to prevent oxidation. Deposition of both layers is done in a single session (without venting the system) using the Leybold Heraeus Z406 sputtering system in Leiden. Before sputtering, we do a 4-5 second long dip in hydrofluoric (HF) acid to remove oxides from the surface of the detection chip, followed by three H₂O baths. The chip is dried using nitrogen.

The chip is then loaded into the Z406 within minutes to prevent re-oxidation of the surface as much as possible. The chip is glued to the sample holder using silver paint to improve cooling. Sputtering is started from a background pressure of $5.5 \cdot 10^{-6}$ mbar. We use the following sputtering parameters:

- Cu layer: $5 \cdot 10^{-3}$ mbar argon, flow 49 sccm. RF voltage 1 kV. Pre-sputtering for 3 minutes, final sputtering for 10 minutes.
- Au layer: $5 \cdot 10^{-3}$ mbar argon, flow 49 sccm. RF voltage 1 kV. Pre-sputtering for 3 minutes, final sputtering for 90 seconds.

Measured thickness of the combined Cu/Au layer using DektakXT: 130 nm.

D.3.2 Calcium fluoride

We start from a sample ordered from Kurt J. Lesker, containing crystalline calcium fluoride pieces (1-4 mm) with a purity of 99.99%. The deposition was done using resistance evaporation, inspired by earlier work by Mamin et al. [14]. To improve thermalization, the CaF₂ is deposited on top of a thin layer of copper and gold. The copper and gold are deposited using e-beam evaporation in the Leybold Heraeus L560 at the Kavli Nanolab. The parameters for the different materials are:

- Cu layer: e-beam, 12 kV. Evaporation time 100 seconds at a rate of 0.8-1.2 Å/s.
- Au layer: e-beam, 12 kV. Evaporation time 375 seconds at a rate of 1.0-1.2 Å/s.
- CaF₂ layer: resistive heating, 25% power. Evaporation time 610 seconds at a rate of 3-5 Å/s.

Measured thickness of the Cu/Au layer using DektakXT: 40 nm. Measured thickness of the CaF₂ layer: 240 nm.

D.3.3 Palladium

Target ordered from ESPI: purity 99.99%, with less than 2 ppm Fe. The sputtering is done in the UHV sputtering system in Leiden. Before sputtering, we do a 4-5 second long dip in hydrofluoric (HF) acid to remove oxides from the surface of the detection chip, followed by three H₂O baths. The chip is dried using nitrogen. After the HF dip, the chip is loaded into the vacuum as soon as possible (< 20 minutes).

Sputtering is started at a chamber pressure of $7.7 \cdot 10^{-9}$ mbar. Sputtering is done using an argon pressure of $3.3 \cdot 10^{-3}$ mbar. The RF power is set to 100 mA, 401V. We pre-sputter for 5 minutes to clean the target, then do real sputtering on the sample for 20 minutes. The measured thickness of the palladium layer using the DektakXT is 108 nm.

D.4 CONSIDERATIONS FOR DOUBLE-LAYER DETECTION CHIPS

As discussed in Ch. 2, it is worth to invest time in the development of double-layer detection chips. This would enable the fabrication of on-chip transformers to boost the coupling efficiency between the pickup loop and SQUID input coil, and would also open the possibility to fabricate gradiometric pickup loops that cross the RF wire to reduce flux crosstalk. The first attempts to fabricate these double-layer devices were done by de Voogd [59]. In these attempts, the first layer was fabricated following a similar recipe as described in Sec. D.1 (RIE etching), while the second layer was made using a lift-off process. At the visual inspection after the sputtering of the second NbTiN layer, it was found that the resist was cracked. The measured critical current of the second layer, which was much lower than expected with only several

μA , confirms that the second layer was contaminated, probably from the resist which could not stand the high temperatures during sputtering.

From this we conclude that structures made by sputtering and lift-off might be more susceptible to contamination, resulting in a material with a lower critical current density. Hence, it is preferable to make the RF wire and the secondary coil of a transformer from the first (reactive-ion-etched) layer of high thickness NbTiN, as especially the RF wire should be able to carry large currents when used for an NMR experiment.

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