Nuclear magnetic resonance force microscopy at millikelvin temperatures

Haan, A.M.J. den

Appendices

Appendix A

Copper sample fabrication

In this appendix, we will give a detailed description of the fabrication procedure of the copper sample and the pick-up coil.

We used a wafer with a 375 nm NbTiN film with a resistivity of 92 $\mu\Omega$cm and a critical temperature of 15.3 K.

A.1 Patterning pick-up coil and RF-line

The following procedure was used to pattern a pick-up coil into the superconducting film.

1. Cleaned the sample with acetone, then isopropanol, dried with nitrogen gas.
2. Spin coated chemically enhanced negative resist AR 7700.18 at 4000 rpm
3. Baked at hot plate at 85°C for 1 min.
4. e-beam at 14 μC/cm^2 at 30 kV
5. Baked at hot plate at 105 °C for 2 min.
6. 60 s developer 300-46
7. 30 s rinsed with stopper millipore H_2O, dried with N_2
8. Baked hot plate at 120°C for 2 min.

A.2 Reactive ion etching

The film and resist was etched in Delft (Kavli Institute of NanoScience) with David Thoen, using a reactive ion etcher, with the following settings:

1. Pre-etch with Gas flow: 12.5 sccm SF_6 and 5 sccm O_2 and RF power 50W$_f$ and 5W$_r$, Bias Voltage 334 Vb, 8 nT

2. Current stable after 30 seconds.
3. Etched for 7 min and 20 sec plus 20 sec. overetch.
4. Oxygen cleaned 50WF, 5Wr, 445Vb, 6mT, 20sccm for 100 sec.
5. Extra overetch step for 60 sec. We saw a shortcut at the pick-up coil
6. with these settings: SF6/O2, 50Wf, 10Wr, 400Vb

A.3 Fabricating the second copper layer

For the second copper layer, we used the following procedure for patterning the structure:

1. Ultrasonic cleaning with acetone and, several times in new acetone (>3 times)
2. Spin coat 200k PPMA from AllResist 642.12 at 4000 rpm
3. Baked at hot plate at 160 degrees for 3 min.
4. Spin coat 950k PPMA (AllResist 672.045) at 4000 rpm
5. Bake at hot plate at 160 degrees for 3 min.
6. e-beam at 200 μC/cm$_2$ at 30kV.
7. 3 min. developer (AllResist 600.56)
8. 30 seconds stopper isopropanol
9. Dried with nitrogen gas N_2

A.4 Sputtering copper and gold

After the preparation of the sample, the chip was sputtered with a 300 nm copper layer and a 16 nm gold layer with an ATC-sputtermachine, operated by D. Boltje. We used the following parameters for sputtering copper:

1. Argon pressure: 5 mTorr ($7{\cdot}10^{-}3$ mbar)
2. Flow: 25 sccm
3. Current: 400 mA
4. Duration: 18 min, yielding an expected 300 nm with approximately 10 nm roughness.

The sputter parameters for gold are:

1. Argon pressure 10 mTorr ($13 \cdot 10^{-}3$ mbar)
2. Flow: 25 sccm
3. Current: 200 mA
4. Duration: 1 min (16 nm)

A.5 Lift off

Using acetone and an ultrasonic bath, the resist with the copper on top dissolves and the remaining copper stays on the chip. The whole process of applying the second layer of copper was done twice, because the part of the copper structure that had to stick to the silicon had come loose while being in the ultrasonic bath. Although, using a 50°C acetone bath for the second time, it was still needed to use an ultrasonic bath to dissolve the remaining resist with the copper. This second lift-off attempt was successful however.

Appendix B

Transformation algorithm for cantilever positioning

In this appendix, the calculations of the transformation from the heights at the piezoknobs to the xyz-position of the cantilever tip are given. These calculations were used (in chapter 6 and chapter 7) to determine the positions of the cantilever above the samples. The readout of the heights was performed by capacitance sensors, which were calibrated at room temperature.

In figure B.1, the MRFM positioning system and several viewpoints of the piezoknobs are shown. Figure B.1a shows a platform with a cantilever holder attached to it. This platform, shown in figure B.1c can be moved by the piezoknobs to several orientations in comparison with the MRFM cover plate. Since the cantilever holder with cantilever is attached to the platform (sticking out in the figure), the cantilever can be put in any position in the range defined by the piezoknobs. For example, when turning piezoknob 1 to the right (assuming a right-turning screw thread), the platform moves up at piezoknob 1, which results in a movement of the cantilever to the right (in the x-direction). Note that the distance between the contact points may change if piezoknobs are turned. Therefore, degrees of freedom need to be added to the contact points while keeping the platform fixed. This is accomplished by having three different contact points; The contact point of piezoknob 2 is fixed, the contact point of piezoknob 1 can move in only one direction (x-direction) and the contact point of piezoknob 3 can move in xy-direction (any direction in plane), see figure B.1d.

The problem is to find the xyz-position of the cantilever from the heights at the piezoknobs. In reality, the height at piezoknobs is derived from the height at the capacitance sensors, which are positioned close but not exactly at the place of the piezoknobs. The input parameters are therefore the distances (not the directions) at the piezoknobs, d_1, d_2 and d_3. In addition, many fixed parameters, set by the design of the MRFM positioning system (design parameters) have to be incorporated for the final calculation. A summary of the input and design parameters are shown in table B.1.

It is convenient to put the origin of the coordinate system at point p_2, since this

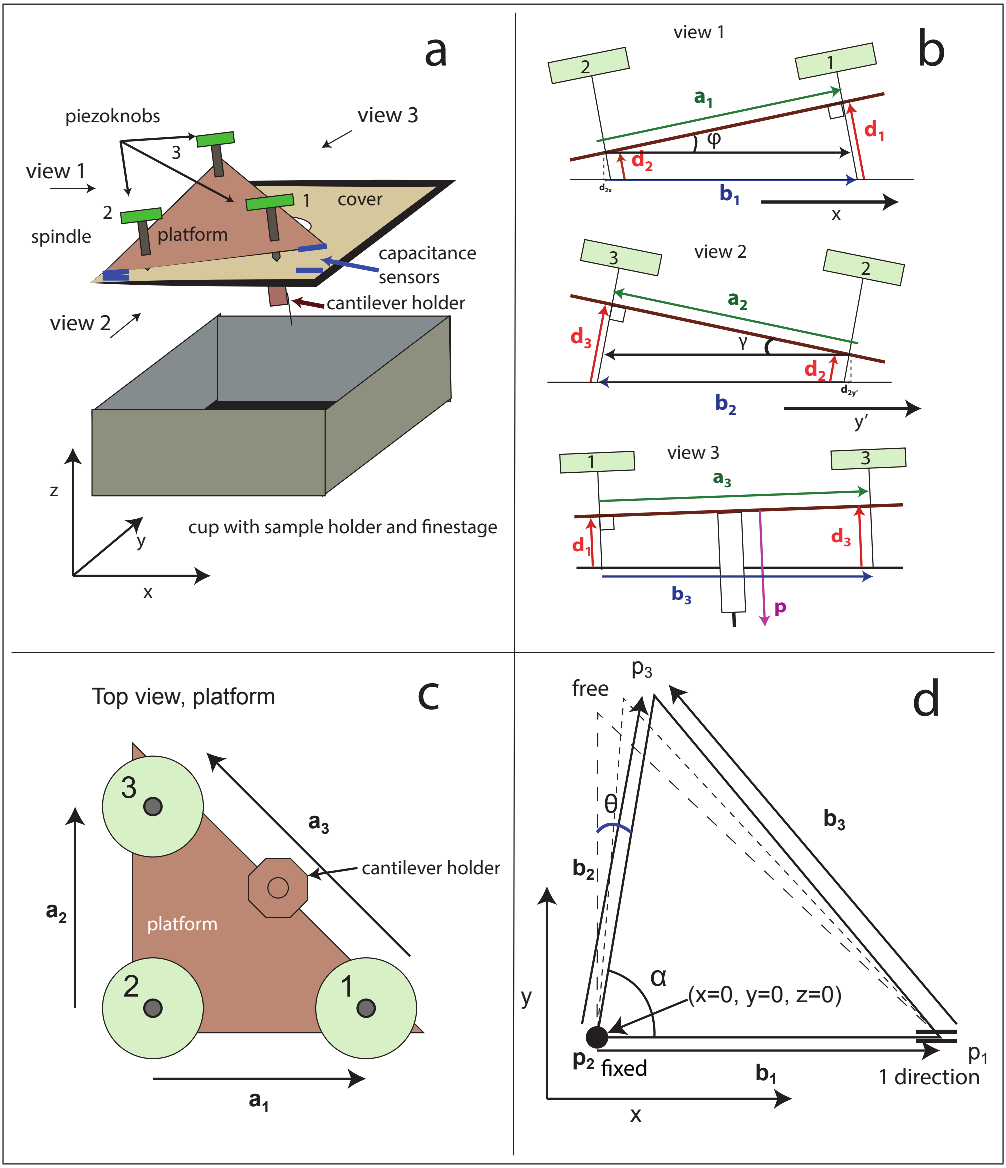

Figure B.1: MRFM positioning system and drawings for the piezoknob transformation calculations. (a) three-dimensional drawing of the MRFM positioning system, the piezoknobs move the platform in comparison with the cover plate. (b) three different views as shown in [a] with the definitions of $\mathbf{d_1}$, $\mathbf{d_2}$ and $\mathbf{d_3}$. All three views are perpendicular to the platform. (c) Top view of the platform with the definition of vectors $\mathbf{a_1}$ and $\mathbf{a_2}$. (d) The contact points of the endpoints of the piezoknobs, indicated by p_1, p_2 and p_3. At p_1, the contact point of the piezoknob can only move in the x-direction, at p_2, the contact point is fixed, which is therefore chosen as the origin, and at p_3, the contact point can move both in x and y. The vectors $\mathbf{b_1}$ and $\mathbf{b_2}$ point to the contact points p_1 and p_2 from the origin (p_2). $\mathbf{b_3} = \mathbf{b_2} - \mathbf{b_1}$.

Table B.1: parameters of MRFM positioning system

parameters	meaning	defined in fig.	value
input:			
d_1	distance at piezoknob 1	B.1b view 1, 3	-
d_2	distance at piezoknob 2	B.1b view 1, 2	-
d_3	distance at piezoknob 3	B.1b view 2, 3	-
design:			
a_1	distance between p1, p2 on platform	B.1b,c	38 mm
a_2	distance between p3, p2 on platform	B.1b,c	38 mm
$a_3 = \sqrt{a_1^2 + a_2^2}$	distance between p1, p3 on platform	B.1b,c	54 mm
p	length of the cantilever holder	B.1b view 3	24 mm

point does not move. The route in solving this problem is by finding the vector from the origin to the cantilever in Cartesian coordinates. We first want to find the length of the vectors of the contact points, $b_1 = \| \mathbf{b_1} \|$, $b_2 = \| \mathbf{b_2} \|$ and $b_3 = \| \mathbf{b_3} \|$ in terms of the input- and design-parameters (d_1, d_2, d_3 and a_1, a_2) as defined in figure B.1b, B.1c and B.1d:
From figure B.1b, view 1, we see:

$$b_1 = \sqrt{a_1^2 + (d_1 - d_2)^2} \tag{B.1}$$

Similar for view 2:

$$b_2 = \sqrt{a_2^2 + (d_3 - d_2)^2} \tag{B.2}$$

Similar for view 3:

$$b_3 = \sqrt{a_1^2 + a_2^2 + (d_3 - d_1)^2} \tag{B.3}$$

Hence we can determine the vectors **b1**, **b2**, and **b3**, which are in the xy-plane. Since the contact point p_2 is fixed and p_1 can only move to the right:

$$\mathbf{b_1} = b_1 \hat{\mathbf{x}} \tag{B.4}$$

For determining $\mathbf{b_2}$, we first obtain α and θ from the cosine rule, which are defined in figure B.1d.

$$\alpha = \arccos\left(\frac{b_1^2 + b_2^2 - b_3^2}{2b_1 b_2}\right) \equiv \arccos(D) \tag{B.5}$$

Therefore:

$$\theta = \frac{\pi}{2} - \alpha = \arcsin\left(\frac{b_1^2 + b_2^2 - b_3^2}{2b_1 b_2}\right) \tag{B.6}$$

Hence:

$$\mathbf{b_2} = b_2 \left(\sin\theta \hat{\mathbf{x}} + \cos\theta \hat{\mathbf{y}}\right) \tag{B.7}$$

$$= b_2 (D\hat{\mathbf{x}} + \sqrt{1 - D^2}\hat{\mathbf{y}}) \tag{B.8}$$

We can derive $\mathbf{b_3}$ by:

$$\mathbf{b_3} = \mathbf{b_2} - \mathbf{b_1} \tag{B.9}$$

In the following, we determine the vectors which follow the direction of the piezoknob spindles $\mathbf{d_1}$, $\mathbf{d_2}$ and $\mathbf{d_3}$, see figure B.1b and B.2a. They all have the same direction, since these vectors are all perpendicular to the platform. Therefore we only need to determine the directions for one of the three vectors. It is convenient to calculate the direction of $\mathbf{d_2} = d_{2x}\hat{\mathbf{x}} + d_{2y}\hat{\mathbf{y}} + d_{2z}\hat{\mathbf{x}} \equiv d_2\boldsymbol{\epsilon}$. The projection of $\mathbf{d_2}$ on the x-axis, (d_{2x}), can be found by using figure B.1b:

$$\begin{aligned} d_{2x} &= -d_2\sin(\phi) && \text{(B.10)}\\ &= d_2\frac{d_2 - d_1}{b_1} && \text{(B.11)}\\ &\equiv d_2\epsilon_x && \text{(B.12)} \end{aligned}$$

Since piezoknob 1 is fixed in the y-direction (moves only in x), d_{2x} does not change when piezoknob 3 changes. In other words, trivially, rotation of the stage around the x-axis does not change d_{2x}. This is not the case in the y-direction, since the contact point of piezoknob 3 may also move in the x-direction. For the projection in the y-direction d_{2y}, we use figure B.2. We can easily calculate the projection of d_2 along the line following $\mathbf{b_2}$, which we define as $d_{2y'}$. From figure B.1b, view 2, we find:

$$\begin{aligned} d_{2y'} &= d_2\sin(\gamma) && \text{(B.13)}\\ &= d_2\frac{d_2 - d_3}{b_2} && \text{(B.14)} \end{aligned}$$

With two projections on the xy-plane and the known magnitude of $\mathbf{d_2}$, the vector is uniquely defined. By drawing two lines that intersect perpendicularly at d_{2x} and $d_{2y'}$, the projection of $\mathbf{d_2}$ on the xy-plane, $\mathbf{d_{2r}}$, is found (see figure B.2a). The y-component of this vector is equal to d_{2y}. Therefore, using figure B.2a:

$$\begin{aligned} d_{2x'} &= \frac{d_{2y'}}{\sin(\theta)} = \frac{d_{2y'}}{D} && \text{(B.15)}\\ d_{2y} &= \Delta d_{2x}\tan(\theta) = (d_{2x'} - d_{2x})\tan(\theta) && \text{(B.16)}\\ &= d_2\left(\frac{d_2 - d_3}{b_2} - D\frac{d_2 - d1}{b1}\right)\frac{1}{\sqrt{1 - D^2}} && \text{(B.17)}\\ &\equiv d_2\epsilon_y && \text{(B.18)} \end{aligned}$$

We can find the z-component of $\mathbf{d_2}$ by using the x- and y-values:

$$\begin{aligned} d_{2z} &= \sqrt{d_2^2 - d_{2x}^2 - d_{2y}^2} && \text{(B.19)}\\ &= d_2\sqrt{1 - \frac{(d_2 - d_1)^2}{b_1^2} - \left(\frac{d_2 - d_3}{b_2} - D\frac{d_2 - d_1}{b1}\right)^2\frac{1}{1 - D^2}} && \text{(B.20)}\\ &\equiv d_2\epsilon_z && \text{(B.21)} \end{aligned}$$

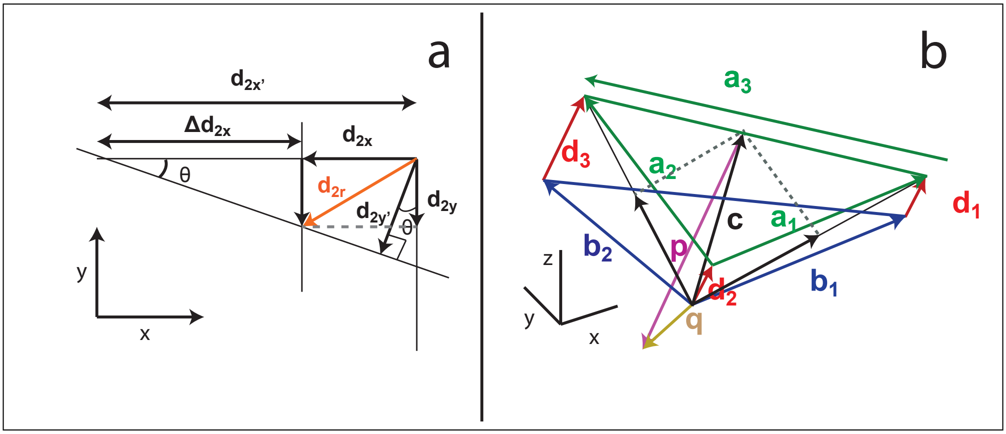

Figure B.2: Vector drawings. (a) Drawing for the calculation of d_{2y}. The components d_{2x} and $d_{2y'}$ are also shown in figure B.1b. The vector $\mathbf{d_{2r}}$ can be composed from d_{2x} and $d_{2y'}$ and represents the projection of $\mathbf{d_2}$ on the xy-plane. The y-component of this vector is d_{2y}. (b) All vectors that compose the configuration of the positioning system.

Since the vectors $\mathbf{d_1}$ and $\mathbf{d_3}$ also have the same direction, we can write:

$$\mathbf{d_1} = d_1\boldsymbol{\epsilon} \tag{B.22}$$
$$\mathbf{d_2} = d_2\boldsymbol{\epsilon} \tag{B.23}$$
$$\mathbf{d_3} = d_3\boldsymbol{\epsilon} \tag{B.24}$$

In figure B.2b, all vectors that compose the configuration of the MRFM positioning system are shown. With the calculated vectors ($\mathbf{b_1}$, $\mathbf{b_2}$, $\mathbf{b_3}$, $\mathbf{d_1}$, $\mathbf{d_2}$ and $\mathbf{d_3}$) we have sufficient information to find the other vectors. We can for example calculate the vectors $\mathbf{a_1}$ and $\mathbf{a_2}$, which follow the sides of the platform, see also figure B.1c:

$$\mathbf{a_1} = \mathbf{d_1} + \mathbf{b_1} - \mathbf{d_2} \tag{B.25}$$
$$\mathbf{a_2} = \mathbf{d_3} + \mathbf{b_2} - \mathbf{d_2} \tag{B.26}$$

These vectors are perpendicular to each other and have values, a_1 and a_2, given by the design. A check for validity of the above calculations is therefore the calculation of the inproduct: $\mathbf{a_1} \cdot \mathbf{a_2} = 0$.

We have defined the vector $\mathbf{q}$ as the vector that points to the cantilever, see figure B.2b. To determine $\mathbf{q}$, we need to find the vector that points to the attachment of the cantilever holder to the platform, vector $\mathbf{c}$, and the vector that points from this attachment to the cantilever, vector $\mathbf{p}$. Since the cantilever holder is perpendicular to the platform, like the spindles, the vector $\mathbf{p}$ is given by:

$$\mathbf{p} = -p\boldsymbol{\epsilon} \tag{B.27}$$

Where p is the length of the cantilever holder. We can find the vector $\mathbf{c}$ by looking at figure B.2b:

$$\mathbf{c} = \frac{1}{2}(\mathbf{d_3} + \mathbf{b_2}) + \frac{1}{2}(\mathbf{d_1} + \mathbf{b_1}) \tag{B.28}$$
$$= \frac{1}{2}(d_3 + d_1)\boldsymbol{\epsilon} + \frac{1}{2}(\mathbf{b_1} + \mathbf{b_2}) \tag{B.29}$$

Hence, we find for the cantilever position:

$$\mathbf{q} = \mathbf{c} + \mathbf{p} \tag{B.30}$$
$$= \frac{1}{2}(d_3 + d_1 - 2p)\boldsymbol{\epsilon} + \frac{1}{2}(\mathbf{b_1} + \mathbf{b_2}) \tag{B.31}$$

Decomposed in x, y and z yields:

$$x = \frac{1}{2}(d_3 + d_1 - 2p)\frac{d_2 - d_1}{b_1} + \frac{1}{2}(b_1 + b_2 D) \tag{B.32}$$
$$y = \frac{1}{2}(d_3 + d_1 - 2p)\left(\frac{d_2 - d_3}{b_2} - D\frac{d_2 - d1}{b1}\right)\frac{1}{\sqrt{1 - D^2}}$$
$$+ \frac{1}{2}b_2\sqrt{1 - D^2} \tag{B.33}$$
$$z = \frac{1}{2}(d_2 + d_1 - 2p)\cdot$$
$$\sqrt{1 - \frac{(d_2 - d_1)^2}{b_1^2} - \left(\frac{d_2 - d_3}{b_2} - D\frac{d_2 - d1}{b1}\right)^2 \frac{1}{1 - D^2}} \tag{B.34}$$
$$D = \frac{b_1^2 + b_2^2 - b_3^2}{2b_1 b_2} \tag{B.35}$$
$$b_1 = \sqrt{a_1^2 + (d_1 - d_2)^2} \tag{B.36}$$
$$b_2 = \sqrt{a_2^2 + (d_3 - d_2)^2} \tag{B.37}$$
$$b_3 = \sqrt{a_1^2 + a_2^2 + (d_3 - d_1)^2}. \tag{B.38}$$

To position the cantilever at a desired location, the reverse transformation, which is the transformation from x, y and z to the heights at the piezoknobs, is useful. For this we can use a linear approximation, since usually the relative movements are small in comparison with the size of the MRFM positioning system. A 30 micrometer movement yields angle changes of less than 0.1 degrees.

We want to approximate the solution around a point where the movements have to take place. In other words, we approximate around the setpoint d_1^0, d_2^0 and d_2^0, corresponding to positions x^0, y^0 and z^0. A linear approximation near these points can be found via a multidimensional Taylor approximation:

$$\mathbf{q} = \begin{pmatrix} x \\ y \\ z \end{pmatrix} \qquad \mathbf{q^0} = \begin{pmatrix} x^0 \\ y^0 \\ z^0 \end{pmatrix} \qquad \mathbf{d} = \begin{pmatrix} d_1 \\ d_2 \\ d_3 \end{pmatrix} \qquad \mathbf{d^0} = \begin{pmatrix} d_1^0 \\ d_2^0 \\ d_3^0 \end{pmatrix} \tag{B.39}$$
$$\mathbf{q} - \mathbf{q^0} = \mathbf{A}(\mathbf{d^0})(\mathbf{d} - \mathbf{d^0}) \tag{B.40}$$

with

$$\mathbf{A}(\mathbf{d^0}) = \begin{pmatrix} \frac{\partial x}{\partial d_1}(d^0) & \frac{\partial x}{\partial d_2}(d^0) & \frac{\partial x}{\partial d_3}(d^0) \\ \frac{\partial y}{\partial d_1}(d^0) & \frac{\partial y}{\partial d_2}(d^0) & \frac{\partial y}{\partial d_3}(d^0) \\ \frac{\partial z}{\partial d_1}(d^0) & \frac{\partial z}{\partial d_2}(d^0) & \frac{\partial z}{\partial d_3}(d^0) \end{pmatrix} \tag{B.41}$$

$$d^0 \equiv (d_1^0, d_2^0, d_3^0)$$

Where $\mathbf{d}$ is the vector with the new measured heights at the piezoknobs and $\mathbf{q}$ is the corresponding new position vector of the cantilever. In order to find the linear transformation matrix for the relative heights at the piezoknobs from the relative xyz-position of the cantilever, we just have to take the inverse of this matrix:

$$\mathbf{d} - \mathbf{d^0} = \mathbf{A^{-1}}(\mathbf{d^0})(\mathbf{q} - \mathbf{q^0}) \tag{B.42}$$

These calculations are implemented in a Labview program, such that positions can be read out and also new positions can be set.