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## **Nuclear magnetic resonance force microscopy at millikelvin temperatures**

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# Chapter 1

## Introduction

Magnetic resonance force microscopy (MRFM) has been introduced as a possible tool to obtain nanometer 3D resolution or even the 3D atomic structure of biological samples and proteins for structural biology [1]. The resolution of this technique is still limited by experimental factors, such as thermal force noise (see chapter 2) and the interaction with fluctuations in the surface. Although a resolution of  $< 10$  nm has been achieved with nuclear magnetic resonance force microscopy [2], improvement of orders magnitude in sensitivity is required to efficiently measure the 3D atomic structure of a sample.

To put this into perspective, several other techniques have been successfully implemented to uncover biological structures [3, 4, 5]. The most prominent techniques are X-ray crystallography, in which many identical proteins are coaxed to form a crystal, nuclear magnetic resonance (NMR) and cryo-electron microscopy (cryo-EM). These tools have delivered detailed information on the 3D atomic composition of many protein structures [6, 7] and play an essential role in the current development of pharmaceutical drugs [8, 9, 10] and research in protein misfolding [11]. The knowledge of the mechanisms and the use of computer models is highly dependent on the data (and also the availability of the data [10]) provided by these tools.

Despite the large database of proteins, only a small fraction of the revealed proteins are membrane proteins, which are targets for 50% of the drugs [12]. Moreover, they comprise 20-30% of the total number of proteins in a genome [13, 14]. This relative low number of generated 3D-structures of membrane proteins is due to the technical limitations of the current available techniques [15].

Given the importance of structural biology in current research and the possibility to image membrane proteins, investigation and development of magnetic resonance force microscopy as an extra tool may have significant value. In addition, given the high risks (of invested capital) in pharmaceutical research and the reduction of this risk by new techniques uncovering 3D-protein structures in membranes, shows this increasingly well.

Most of the MRFM-experiments are performed at temperatures near the boiling point of liquid helium (4.2 K). These low temperatures provide a considerable improvement in comparison with room temperature measurements, since the minimal

detectable magnetic moment  $\mu_{min}$  (as explained in chapter 2 and chapter 3) is given by:

$$\mu_{min} = \frac{1}{G} \sqrt{4\Gamma k_B T \Delta f} \quad (1.1)$$

Where  $\Gamma = \frac{\sqrt{km}}{Q}$  is the damping,  $G = \partial B / \partial x$  is the gradient of the magnetic field,  $k_B$  is the Boltzmann constant,  $T$  is the temperature,  $\Delta f$  is the bandwidth and  $k$ ,  $m$  and  $Q$  are the spring constant, mass and quality factor of the cantilever respectively.

We try to improve this force sensitivity by more than a factor 10 by measuring at millikelvin temperatures. Moreover, in this low millikelvin temperature regime, many unclear phenomena in condensed matter physics are present. The use of MRFM as a tool for condensed matter physics at millikelvin temperatures is focused on another direction in research, which may contribute on its pathway to the development of MRFM as a tool for the imaging of 3D biological samples.

In this thesis, several other implementations aimed at improving the sensitivity, and thus lowering the minimal detectable magnetic moment, will be introduced. As a milestone, we show magnetic force measurements and nuclear magnetic resonance force measurements below 50 mK temperatures in chapter 6 and chapter 7. Requirements in terms of sensitivity for these measurements are less stringent than for the imaging of biological samples, which makes other (advanced) condensed matter measurements with the current setup possible in the near future.

Recently, diamond cantilevers with high quality factors and low force noise have been developed [16], which may (using the optimal cantilever shape) reduce the force noise by a factor of ten. In addition, a significant improvement of sensitivity may be obtained by using smaller magnets. In chapter 4, the fabrication of a new and smaller magnetic particle will be discussed. Another important factor is the dissipation due to the interaction of the cantilever with the paramagnetic spins in the material, which will be discussed in chapter 6. By using different substrate materials this influence can be reduced as well.

The construction of the thesis is such that the first two chapters (chapter 2 and chapter 3) form the foundation of the thesis in which the experimental setup and the theory will be discussed. Subsequently, chapter 4 and chapter 5 show experimental improvements for MRFM. Several of these improvements enabled the results in magnetic force measurements and nuclear magnetic resonance force measurements, as discussed in chapter 6 and chapter 7. Finally, in chapter 8, the use of different RF-pulses and adiabatic inversion, which may be helpful for future experiments with our current setup, will be discussed.

To be more specific:

- In chapter 2, the focus is directed to the experiments, in which the experimental setup, sample preparation and experimental limitations will be described.
- In chapter 3, the experimental limitations, focused on noise sources will be further evaluated.
- In chapter 4, the design, fabrication and measurement of a smaller magnet with higher field gradients ( $G$ ) will be discussed. This higher field gradient would yield significant better sensitivity (see Eq. 1.1).

- In chapter 5, vibration reduction measures implemented in our cryogen free dilution refrigerator will be discussed. Moreover, the low vibrations in the cryostat enabled us to perform a benchmark atomic resolution scanning tunneling microscopy experiment on graphite. With these low vibrations, the effective mode temperature (at the eigenfrequency of the cantilever) is dominated by the thermal bath of the cantilever, i.e. the effective mode temperature is thermally limited. When performing cantilever cooling, these low vibration become even more important. We were able to cool the cantilever to  $160 \mu\text{K}$ , partly due to the efforts in vibration reduction [17].
- In chapter 6, magnetic force measurements on the Si/SiO<sub>2</sub>-interface will be discussed. With these measurements a new semi-classical description of the interaction of a para-magnetic spin, having longitudinal and transverse relaxation, with the cantilever is tested.
- In chapter 7, nuclear magnetic resonance force microscopy measurements on a copper sample are described. In these experiments we performed saturation/recovery experiments as a function of frequency, distance and temperature. The temperature dependence measurements correspond to the Korringa relation [18, p. 363].
- Finally in chapter 8, an introduction to NMR and adiabatic pulses will be given. This chapter can also be read first when not familiar with this subject.

