# MULTI-MODE LOW TEMPERATURE SCANNING PROBE MICROSCOPY

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Signature of Author

*To my Family.*

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# Abstract

This thesis describes the construction and operation of a multi-mode low temperature scanning probe microscope (SPM) using a quartz tuning fork as the sensor. Atomic force microscopy (AFM), magnetic force microscopy (MFM) and scanning capacitance microscopy (SCM) images of test samples at different temperatures will be demonstrated. A multi-layer Co/Pd thin film sample has also been studied via AFM and MFM imaging to illustrate the power of the microscope.

After a short introduction to the system and reviews of history and applications of SPMs, the basic operation and theories of SPM will be introduced.

Chapter 2 briefly describes the design of the instrument and most of the commercially available parts of the microscope. It includes the descriptions of the scanning probe microscope head, electronics, basic control software, the system temperature control, the magnet and the vibration control.

The key component of the microscope — the homebuilt tuning fork multi-mode microscope probe and the software control and analysis of lift-mode for MFM operation are described in the following chapter: Chapter 3.

Due to wide fields covered by this work, AFM, MFM and SCM are described in separated chapters, and each chapter will start with an introduction of this area followed by experimental results and end with the theory and explanation of the results.

Important theoretical and practical aspects of atomic force microscopy are discussed in Chapter 4. This chapter also presents calculations of the interaction forces between a tip and a sample and also an explanation of the large shifts in the resonant

frequency of the probe when immersed in superfluid helium. Different modes of AFM operations are discussed. AFM images of the test samples at room temperature and in superfluid helium can be found in this chapter as well.

Chapter 5 includes the calculation of the interaction forces between an MFM tip and a magnetic field and some test sample images. The explanation of hydrodynamic effects in lift mode and practical application of Q control are also described.

The study of multi-layer Co/Pd thin films with the MFM is described in Chapter 6. This is the first time that this kind of sample being investigated at low temperature and in high magnetic field with MFM. Experimental data on system noise levels and discussions of improvements by further noise reduction are also included in this chapter.

The experimental sensitivity limit and operations of the SCM are described in Chapter 7 followed by the test sample images compare to DI's images and the images of a Ga(Al)As semiconductor sample at low temperature and in superfluid helium.

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# Introduction

The physical characterisation of semiconductor devices is a challenging task for device engineers and researchers. Until recently, standard methods for characterising semiconductors did not provide an effective means of determining two-dimensional quantities on a sub-micron scale. These methods include SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), SIMS (Secondary Ion Mass Spectroscopy), SRP (Spreading Sheet Resistance Profiling), and 1D C-V (one-dimensional Capacitance-Voltage).

With the advent of smaller device geometries and high reliability requirements, new characterisation tools are needed. Alternatives tools such as the various types of scanning probe microscopy (SPM), have been applied to not only characterising semiconductor devices but also monitoring semiconductor device processes.

This thesis reports the development of a novel scanning probe microscope (SPM) which can operate at temperatures between 300K and 1.5K in magnetic fields as high as 12T. The intrinsic resolution of the instrument is better than 20nm with abilities to map variation of magnetic fields and capacitance variations as small as $10^{-19}$F.

In the early 1980's scanning probe microscopes (SPMs) dazzled the world with the first real-space images of the surface of silicon. The first scanning tunnelling microscope (STM), the ancestor of all scanning probe microscopes, was invented in

1981 by Gerd Binning and Heinrich Rohrer at IBM Zurich [1]. Five years later, they were awarded the Nobel Prize in Physics for this invention. The first atomic force microscope (AFM) was developed by Binnig, Quate and Gerber in 1986 using STM technology [2].

The AFM has become a widely used technique in condensed matter physics, nanotechnology and biology. AFM is most commonly performed using non-contact mode or tapping mode [3] for soft samples and biological use [4]. Piezoelectric quartz tuning forks were introduced into AFM by Günther *et al.* [5] for use in a scanning near field acoustic microscope (SNAM), and later by Karrai and Grober [6] as a distance sensor for a scanning near field optical microscope (SNOM). Recently Giessibl has employed them for atomic resolution AFM imaging [7]. The first application of tuning forks in high magnetic fields at liquid helium temperatures was by Rychen *et al.* in 1999 [8].

Some other important recent examples using low temperature AFM include the study of superconductors [9], two dimensional electron gases [10] and atomic resolution force microscopy [11] [12] [13].

The magnetic force microscope (MFM) is very closely related to the atomic force microscope and developed directly from it in 1987 [14][15]. It was recognized that if a sharp magnetic tip on the end of a flexible cantilever was scanned near the surface of a magnetised sample, the magnetic interaction between the tip and sample would cause deflections of the cantilever which could be measured as a function of tip position.

Scanning capacitance microscopy was developed from a capacitive videodisc reader which was made by RCA in 1982 [16]. Matey and Blanc [17] showed the first demonstration of the SCM concept in 1985. Another early SCM was that of Bugg and King [18] built in 1988.

The low temperature scanning capacitance microscope (SCM) [16][17][18] combined with quartz tuning-fork atomic force microscope (AFM) [2] techniques enable us to carry out nano-scale studies of physical systems and phenomena in high magnetic fields at liquid helium temperatures. For example, it can be used for profiling of quantum Hall effect edge states [19], and how they evolve with magnetic field. In the conventional theory of the QHE, strong-field localization is associated with a single-particle drift motion of electrons along contours of constant disorder potential. However recent experiments are still testing the details of this effect with different detection methods. Such methods include scanning single-electron-transistor (SET) microscopy [20][21][22], scanned gate microscopy (SGM) [23][24] and low temperature AFM [25][26].

Capacitance spectroscopy of single InAs quantum dots, and mapping carrier concentrations in GaN and relating the surface topology to the dopant profiles are further examples of possible applications. Combined with AFM, it would provide much insight into the behaviour and locations of dopants in GaN, and will hopefully lead to the development of better GaN material.

# Chapter 1

# A Brief Introduction to SPM

In general, SPMs contain the components illustrated in Figure 1.1. The differences between them are the different probes and different probe-sample interactions under investigation.

Since the Scanning Tunnelling Microscope (STM) is the ancestor of all SPMs, it is a good example to introduce first. STMs use a sharpened, conducting tip with a bias voltage applied between the tip and the sample. When the tip is brought to within about 1nm of the sample, electrons from the sample begin to "tunnel" through the 1nm gap into the tip or vice versa, depending upon the sign of the bias voltage. (See Figure 1.2) The resulting tunnelling current varies with tip-to-sample gap, and it is the signal used to create an STM image. For tunnelling to take place, both the sample and the tip must be conductors or semiconductors. Unlike AFMs, it is hard for STMs to image insulating materials.

Two main modes of operation exist in STM: constant height mode and constant current mode (See Figure 1.3). Constant height mode, which records the change in the tunnelling current, has a higher resolution, but it is only suitable for very flat samples. Constant current mode yields quantitative information on sample topography as a

Figure 1.1: Schematic of generalized SPM (from [27])



Figure 1.2: Schematic of tip and sample interaction of STM (from [27])

Figure 1.3: Two STM modes of operation (from [27])

feedback loop is used to make the tip follow changes in surface height.

The basic operation principles of AFM, MFM and SCM are very similar to the STM and will be introduced at the beginning of the relevant chapters.

# Chapter 2

# Experimental Apparatus

## 2.1   System Overview

The helium temperature and high magnetic field scanning operations take place in the cryostat about one metre below ground level. The cryostat is a vacuum insulated, liquid helium vapor shielded dewar of an all metal construction supplied by Oxford Instruments [28]. The superconducting magnet is located at the bottom of the dewar. The cryostat has a reservoir which holds about 40 litres of liquid helium above the magnet, that enables us to run the system for up to four days (100 hours) before refilling.

The sample space is insulated from the reservoir by the inner vacuum chamber (IVC). Two methods of controlling the temperature in the sample space are provided. One is a needle valve on the IVC below the reservoir to let the liquid helium into the sample space to cool down the system or fill the system with liquid helium. The other is a heater with a temperature sensor. A temperature controller, Oxford Instruments ITC (Intelligent Temperature Controller, model 503) is used to balance the heater and helium gas flow or sweep the temperature automatically. Further details can be found in section 2.5. A schematic diagram of the cryostat, magnet and the sample

Figure 2.1: A Diagram of Solenoid, Helium Reservoir, Sample space, OVC and IVC of the Cryostat

space can be found in Figure 2.1.

The cryostat hangs from an anti-vibration table over a two metre deep pit. The top of the cryostat is covered by a very heavy box with cavity walls filled with sand. The pumping lines and the return line pass through another sand box on the way to the vacuum pump housed in a service room adjacent to the laboratory. All the electrical wiring that connects to the cryostat enters through the top of the sound-proof box and is suspended over the instrument with bungee ropes. This design attempts to minimize the acoustic vibration from the surroundings which is transmitted to the microscope. A further discussion of the vibration control can be found in section 2.7. Figure 2.2 illustrates the layout of the whole system.

Figure 2.2: A Diagram of System Overview

## 2.2   Scanning Probe Microscope Head

### 2.2.1   Overview

The core of the system is the head of the microscope. This is where the physical interactions which produce the data take place. Figure 2.3 is a diagram of the head: the sample holder, the scanning probe mount (a scanning tunnelling microscope probe is shown in this picture), the scan tube and the sample to probe coarse approach PZT (lead zirconate titanate ceramics) tubes. All of these will be described in this section.

This microscope head is a conventional design for low temperature operation purchased from Oxford Instruments. The sample is scanned relative to a fixed scanning probe. The advantage of this design is that the scanning probe is not moved by the fragile scan tube, the only object moved by the scan tube is the sample, which is normally small and light. This results in better reliability and linearity, and a complex

CONNECTION
TO INSERTION
ROD

SOCKET

PLUG

SPLIT SHIELD

SCAN TUBE

Z DRIVE TUBE

MAIN FLANGE

SAMPLE PUCK

SHIELDED
STM PROBE
HOLDER

COOLING AND
SUPPORT LEG

Figure 2.3: Microscope Head with a STM probe

scan probe mount can be used.

Almost all the parts of the microscope head are made from copper, which has a different thermal expansion coefficient (about 18ppm/K) [29] as compared to PZT tubes (about 5 ppm/K) [30]. Thus a 1 mm minimum tip and sample gap must exist when the microscope head is moved into the cryostat. When the head is moved from horizontal to vertical, the tip-sample gap is reduced by approximately 0.5 mm (probably due to slippage of the scan tube due to the springs not holding the scan tube tightly enough). The gap will also be reduced when the system is cooled down from 300K to helium temperature due to the difference in the thermal expansion coefficients. An estimate of this reduction without considering the thermal expansion variation with temperature is as follows:

$$25.4mm \times (18ppm/K - 5ppm/K) \times (300K - 4.2K) \simeq 0.1mm. \qquad (2.2.1)$$

where 25.4mm is the length of the scan tube. This is only a very rough guess, but the experiment tells us that the tip-sample distance reduces when the temperature is reduced.

## 2.2.2   Approach mechanism

There are two sets of PZT tubes in the microscope head. One set of 3 PZT tubes is designed to move in the vertical, Z direction only. The other is the scan tube which can move both in the Z direction and in the X and Y directions. The scan tube is attached onto the Z drive tubes by 3 beryllium-copper springs (clamp to the Z drive tubes) which can be seen in figure 2.3. The Z drive tubes work as follows: To move forward, all 3 Z drive tubes expand slowly to enable the scan tube to follow the movement, and then jerk back suddenly when they reach maximum extension. The

scan tube will remain in position due to its mass. By repeating the above actions, the scan tube has a Z range of about 20 millimeters which is limited by the length of the scan tube. To move backwards, the motion is similar to the forward actions apart from interchanging the direction of expansion and contraction.

This kind of Z drive mechanism is not ideal since it is very hard to determine the step size, which depends on the tightness of the springs and gravity. An improved Z driver mechanism has also been developed as shown in Figure 2.4 [31]. It was designed by S. H. Pan in 1993 [32] and has been widely used in low temperature and high vacuum scanning probe applications [31] [33]. The mechanism can be explained in Figure 2.5 [31]: six piezo legs hold a moving shaft made in sapphire with the scan tube in the center. When a voltage is applied to one leg, it slides backwards along the surface of the shaft, as friction between the other three legs and the shaft holds the shaft stationary. After an appropriate delay, the same voltage is applied to the other legs in sequence and then the voltage is ramped down simultaneously, as shown in Figure 2.5(from [31]). Consequently, all the legs together carry the shaft one step forward (to the left). This design is reliable with reproducible step sizes [34] and can be operated in a vertical direction with minimal step-size differences due to gravity, since it does not rely on inertia.

The automatic coarse approach process uses both sets of the PZT tubes. Figure 2.6 illustrates the Z movements of the tubes and the sample. First of all, the system extends the scan tube slowly to 'feel' the surface: the process stops if the control signal reaches the set point during the approach. Otherwise, the scan tube is brought back and the Z drive will carry the scan tube one coarse approach step forward. The process is then repeated until the set point is reached.

Figure 2.4: A new design of the z drive motor



Figure 2.5: Schematic illustration of the working principle of the new designed Z drive motor. [31]

Figure 2.6: Schematic illustration of the working principle of the coarse approach

However the new mechanism is still at the construction stage and all the results reported in this thesis were made using the Oxford Instruments' mechanism.

### 2.2.3 Scanning tubes

From the very beginning when Binnig and Rohrer designed and built the first scanning tunnelling microscope [1], piezoelectric materials came to be central to the development of scanning probe microscopy as they allow the position of the probe to be manipulated with sub-nanometer accuracy. Nowadays scan ranges from several nanometers up to tens of microns can be achieved routinely. However, stepper motor controlled X-Y stages are still used in some low temperature scanning probe instruments in which the resolution is not critical but a large positioning range is required.

The piezoelectric effect was discovered by Pierre and Jacques Curie in 1880, approximately 100 years before the invention of the STM. Piezoelectric materials deform when an electric field is applied across them (inverse piezoelectric effect) [35]. Conversely, if mechanical pressure is applied to them and they are deformed, a voltage develops across the material (direct piezoelectric effect). This is due to a net electrical

Figure 2.7: Schematic diagram of a PZT tube showing the five electrodes

polarisation and deformation which exists in the unit cell of the material below the Curie temperature.

In the early years of STM instrumentation, tripod piezoelectric scanners were the predominant choice [36]. the displacements along the X, Y, and Z directions are actuated by three independent PZT transducers. Each of them is made of a rectangular piece of PZT, metallized on two sides.

The piezoelectric materials used in SPM nowadays are various kinds of lead zirconate titanate ceramics (PZT). The mechanism of piezo-electricity in PZT is somewhat different from single-crystal piezoelectric materials, such as quartz. The PZT ceramics are made by firing a mixture of $PbZrO_3$ and $PbTiO_3$ together with a small amount of additives at about 1350°C under strictly controlled conditions. The result is a solid solution.

In 1986 Binnig and Smith published a paper which describes a cylindrically symmetric structure in which a single piece of piezo-electric material could be used for three-dimensional tip motion [37]. The tube scanner we are using is similar to the original design, which is shown in Figure 2.7. A tube made of PZT, metallized on the outer and inner surfaces, is poled in the radial direction. Voltages are applied between the inner and outer electrodes to achieve the desired tube motion. The outer electrodes each occupy one quadrant of the outer surface. There are two to control motion in the X direction ($\pm X$) and two for the Y direction ($\pm Y$). If a voltage is applied between the inner electrode and one of the X(Y) quadrants then the resulting deflection in the X(Y) direction is given by [36]

$$\Delta X(Y) = \frac{\sqrt{2}d_{31}V_{X(Y)}L^2}{\pi Dh} \tag{2.2.2}$$

where $\Delta X(Y)$ (m) is the deflection of the tube in the X(Y) direction;

$d_{31}(m/V) = s/E$, where $s$ is the strain due to the applied electric field, E;

$L$ (m) is the length of the tube;

$h$ (m) is the thickness of the tube;

$D$ (m) is the diameter of the tube;

$V_{X(Y)}$ (V) is the applied voltage.

The range of motion can be extended if a voltage is applied to the opposite electrode with an opposite polarity at the same time. It is the voltage difference between the opposite electrodes which determines the scan range. In this case, the deflection is given by

$$\Delta X(Y) = \frac{\sqrt{2}d_{31}(V_{+X(+Y)} - V_{-X(-Y)})L^2}{\pi Dh} \tag{2.2.3}$$

The deformation of the tube in Z direction ($\Delta Z$) on application of a voltage ($V_Z$) to the Z electrode is given by equation 2.2.4:

$$\Delta Z = \frac{d_{31} V_Z L}{h} \tag{2.2.4}$$

However the co-efficient $d_{31}$ varies with temperature since the materials come to be stiffer in a lower temperature. The specifications of the scan tube are listed in Tables 2.1 [38] and 2.2 [38].

Table 2.1: Scanning range of the scan tube [38]

| Temp.(K) | X(Y) range ($\mu m$) | X(Y) cal ($\text{Å}/V$) | Z range ($\mu m$) | Z cal ($\text{Å}/V$) |
|---|---|---|---|---|
| 290 | 46.2 | 1074 | 4.477 | 298 |
| 200 | 31.0 | 721 | 3.004 | 200 |
| 150 | 24.4 | 567 | 2.364 | 158 |
| 100 | 18.2 | 423 | 1.764 | 118 |
| 50 | 12.0 | 279 | 1.163 | 78 |
| 4 | 8.4 | 195 | 0.814 | 54 |

Table 2.2: Specifications of the scan tube [38]

| Tube scanner details | | HV Range | Min (V) | Max(V) |
|---|---|---|---|---|
| Length(mm) | 50.8 | X-axis | -215 | 215 |
| Outer Diameter(mm) | 12.7 | Y-axis | -215 | 215 |
| Wall Thickness(mm) | 0.508 | Z-axis | -75 | 75 |

## 2.2.4 Non-linearity and Hysteresis

Because of differences in the material properties and dimensions of each piezoelectric element, each scanner responds differently to an applied voltage. This response is conveniently measured in terms of sensitivity, a ratio of piezo movement-to-piezo voltage, namely how far the piezo extends or contracts per applied volt (see the last column of table 2.1). As piezo scanners exhibit greater sensitivity at the end of a

Figure 2.8: Hysteresis and nonlinearity of the scan tube

scan line than at the beginning, the relationship of movement vs. applied voltage is nonlinear. This causes the forward and reverse scan directions to behave differently and display hysteresis between the two scan directions. The effect of nonlinearity and hysteresis can be seen from the curve in figure 2.8. As the piezo extends and retracts throughout its full range, it moves less per applied volt at the beginning of the extension than near the end. The same is true when the piezo is retracting – the piezo moves less per applied volt at the beginning of its extension than near the end.

Nonlinearity and hysteresis result in differences between trace and retrace line and can cause feature distortion in SPM images. This nonlinear relationship can be corrected by applying a nonlinear voltage in real-time to produce a linear scan in X and Y in both trace and retrace scan directions by calibration [39]. Whilst manufacturers such as Digital Instruments implement this in their microscopes, TOPS3 has not implemented this. As the corrections depend on temperature it would be extremely complex to implement it on the 'CryoSxm' head. At low temperatures the

Figure 2.9: A three dimension view of assembling the probe mount

scan range decreases, and fortunately so does the non-linearity.

## 2.2.5   The Probe Mount

The head of the scanning probe microscope is the core of the system, and the quartz tuning fork probe is the core of the head, since the signal from the probe controls the microscope and generates the physical data. Different types of scanning probe microscopes may only differ in the type of probe that is used.

The system uses the same type of quartz tuning fork as the force sensor in all modes but different cantilevers and different system settings implement three different modes of operation (AFM, MFM, SCM). These three modes of operations are even able to be run at the same time. The details of the tuning fork scanning probe will be discussed in section 3.2.

Since the quartz tuning fork is much bigger than conventional cantilever chips, a

Figure 2.10: A three dimension view of the probe mount assembled

special probe mount has been designed to fit it into the original system. As shown in figure 2.9, one tine of a quartz tuning fork is glued down onto a copper probe base with non-conducting epoxy resin with fillers. The probe platform has a slope of 11 degrees to ensure that the tip is the highest point of the assembly. After putting a normal cantilever on the end of the tine, the probe base is screwed onto a pair of clamps to clamp onto the dither piezo (or a copper cylinder which can replace the dither piezo). For scanning capacitance microscopy, a small set of hand-made coils is fitted in a hole underneath the dither piezo or the copper cylinder. After all the wires have been soldered, the copper base of the probe is slid onto 3 copper rods – the main frame support of the head. Figure 2.10 illustrates the parts of the scanning probe when the system is assembled.

Figure 2.11: A Block Diagram of the Electronics

## 2.3 Scanning Probe Microscope Electronics

### 2.3.1 Overview

This section describe the electronic circuits which, when interfaced with the computer software, are used to drive, control and acquire data from the scanning probe microscope.

Figure 2.11 shows a block diagram of the SPM system from the point of view of the electronics.

Basically the circuitry consists of the following components:

1. The scanning circuits which provide the signals for scanning the tip over the sample surface;

2. The function generator which drives the tuning fork at certain amplitude and frequency or can be controlled by the phase lock loop (PLL).

3. The pre-amplifier which amplifies the output signal from the tuning fork sensor.

4. The Q control loop which is used to boost the Q of the tuning fork to increase the sensitivity or depress the Q to increase the scanning speed. Further discussion of the Q control can be found in section 2.3.6.

5. Lock-in amplifier which gives the frequency, amplitude and phase information of the tuning fork.

6. Phase lock loop which keeps the tuning fork running at the resonant frequency or at a certain amplitude by controlling frequency of the function generator. The loop error signal provides a frequency shift signal in phase lock mode. It's an optional device depending on which imaging mode is used.

7. Analog-to-Digital converter which converts the analog data to the digital image data.

These components will be discussed in the following sections.

## 2.3.2 The scanning circuits

A block diagram of the TOPS3 scanning circuits is shown in Figure 2.12. There are two parallel circuits, one is the X scan digital to analog converter(DAC) for motion in the X direction and the other one is Y scan DAC for motion in the Y direction. There is also a scan rotation control circuit which can swap the X and Y signals, thus the system can work in both directions: scanning X direction lines and also Y direction lines. For scanning at other angles the scan area will be reduced due to the limitations of the scan tube. The X scale DAC and Y scale DAC determine the scanning area size, and the X and Y offset DAC controls the X and Y shift of the scan. The X and Y modulation settings allow the application of an external modulation signal to the

Figure 2.12: Schematic Diagram of the Scanning Control Circuit

tip control. The maximum range of the X and Y scan output is from -10V to +10V. It is then amplified to $\pm215$V with a high-voltage amplifier.

## 2.3.3 The Function Generator

The function generator which is used in the experiment is the TG220 made by Yoko-gawa Electric Corp [40]. The maximum output voltage is $\pm10$V with 1mVpp resolu-tion. It provides four types of oscillating voltage (sine,square triangular and pulse) but only sine wave generation is applied in the experiments. In its sweep mode, the frequency and the output amplitude can be controlled by external signals. This function is applied with a phase locked loop (PLL) to control the tuning fork probe working at resonant frequency and certain amplitude. The function generator also provides a modulation function to generate a modulated signal which is used in test-ing the sensitivity of the scanning capacitance microscope. This will be discussed in detail in Chapter 7.

Figure 2.13: Block Diagram of Pre-amplifier

### 2.3.4   The preamplifier

Since the tuning fork detection method is designed for low temperature operation, it needs to have very low energy dissipation during the scan. Namely, the driving voltage and the signal output will both be very small. Thus it's necessary to use a pre-amplifier to boost the signal before further processing. A commercially available current pre-amplifier [41] is used to boost the output signal from tens of nano-amperes to tens of milli-volts ($10^6$ A/V). A block diagram of this pre-amplifier is shown in figure 2.13. This preamplifier is used as it is less affected by large capacitance on input of preamplifier than typical home built versions. Some homemade preamplifiers using operational amplifiers have also been built for the application, the noise level is lower or very close to the commercial preamplifier, but the results in this thesis are all obtained using the commercial one.

### 2.3.5  The phase lock loops

Due to the high mechanical Q of quartz tuning forks, there is a delay of tens to hundreds of ms before the tuning-fork oscillations reach their steady-state condition. As a result, traditional, open-loop methods of AC AFM [42] may not be optimum for tuning-fork feedback, since an AFM might not track a surface fast enough. A phase-locked-loop (PLL) circuit is applied to overcome this limitation by actively tracking the tuning-fork resonant frequency. Basically, as can be seen in figure 4.8, a proportional-integral(PI) loop uses the phase information from the lock-in-amplifier to update the driving frequency, generated by the function generator, to keep the tuning fork running at its resonant frequency (in the same way the tuning fork's amplitude can also be controlled by the other channel of the PID loop). The TOPS3 system then adjusts the height of the probe above the sample to keep the resonant frequency at a preset value.

### 2.3.6  Q-Control circuit

The Q of the tuning fork is determined not only by the material and shape of the tine itself but also several external experimental parameters:

1. Temperature will change the stiffness of all the materials: the mount and the epoxy, thus altering the quality factor.

2. Lower gas pressure around the cantilever increases the Q due to lower energy dissipation.

3. Similarly, tuning forks have far more energy dissipation in liquid than in air.

Figure 2.14: Block Diagram of Phase Lock Loop



Figure 2.15: Schematic of Q-control feedback circuit

Unsurprisingly, the quality factor in superfluid helium is much lower than in vacuum due to acoustic emission by the tine. Experimentally we find that the Q in the helium is still similar to that in air at room temperature which is very convenient from an experimental view point.

Technically, for normal AFM scanning, a quality factor between one or two thousand is sufficient, but for MFM scanning, a much higher quality factor, between 3000 to 5000, is needed to achieve better resolution since the tip-sample interaction is much weaker. However, due to the probes and their mounting, they all have random quality factors between 800 to 2000 in the air and have very different quality factors at different temperature (as shown in figure 4.14). The Q control circuit is essential to optimize the system performance.

A schematic diagram of the Q control circuit can be seen in figure 2.15 [43]. In the diagram, the tuning fork response is shifted in phase and amplified before being added to the drive. The effective damping is altered by feeding back some of the response, with the phase of this feedback signal, relative to the fork velocity, determining whether the damping is effectively increased or decreased. The degree of alteration of the damping (hence of Q) is determined by the gain setting.

Mathematically the differential equation of the one tine tuning fork system (forced oscillator system) is given by [44]:

$$F = F_0 e^{i\omega t} = m\frac{d^2 z}{dt^2} + r\frac{dz}{dt} + kz \qquad (2.3.1)$$

where F is the standard sinusoidal driving force, m is the effective mass of the vibration system, r is the damping constant, k is the stiffness and z is the displacement. The cantilever oscillation in intermittent contact with the sample can be expressed

as:

$$z = z_0 + A_1 e^{i(\omega t - \varphi)} + A_2 e^{i(2\omega t - \varphi)} + ... \quad (2.3.2)$$

The quality factor of the system $Q = \omega_0 m/r$ when damping constant $r$ is small, where $\omega_0$ is the resonant frequency.

For a Q control system, another amplified (G) and $90^o$ phase shifted driving force is added to the system. Ideally if the the positive feedback is working correctly, the feedback force $F_2$ is given by: $F_2 = GA_1 e^{i(\omega t - \varphi + \pi/2)}$. Neglecting the minor contributions of the higher harmonics [45] [46], $F_2$ is proportional to the velocity of the cantilever $dz/dt = \omega A_1 e^{i(\omega t - \varphi + \pi/2)}$. Thus the differential equation can be approximated by:

$$F + F_2 = m\frac{d^2 z}{dt^2} + (r - G/\omega)\frac{dz}{dt} + kz \quad (2.3.3)$$

where $F_2$ is the interaction force between the tip and the sample. In other words, the cantilever oscillates with an effective damping factor electronically tuned of $r_{eff} = r - G/\omega$. Now the Q has been increased to $\omega_0 m/(r - G/\omega)$. However, high effective Q values are unstable for imaging. For gains $G > r\omega$, the effective damping is negative and the cantilever oscillation is unstable. For an ideal electronic Q control, the highest Q value is limited by the changes of the damping e.g. due to viscosity as a consequence of thermal fluctuations [47] [48]. Thus the maximum gain is $G = (r - \Delta r)\omega$, and the minimum effective dampling factor is $r_{eff} = \Delta r$, where $\Delta r$ is the damping factor variation of the fluid as a consequence of the thermal fluctuations. Typically effective Qs of about 10 times bigger or smaller as compared to the ones without Q control can be easily reached. There is no limit with decreasing the Q.

Figure 2.16 shows the result of using Q-control in superfluid helium to increase and decrease the quality factor of tuning fork with one tine immobilized. It can be

Figure 2.16: The result of using Q-control in superfluid helium to increase and decrease the Q of tuning fork with one tine immobilized

seen that the resonant frequency of the tuning fork varies slightly while the Q changes. Over a wide range (two orders of magnitude) of the quality factor, but the variation is only 0.14%. Tamayo et al. [48] showed that, when using Q-control, the resonant frequency of the oscillator varies with the phase of the feedback signal. Therefore it is believed that, for the plots shown in Figure 2.16, the feedback signal was slightly out of phase with the velocity.

## 2.3.7   Analog-to-digital conversion

A National Instrument's 6034E A/D card is also used in the experiment. It offers 16 different channels (8 channels in differential mode) and 16 bit resolution with a sampling rate of 200k samples per second. The measurement range varies from $\pm 50mV$ to $\pm 10V$ depends on the device gain settings. The lift-mode MFM software

obtains the data from this card since TOPS3 does not provide a way for lift-mode operation. The software interface of this card is described in the following chapter.

## 2.4 Scanning Probe Microscope Software

### 2.4.1 Overview

The software automates the coarse approach process and scanning control of the scanning probe microscope, acquires and displays the resulting images and curves, stores the images and scan parameters and facilitates processing and analysis of the data. The direct control of the hardware is handled by TOPS3 software written in Visual C++ supplied with the instrument by Oxford Instruments [28]. An extended control programme written in Borland Delphi [49] uses ActiveX [50] functions supplied with TOPS3 software to implement the 'lift mode' for magnetic force microscopy. The Analog-to-Digital card is also interfaced in the 'lift mode' control panel to gather experimental data. Further details will be discussed in Chapter 3.3. Another computer running lock-in amplifier control software written in Labview [51] monitors and records two channels of the lock-in amplifier to visualize the data flow (tuning fork's amplitude and phase change). A digital oscilloscope is also used to monitor other signal channels. A block diagram of these data and control flows can be seen in figure 2.17.

### 2.4.2 TOPS3

The TOPS3 software consists of a user-control interface and data analysis software running under Microsoft Windows on a Pentium PC. The Control and Acquisition software is linked to the TOPS3 Control Unit via a PC interface card and high

Figure 2.17: A block diagram of software control and data flows

speed interface link, which allows commands and data to be transferred between the software interface and the control unit for the operation of the whole system. The data analysis software is an OEM version called analySIS (Soft Imaging System) [52]. It provides a set of necessary data analysis functions for SPM images. Since they are all very commonly available in conventional SPM software, these functions will not be introduced in detail here.

The TOPS3 control and acquisition software exposes all the settings and system parameters through a collection of programmable objects (known as external functional interfaces), from which an external program is able to invoke the functions. In other words, these exposed objects allow the TOPS3 to be 'driven' by applications written by the user without the need to access or alter the core controller code base. This programmability with TOPS3 is achieved by the Windows' ActiveX [50] technology. The ActiveX programme will be introduced in the next section.

Figure 2.18: The Traces of the EG&G Lock-in Amplifier Labview Control Panel

## 2.4.3 Lock-in amplifier control

The lock-in amplifier is used to monitor the behaviour of the tuning fork, thus it is an essential instrument to generate physical data in the experiment. As mentioned before, the GPIB (General Purpose Interface Bus) protocol connects the lock-in amplifier and a Labview control panel software. This software is based on a standard EG&G lock-in amplifier Labview driver. It provides full support for monitoring and controlling both channels. The data can also be saved to a file for further analysis. With the help of the software, it is very easy to adjust the settings of the lock-in amplifier and very useful to monitor and analyze the characteristic of the tuning fork. The two channels' traces of the Labview control panel can be seen in figure 2.18 (only a part of the control panel).

### 2.4.4   Q measurement

The quality factor, Q, describes the sharpness of the system's response. Q is equal to the ratio of the center frequency to the half power bandwidth. The quality factor comes to be especially important since it is proportional to the sensitivity of the probe in MFM imaging. It is measured by sweeping the drive frequency through the resonant frequency of the tuning fork, and then recording the response of the the tuning fork via the lock-in amplifier control software. Finally the Q is calculated with the help of measurement software written in Delphi.

Since the lock-in amplifier's frequency resolution is 0.5Hz, the measurement will be imprecise especially for a high quality factor tuning fork from the frequency data generated by the lock-in amplifier. This disadvantage can be avoided by assuming the frequency sweep is generated at a constant rate (a new feature of labview 6 (which allows to record the time of the data being obtained) is able to overcome this limitation). In the Q measurement software, after determining the two ends of the sweeping data and entering the sweep range from the function generator, a linear normalization of the frequency is carried out on all the amplitude data. Thus a more precise quality factor can be calculated.

This software also includes a best fit curve for the data. Two methods are used in the application, both based on an electrical resonant RLC (Resistor, Inductor, Capacitor) circuit model [53]. The only difference between them is that one of them considers the additional parallel capacitance in the tuning fork. The details about the RLC equivalent circuit can be found in section 3.2.1.

The software's user interface can be seen in figure 2.19.

Figure 2.19: The User Interface of the Q measurement software

## 2.5 Temperature Control in The Sample Space

The heat exchange between the microscope and its environment occurs in three ways: conduction, radiation and convection. There are two heat exchange sources for the sample space. One is the radiation from the VTI (Variable Temperature Insert) and the other one is constant gas flow from the needle valve. When the helium reservoir is filled with liquid helium, the sample space will slowly reach an equilibrium temperature close to 130K. By adjusting the temperature of the VTI, this equilibrium temperature can be raised up to 220K. However, this method is too slow, although it's very stable. To control the temperature we can use the needle valve on the side of the IVC (Inner Vacuum Chamber) and the VTI. Allowing helium into the sample space through the needle valve and pumping it out with the rotary pump is a very efficient way to cool the sample space. Normally, 2-6 mbar constant pressure measured in the pumping line is used as a constant gas flow to cool the head. To increase the head's

temperature, the same method is used but the VTI is heated up so that the gas flow will be warmed up before passing the microscope head.

The temperature of the head and the VTI can be measured and controlled by the ITC (Intelligent Temperature Controller). The ITC reads the temperature from the sensor in the head and VTI and is also able to heat them up with the heaters inside. It also takes control of the needle valve to manage the gas flow. Since there are too many variable parameters to control the temperature, the ITC may not be able to handle all of them well. To make it simple, a constant rate of the gas flow is set (2-6 mbar measured in the line, 2 mbar is an economic way to maintain the temperature up to 280K and 6 mbar is the maximum gas flow at which the heater of the VTI can be balanced), only the VTI's heater is left to be controlled by the ITC. This method works down to a temperature of 10 kelvin. However below this temperature no method has yet been found to control the temperature with sufficient stability to allow scanning in cold gas. Scanning in superfluid helium has been achieved (1.7K-2K).

## 2.6   The Magnet

The magnet consists of a number of coaxial solenoid sections wound using multi-filamentary super-conducting wire. With a super-conducting magnet power supply (model IPS120-10), which is capable of delivering up to 120 amps at 10V, the magnet provides the ability to achieve 12T at a 4.2K bath temperature. The main coil specifications are shown in the Table 2.3:

Table 2.3: Specification of the magnet

| Maximum central magnetic field at 4.2 K | 12 tesla |
|---|---|
| current for full 4.2 K field | 99.649 amps |
| field / Current ratio | 0.120 tesla/amp |
| Homogeneity (in 10mm DSV) | 0.864 parts in $10^3$ |
| Magnet clear bore diameter | 64 mm |
| Nominal inductance | 27.6 henries |
| Switch heater current for open state | 67 mA |
| Nominal weight | 20kg |
| Current decay in persistent mode | 0.942 parts in $10^4$ per hour |
| Distance of magnetic field centre from base of magnet | 135.2 mm |



Figure 2.20: Schematic of the Pit

## 2.7   Vibration Control

Vibration control system is essential as the space between the tip and sample is less than a few nano-meters during the scan. For this reason, the whole system is supported by Newport pneumatic isolators [54] over a special designed pit.

As can be seen in figure 2.20 and figure 2.2, the outer concrete of the pit will cut off the low-frequency vibration while the inner loose sand layer will absorb high frequency vibration, and acoustic insulation foam is wrapped all around the cryostat to deaden resonances. The vacuum pumping system rests, in an adjacent room, on

rubber feet, as do the sandbox frames. The pumping lines pass through sandboxes to isolate the cryostat from vibrations from the pumps. All the electrical leads to/from the cryostat will pass through and clamp onto another special made sandbox over the top of the cryostat and suspended loosely from the ceiling to prevent transmission of vibrations to the microscope and cryostat. To summarize, the whole system is well isolated from acoustic vibration from ground and other possible directions.

## 2.8  Summary

The descriptions of the microscope head, electronics, basic control software, the system temperature control, the magnet and the vibration control are included in this chapter. Thus the design of the instrument and most of the commercial made parts of the microscope were introduced.

In the following chapter, those key parts which were home-made, such as the microscope probes, and homebuilt software control and analysis of lift-mode for MFM operations are described.

# Chapter 3

# Quartz Tuning Fork Probes and Lift-mode Control

## 3.1  Introduction

Two more important parts of the experimental apparatus are described in this chapter. The first part is the description of the crucial force sensors which are made from commercially available quartz tuning forks (as shown in figure 3.1) and the second half describes the structures and methods of the lift-mode control software.

## 3.2  Quartz Tuning Fork Probes

It is well established that quartz tuning forks can be used as sensors for acoustic and force microscopy [5][6][55]. These self-oscillating high mechanical quality factor sensors provides a good method of detecting sub-pN forces especially at low temperature.

Previous papers have reported difficulty in obtaining images with tuning forks in liquid helium due to instabilities in the resonant frequency [8]. Since the base part and prongs are made out of a single quartz crystal, internal dissipation is low in this oscillation mode and the Q value is extremely high. However, the symmetry of

Figure 3.1: SEM image of a quartz tuning fork with a tungsten wire attached on the end of one tine

the prongs is broken when the tip is attached to one tine and even more seriously compromised when one tine is subject to a tip-sample interaction. We use a similar method to that of Giessibl [56], in which one tine is glued down, to overcome this problem.

### 3.2.1  Immobilizing one tine

The tuning fork's electrical behaviour in the circuit can be described as a combination of a series resonant circuit of a resistor (R), an inductor (L) and a capacitor (C) in parallel with a capacitor ($C_0$) due to the capacitance between the electrodes. The equivalent circuit is shown in Figure 3.2. The tuning fork is driven by AC voltage and the current output is amplified and measured.

The immobilization of the tuning fork tine has two effects: firstly, the Q of the fork is reduced. Secondly, the capacitive current due to $C_0$ is more significant due to the reduced Q.

This capacitive current ($I_0$ in figure 3.2) causes a dip in the I vs f relationship just

Figure 3.2: Equivalent circuit for the piezoelectric quartz tuning fork resonator

above $f_0$. Below $f_0$, the capacitive current ($I_0$) and the current due to the motion of the quartz tines ($I_p$) are in phase and therefore their magnitudes add algebraically. However as the frequency is swept through resonance, $I_p$ changes phase by 180° and therefore their magnitudes effectively cancel each other. This results in the dip which is significant when one tine is glued down. See figure 3.3 for an I vs f plot of a fork with one tine glued.

A remedy for this is to subtract $I_0$ from the total current, so that we are just left with $I_p$. This is done by adding a current equal in magnitude to $I_0$ but 180° out of phase, as shown in figure 3.4 [57]. The centre tapped transformer results in a 180° phase difference between the currents in the upper and lower branches. $C_x$ is a trimmer capacitor which can be tuned to equal the capacitance of the fork electrodes (about 2pF). $I_{C_x}$ is then equal in magnitude, but 180° out of phase with $I_0$. The net current to the current preamplifier is therefore just the current due to the motion of the fork.

The lower curve in figure 3.3 is a plot of I vs f for the same fork, the difference between the two traces being that "capacitance subtraction" has now been implemented for the lower resonance. The dip is now gone and the peak looks much more like that of a driven harmonic oscillator. The Q is determined to be about 2000.

Figure 3.3: Tuning fork glued one tine before and after capacitance compensation



Figure 3.4: The circuit to cancel the effect of tuning fork's stray capacitance

**The AFM/SCM Probe**

Figure 3.5: The AFM/SCM probe

## 3.2.2 Specifications

Our force probe consists of a modified quartz crystal tuning fork [58] with an un-modified resonance frequency of $f_0^{bare} = 2^{15} Hz$ and $Q^{bare} \simeq 10000$ under ambient conditions. (See Figure 3.5)

From the dimensions of this tuning fork cantilever (width $w = 320\mu m$, thickness $t = 380\mu m$, and length $l = 3.2mm$), we estimate its spring constant to be $k = 0.25Ew(t/l)^3 \simeq 10.5kN/m$ where $E = 7.87 \times 10^{10} N/m^2$ is the Young's modulus of quartz. Using the density of quartz, $\rho = 2650kg/m^3$, the theoretical eigenfrequency $f_0^{theory}$ is given by [59] [60]:

$$f_0^{theory} = \frac{(1.8751)^2}{2\pi\sqrt{3}}\sqrt{k/m} = \frac{1.01498t}{2\pi l^2}\sqrt{E/\rho} = 32668Hz \qquad (3.2.1)$$

which is in good agreement with the nominal eigenfrequency – 32768Hz(0.3% difference). This difference is within the uncertainty caused by the measurement of the fork dimensions and also the complexity of the tuning fork's shape (not a perfectly

Figure 3.6: Tuning Fork on the Platform

rectangular beam).

After extracting the fork from its hermetically sealed container, we glue down one tine onto the platform, which makes the other tine a self-exciting, high-Q, stiff cantilever [61]. For good performance, it's essential that one tine of the fork is glued down onto a hard, rigid base with non-conducting epoxy resin with fillers (we use J-B Weld [4]). The platform has a $11^o$ slope to make sure that the tip on the tuning fork will be the highest point. (See Figure 3.6 ). This stage of gluing reduces the resonant frequency to $f_0 \sim 32.4kHz$ and the quality factor to $Q \sim 2000$.

With the help of a micromanipulator and an optical microscope [62], different types of tips from commercially fabricated cantilevers can be glued to the end of the free tine. This step causes a slight change of Q and a frequency shift of about 30 to 50Hz due to the additional mass of the cantilever and the glue. From the dimensions of the commercial cantilever [63] (width $w = 35\mu m$, thickness $t = 2\mu m$, and length $l = 300\mu m$) and the density of silicon $\rho = 2.33g/cm^3$, we estimate the mass $m_{cantilever} \simeq 50ng$. The mass of the epoxy is estimated to be much heavier than the cantilever from the dimensions, since the undetermined mass of the epoxy, the total mass can only be estimated $\gg 100ng$.

The additional mass has also been estimated from the frequency change [64]. The tuning fork tine can be approximated as a uniform cantilever of rectangular shape with a spring of stiffness k and an effective mass $m^* \simeq 0.24m = 0.24\rho lwt = 250\mu g$ ($f = \frac{1}{2\pi}\sqrt{k/m}$ c.f. equation 3.2.1). When an end mass M is added, the resonant frequency is given by [65]:

$$f_{shift} = \frac{1}{2\pi}\sqrt{\frac{k}{m^* + M}} \tag{3.2.2}$$

with the eigenfrequency $f_0 = 32.40kHz$ and the shifted frequency $f_{shift} \sim 32.37kHz$ the added mass M can be estimated as:

$$\begin{cases} f_0 &= \frac{1}{2\pi}\sqrt{\frac{k}{m^*}} \\ \\ f_{shift} &= \frac{1}{2\pi}\sqrt{\frac{k}{m^* + M}} \end{cases} \tag{3.2.3}$$

$$\Longrightarrow M = \frac{k}{4\pi^2}\left(\frac{1}{f_{shift}^2} - \frac{1}{f_0^2}\right) \simeq 490ng \tag{3.2.4}$$

in reasonable agreement with the previous estimate.

The SCM probes can be made with metal-coated cantilevers and use silver epoxy to link the cantilever to the electrodes (See Figure 3.7). By insulating with a layer of polydimethylsiloxane (PDMS), these probes may also be suitable for use in water for biological research [66].

Figure 3.7: Top view of a tuning fork SCM probe

### 3.2.3   Tip etching

Using a tip from a commercially available silicon SPM probe chip is a good choice since these probes give a standard performance and are easy to obtain. However using these tips leads to some difficulties in the tuning fork sensor. First of all, silicon is not conductive. A metal coated tip will have difficulty in connecting to the tuned circuit in SCM applications. Although the tip sticks out from the tine of the tuning fork, the distance between the sample and the tuning fork tine is still too close since the probe is too small when the tip scans over the sample. The interaction between the tuning fork tine and the sample surface affects the result especially when the sample is in the air and the system is running at lift mode. The long range force interaction has been discussed in detail in Chapter 4 section 4.2.4, 4.2.5 and 5.1.2.

A homemade tungsten wire tip may overcome these problems. A standard technique for fabricating sharp tips has been the "drop off" method [67], based on electrochemical etching in which the etching current is stopped immediately after part of the wire immersed in the electrolyte drops off due to it's own weight (see Figure 3.8). The chemical reactions that occur during etching of a tungsten tip are shown in table 3.2.3:

Table 3.1: Chemical reactions for tungsten wire etching

| Cathode | $6H_2O + 6e^- \longrightarrow 3H_2(g) + 6OH^-$ | SRP=-2.48V |
|---------|------------------------------------------------|------------|
| Anode | $W(s) + 8OH^- \longrightarrow WO_4^{2-} + 4H_2 + 6e^-$ | SOP=+1.05V |
| | $W(s) + 2OH^- + 2H_2O \longrightarrow WO_4^{2-} + 3H_2(g)$ | $E_0$=-1.43V |



Schematic diagram of the control system

Figure 3.8: Schematic diagram of the control system (from [68])

The SRP is Standard Reduction Potential and SOP is Standard Oxidation Potential.

The voltage applied to the chemical cell must be higher than $E_0$ to enable this reaction to happen. The chemical cell is shown schematically in Figure 3.8:

Previous experiments have shown that the cut-off time has the most significant effect on the sharpness of the tip; a shorter cut-off time results in a sharper tip (Figure 3.9) [69]. A circuit has been built that has an adjustable cut-off time less than 50ns [70]. (See Appendix B)

Our investigation shows that critical initial voltage ($E_0$), low current ($\sim 7.5mA$) and about 1.5mm length of the 0.02mm dia. tungsten wire dipt into the 2mol/L $OH^-$

Figure 3.9: Cut-off time (from [69])

solution will result in the best result: about 80% chance of getting a <30nm dia. tip. SEM(Scanning Electron Microscope) is needed to characterize the tips, in particular their sharpness, although, the tips are often so sharp that we must image at the limit of resolution of the SEM. Figure 3.10 is SEM picture of a typical tip we have made (about 30nm dia.).

## 3.3 Lift mode control

### 3.3.1 Overview

Magnetic force microscopy and electro-static force microscopy both measure long distance tip-sample interaction forces. These techniques rely on removing topographical information from some other signals. They work by first determining the topographic information along a scan line and then lifting a pre-determined distance above the surface to retrace the line following the contour of the surface, which is commonly known as 'lift mode'. In this way, the tip-sample distance should be unaffected by

Figure 3.10: A typical etched tungsten tip

topography, and an image can be built up by recording changes which occur due to longer range force interactions, such as magnetic forces.

The lift mode is implemented in software by scanning over each line of the image for four times (due to hysteresis) which can be seen in figure 3.11. Since this mode is not implemented in TOPS3 control and acquisition software, it has to be implemented by custom-written software. Basically, the tip motion and settings of all the scanning parameters will be controlled via ActiveX interface provided by TOPS3 software, and all the physical data will be acquired via an analog-to-digital card. The imaging display and some simple data analysis is also be processed through this lift mode control panel.

Figure 3.12 shows the a control flow diagram for obtaining a 256x256 resolution scanning probe image. For both AFM and MFM scanning mode, the tip actually has to scan trace and retrace lines to get rid of the hysteresis. Normally the retrace lines are more stable and linear than the trace lines so that they have the 'correct' scales.

Figure 3.11: Lift mode scan for Magnetic Force Microscope

The PID (Proportional, Integral, Differential) loop shown in figure 3.12 is the one used in TOPS3 to control the tip's distance over the surface to maintain a constant controlling signal. When the PID loop is switched on, the system will automatically move the tip up and down to follow the topography when the tip is scanned over the surface, and the channel of the controlling signal is just the error data. When the PID loop is off in the lift mode, the tip follows the pre-recorded topographic information, and this time the control signal provides the magnetic force data.

At the end of the scanning progress, the tip has to be put back to the centre of the scanning area. This has to be done to reduce the voltage applied to the scan tube after the scan. High voltages can be harmful especially for low temperature operation, as in low pressure helium gas, they may cause a high voltage discharge problem between the electrodes of the scan tube.

### 3.3.2 ActiveX interface programming

As mentioned in Chapter 2.4.2, motion control of the scan tube can be achieved through the ActiveX interface provided by TOPS3 control and acquisition software.

Figure 3.12: A software control flow diagram for lift mode

All the settings and system parameters are embedded into a collection of programmable objects. After the ActiveX objects have been created by the external controlling software, all the functions and settings of the TOPS3 can be invoked by the 'methods' and 'properties' of the objects. These 'methods','properties' and also 'objects' information are required by the client application in order to access them. The type information is contained in a 'type library' file. The TOPS3 installation includes a type library file called 'Tops.tlb' which is placed in the same directory as the other system files. This file can be viewed and converted into a Delphi source file by Borland Delphi's ActiveX tools. A partial listing of the objects used in the lift mode control software is given below [71]:

**Server** Top level TOPS3 server object. Use the methods of this object to obtain references to other TOPS3 Server objects.

**Acquire** Object giving access to acquisition related commands.

**FeedbackLoopParams** Object giving access to the Feedback Loop parameters to switch on/off the PID loop.

**Hardware** Object giving access to various hardware components and settings within the TOPS 3 electronics. It is used to move the tip up and down.

**HeadParams** Object giving access to Head related parameters.

**ScanParams** Object giving access to the Scan Parameters. It is used to set the scanning range and scanning speed in the application.

**System** Object giving access to system wide information and settings.

**StatusPanel** Object giving access to the Status Panel parameters. It is used to withdraw the tip after the scan.

**TipControl** Object giving control over tip position. This object is used to position the tip in nanometer scale.

The methods and properties of the objects used in the application are shown in the list (incomplete) below:

- **ScanParams.XScanAmp:Single**: This property determines the size of the scan area in the X direction. (A similar property for Y direction is Scan-Params.YScanAmp.) The value can be set from 0 to twice the maximum X piezo deflection. It sets or returns the X Scan Amplitude in Å or nm depending on the currently selected units mode. The units mode can be set using the UnitsMode property of the Acquire object. The maximum scan amplitude is fixed by the value of the X piezo deflection coefficient. This deflection coefficient can be set using the XpiezoMax Property of the HeadParams object. The calibrated X maximum piezo deflection against temperature data can be found in the table in section 2.2.3.

- **ScanParams.Surfacevel:Single**: A very important property which controls the speed of the tip. It sets or returns the tip surface velocity in Å/$s$ or nm/s depending on the currently selected units mode. All tip movement is limited to the rate determined by this value. Further discussions about the tip movement are in section 3.3.3.

- **TipCtrl.Tipmoving:Boolean**: The property returns TRUE if tip is moving else FALSE. This read-only property is used to determine if the tip is still

moving in response to a recent move tip command. For low surface velocities, a tip move may take several seconds or longer to execute. Any further tip move commands issued while the tip is still moving will be ignored. The application will typically poll the TipMoving property and issue further tip move commands only when it becomes FALSE. And it can be used to calibrate the tip moving velocity (Section 'Scan Speed Control' in Chapter 3.3.4).

- **TipCtrl.EnterTipControlMode:Boolean**: This method is called to place TOPS3 into 'Tip Control' mode. Any tip move commands issued using the MoveToAbs method (described below) are ignored unless the EnterTipControlMode method has been called first. When this method is first called the tip will be positioned at the centre of the scan area (scan DAC position 2048,2048).

- **TipCtrl.ExitTipControlMode:Boolean**: This method is called to exit 'Tip Control' mode. This method must be called after issuing any required tip move commands to return TOPS3 to normal mode. In a typical application, the EnterTipControlMode method will be called first followed by a series of tip move commands and finally a call to ExitTopcontrolMode. When ExitTipControlMode is called the tip will remain at the latest position specified, requiring the user to move the tip back to the centre of the scan area after the scanning has been completed before leaving 'Tip Control' mode. ExitTipControlMode will fail and return FALSE if the controller is not in 'Tip Control' mode or if the tip is currently moving.

- **TipCtrl.MoveToAbs(XScanDAC, YScanDAC:Longint(DAC position**

**0-4095)):Boolean**: This method is called to issue a particular tip move command. Prior to calling this method the controller must be placed into 'Tip Control' mode using the EnterTipControlMode method. MoveToAbs will fail and return False if the controller is not in 'Tip control' mode or if the tip is currently moving. Note that the bottom left corner of the scan area has scan DAC co-ordinates 0,0 and the top right hand corner co-ordinates 4095,4095. After this method is executed, the velocity of the tip movement is determined by the setting of ScanParams.Surfacevel property and the absolute movement steps are determined by the size of scanning area: ScanParams.XScanAmp and ScanParams.YScanAmp.

- **FeedbackloopParams.LoopLocked:Boolean**: This property allows the user to 'switch' the feedback loop on and off. Note that when it is TRUE, the Feedback loop is actually switched off.

- **StatusPanel.WithdrawTip(Withdraw:Boolean):Boolean**: When this method is called with Withdraw = TRUE, the tip is withdrawn fully using the Z Voltage (Z DAC). When called with Withdraw = FALSE, the tip is released.

- **Hardware.CalibDACVal[1]:Single**: This property is used to set or return the calibrated value for the Z DAC. The calibration units are in Å or nm depending on units mode currently selected. It is used to update the pre-determined tip sample distance in the lift mode.

### 3.3.3   Data acquisition

The detailed control flow diagram of obtaining one line of data can be seen in figure 3.13. For each line scan, the tip will only be issued the command to move over

Figure 3.13: A software control flow diagram for lift mode



Figure 3.14: A Diagram of obtaining data during a line scan

the surface once from one end to the other. Whilst the tip is moving by the ActiveX tip control, a carefully time controlled data sampling is progressing (see figure 3.14). Since the tip moving velocity is constant which is set by ScanParams.Surfacevel, a data sample rate can be calculated to obtain the data of 256 points along the line during the line scan. It will take about 5 seconds for a normal scan line, which means the interval between two sample points is about 20ms (5s/256). Due to a limitation of the normal Windows time controlling APIs, the minimal 'time slice' is 50ms, therefore a new method of controlling the 'time slice' to 5 ms is used in the application.

There are a new group of time functions in the Windows Win32 API [72] to enable better time control. There are two functions of this group used in the application. One is QueryPerformanceCounter: this function retrieves the current value of the high-resolution performance counter, if one exists (it depends on the hardware of the PC). The other is QueryPerformanceFrequency: this function retrieves the frequency of the high-resolution performance counter. The actual time interval between the QueryPerformanceCounter function has been executed can be calculated as: (Current Performance Counter - Last Performance Counter)/(Performance Frequency) in seconds. The fine time control works in this way: the application keeps polling the current 'Performance Counter' and compares it with the already calculated 'trigger-time Counter', and will only issue further commands when it comes to be equal or bigger than the value of 'trigger-time counter'.

### 3.3.4   Scan speed control

There are two key parameters for a line scan control: one is the tip moving velocity, another one is well calculated and controlled sampling rate. It also needs

to be mentioned here that the distance scanned over a line is determined in Scan-Params.X(Y)ScanAmp settings and the parameters (XScanDAC and YScanDAC) in TipCtrl.MoveToAbs function. A full range scan (X(Y)ScanDAC from 0 to 4095) results in the tip scan over a distance in the ScanParams.X(Y)ScanAmp settings.

As can be seen in the last section, the tip moving velocity is set in ScanParams.Surfacevel. However the setting of the surface velocity is not in 100 percent agreement with the calibration. To calibrate the tip's moving speed, the time between tip start and stop (determined by polling the TipCtrl.Tipmoving property) are measured carefully. A large area scan (result in a longer time scan) is performed to calibrate the speed. The result shows that the actual time taken by scanning from one end to the other is about 1% faster than expected. This can be avoided by using a smaller setting in ScanParams.Surfacevel.

Although using the hardware supported better time control functions, the windows minimal 'time slice' is still quantized to be 5ms or 10ms (depends on Win9x kernel or WinNT kernel). This time cannot be altered due to the system core. Thus the full line scan time will also be quantized. Due to the same reason, the setting of the scanning speed needs to be considered carefully. Of course, these will all be done in the application automatically after the user sets up the time per scan line.

### 3.3.5   Analog-to-Digital conversion

A National Instrument 6034E card is used as the Analog-to-Digital converter (ADC) to gather data for the lift mode application. It has 16 single-ended or 8 differential channels and 16 bit resolution with a sampling rate up to 200k per second. The measurement range varies from $\pm 50mV$ to $\pm 10V$ depending on the device's gain settings.

$$V_m = [V_{in+} - V_{in-}]*Gain$$

Figure 3.15: Programmable Gain Instrumentation Amplifier

The input of the ADC is connected to a PGIA (Programmable Gain Instrumentation Amplifier)(see figure 3.15 [73]). Each input signal is connected to the positive input of the PGIA and its reference is connected to the negative input. The card is programmed to operate in differential input mode which is suitable for signals which have their own reference. Since the output of the PGIA is a subtraction of the two terminal inputs, all multiplied by a pre-programmed gain, any noise that is common to the signal and its ground is eliminated. This is known as common-mode noise rejection and is an advantage of operating in differential mode. This noise may, for example, have been picked up by the signal input lead to the ADC and be common to both the signal and ground leads.

A group of functions are supplied with the card called NI-DAQ (National Instrument, Data Acquisition). Two functions are introduced below:

**AI_Configure(deviceNumber, chan, inputMode, 1, polarity, 1)**: Informs NI-DAQ of the input mode (single-ended or differential), input range, and input polarity selected for the device.

**AI_Read(deviceNumber, chan, gain, reading)**: Reads an analog input channel (initiates an A/D conversion on an analog input channel) and returns the unscaled

result.

The parameters are discussed below:

**deviceNumber** assigned by Measurement & Automation Explorer.

**chan** channel to be configured.

**inputMode** indicates whether channels are configured for single-ended or differential operation(0).

**polarity** indicates whether the ADC is configured for unipolar or bipolar operation.

**gain** gain setting for the channel. (-1:$\pm$10V; 1:$\pm$5V; 10:$\pm$500mV; 100:$\pm$50mV)

**reading(Output)** the integer result of the A/D conversion.(-32768 to 32768)

### 3.3.6 Lift mode data analysis

The topographic images and magnetic force images which are acquired in lift mode are all referenced to the ADC captured voltages with a centre position of 0V. For topographic images, it is the middle of the overall vertical movement. In other words, when the system shows you an image, it centers the data at a Z-Centre Voltage value of 0V. Therefore, if the data is captured at an average value of 5V, a Z offset of 5V will have to be removed from the image in order to observe the data. Another correction is required due to the slope of the sample. For an example, a slope of 1 degree for a 10 micron scan area will result in 175nm height difference between two ends, however the roughness of the sample may be less than 40nm. Therefore the contrast of the trace will be lost. This can be avoided by the line fit process shown in figure 3.16: First of all, a standard method is used to calculate the best fit line to the original

Figure 3.16: Data processes for trace and retrace lines

Figure 3.17: A khaki based color table and a typical magnetic force image

data. By subtraction of the best fit line, a relative offset has been subtracted from the data. Therefore the data is now referenced to the average value of all the data. The curve will then be re-scaled to the display range settings, 50nm in this case.

In order to create a conspicuous image to reflect the height information in a two dimension image, a khaki based color table is selected (see figure 3.17). The darkest side is mirrored to the minimal value and brightest side shows the maximal value depends on the scale range settings of the data. A typical magnetic force image can also be found in figure 3.17. All these processes are carried out in a temporary memory location and will not change the original data saved to disk.

## 3.4   Summary

In this chapter, the homebuilt key parts of the microscope were described. The specifications of quartz tuning forks were discussed. These probes enable AFM/MFM and SCM to be preformed. The further calculation of interaction forces of those probes in different modes and performances at low temperature will be described in the next few chapters.

The lift-mode programs enable us to do AFM and MFM with better real-time data analysis. However the program source code is too long to list in the appendix. Hopefully a cd including the source code and related documents will be included with the thesis.

# Chapter 4

# Tuning Fork Atomic Force Microscopy at Low Temperature and in Superfluid Helium

## 4.1 Basic Operation Principles of AFM

The atomic force microscope (AFM) probes the surface of a sample with a sharp tip, a couple of microns long and often less than $200\mathring{A}$ in diameter. In conventional AFM, the tip is located at the free end of a cantilever that is 100 to $500\mu$m long. Forces between the tip and the sample surface cause the cantilever to bend, or deflect (shown in Figure 4.1). A detector measures the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under the tip. The measured cantilever deflections allow a computer to generate a map of surface topography. AFMs can be used to study insulators and semiconductors as well as electrical conductors.

In AFM, constant force mode is analogous to constant current mode in STM. In constant force mode, the cantilever deflection (and hence the tip-sample interaction force) is kept constant via a feedback loop. Quantitative information on surface topography is obtained using this mode.

Figure 4.1: AFM with Laser Detection (from [27])

Several forces typically contribute to the deflection of an AFM cantilever (Van der Waal's forces, capillary forces, Coulomb repulsion, ionic repulsion, etc.[74]). A typical force-distance curve is shown in Figure 4.2.

AFM can be operated in 2 different modes: DC mode (which is known as contact AFM) and AC mode (tapping mode and non-contact mode). In contact-AFM mode, as the scanner traces the tip across the sample, the contact force causes the cantilever to bend to accommodate changes in topography. In tapping-mode, the tip is oscillating in both the repulsive force region and attractive force region (see Figure 4.2), the tip-sample interaction can be obtained by measuring the change in amplitude or phase.

An AFM is commonly used in the contact mode, that is, it is operated in the so-called repulsive force regime between the tip and sample. (See Figure 4.2) The tip is usually attracted to the surface due to capillary condensation (because the tip dips into the liquid surrounding the sample). Therefore, in the contact mode, the sample

Figure 4.2: Inter-atomic Force VS. Distance curve (from [27])

experiences both compressive forces that originate from the tip-sample contact, and shear forces that are attributed to the lateral scan movement. Both forces could induce elastic and/or plastic sample deformation. Furthermore, the resolution of the resulting images could be poor (but better than non-contact mode), because of the stick-slip motion of the tip caused by the lateral shear force and changes in the tip radius due to the tip-sample interaction.

The motivation for the tapping AFM is to overcome the difficulty of operating the non-contact mode AFM which has poorer resolution and to minimize the contact and lateral forces between the tip and sample. Although the non-contact mode AFM is ideal in the study of material systems that are susceptible to damage, it has a number of practical drawbacks. In the non-contact AFM, the tip oscillates with a small amplitude (<5 nm) near the surface. There is only a narrow region where the oscillation amplitude is affected by the short-range van de Waals interaction before the tip becomes abruptly captured by the surface liquid layer. Once the tip is captured, it has to be set free to resume a "free" oscillation such that the feedback system can

respond correctly to variations in the surface topography.

The tapping mode AFM differs principally in that as the probe oscillates at ~300 kHz (its resonant frequency), the tip is made to strike the surface on each oscillation. The oscillation amplitude, and therefore, the energy associated with the oscillation, is made to be sufficient to overcome the stickiness of the surface for a wide range of vertical positions of the probe. In the other words, it is more sensitive and the sharp tip's life is longer.

## 4.2   Tip - Sample Interaction Force

### 4.2.1   Introduction

Since the first invention of AFM in 1986 by Gerd Binnig, Calvin Quate, and Christopher Gerber [2], applications of AFM have provided novel directions for research in nano-science. The atomic force microscopy (AFM) and its descendants: magnetic force microscopy (MFM) and Electric Force Microscopy (EFM) are all based on force detection but on different forces. In this section, the interaction forces will be discussed in detail. These forces include: atomic attractive, repulsive forces and hydrodynamic effects. The effect of magnetic forces will be described in Chapter 5.

### 4.2.2   Tuning Fork Force Detection

The tuning fork used as the sensor is a commercially available quartz crystal tuning fork with an unmodified resonant frequency of 32768 Hz and Q about 10000 when removed from the can. It is driven by an ac voltage at the frequency of its mechanical resonance. The displacement of the piezoelectric crystal induces charge on the electrodes of the tuning fork, and the resulting current is proportional to the velocity

of the tine. The current is converted to a voltage by a transconductance amplifier ($10^6$ V/A) and the phase and amplitude of the voltage are measured with a lock-in amplifier. Excitation amplitudes are typically of the order of 10 mV peak-to-peak. The mechanical oscillation amplitudes can be estimated (as a model for a rectangular cantilever) by [7] [75]:

$$S = 3d_{12}E_Y w \frac{L_e t}{L^3}(\frac{L_e}{2} - L)2\pi f(nA/nm) \qquad (4.2.1)$$

where $E_Y$ is the Young's modulus, L the length, $w$ the width and t the thickness of the prongs, $L_e$ the length of the electrodes of the tuning fork and $d_{12} = 2.1pC/m$ is the piezoelectric coupling constant for quartz [76]. The tuning fork used in the experiment are 0.32mm long, 0.38mm thick and 3.2mm long. From the dimensions of the tuning fork, equation 4.2.1 gives a theoretical sensitivity of 1.7nA/nm at 32 kHz. A typical detection current is about 50nA and then be amplified to 50mV with the pre-amplifier ($10^6 V/A$). This gives an amplitude of about 30nm and a sensitivity of about 0.01nm with 1mV detection resolution.

When one tine is immobilized to the copper probe mount, the symmetry of the prongs is broken, thus the quality factor drops from about 10000 to about 2000 at room temperature in air (and similarly in liquid Helium at 1.7K) as the stress at base of the tines is no longer symmetric. However it is more stable and more faithful in reporting the force change since one tine sensor behaves as a simple, self-exciting, high-Q and stiff cantilever sensor. The quality factor is high enough to operate in AFM mode and Q control can be applied to change the Q. The specification of the tuning fork probe was discussed in Chapter 3.

The effective mass theory can be used to study the frequency change of the tuning

fork [64]. The one tine tuning fork can be approximated as a uniform cantilever of rectangular shape with a spring constant k and an effective mass $m^*$. From equation 3.2.4:

$$M = \frac{k}{4\pi^2}\left(\frac{1}{(f_0 + \Delta f)^2} - \frac{1}{f_0^2}\right) \qquad (4.2.2)$$

since $\Delta f \ll f_0$ the equation can be re-written as:

$$M = \frac{k}{4\pi^2}\left(\frac{f_0^2 - (f_0 + \Delta f)^2}{f_0^2(f_0 + \Delta f)^2}\right) = -\left(\frac{k}{2\pi^2 f_0^3}\right)\Delta f \qquad (4.2.3)$$

It shows that the change of the effective mass is proportional to the frequency shift and adding mass will decrease the resonant frequency. Using the spring constant $k = 10.5kN/m$ and a typical resonant frequency $f_0 = 32.4kHz$, the coefficient equals 15.7ng/Hz.

### 4.2.3 Frequency-Distance Curve

All the experimental data taken by the AFM probe are determined by the interaction between the tip and sample. Thus the force-distance curve is very important to the understanding of the tip-sample interaction.

For a tuning fork AFM probe, we study the change in the tip-sample force gradient by looking at the frequency shift of the resonant frequency of the tuning fork, so the investigation of the frequency-distance curve is very important in order to understand the principles of the tuning fork AFM. For the case when the tip-sample distance, d, is much greater than the amplitude of the tuning fork $A_0$, the frequency shift $\Delta f$ can be described as the gradient of the tip-sample force: $\Delta Freq_{ts} = \frac{f_0}{2k}\partial F_{ts}/\partial d$, as described in the work by Albrecht et al. [77] and Giessbl et al. [61].

A simple model of how the force ($F_{LJ}$) varies with distance($d$) between tip and sample atoms can be described by a Lennard-Jones (LJ) potential with bond energy $E_{bond}$ and equilibrium distance $\sigma$ for simplicity:

$$F_{LJ}(d) = -12\frac{E_{bond}}{\sigma}\left[(\frac{\sigma}{d})^7 - (\frac{\sigma}{d})^{13}\right]. \tag{4.2.4}$$

Since the tuning fork cantilever is operating in dynamic-force mode, a force component, caused by dynamic tip-sample distance, needs to be taken into account. With the results above, the total $F_{LJ}$ for an idealized pyramidal silicon tip that is bounded by (111) planes (full tip angle $\alpha = 70.5°$ [78]) at a distance $q$ to a flat silicon surface can be calculated explicitly. It is instructive to express $F_{LJ}$ as a function of $d/\sigma = q$ (where $\sigma$ is the distance at which the force is zero) [61]:

$$F_{ts}(q) = -0.1/q - 18.9\left[1/q^7 - 1/q^{13}\right] nN. \tag{4.2.5}$$

as $\Delta Freq_{ts}(q) = \frac{f_0}{2k}\partial F_{ts}(q)/\partial q$:

$$-\Delta Freq_{ts}(q) = \frac{f_0}{2k} \times \left[\frac{-0.1}{q^2} - 18.9\left(\frac{7}{q^8} - \frac{13}{q^{14}}\right)\right] Hz. \tag{4.2.6}$$

and the potential energy $E_{ts} = \int F_{ts}(q)dq$:

$$E_{ts}(q) = -0.1ln(q) + 18.9\left[\frac{1}{6q^6} - \frac{1}{12q^{12}}\right](10^{-18}J). \tag{4.2.7}$$

the $F_{ts}(q)$, $E_{ts}(q)$ and $-\Delta Freq_{ts}(q)$ can be plotted in Figure 4.3. Although the tip geometry is usually not regular and no dissipation was included into the calculation, the predicted 10 Hz frequency shift dip is still in good agreement with the experimental data (4Hz) shown in Figure 4.4. Experiment shows that the minimum

Figure 4.3: Tip-sample Frequency Shift,Force,Energy against Distance

occurs at longer distances than the theory suggests. This is due to the fact that the measurement was taken with a large amplitude (about 30-50nm) of tuning fork oscillation which was averaging the force interaction.

The interesting thing shown in figure 4.3 is that the frequency shift comes to be positive in the attractive force region as the frequency shift is responsive to the gradient of the force change. In principle, $F_{ts}$ is attractive until the front atom of the tip meets the sample. In high vacuum, it is even possible to obtain atomic resolution by probing the attractive force [79], and carrying out faster and more stable scanning whilst avoiding damage to the tip (See Figure 4.5, 1s/line, the blurred image is due to the 25ms time constant limitation of the TOPS3 and maybe also because the tip is

Figure 4.4: Tip-sample Frequency Shift against Distance (Data)

Figure 4.5: Negative frequency shift mode AFM ($\Delta f = -2Hz$) on 20nm steps gratings at room temperature. ($5\mu$m by $5\mu$m area $5\mu m/s$)

far away from the sample). However, attractive force mode depends on a tip-sample negative frequency shift of at least 2 Hz, and the frequency change is relatively small. It is much easier to work with the repulsive forces region which arise upon contact at room temperature and in the liquid (See Figure 4.6).

## 4.2.4 Resonant Frequency Change in Liquid Helium

Stable operation cannot be achieved in liquid helium at temperatures above the superfluid transition due to the boiling of the liquid. The resonant frequency drops by several hundred hertz on immersion in superfluid helium. Chu [80] showed that the frequency of a cantilever beam immersed in an inviscid fluid, $f_f$, is given by

$$f_f = f_v(1 + \frac{\pi \rho_f w}{4\rho_c t})^{-1/2} \tag{4.2.8}$$

where $f_v$ is the resonant frequency in vacuum, $\rho_f$ the density of the fluid and $\rho_c$, t, and w the density, thickness, and width of the cantilever, respectively. Experimentally we find $f_v = 32245Hz$ and $f_f = 31551Hz$, i.e., a drop in $f_0$ of 2.15% on immersion

Figure 4.6: Positive frequency shift mode AFM ($\Delta f = 2Hz$) on 20nm steps gratings at room temperature. ($5\mu$m by $5\mu$m area $1\mu m/s$)

in liquid helium. Taking $\rho_f = 145kg/m^3$ for the density of liquid helium and $\rho_c = 2650kg/m^3$ for quartz, the expression derived by Chu predicts a drop in $f_0$ of $1.80\%$, which is in close agreement with our experimental result.

This model can also be applied to the case of the frequency change of the tuning fork when the sample space is pumped out from a pressure of 1 bar to vacuum. Taking $\rho_f = 0.1785kg/m^3$ for the density of helium gas at $0^oC$ at 1 bar, and the expression predicts a 0.7Hz resonant frequency difference between the 1bar helium and vacuum. Experimentally a half hertz change is found which fits the theory reasonably well. Further more, if the $\rho_c >> \rho_f$ and the width and the thickness are smilar, the equation can be rewritten:

$$
\begin{aligned}
f_f &= f_v(1 - \frac{\pi \rho_f w}{8\rho_c t}) \\
f_f - f_v &= -f_v(\frac{\pi \rho_f w}{8\rho_c t})
\end{aligned}
$$

(4.2.9)

which implies that the frequency change is proportional to the density of the gas

or the liquid which agrees with the experiment taken in argon gas as shown in the figures in section 4.4.2.

## 4.2.5 The Calculation of the Long Distance Forces

A very different frequency-distance curve is observed when operating in superfluid helium as compared to gas (See figure 4.7). In this case, the tuning fork begins to sense the sample surface at a distance of several hundred microns. Such long-range probe-surface interactions have been reported previously for tuning forks in gas [81] and for conventional cantilevers in liquid [82] [83] [48]. Since the resonant frequency of the tuning fork in an essentially inviscid fluid is insensitive to changes in damping, we were able to study long-range changes in the effective mass of the cantilever in a controlled and precise fashion. Changes in the effective force constant could also produce frequency shifts, however at such large distances the required force gradients are unlikely. Also as we observe the velocity resonance, the resonant frequency does not alter with a change in dissipation.

The theoretical analysis of the frequency-distance curve is inspired by the description of a sphere, immersed in an inviscid fluid, moving perpendicularly to a wall. Milne-Thomson [84] calculates that as the sphere approaches the wall, its hydrodynamic effective mass $m'$ increases to $\frac{1}{2}[1 + \frac{3r^3}{8(r+x)^3}]m'$, where r is the sphere radius and x is the distance from the wall to the surface of the sphere. The total effective mass as a function of the distance $z$,can be written as:

$$m^*(z) = m_0^* + \frac{1}{2}[1 + \frac{3r^3}{8(r+z)^3}]m' \qquad (4.2.10)$$

If we make the assumption that an expression of similar form is valid for the case

Figure 4.7: The Long Distance Frequency-distance Curve in Superfluid Helium

of a rectangular beam approaching a surface, we can write the following expression

for the resonant frequency of the beam near a surface in an inviscid fluid:

$$f_0 = \frac{1}{2\pi} \sqrt{\frac{k}{m^* + m'_0[1 + \frac{ca^b}{(a+z)^b}]}} \tag{4.2.11}$$

where k and $m^*$ are the force constant and effective mass of the beam, respectively, $m'_0$ is the additional effective mass of the fork on immersion in superfluid, a is now a characteristic length, related to the fork dimensions, z is the tip-sample distance and c and b are constants.

Thus the frequency of a beam an infinite distance from the surface $f_\infty$ is given by:

$$f_\infty = \frac{1}{2\pi}\sqrt{\frac{k}{m^* + m_0'}} \qquad (4.2.12)$$

The equation 4.2.11 can be rewritten to get the expression of the resonant frequency shift $\Delta f$ by comparing to equation 4.2.12:

$$\Delta f = f_0 - f_\infty = -\frac{c}{2}f_\infty \left(\frac{m_0'}{m^* + m_0'}\right)\frac{a^b}{(a + z)^b} \qquad (4.2.13)$$

In fitting the data in figure 4.7 we have assumed this functional form, taking a, b and c to be free parameters. $m^* = 250\mu g$ is calculated from the dimensions and density of the tine with the equation in the section 4.2.2, and $m_0'$ is determined from the change in resonant frequency of the fork on immersion in superfluid. With a typical resonant frequency change 32245Hz (vacuum) to 31551Hz (superfluid helium), the equation 4.2.2 finds that the effective mass $m_0' = 11.4\mu g$. The curve through the data points in figure 4.7 is the best fit to the data, with the parameter values $a = 130 \pm 60\mu m$, $b = 2.0 \pm 0.8$ and $c = 0.112 \pm 0.006$. These values are in surprisingly close agreement with those predicted for a sphere (about 0.2). For example, a is found to be approximately equal to half the thickness (or width) of the tuning fork tine, whereas in the case of a sphere it is the radius. The pre-factor c is of the same order of magnitude as that found for a sphere. b is slightly lower and this may be due to the different geometries of the two cases. It is believed that the success in fitting the data to this model is strong evidence that the long-range fork-sample interaction is due to the increasing hydrodynamic mass of the cantilever as the gap between the two bodies is narrowed. These effects may become significant for other fluids

and cantilevers where Q-control is used to reduce the width of the resonance of the cantilever.

## 4.3 Operation modes

### 4.3.1 Overview

Several modes of operation are available to the operator of the SPM. These modes include phase lock mode (using a proportional-integral (PI) feedback loop to always work at resonant frequency), amplitude lock mode (working at a constant amplitude of the tuning fork) and phase detect (scan with a constant lift height at a constant frequency, look at the phase change). The lift mode is actually a combination of amplitude lock mode and phase detect mode. Each of these operational modes will now be described in more detail.

### 4.3.2 Phase lock mode

As shown in Figure 4.8, the TOPS3 [85] SPM controller controls the AFM probe to approach and scan the sample. The probe is driven by the function generator oscillating over the sample at its resonant frequency. A phase-locked-loop (PLL) is used to track changes in the fork's resonant frequency, $f_0$, as it interacts with the sample. The phase signal and amplitude (A) are then sent to TOPS3 by the PLL, which will be used by TOPS3 to maintain a constant tip-sample distance. The vertical movement of the scan tube will be recorded as the topograph of the sample.

The control circuit of the phase lock mode AFM is illustrated in Figure 4.9. On the one hand, it's obvious that faster data collection will result in a faster scan speed, on the other hand, the lock-in amplifier requires at least several cycles of the tuning

Figure 4.8: Block Diagram of Tuning Fork AFM

fork oscillation to collect information, to lock to the right phase and give out the correct data. Furthermore, the time constant (TC) settings of the feedback loop must be greater than the lock-in amplifier to avoid instabilities.

As shown in the broken-line box 'A' in Figure 4.9, the tuning fork is profiling the sample and sending the data to the lock-in amplifier, and the lock-in amplifier will lock to the reference frequency and pass the phase to the phase lock loop (PLL). The PLL provides the correction of the frequency back to the tuning fork. Thus they form a control loop. The period of the tuning fork oscillation is about $1/32kHz \simeq 30\mu s$, so the lock-in amplifier's time constant (TC) is set to be $320\mu s$ and the PLL's TC is about 5 ms. This factor of ten difference in time constant avoids instability in the feedback loop.

The low-pass filter's TC has been set to be 5ms to cut off higher frequency noise from the PLL loop. The 5ms TC enables the data collection rate to be $1/5$ms $/2 =$

Figure 4.9: Schematic diagram of phase lock mode AFM control circuit

100 Hz (sampling rate = 2 × data). The maximum speed is $256/100 = 2.56$ s/line for a 256 pixel line, and it will take about $2.56 \times 2 \times 256/60 \simeq 22$ minutes (counting the reverse lines) to take an image of 256 by 256 pixels. From the calibration, the TOPS3's digital PID loop's TC (i.e TC of tip-sample distance control, the time of moving can be ignored) is 25ms (40Hz). Therefore, the best quality of the image can be achieved at a scanning speed of 256/40=6.4s/line.

Since it is always working at its resonant frequency, the phase lock mode provides a direct method of measuring the frequency-shift versus distance curve. It's very easy to track the resonant frequency, thus it is very useful for the resonant frequency measurements which require an instant and accurate result and especially in situations when Q changes, such as determining resonant frequency against the pressure and resonant frequency against the temperature etc. The phase lock mode also provides the possibility of a fast scan since the measurement is not limited by the mechanical settling time (The scanning speed is currently limited by the 25ms TOPS3 feedback response delay). A change in the Q of the tuning fork during the measurement also

affects this mode less than in other modes, so that the phase lock mode might be more stable in superfluid helium except that if the density ($\rho$) is altering in HeII might get incorrect value. The first successful image taken in superfluid helium was taken using this method.

### 4.3.3   Constant amplitude mode

The constant amplitude mode is actually simpler than the phase lock loop method. If we remove the PLL loop from figure 4.9, the tuning fork's output amplitude signal can be kept constant using the TOPS3 controller instead of the phase lock loop. This method is similar to the conventional laser cantilever non-contact mode AFMs, the only difference is that a much higher spring constant cantilever is used.

The advantage of using constant amplitude mode is that there is only one parameter to be adjusted – the distance between the tip and the sample, so that the time constant can be set easily without worrying about two parameters interacting with each other as in phase lock loop mode. Another advantage is that the amplitude of the tuning fork is constant which is very necessary for the operation of scanning capacitance microscopy since the capacitance change is related to the amplitude of the tuning fork. The noise level of this method ($<0.1$mHz for $f_0 = 32kHz$ and Q=5000) is much lower than the phase lock mode which is about 3mHz peak to peak (due to the feedback circuit). However the scan speed is now limited by the TC of the TOPS3's feedback loop (5s/line), and there is a problem only in very high Q ($>$8k) in lift mode.

Figure 4.10: Schematic diagram of phase change due to resonant frequency shift

## 4.3.4 Constant distance mode and lift mode

The lift mode was invented for magnetic sample imaging. The basic idea is to try to get rid of the topographic information during magnetic force gradient detection. It contains two modes together: the constant amplitude mode and the constant distance mode – the magnetized probe scans over the sample with the constant amplitude mode to gather the topographic information and then it is lifted up to scan over the sample again, using the previously determined topograph to maintain the distance between the tip and sample to a constant value and record the phase change due to the magnetic force interaction which is called constant distance mode.

The phase shift is due to the interaction force gradient altering the force constant of the cantilever which results in a shift of the resonant frequency of the tuning fork. As shown in figure 4.10, the phase component (experimentally achieved using the Y

output when it's in phase at resonant frequency) change is much steeper than the amplitude component (experimentally measured using the X output when it is in phase at resonant frequency) at the resonant frequency and locked at the correct phase. Therefore a small resonant frequency shift will result in a big change in the quadrature component (Y of the output) when the system is working at the tuning fork's resonant frequency. The sensitivity of using the constant height mode is much higher than the other two methods (0.1mHz) so that it's suitable to detect small force gradients such as those due to magnetic or electric force gradients, but it only works to detect small force gradients as the phase change is linear (positive) in a small frequency range. The phase lock mode causes too much noise so it can not be used with the constant distance mode. However the constant distance mode can still pick up the topographic information when it's working in the air or in the liquid due to the previously discussed hydrodynamic effect.

## 4.4  Low Temperature and High Magnetic Field Operations

### 4.4.1  Overview

To scan at low temperatures rather than at the room temperature, there are extra factors to be considered. First of all, since the microscope head has to be put in the cryostat, the sample and probe are not visible directly. This causes problems such as alignment of the probe and the sample, especially for a very small scanning area. It also leads to a problem estimating the distance between the tip and the sample — it might take ages to do the approach but you still can not know how far away the probe is until the tip luckily reaches the sample gently or unfortunately crashes.

This problem can be solved by using a video camera combined with a microscope at room temperature (e.g. Digital Instrument's Dimension 3100), but this method is impractical at liquid helium temperatures. Secondly the temperature and the pressure changes will also shift the tuning fork's resonant frequency, so that there will be noticeable artifacts adding into the data if the environmental conditions are not strictly controlled. Therefore the only possible way to scan at helium temperature will be in the superfluid helium since we have not been able to control the temperature between 10K and 4.2K in our cryostat. The third problem is the high-voltage discharge at low temperature in helium gas. The system applies up to $\pm215V$ to the scan tube's electrodes which means there will be up to 430V difference between the two thin electrodes with a gap less than 1mm. At low temperatures with low pressure helium gas, this situation may cause an electrical discharge on the scan tube during the scan. The discharge interrupts the tube movement and the sparks etch the scan tube badly. These disadvantages make low temperature operation more difficult compared to operation at room temperature. In the following sections, details of these topics and methods to overcome them will be discussed.

## 4.4.2 The dependence of the resonant frequency change with temperature and pressure

The probe used in the scan is a quartz tuning fork. The detection methods have been described previously. Due to the difficulties of controlling the temperature at low temperature, instability of the temperature and the pressure will affect the result. Therefore study of the resonant frequency change against temperature and pressure will be very helpful in guiding us to choose the best conditions for the scanning process.

Figure 4.11: Plot of resonant frequency (dots) and Q (square blocks) against pressure (>20mbar) at room temperature

Figure 4.12: Plot of resonant frequency and Q against pressure (<20mbar) at room temperature for argon gas

As shown in Figure 4.11 and Figure 4.12 (they are two different tuning forks), the tuning fork's resonant frequency was plotted against the pressure in argon gas at room temperature. The resonant frequency was recorded at the maximum current response of the tuning fork. These tuning forks have two free tines suspended by the leads in the vacuum chamber, since the pressure change is symmetric to both tines this will not affect the result, and the quality factor is extremely high so that the current response at the resonant frequency will be much higher and sharper which makes it easier for us to determine the resonant frequency.

The blue straight line in Figure 4.12 and Figure 4.11 is the best linear fit of the experimental data. As we will discuss in Chapter 5.1.2, the tuning fork's resonant frequency change is linear in the density change of the surrounding gas. For an ideal gas, the density of the gas is proportional to the pressure at a constant temperature. To summarize, the resonant frequency of the tuning fork is proportional to the pressure which is in good agreement with the experimental data.

At the lower pressure end of figure 4.12, it is noticed that the slope of the resonant frequency change increases. A change of 0.15 Hz in the resonant frequency corresponds to about 2.5mbar of the pressure in the plot which is 60mHz/mbar. This noticeable frequency change suggests that there is a need to control the pressure. In the normal experimental situation the pressure can be controlled fairly well or a manostat can be applied to regulate the pressure as required.

Figure 4.13 [86] shows an easily-constructed and effective manostat that employs a rubber diaphragm, which, when there is a pressure difference across it, bows out and blocks the pumping orifice. Thus the cryostat's pressure can be automatically maintained as the pressure of the gas pre-trapped in the chamber. The performance

Figure 4.13: A simple homemade manostat

of this manostat is greatly improved if the orifice is cut slightly on a slant as shown. This manostat is effective down to a cryostat pressure of about 8mbar to maintain the pressure change better than 0.1mbar.

For low temperature operation the temperature control is much harder and more important than the pressure. The resonant frequency and Q of a typical one-tine immobilized quartz tuning fork as a function of temperature is shown in figure 4.14. The data were taken under vacuum during the warm up period from 10K. The first point at 20K might be incorrect since the system might not have yet reached the equilibrium.

The resonant frequency plot (blue) in figure 4.14 shows a big valley around 50K close to the freezing temperature of oxygen (54.2K at 1bar) and a small spike close to the freezing temperature of nitrogen (63.2K in 1bar). There is also another valley in the Q plot close to the normal boiling point of nitrogen (77.2k in 1bar). Below 40K the Q and frequency all climb up to their highest point due to the fact that there should

Figure 4.14: One tine immobilized tuning fork's Q and Frequency against temperature in vacuum

be no more phase changes for nitrogen and oxygen and everything is getting stiffer. Whilst the observations suggest that adsorption of gases onto the fork is affecting the resonant frequency, the reason for these changes are still not determined. Whatever the reason, the one-tine-immobilized tuning forks all show large frequency and Q changes in the 50 kelvin range. The steepest slope in the figure 4.14 is -1.26Hz/K between 40K to 47K and the Q also changes correspondingly in this region. Therefore the temperature control is more important than the pressure.

### 4.4.3 Temperature control

A good vacuum is the best condition for MFM lift-mode scanning as discussed in section 5.1.2, it also benefits from there being no need to worry about the high-voltage discharge problem at low temperature. However there is no good way to prevent the sample space's temperature slowly approaching to an equilibrium temperature of approximately 150K when the helium reservoir is filled with liquid helium. This equilibrium is a balance of heat conduction from room temperature and radiation loss to the 4.2K bath temperature. Between 100 to 250 K there is enough time for the SPM to capture an image during the warming up or cooling down period in vacuum before the temperature has changed too much. By using the heater, the radiation loss of the VTI can be balanced to reach a higher equilibrium temperature. Thus in this temperature range is recommended to operate in vacuum for the lift mode scan process which guarantees a good resolution and quality. For the temperature range from 20K to 100K, there must be some cool helium gas flowing past the sample to cool the system and the ITC can be used to automate the temperature controlling as introduced in Chapter 2.5. Either full pumping on low pressure gas flow (a few mbar measured at the exit of the sample space) or gentle pumping (or control with

manostat) on higher pressure gas flow (about 1 bar) will both be able to control the temperature well enough in this temperature range (<10mK/hour). The system will approach the stable temperature point within a few hours. A gas pressure should be carefully chosen to prevent high voltage discharge in this range as well. For an ideal condition the lower pressure is better not only for lower noise in lift mode scan but also for low helium consumption. A gas pressure of 2-3mbar is recommended for this cryostat.

### 4.4.4   High-voltage discharge at low temperature

Liquid helium is a fluid of low conductivity. Gaseous helium has, at room temperature, a low dielectric strength, about 1/12 the dielectric strength of air at atmospheric pressure. But at its boiling point at one bar, helium gas is sufficiently dense to have a dielectric strength about 3.3 times that of air at room temperature at the same pressure.

In figure 4.15 [87] [88], both the room temperature, $20^oC$, and the cryogenic temperature breakdown voltages, are plotted together as a function of both bar-mm(for the $20^oC$ values) and (g/cm3)-mm, which apply to both the $20^oC$ and the low temperature values. Within the scatter of the data, there does not appear to be a temperature difference in the breakdown voltage for the same density-spacing product.

Reference  [88] [89] [90] and [91] are references which present data only at low voltages near the Paschen minimum but apparently using, in most cases, carefully purified helium.

The consensus of the apparently most correct data, places the Paschen minimum for pure helium at approximately 160 volts at 50-70 mbar.mm [88] [89] [91]. Since the

Figure 4.15: Paschen curve for helium in log log scale at $20^oC$. (from [87])

purity of the helium gas used in our work is not especially high (it will be higher when < 10k since all other materials are solid), the breakdown point could be lower than the Paschen minimum. The pressure around the scan tube should be higher than at the exit of the sample space where the pressure is measured, and the maximum voltage difference between two thin electrodes of the scan tube is 430V. It is therefore very likely that a discharge will occur when controlling the system temperature using low pressure gas flow at low temperature. The way to prevent the discharge is to use higher pressure helium gas and always park the scan tube at the center where there is no applied voltage on all the electrodes after the scan.

Scanning in superfluid helium is fine when the head and the scan tube are all immersed in the liquid. No electrical discharge has been found in superfluid helium since the density of the liquid is too high for breakdown. Also at the lowest temperature of 1.6K the vapour pressure is fairly high (about tens mbar) so even if the scan tube is above helium surface, breakdown is unlikely.

## 4.5   Test Sample Results

Figure 4.16 shows scanning tunnelling microscopic (STM) images taken at room temperature of gold monatomic steps in order to test the vertical resolution of the scan tube and also the control software. The sample is gold on glass and the surface consists of flat terraces separated by monatomic steps. This "flat" surface was obtained by subjecting the sample to high temperatures for a several seconds with a miniature blowtorch. The monatomic steps demonstrate the high vertical resolution obtainable with the microscope.

The left image and the right image are of the same region, while the right one has

Figure 4.16: STM images of gold monatomic steps at room temperature ($4\mu$m by $4\mu$m area)

a pit caused by the crash of the probe. There is also a very clear scratch on both pictures.

Figure 4.17 demonstrates the ability of the tuning fork AFM in different conditions with the test grating sample. Figure 4.17(a) shows a $6\mu m$ by $6\mu m$ area of a silicon calibration grating using a silicon cantilever acquired at room temperature in air. The Q is about 2000 which is a good value for AFM imaging. Figure 4.17(b) shows a similar image acquired at 50 K in vacuum without Q control. The Q of the fork in vacuum is about 20 000 at that temperature and such high sensitivity makes microscope control more difficult, as shown by the poorer quality of this image. This can be solved by using the Q control circuit to decrease the Q. Additionally, experiments tell us that silicon tips scanning on silicon sample will wear much faster on a silicon surface. This may be caused by the similarity of the atomic structures which introduces strong interactions between the tip and the sample. Figure 4.17(c) shows a 500nm by 500nm image of a gold thin film acquired in superfluid helium at 1.7 K and in a magnetic field of 10 T. It seems that there are no strong effects of the magnetic field although the tuning fork's leads are made with ferromagnetic material.

Figure 4.17: Topographic images of (a) silicon grating acquired in air at room temperature, (b) silicon grating acquired in vacuum at 50 K, (c) gold thin film acquired in superfluid helium at 1.7 K and in a magnetic field of 10 T. (d) is a cross section take along dotted line in (c) [43]

Figure 4.18: Q control in superfluid helium

Surprisingly, imaging could also be performed while the field was being swept from 0 to 10 T, with negligible shift in the relative lateral tip-sample position. The most important point is that stable topographic imaging utilizing a piezoelectric quartz tuning fork in superfluid helium has been achieved. This success is due to the fact that one tine has been immobilized, as discussed earlier.

All of the last three images are obtained in phase lock mode. Constant amplitude mode has also been performed in superfluid helium. The images will be shown with the capacitance information in Chapter 7. However the amplitude of the tuning fork probe drifts a lot due to the change of Q with temperature and resonant frequency, thus the phase lock mode is easier to operate in superfluid helium than the constant amplitude mode.

Figure 4.18 demonstrates the ability of the Q control circuit in superfluid helium

although the Q=2900 without Q control is suitable for scanning.

# Chapter 5

# Magnetic Force Microscopy at Low Temperature with Tuning Forks

## 5.1 Tip-Sample Interaction Force

The method of magnetic force detection, known as lift-mode, has been introduced in Chapter 3.3. In this section, the interactions between tip and sample in lift mode are described. These interactions include the magnetic force gradient and the influence of hydrodynamic effects when the system is working in liquid or air.

### 5.1.1 Magnetic Force Gradient Estimation

In MFM, as shown in figure 3.11, a tiny magnetized tip on the end of the cantilever is brought to within several tens to hundreds of nanometres of the surface of the sample. The tip is then scanned over the surface of the sample following the previously obtained topography. The stray magnetic field of the sample causes a force gradient at the tip which will cause a frequency change in the tuning fork sensor, the phase change of the tuning fork response will be recorded as a map of the magnetic information of the scan area.

From equation 4.2.4, it is clear that atomic force in the attractive force region

decays with a power of seven. However, the magnetic force gradient decays with a power of three – related to the decay of magnetic field, much slower than the atomic forces. So the magnetic force dominates the force interactions in the distance between tens to a few hundred nanometres over the magnetic sample. Therefore the magnetic force gradient can be measured within this region.

The nature of the magnetic force gradient will now be explained in more detail. When a magnetic point dipole, $\boldsymbol{m}$, is brought into a magnetic field of flux density $\boldsymbol{B}$, the magnetostatic energy of the system is given by:

$$E = -\boldsymbol{m} \cdot \boldsymbol{B} \tag{5.1.1}$$

$$= -\mu_0(\boldsymbol{m} \cdot \boldsymbol{H}) \tag{5.1.2}$$

The force on the dipole due to its presence in the field is given by:

$$\boldsymbol{F} = -\nabla E \tag{5.1.3}$$

$$= \mu_0 \boldsymbol{\nabla}(\boldsymbol{m} \cdot \boldsymbol{H}) \tag{5.1.4}$$

It is the force exerted on a point dipole, $\boldsymbol{m}$, due to its presence in a magnetic field of strength $\boldsymbol{H}$. Since a real MFM tip is not a point dipole, the force on a tip in the presence of a magnetic field can be calculated by summing equation 5.1.3 over all the dipole moments of the tip. This method of calculating the force on an MFM tip was introduced by Wadas and Grütter in 1989 [92] . (An alternative method is to sum the interaction between each dipole in the tip and each dipole in the sample [93] [94].)

Consider a MFM tip divided into tiny regions of volume $dV$ and magnetic moment $\boldsymbol{m}$. The magnetic moment of such a volume element is related to the magnetization, $\boldsymbol{M}$, by $\boldsymbol{m}{=}\boldsymbol{M}dV$. So, according to equation 5.1.3, the force, $\boldsymbol{dF}$, on this region of the

tip in the presence of a magnetic field is given by:

$$\boldsymbol{dF} \quad = \quad \mu_0 \nabla(\boldsymbol{m} \cdot \boldsymbol{H})dV \tag{5.1.5}$$

The total force on the tip is then obtained by calculating the force, $\boldsymbol{dF}$, on each volume element, $dV$, in the tip and summing them together. Letting $dV$ approach zero, we can then write the total force, $\boldsymbol{F}$, on the tip as:

$$\begin{aligned} \boldsymbol{F} \quad &= \quad \mu_0 \int_{V_{tip}} \nabla(\boldsymbol{m} \cdot \boldsymbol{H})dV \\ &= \quad \mu_0 \int_{V_{tip}} (\boldsymbol{i}\tfrac{\partial}{\partial x} + \boldsymbol{j}\tfrac{\partial}{\partial y} + \boldsymbol{k}\tfrac{\partial}{\partial z})(M_x H_x + M_y H_y + M_z H_z)dV \\ &= \quad \boldsymbol{i}F_x + \boldsymbol{j}F_y + \boldsymbol{k}F_z \end{aligned} \tag{5.1.6}$$

If the cantilever is parallel to the sample, then it is the vertical component of the force, $F_z$, which is detected. It can be seen that

$$F_z \quad = \quad \mu_0 \int_{V_{tip}} (\tfrac{\partial}{\partial z}M_x H_x + \tfrac{\partial}{\partial z}M_y H_y + \tfrac{\partial}{\partial z}M_z H_z)dV \tag{5.1.7}$$

If the tip is magnetized solely along the z-axis (so that $M_x = M_y = 0$), as is usual in MFM experiments, then the equation reduces to:

$$F_z \quad = \quad \mu_0 \int_{V_{tip}} (\tfrac{\partial}{\partial z}M_z H_z)dV \tag{5.1.8}$$

So, if the dimensions and magnetization of the tip are known and the stray field of the sample has been calculated, equation 5.1.8 can be used to calculate the z-component of the force on the tip. Equation 5.1.8 shows clearly that it is the gradient of the stray field that is the force interaction between the tip and sample if assuming the gradient of the fields are regular in the small area (around the tip). However in lift-mode the phase image maps the gradient of the magnetic force which is the 2nd order differential of the magnetic field.

Figure 5.1: The Lift Mode with the Tuning Fork Tine

## 5.1.2 The Influence of Hydrodynamic Effects in Lift Mode

The increasing hydrodynamic mass of the cantilever in the liquid as the probe approaches a surface can also be applied to the case of gas. A similar force exists but is about 1000 times less depending on the density of the medium. It can be used to explain the extra features seen in the magnetic force data. As we measure the velocity resonant frequency which is independent of Q even in viscous fluid we can hope to distinguish damping from the effect of a mass change. However we may see a change in mass due to the viscous boundary layer.

As shown in figure 5.1, the tuning fork tine is much bigger than the tip. If it gets too close to the sample in the liquid or in the gas, the tine can also sense the sample via the hydrodynamic interaction discussed in the last section.

For this interaction between the tuning fork tine and the sample, it is only a function of the average distance between them since the tine is too big to see the small features. In other words, the sample is relatively 'flat' from the perspective of the 'huge' tine. In lift mode, the tine is moved so that it follows pre-obtained topographic information in order to keep the tip-sample distance constant to get rid

of the topographic effects to the tip, however this may cause the tine to sense the change of the distance between the tine and the sample since the tine is insensitive to the small features but to the average of the big area. Thus the topography will appear in the magnetic picture if the long distance force gradients are comparable to the frequency shift due to the change in effective mass.

The cases of 1 bar helium gas and liquid helium are studied to estimate the effect by comparing the magnitude of the frequency shifts with the magnetic sample signal and system noise. The magnetic force test sample — an Iomega Zip disk typically gives a signal of about 3 mHz difference peak to peak and a noise level about 0.1mHz (equivalent to a force gradient of $1.5 \times 10^{-5}$ N/m ) difference peak to peak with a tuning fork Q of 5000.

Two different models will be applied. First of all, with the best fit coefficients b=2 in the expression 4.2.12 in the last section, giving:

$$\Delta f(z) = -\frac{c}{2}f_\infty(\frac{m'_0}{m^* + m'_0})(\frac{a}{(a + z)})^2 \qquad (5.1.9)$$

when the distance is extremely close, for example, when doing the lift mode scan, $z \ll a$.

$$\Delta f_{z21} = cf_\infty(\frac{m'_0}{m^* + m'_0})(\frac{\Delta z}{a}) \qquad (5.1.10)$$

since $m'_0 << m^*$, the long distance resonant frequency shift due to lifting from tip sample distance $z_1$ to $z_2$ – $\Delta f_{z21}$ is proportional to the additional effective mass load ($m'_0$) due to the immersion in the liquid(gas) and also proportional the distance change $\Delta z$ ($z_2 - z_1$). Since the additional effective mass load due to the immersion is

Figure 5.2: Lift Height Against Phase Shift

proportional to the density of the liquid(gas) as discussed in section 4.2.2, the higher vacuum results in less long distance force interaction.

With the experimental data for $m^*$ and $m'_0$, lifting from 3.1 $\mu m$ to 3.2$\mu m$ ($z_1 = 3.1\mu m$, $z_2 = 3.2\mu m$, the 3$\mu m$ here is for the height of the tip (estimated from a typical tip's SEM picture), 100nm is a typical lift height for lift mode and $z_2 - z_1 = 100nm$ is the typical sample roughness), the expression gives a frequency shift of 0.11Hz in superfluid helium, 0.65mHz in 1 bar of air and 0.11mHz in 1 bar of helium gas. Since the frequency shift is proportional to the density of the gas/liquid, the resonant frequency shift is 112 times bigger in superfluid helium than in the 1bar air and about 1/7 in the 1bar helium gas compared to 1bar air. Compared to the strength of the magnetic signal, the calculation implies that the lift mode might never be successful in superfluid helium unless we use a long enough tip, and there may be some problem doing lift mode in the air.

These results are surprisingly close to what we found in the experiment. A ghost image of the topograph quite often exists in the lift mode image and the situation is much better when it's in 1 bar of helium gas or in vacuum. Figure 5.2 was taken in 1 bar of air. The left half of the picture is the topographic features of the sample and the right half is the phase shift data in lift mode. The lift height was kept changing along the y axis as marked on the right side. The figure shows the lift height change

Figure 5.3: Comparison of topography and magnetic force images with and without Q control at room temperature in air

causes the losing of the topographic details may be due to the interaction resulting from an average over a bigger area.

## 5.2 Testing Sample and Q Control

As shown in figure 5.3, (a1), (a2), (b1) and (b2) are topography and magnetic force images of a $10\mu m$ by $10\mu m$ zip disk area respectively. Group (a) images were taken at Q=2000 without Q control at a scan speed of 5sec/line, correspondingly group (b)

Figure 5.4: Comparison of magnetic force line traces with and without Q control at room temperature

images were taken at Q=9200 with Q control at scan speed of 10sec/line in the same area. Apparently high Q images have some problem of scan speed and topographic image since the tuning fork are not be able to trace the topography due to the high quality factor (this can be solved by decreasing the scan speed) but they have a better contrast in the magnetic force image. This can be explained in the comparison of magnetic force line traces as shown in figure 5.4. The high Q magnetic force signal is about 5 times stronger than the one without Q control. This increase in signal is proportional to the Q.

Figure 5.5 demonstrates the topography and magnetic force images of a $10\mu m$ by $10\mu m$ zip disk area taken at 140K in vacuum. The left two images are topography and the right two are magnetic force images. Group (c) images were taken at Q=8400 without Q control at scan speed of 10sec/line and group (d) were taken at Q lowered to 1800 by Q control at scan speed 5sec/line. Similarly, the high Q one has about 5

Figure 5.5: Comparison of topography and magnetic force images with and without Q control at 140K in vacuum

Figure 5.6: Comparison of magnetic force line traces with and without Q control at 140K in vacuum

times stronger magnetic force signal as shown in figure 5.6.

A calculation of the SNR(signal-noise ratio) has been carried out with Matlab for the MFM results shown in figure 5.6. Basically a spectrum analysis was done on the line trace data, and then the signal was separated into 'signal' part and 'noise' part by setting the cut-off frequency in the spectrum. The SNR was determined by the sum of 256 lines of the integrated area of the 'signal' part and 'noise' part. The SNR for the data with Q control are 26.6dB and are 18.3 dB without Q control. The signal strengthens by 16.0dB and the noise increases by 7.8dB correspondingly by increasing Q by 4.65 times. We conclude that the signal improves more than the noise and therefore the SNR was also increased by using Q control.

In summary, topographic and lift mode magnetic force imaging have been demonstrated at room temperature in air and at low temperature in vacuum. Q control has also been performed both increasing or decreasing the Q. The magnetic signal is

proportional to the Q but high Q result in slower scan speed due to a longer settle time for topographic imaging.

The noise is not only thermal in origin but also due to electrical pickup and ground loops (which has been got rid of by using well-arranged ground).

In the following chapter, the MFM technology will be applied to study multi-layer Co/Pd thin films. It provides a good demonstration for the various applications of this microscope. The topics of noise level and sensitivity will also be discussed in more details.

# Chapter 6

# Images of Multi-layer Co/Pd Thin Film Samples

## 6.1 Overview

Magnetic thin films with perpendicular magnetic anisotropy have been extensively studied since the 1960s because of their potential application as magnetic storage devices. In the case of the garnets (the best known example) the magnetic storage integration capacity is limited by the micron size of the magnetic domains. Recently there has been a revival of interest in this area with the synthesis of new magnetic layers with very strong perpendicular anisotropy using either ordered alloys [95] or multi-layers [96]. In particular, cobalt-based multi-layers, with either palladium or platinum as the non-ferromagnetic spacer layer, have attracted much interest recently since they offer the possibility of high storage density. The magnetic properties of the multi-layers can be manipulated easily by varying the number of layers, the thickness of the layers, and the growth conditions [97].

This chapter presents a study of Co/Pd multi-layers which are known to have perpendicular anisotropy when the Co layer thickness is less than 8 Å [98]. The sample was studied by the MFM images captured with our multi-mode microscope

both at room and low temperature. The super-conducting solenoid has been used to study the sample at different magnetic fields. MFM images captured by a Digital Instrument Dimension 3100 and MFM images of ordered alloys are also presented.

## 6.2 Room temperature images with Dimension 3100

The MFM technique offers certain advantages over other techniques that have been used previously to study thin ferromagnetic films, such as Lorentz force microscopy [98], the Faraday effect [99] and Scanning Hall probe microscopy (SHPM) [100] [101]. For example, it allows imaging of films of varying thickness, it can resolve lateral features down to 10 nm in principle [102], and there are no special requirements for the substrate. SHPM has higher sensitivity to magnetic signals (about $0.3G/\sqrt{Hz}$ [100]) and the probe is non-magnetic, but it normally has a poorer spatial resolution (about 0.3 $\mu m$ [100]).

The multi-layer films were grown by Dr. Marrows at Leeds University on GaAs substrates by dc magnetron sputtering at an argon pressure of 0.4 Pa. The nominal layer thickness was 4.7 Å for the Co layers and 14.2 Å for the Pd layers. The thickness of the films was measured using a Dektak profilometer capable of measuring the step height to an accuracy of 12 Å. The measurements indicate that the thickness of the layers was approximately 30 percent greater than the nominal thickness. Therefore a thickness of 6.2 Å for the Co layer and 18.6 Å for the Pd layers were assumed. Three samples (a,b,c) were produced with 20, 200 and 300 bilayers, respectively. The MFM images shown in Fig.6.1&Fig.6.2 were obtained using a Digital Instruments Dimension 3100 AFM at room temperature with lift mode. The lift height of tip was 50nm above the sample surface.

Figure 6.1: (top)MFM image of sample (a) with 20 bilayers, phase shift scale of 2 degrees, 5 micron by 5 micron area; (bottom) Inverse FFT image after filtering the image with a high-pass filter (see figure 6.3). (Images obtained by DI3000)

Figure 6.2: (top)MFM images of sample (b) with 200 bilayers, phase shift scale of 25 degrees, 10 micron by 10 micron area; (bottom)MFM image of sample (c) with 300 bilayers, phase shift scale of 40 degrees, 10 micron by 10 micron area. (Images obtained by DI3000)

Figures 6.1 and 6.2 show the MFM images of the domain structure of the multi-layer thin films with thicknesses ranging from 20 to 300 bilayers. The sample is obviously very flat (roughness < 10nm) apart from some dirt (about 70nm high). The few blurred areas in upper image of figure 6.2 are due to regions of dirt in the topography. The domains show a distinctive pattern, commonly referred to as stripe domains. These arise because this shape minimizes the demagnetization energy of the film after the vestigial domains become unstable. The apparent random nature of their wandering is due to local variations in coercivity. That is, local pinning centres fix the domain walls at various points as they move and domain patterns form around and between them [102].

By comparing the strength of the signal (see signal amplitudes listed in table 6.1, it is clear that the magnetic signal is linearly related to the numbers of bilayers. This can be easily explained by considering the number of Co atoms per unit area.

Table 6.1: Properties of MFM images of Co/Pd multi-layers

| Sample | (a) | (b) | (c) |
|---|---|---|---|
| Bilayers | 20 | 200 | 300 |
| Signal scale (phase)(degree) | 2 | 25 | 40 |
| Signal amplitude (phase)(degree) | 2.29 | 23.7 | 40.7 |
| Scan range (micron) | 5 | 10 | 10 |
| Scan speed | 1s/line | 1s/line | 1s/line |
| Average freq. of domains (per $\mu m$) | 3.8 | 1.3 | 1.1 |
| Average repetition length ($d$ $\mu m$) | 0.2632 | 0.7692 | 0.9091 |

The images of the domains were analyzed using two dimensional Fast Fourier Transforms (FFTs). In Figure 6.3, the top left image is a 2d FFT (256 by 256) of the image at the top of figure 6.1, the top right image is the inverted FFT image filtered by the circle in the FFT image with an ideal high-pass filter (a bigger and clearer image can be seen at the bottom of figure 6.1)and the lower image is a cross

Figure 6.3: (Top left) 2d FFT of figure 6.1, (Top right) inverted FFT with high-pass filter, also in figure 6.1, (Bottom) a cross section of FFT image in direction of line shown in top left.



Figure 6.4: Illustration of the angle average calculation

section taken along the white line in the FFT image. The repetition length $d$ is determined by the highest peak of the cross section. For stripe domains which are not isotropic, an evaluation of FFT data along polar coordinates can be done using Matlab to calculate the average repetition length. This is explained in figure 6.4. The point 'o' (128.5,128.5) are the centre coordinates of all 256 by 256 points FFT data. The distance between the data and center coordinates is 'r'. However, this 'r' will be rounded to an integer thus all $256 \times 256$ data can be sorted into hundreds of rings with integral radius. Therefore the averaged FFT values on the same ring is angle averaged FFT values sorted by the ring radiuses.

The repetition length $d$ of the stripe domains measured by FFT for the various films is summarized in Table 6.1. Variations in $d$ occur because of local pinning of the domain walls. Qualitatively, the domains can be seen to reduce in size as the films decrease in thickness. This behavior is driven by the wall energy minimization process. Draaisma *et al.* [103] have modelled these processes for perpendicularly oriented multi-layer films.

## 6.3 Room temperature MFM images in variable magnetic field

Sample (c) which has 300 bilayers was selected for a low temperature experiment as it has the strongest magnetic signal among the samples.

Figure 6.5 shows MFM images of sample (c) as the external field is swept from 0T to 0.6T (passed the saturation point) and then back to 0T as specified in Table 6.2, following the magnetic hysteresis loop of the sample. Figure 6.7 shows the MFM image with reverse external magnetic field applied as detailed in Table 6.3.

Figure 6.5: MFM images(5 $\mu$m square) of sample (c) for external magnetic fields of (1)0T, (2)0.2T, (3)0.4T, (4)0.4T, (5)0.2T, (6)0T.(Specifications detailed in Table 6.2)



Figure 6.6: MFM images(5 $\mu$m square) of sample (c) for external magnetic fields of (1)0T(inverted), (2)0.2T, (3)0.2T, (4)0T(inverted).

Table 6.2: Specifications of figure 6.5

| Image | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Scan size ($\mu m$ square) | 5 | 5 | 5 | 5 | 5 | 5 |
| Lift height (nm) | 100 | 100 | 100 | 100 | 100 | 100 |
| Temperature (K) | 300 | 299.5 | 299.4 | 299.3 | 299.3 | 299.3 |
| External field (T) | 0 | 0.2 | 0.4 | 0.4 | 0.2 | 0 |
| Signal scale (software unit) | 10 | 10 | 10 | 10 | 15 | 10 |
| Topography scale (nm) | 10 | 10 | 10 | 10 | 10 | 10 |
| Pressure (mbar) | 5.0 | 4.6 | 4.5 | 4.6 | 4.5 | 4.6 |
| Plane fit | off | on | off | off | off | off |
| Q (k) | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 |
| Scan speed (second/line) | 5 | 5 | 5 | 5 | 5 | 5 |

Table 6.3: Specifications of figure 6.7

| Image | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Scan size ($\mu m$ square) | 5 | 5 | 5 | 5 | 5 | 5 |
| Lift height (nm) | 100 | 100 | 100 | 100 | 100 | 100 |
| Temperature (k) | 298.2 | 298.2 | 298.2 | 298.3 | 298.3 | 298.4 |
| External field (T) | 0 | -0.2 | -0.4 | -0.4 | -0.2 | 0 |
| Signal scale (software unit) | 10 | 20 | 20 | 20 | 25 | 7 |
| Topography scale (nm) | 40 | 40 | 40 | 40 | 40 | 40 |
| Pressure (mbar) | 3.4 | 3.4 | 3.4 | 3.5 | 3.5 | 3.5 |
| Plane fit | off | off | off | off | off | off |
| Q | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 |
| Scan speed (second/line) | 5 | 5 | 5 | 5 | 5 | 5 |

In figure 6.5, the images (1) and (6) look very different from (2) and (5). This is because the tip magnetic moment has been flipped between 0 to 0.2T. As shown in figure 6.6, the left two images are inverted images from original data which agree with the two images on the right. Since it is much easier to study the images without the effect of the reversal of the tip magnetisation, the rest of the images were all taken in negative magnetic fields.

Previous MFM studies of this material were unable to reach saturation fields [104] (-0.3T to 0.3T by using permanent magnet, the saturation point is about 0.55T [104]).

Figure 6.7: MFM images(5$\mu$m square) of sample (c) for external magnetic fields of (1)0T, (2)-0.2T, (3)-0.4T, (4)-0.4T, (5)-0.2T, (6)0T.(Specifications detailed in Table 6.3)

However our super-conducting solenoid is able to apply $\pm 12$T to the sample volume. When the external magnetic field was swept to 0.4T, the stripe domains broke up and formed bubble domains. This is well known in bubble-type materials [105] and it is likely that a similar process has occurred here. As far as we know, it is the first observation of bubble domains with MFM for this material at room and low temperature.

In acquiring these images it is obviously necessary to bring the tip close enough to the surface so that it interacts with the stray field of the sample. In this sense MFM is an invasive technique and raises the question of whether the tip creates a local field which modifies the sample stray field and affects the image. The tip-sample interaction becomes increasingly strong as their relative separation is reduced. However images taken at 0T were very similar indicating that the stray field of the tip was insufficient to cause any modification to the domain structure of the multi-layer

films [102]. In this case, there are no sudden shifts in the MFM images taken by the DI Dimension 3100. Nevertheless in higher external magnetic fields the tip was seen to cause local magnetization changes in the sample such as domain switching and deformation of domain walls. Such sudden domain shifting can be found in MFM images taken in external fields higher than 0.4T and the stronger the field the higher the probability of deformation. It was reported by Klein *et al.* [106] earlier with a MFM image of a Fe/Pd sample obtained at 0.467T. This interaction would have been expected to occur during the topographic scan since the tip-sample distance is much smaller than during the magnetic force scan (50-250nm). Unfortunately this can not be prevented. In lift mode, it is necessary to determine the topograph of the sample before the MFM scan, and this will necessarily bring the tip much closer to the sample.

As the applied field is increased from zero, the domains grow and the magnetization increases almost linearly to saturation. Since the magnetization of both the sample and the tip was greatly increased, the magnetic signal was greatly increased in the magnetic fields. Therefore the signal-noise ratio was enhanced at higher magnetic field, as clearly demonstrated in the MFM images.

At saturation the film is completely magnetized in the direction of the applied field, except for small, vestigial, domains. These are closed wall domains with a vanishingly small dimension held by strong local pinning forces [97]. They are also important in the nucleation of domains when the applied field decreased from saturation. As the applied field is reduced the vestigial domains remain stable until a critical field strength, the nucleation field, is reached. They then become unstable and form into stripe domains [105]. The driving force behind this process is the minimization of

the demagnetization energy in a uniformly magnetized thin film. However there are still some random factors so that the patterns are entirely different as compared with those taken before saturation.

## 6.4  Low temperature MFM images in variable magnetic field

The series of MFM images shown in figure 6.8 and 6.9 were taken at low temperature (113K). The scan size was increased to 10 $\mu m$ and the images were taken every 0.1T following the magnetic hysteresis loop to study it more carefully. There is a clear vertical line in most of the low temperature MFM images which was unfortunately caused by a fault of the scan tube at low temperature. The fault only happens at low temperature, at certain pressures, and is caused by a damaged +X electrode of the scan tube. Voltages applied to the electrode can cause a small discharge above a certain voltage and thus cause a slight instability and non-linearity in the height control. As shown in the traces/retraces in figure 6.10, this flaw did not affect the topographic image much but it is significant in the MFM image due to the greater signal sensitivity. This artifact is also confirmed in image 6 of figure 6.9. The stripe shifted as the offset shifted. Replacing the scan tube will solve this problem.

Stripe domains break up into bubble domains when the external field is swept up to 0.4T and the process looks very similar to the process at room temperature. However the bubble domains at low temperature are somehow different from the ones at room temperature. They seem to form in pairs instead of simple dots and are aligned in one direction as shown in figure 6.13. Since the tuning fork MFM is actually imaging the second differential of the magnetic field with respect to height, these paired dots

Figure 6.8: MFM images(10$\mu$m square) of sample (c) for external magnetic fields of (1)0T, (2)-0.1T, (3)-0.2T, (4)-0.3T, (5)-0.4T, (6)-0.5T.(Specifications detailed in Table 6.4)



Figure 6.9: MFM images(10$\mu$m square) of sample (c) for external magnetic fields of (1)-0.5T, (2)-0.4T, (3)-0.3T, (4)-0.2T, (5)-0.1T, (6)0T.(Specifications detailed in Table 6.5)

Figure 6.10: Topograph (Top left), MFM image (Top right) and their cross sections (upper: topographic traces; lower: magnetic force traces; blue: trace, red: retrace) along the write line of the VSM sample (not plane-fitted, same data as image (1) of figure 6.8)

Table 6.4: Specifications of figure 6.8

| Image | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Scan size ($\mu m$ square) | 10 | 10 | 10 | 10 | 10 | 10 |
| Lift height (nm) | 100 | 100 | 100 | 100 | 100 | 100 |
| Temperature (K) | 113.39 | 113.03 | 113.15 | 113.08 | 113.07 | 113.09 |
| External field (T) | 0 | -0.1 | -0.2 | -0.3 | -0.4 | -0.5 |
| Signal scale (software unit) | 25 | 40 | 50 | 40 | 40 | 10 |
| Topography scale (nm) | 25 | 30 | 30 | 30 | 30 | 30 |
| Pressure (mbar) | 2.5 | 1.7 | 1.7 | 1.8 | 1.7 | 1.7 |
| Plane fit | on | on | on | on | on | off |
| Q (k) | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Scan speed (second/line) | 8 | 8 | 8 | 8 | 8 | 8 |

Table 6.5: Specifications of figure 6.9

| Image | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Scan size ($\mu m$ square) | 10 | 10 | 10 | 10 | 10 | 10 |
| Lift height (nm) | 100 | 100 | 100 | 100 | 100 | 100 |
| Temperature (K) | 113.09 | 113.08 | 113.08 | 113.08 | 116.72 | 116.88 |
| External field (T) | 0 | -0.1 | -0.2 | -0.3 | -0.4 | -0.5 |
| Signal scale (software unit) | 10 | 25 | 25 | 25 | 25 | 10 |
| Topography scale (nm) | 30 | 30 | 30 | 30 | 30 | 30 |
| Pressure (mbar) | 1.7 | 1.7 | 1.7 | 1.8 | 0 | 0 |
| Plane fit | off | off | off | off | on | on |
| Q (k) | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Scan speed (second/line) | 8 | 8 | 8 | 8 | 8 | 8 |

could actually be short rods. As shown in figure 6.11, the left image is the magnetic field distribution and the image on the right is the simulation of the left one's MFM image. It is clear that at the edge of the domain the signal has been emphasized. The alignment of the 'dots' or 'rods' is probably related to the interactions between tip and sample since all of them were aligned to the slow scan direction. Viret *et al.* [107] show a similar MFM image in which the domains lie in parallel stripes, but it was not recovered once the sample had been saturated by application of a perpendicular field. A theoretical model to explain this feature has not yet been found apart from a

Figure 6.11: Computer simulation of the MFM image. Left:Magnetic field distribution; right:MFM image of the left image in theory [108]

reasonable guess that it is because the plane of the sample is not quite perpendicular to the field.

When the sweep was decreased from saturation, it is quite dramatic to see the spirals in figure 6.9. The images are also very different in corresponding high fields (-0.5T,-0.4T,-0.3T) between figure 6.8 and 6.9. The perpendicular magnetization curve is able to explain it well. Figure 6.12 (a) is a perpendicular magnetization curve of a similar multi-layer thin film sample presented by Barnes *et al.* [102] measured by VSM (vibrating sample magnetometry). The shape of the hysteresis loop is very similar to our samples presented in reference [104]. Figure 6.12 (b) shows a suggested schematic magnetization curve for the multi-layer films (other reports [106][109][110][111][112] also find this hysteresis shape). The horizontal line at A and B as the external magnetic field is reduced from saturation shows that the magnetization remains too high to allow the appearance of the bubble domains. However it is followed by a very steep drop of magnetization which suddenly bypasses the 'bubble domain' region and leads to the creation of spiral domains. Due to the sudden change of the magnetization at this point, only very few of bubble domains could have appeared and

Figure 6.12: (a)Perpendicular magnetization curve for a 100 bilayer Co/Pd film (from [102]) (b)Schematic magnetization curve.

it is not possible for them to connect to each other due to the large spacing. Therefore the domains were able to expeditiously grow as the field was reduced. The sudden appearance of the spirals is probably caused by the curved domains trapped by itself as in the left spiral in image(2) of figure 6.9. This special phenomenon can not be seen at room temperature (figure 6.5 and 6.5) might be due to the differences in the hysteresis loop. It could be confirmed by a VSM (vibrating sample magnetometry) measurement at low temperature. When the magnetization was reduced further, the spiral grew at both ends, then forked (image(3)) and finally joined with each other (image(4)(5)(6)).

To summarize, the differences in MFM images at various magnetic fields between room and low temperature are probably caused by the differences in the hysteresis loop.

The movement of the bubble domains at fields close to saturation is another interesting topic. By scanning the same area under the same conditions for several times, there is a kind of 'movement' which is very small($< 0.5 \mu m$) (shown in Figure 6.13)

Figure 6.13: AFM (left 2) and MFM (right 2) images(10 $\mu m$ square) of sample (c) for external Magnetic fields of -0.4T.

and is probably due to the magnetic moment on the tip exerting a force in excess of the pinning force on the domain. As mentioned earlier, this probably occurs during the topographic scan when the tip is in close proximity to the sample. However the possibility that the domains move with time, independent of the tip position cannot be ruled out.

Some other MFM pictures of this sample taken at 123K can be seen in Figure 6.14 and with its specification listed in Table 6.6. They were also taken along the hysteresis loop and came back after passing the saturation point. Similar 'paired dots' or 'short rods' can be seen in image 3, but no 'bubble' domains can be obtained when the magnetic field is decreased from saturation either. These MFM images also show a

Figure 6.14: MFM images($10\mu$m square) of sample (c) for external magnetic fields of (1)0.2T, (2)0.4T, (3)0.5T, (4)0.4T(came back from saturation), (5)0.3T, (6)0.2T.(Specifications detailed in Table 6.6)

lag in the magnetization when it came back from the saturation. At 0.4T, instead of spirals it formed similar domain stripes. These observations lend support for the theoretical descriptions discussed above.

Table 6.6: Specifications of figure 6.14

| Image | 1 | 2 | 3 | 4 | 5 | 6 |
|---|---|---|---|---|---|---|
| Scan size ($\mu m$ square) | 10 | 10 | 10 | 10 | 10 | 10 |
| Lift height (nm) | 100 | 150 | 150 | 150 | 150 | 150 |
| Temperature (K) | 122.89 | 123.39 | 125.00 | 124.64 | 124.09 | 124.00 |
| External field (T) | 0.2 | 0.4 | 0.5 | 0.4 | 0.3 | 0.2 |
| Signal scale (software unit) | 40 | 15 | 15 | 25 | 25 | 25 |
| Topography scale (nm) | 30 | 30 | 30 | 30 | 30 | 30 |
| Pressure (mbar) | 0 | 0 | 0 | 0 | 0 | 0 |
| Plane fit | off | off | off | off | off | off |
| Q (k) | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Scan speed (second/line) | 8 | 8 | 8 | 8 | 8 | 8 |

## 6.5   The theory of the domain repetition length of the Co/Pd thin-films

The study of the domain repetition length is based on a model by Draaisma and de Jonge[103]. Barnes *et al.* compared this model to MFM images of similar Co/Pd multi-layer samples by varying the number of bilayers without the presence of an applied field [102], and Rushforth *et al.* compared this model to room temperature MFM images (Co/Pd samples) using a permanent magnet to provide up to 0.3T [104]. The way that they defined the 0T values to analyze the MFM image data to produce 'bubble' domains are not quite convincing since any domains are able to break to 'bubbles' by increasing the zero field values. However there is only relative magnetic field strength during the scan and our 0.3T MFM images do not show evidence of break up compared to the lower field images.

Klein *et al.* [106] reported magnetization processes of Fe/Pd thin films with MFM at room temperature. They used a homemade MFM instrument to image the sample up to the saturation field with an electromagnet (around 0.5T). Their results were compared with the model of Kooy and Enz [99]. Viret *et al.* [107] also studied Fe/Pd alloy with MFM but varied the temperature. They plotted the domain wall resistance versus temperature. However, the study of Co/Pd multi-layer thin film along the hysteresis loop with MFM at low temperature has not yet been reported so far.

The model of Draaismia & de Jonge's model considers a system of stripe domains with alternating orientation perpendicular to the plane of the multi-layers. The repetition length $d$ is defined as the total width of two adjacent stripes oriented in opposite directions. The other parameters in the model are the thickness of the layers $t$, the

number of bilayers $N$, the external field $H$, the magnetization of the ferromagnetic material $M$, and a material dependent characteristic length, $\tau = \sigma_w/(0.5\mu_0 M_s^2)$, where $\sigma_w$ is the domain wall energy per unit area and $M_s$ is the saturation magnetization per unit volume of the ferromagnetic material.

According to the model, the observed domain repetition length will allow the system to attain the minimal total magnetic energy of the system. The magnetic energy of the domain structure contains three terms: the magneto-static or demagnetizing energy $E_d$, originating from the poles at the interfaces between the ferromagnetic and non magnetic layers, the wall energy $E_w$ of the domain walls between neighboring domains, and the field energy $E_h$ arising from the interaction of the magnetization with the applied magnetic field. All the energies are calculated per unit volume of the ferromagnetic material and will be normalized to the maximum magneto-static energy $\frac{1}{2}\mu_0 M_s^2$.

The magneto-static energy can be written as

$$e_d = \frac{E_d}{\frac{1}{2}\mu_0 M_s^2} = m^2 + \sum_{n=1}^{\infty} \frac{4}{(n\pi)^3} \frac{d}{t} \times sin^2[\frac{1}{2}\pi n(m+1)]f_n(d), \qquad (6.5.1)$$

where $m = M/M_s$ is normalized magnetization of the film, and with

$$f_n(d) = 1-exp\left(-2\pi n\frac{t}{d}\right)+\frac{sinh^2[\pi n(t/d)]}{sinh^2[\pi n(D/d)]}\times\left\{\frac{1}{N}\left[1-exp(-2\pi n\frac{ND}{d})\right]-\left[1-exp(-2\pi n\frac{D}{d})\right]\right\}$$

$$(6.5.2)$$

where $D = t + s$ ($s$ is the thickness of the spacer layer) is the total thickness of a single bilayer.

Assuming a specific wall energy of $\sigma_w$ per unit area of a domain wall, we can write

$$e_w = \frac{E_w}{\frac{1}{2}\mu_0 M_s^2} = \frac{2\sigma_w}{d(\frac{1}{2}\mu_0 M_s^2)} = \frac{2\tau}{d}, \tag{6.5.3}$$

in which $\tau = \sigma_w / \frac{1}{2}\mu_0 M_s^2$ is a length characteristic for the ferromagnetic material under consideration. $\tau$ can assume values ranging from a few nanometers to many micrometers.

Finally, we have

$$e_h = \frac{E_h}{\frac{1}{2}\mu_0 M_s^2} = \frac{-\mu_0 HM}{\frac{1}{2}\mu_0 M_s^2} = -2hm, \tag{6.5.4}$$

where $h = H/M_s$ is the normalized field applied perpendicular to the film.

The calculation procedure is to choose a magnetization $m$, calculate repetition length $d$ which minimizes the system total energy, and then find a magnetic field $h$ which the system will stay at. This is done by programming in Matlab.

To verify the Matlab program which will carry out the curves to fit our data, figure 6.15 was plotted to compare with the results in Draaismia's paper [103]. The agreements of the figures demonstrated that the Matlab program was correct.

To measure the repetition length of our low temperature MFM images we use numerical analysis of the images. But in some of the images the stripes tend to align in a preferred direction, most of them align to the slow scan direction although the sample was saturated and the change of the external magnetic field was taken with the scan tip withdrawn. As mentioned before, this alignment of the domains might be caused by the tilt of the sample. However, these domain stripes differences in direction need to be averaged. Thus a mathematical averaging method has to be performed as well as simple 2d FFTs.

Therefore we use 2d FFTs to process the images (with a Hanning window to

Figure 6.15: (a) Perpendicular magnetization curves for multilayers with 25 bilayers(N=25), a nonmagnetic layer thickness $s/\tau$ =5, and different ferromagnetic layer thicknesses $t/\tau$; (b) domain repetition length $d/\tau$ corresponding to the magnetization curves.

minimize the effects of the finite image size) and then use Matlab to perform angular averages to the FFT data as introduced earlier. These angle-averaged data shown in figure 6.16 corresponds to the MFM images in figure 6.8 and 6.9.

The average repetition length $d$ was finally determined by the peaks in the angle-averaged FFT data. Most figures have a broad peak, these were due to differences of stripe alignment in different directions. Table 6.7 listed estimated average repetition lengths for the MFM images.

Figure 6.17 shows the domain repetition lengths along the hysteresis loop at low temperatures listed in Table 6.7 and a theoretical fit with $\tau = 25nm$. The parameter values $t = 6.2\mathring{A}$, $s = 18.6\mathring{A}$ and saturation external magnetic field $H_s = 0.55T$ measured by Rushforth $et$ $al.$ [104]. The error bar was determined by varying $\pm 1$ of counted domain numbers in the $10\mu m$ by $10\mu m$ MFM image. The magnetization $m$ was determined by a theoretical m vs h plot as shown in figure 6.18. The plot when magnetic field is increasing to the saturation point (black dots in the figure) agrees

Figure 6.16: Angle-averaged FFT data for MFM images in figure 6.8 and 6.9. The x coordinates are the radius of the 2d FFTs data rings (see figure 6.4), and the y coordinates are the averaged values

Table 6.7: The repetition length data of strip domains determined by angle-averaged FFT data in figure 6.16

| Figure | Field(T) | Average domain stripes number | Average repetition length($\mu$m) |
|--------|----------|-------------------------------|-----------------------------------|
| 1 | 0 | 10 | 1 |
| 2 | 0.1 | 9 | 1.111 |
| 3 | 0.2 | 9 | 1.111 |
| 4 | 0.3 | 7 | 1.429 |
| 5 | 0.4 | 6 | 1.667 |
| 6 | 0.5 | 4 | 2.5 |
| 7 | 0.5 | 1 | 10 |
| 8 | 0.4 | 4.5 | 2.222 |
| 9 | 0.3 | 6.5 | 1.538 |
| 10 | 0.2 | 9 | 1.111 |
| 11 | 0.1 | 9.5 | 1.052 |
| 12 | 0 | 11 | 0.909 |

with the theory very well but the retrace plot (red triangles in the figure) is far from the theory. This can be explained by the hysteresis loop in figure 6.12 as discussed above. The reason is that the plot at high magnetic field stays at higher magnetization which is not correctly calculated, and the retrace plot at low magnetic field comes back to the theoretical track also follows with the hysteresis loop. Therefore the theory and experiment still agree with each other.

One set of the room temperature MFM images were also processed in the same way. A similar result is shown in figure 6.19. The theory plot still fit the data well. Since the images were taken in a smaller area ($5\mu$ by $5\mu$m), there are much larger error bars. The retrace plot is slightly above the trace plot and the theory plot as the reason just discussed.

Draaismia's model gives a good fit to our data despite being based on certain assumptions which are not strictly valid for our sample. The model assumes that only the cobalt layer is magnetic. However in reality the palladium layers are also

Figure 6.17: Domain repetition lengths plots of low temperature MFM images along the hysteresis loop. The line represent the theoretical fit for $\tau$=25nm

Figure 6.18: Theory plot of magnetic field $h$ vs magnetization $m$.

polarized. The model also assumes that the domains have perfect perpendicular anisotropy, that they are arranged in infinitely long parallel stripes, and that the domain walls can move freely due to the absence of any pinning sites. Our samples, produced by sputtering, are unlikely to have perfect perpendicular anisotropy, and the random pattern of domain indicates that there are many pinning sites present.

A further study on the temperature dependance of this material will be recommended. However, at least, the hysteresis loop are possibly temperature dependant which may have caused the spiral images at low temperature.

## 6.6   Noise Level

The sensitivity limit of the magnetic force microscopy is determined by the noise level. Our MFM noise level was measured by looking at the peak-to-peak variation

Figure 6.19: Domain repetition lengths plots of room temperature MFM images along the hysteresis loop. The line represent the theoretical fit for $\tau$=25nm.

Table 6.8: The repetition length data of strip domains determined by angle-averaged FFT data in figure 6.5

| Figure | Field(T) | Average domain stripes number | Average repetition length($\mu$m) |
|--------|----------|-------------------------------|-----------------------------------|
| 1 | 0 | 5 | 1.0 |
| 3 | 0.2 | 4 | 1.25 |
| 5 | 0.4 | 3 | 1.667 |
| 8 | 0.4 | 2.5 | 2.0 |
| 10 | 0.2 | 3 | 1.667 |
| 12 | 0 | 4 | 2.0 |

of the 'Y' output of the lock-in amplifier (the change in phase can be regarded as the change in 'Y' since they have a linear relationship for small phase shifts when the lock-in amplifier is in phase with the tuning fork oscillation). Then, by varying the frequency slightly, dY/df and dY/dPhase can be determined, and thus the voltage noise can be converted in terms of frequency or phase. But the value of dY/df and dY/dPhase will be different for different values of Q and has to be remeasured each time.

The system noise comes from many sources which includes self noise generated by the scan tube, ambient noise that exists in the sample space, and vibrational noise which is a function of the propagation paths and configuration of the acoustic system. These noise levels have been carefully reduced by the design of the anti-vibration system. The most serious background noise came from the electronic circuit of the detection system:

The frequency synthesizer has a few mV of noise, so by keeping the signal large and then by attenuating it by 1/10 or even 1/100 will minimize the noise level. Especially for low drive amplitudes, attenuation is very necessary.

The capacitance compensation circuit for cancelling the fork's stray capacitance can pick up a lot of noise, so it has to be well shielded. Integrating the capacitance

compensation circuit and the current amplifier into the same shielded box helps to decrease the noise level. This has been done for further research.

The tuning fork probe generates signals which are only a few nano-amps through a two metres long coaxial cable and then to be amplified. It is very likely to pick up some noise from the cryostat if the cryostat is not correctly grounded. However a pre-amplifier in the sample space close to the probe is not practical due to difficulties of lack of space and heating at low temperature and the complexity of the design.

The commercial current pre-amplifier seems much noisier than our home-made amplifier. Another serious problem is that the ground connected to our equipments is not 'clean'. It is always 'polluted' by other switch mode power supply noise which has a strong Fourier component around 30kHz. It affects the MFM data and also causes ripples in SCM data, but it is too small to affect the topographic data. Therefore the MFM images taken outside 'working hours' are much better than those taken during the normal working day. An example of this can be seen in image 2 of figure 6.7, the bottom half image shows less noise as it was acquired out of office hours. Image 3, shown in Figure 6.9, has lower noise in the middle section which coincides with the morning tea break.

The value of AC gain should be set as high as possible to make good use of the lock-in amplifier's ADC range. This results in 'less' digitization of the signal and lower noise. A value of 40 or 50 dB is normally applied. Furthermore the longer the lock-in amplifier and filter's time constants the lower the noise level it will be. However, an unnecessarily high time constant will slow down the scan speed. A typical value of 20ms to 25ms time constant was set to meet the basic requirement (for scan speed 6s/line).

Figure 6.20: Fe/Pd alloy sample, $3\mu m$ by $3\mu m$ at 190K, (left) 0.15T; (middle) 0.2T; (right) 0.3T

Noise from the magnet power supply dominates in the low temperature MFM images shown in figure 6.8 and 6.9. However it can be turned off during the scan and the magnet remains running in persistent mode. This cuts the total noise level in half. Furthermore, by using a homemade current pre-amplifier (with built-in capacitance compensation circuit) the noise level can be reduced by a further 30%. Scanning in off-peak time is another way to reduce the noise. By applying the last three methods together, the noise level was cut by a factor of 4. As shown in figure 6.20, these MFM images are taken with a scan size of $3\mu m$ by $3\mu m$ from Fe/Pd alloy thin film sample (sample 1724b, about 50nm thickness, also provided by Dr. Marrows at Leeds University) at 190K. Since the signal is much weaker than the last sample, the last three methods were applied together to obtain these images.

The Q control provides another way to improve the signal/noise ratio. Unfortunately high Q will result in much slower scan and also introduce oscillations in topographic scan which will add some artifacts into MFM imaging. Longer scan times also increase the problem of temperature drifting. Thus it is not advantageous to increase the Q to values above 10,000.

Recently the home made current pre-amplifier with the built-in capacitance compensation circuit was built, the signal source and detection circuit are using the same ground source which is insulated from the cryostat and rest of the instruments such as the magnet controller. Therefore the noise level has been further decreased to the limitation of the instrument. Namely the resolution of the system is brought to 0.01 degree phase shift for the lift mode detection. For a tuning fork with a Q of 3000 and resonant frequency of 32kHz, 0.01 degree phase shift is equivalent to about 1mHz shift at the resonant frequency. Typical force gradients detected by MFM are in the range $10^{-4}$ N/m to $10^{-2}$ N/m, whereas for a typical tuning fork with c = 8.7kN/m and $f_0$ = 32kHz the corresponding frequency shifts are 0.2mHz to 20mHz.

## 6.7   Summary

This chapter demonstrated the capability of the microscope working at room and low temperatures and in different magnetic fields with lift-mode MFM images of the thin film samples. In the end of the chapter, the noise level has been fully discussed and the resolution of the microscope was greatly improved under certain conditions.

It also described a study of the magnetic domain structure of multi-layer Co/Pd thin films. MFM images along the hysteresis loop were taken and numerically analyzed. The theoretical model has also been found to fit the experiment data well. Most interesting phenomenons seen during the experiment were explained, such as 'bubble domains', reversal of the tip magnetisation, sudden domain shifting, scan tube fault, domain alignment and spiral domains etc. However it is only one of the possible applications with this multi-mode microscope. Scanning capacitance microscopy is described in the next chapter.

# Chapter 7

# Tuning Fork Scanning Capacitance Microscopy in Superfluid Helium

## 7.1 Basic Operation Principles of SCM

The SCM consists of a conductive probe tip and a high sensitivity capacitance sensor in addition to the normal AFM components. The SCM measures dielectric coefficient changes of the material (normally semiconductors). In a contact mode SCM [113], the tip in contact with an oxidised silicon sample, forms a MOS (Metal Oxide Semiconductor) capacitor. The MOS capacitor consists of two capacitors in series: one formed by the insulating oxide layer and the other formed by the active depletion layer near the oxide/silicon interface. Figure 7.1 depicts the MOS capacitor formed by the tip and the semiconductor. The total capacitance is determined by the oxide thickness and the thickness of the depletion layer which, in turn, depends on the carrier concentration in the silicon substrate and the applied DC voltage between the tip and the semiconductor. The change of capacitance due to an alternating electric field is illustrated in Figure 7.2.

In SCM, it is usually changes in capacitance (rather than absolute capacitance)

Figure 7.1: Capacitance Detection (from [27])



Figure 7.2: Changing of Capacitance Due to Alternating Electric field(from [27])

that are measured although there are a few microscopes which can measure capacitance directly with reduced sensitivity [114]. This is necessary because the capacitance between, for example, the tip of a one micron diameter probe and a sample surface one micron from the probe-tip is of the order of $10^{-17}$ F ($10^{-5}$ pF), and it is a modulation in this capacitance which must be measured in the presence of the pico-farad of capacitance of the probe and the sensor circuit. The tip-sample capacitance is only a tiny fraction of the overall capacitance in the system. Therefore, it is necessary to modulate a bias voltage applied to the sample (C=Q/V) or the tip-sample distance (C=$\varepsilon$A/d) and detect variations in the amplitude of the resulting capacitance modulation at that frequency.

The changes in capacitance are detected by measuring the resulting changes in resonant frequency of a 100MHz LC circuit, of which the tip-sample capacitance is a component. Our design is based on the work of Bugg and King [18] [115]. This method is conceptually similar to that of C. C. Williams and co-workers [113]. However the detection circuit of Bugg and King is a lumped component circuit whereas Williams design uses a 900 MHz transmission line resonator.

## 7.2  Capacitance modulation

In general, for the study of semiconductors, a bias voltage between the sample and ground is modulated at a frequency in the kilohertz range. The free carriers in the semiconductor beneath the tip (which is near ground potential) are alternately attracted and repelled by the tip due to the alternating electric field. Therefore the tip-sample capacitance is modulated at the same frequency due to the motion of

Figure 7.3: N type CV curve (from [27])

charge under the tip. The amplitude of these modulations (measured with a lock-in amplifier) is dependent on the carrier concentration directly below the tip (See Figure 7.3 and Figure 7.4). By measuring these changes in capacitance as a function of tip position, an image is built up. To obtain quantitative information on the sample carrier concentration, the measurements must be combined with a suitable model [116]. The alternating depletion and accumulation of carriers under the tip can be modelled as an equivalent moving capacitor plate (Figure 7.3). Sophisticated models now exist which can successfully convert dC/dV information into carrier/dopant concentrations [116].

The SCM measures the movement of carriers, which translates into a stronger signal for carrier concentrations around doped Si and a thin oxide layer. The detected SCM signal is, in fact, $\Delta C / \Delta V$; i.e., the change in capacitance (due to depletion or

Figure 7.4: High/Low doping

accumulation) for a unit change in the applied voltage. Because we are applying an AC voltage waveform, dV may be thought of as the peak-to-peak or RMS voltage applied. The dC is related to the change in depletion depth in the semiconductor under the probe.

Mapping the changes in $\Delta C$ at a fixed $\Delta V$ is known as $\Delta C$ mode. It is also possible to use a feedback loop to keep $\Delta C$ constant. The data then consists of the amplitude of bias modulation needed to keep $\Delta C$ constant. This is known as $\Delta V$ mode.

The DC bias to the sample may also be adjusted, thereby moving the point at which the AC bias is applied. This is known as SCS (Scanning Capacitance Spectroscopy) and is a powerful tool for the characterisation of semiconductor devices [117].

An alternative to bias modulation is to modulate the tip-sample distance by placing the probe on the end of a dither piezo. Crudely, from the relation C=$\varepsilon$A/d , this can be used to image variations in the sample dielectric constant $\varepsilon$ or topographic

height variations ($d \equiv$ tip-sample distance). In fact, the early SCM of Bugg and King used this type of modulation [18]. In this way both conductors and insulating dielectrics can be imaged. However it is difficult to deconvolute the changes in topography from the changes in dielectric constant.

## 7.3   Capacitance detection

The capacitance detection principles applied in microscopes can be divided into three groups. The most popular is based on the RCA CED VideoDisc pickup [16]. It contains a stripline resonator, coupled to an oscillator oscillating at 915MHz, which is a frequency on the flank of the resonant curve of the resonator. The frequency of resonance is fine tuned by the tip/sample capacitance, modulating the rf voltage in the resonator [118] [113] [119]. A similar solution was employed at 90MHz by Bugg&King [18] in 1988. Recently Tran et al. [120] demonstrated a $10^{-21}$F resolution (in 1Hz bandwidth) capacitance sensor for SCM in 2001 and Ferrar *et al.* [121] built a SCM based on a coaxial resonator using the same principle (see figure 7.5). The key element of this design is a $\lambda/2$ cable resonator working at 1GHz: one end of the cable terminates with a electrochemically etched tungsten tip while the other end has two variable capacitances in parallel. The above designs detect minute changes of resonant frequency transformed into acceptably large voltage variations by the high Q of a resonant circuit. The higher resonant frequency of the resonant circuit results in higher resolution in capacitance detection. These designs are referred to as RF SCM methods.

Figure 7.5: Ferrar *et al.*'s design: Coaxial cable resonator with the varicaps at one end (T2) and the tip at the other end (T1). (from[121])

The low-frequency SCM uses phase-sensitive measurement of the AC current flowing through the tip/sample capacitance at 2MHz. It offers the possibility of separating the real and imaginary (loss) components of the capacitance [122]. Thus in some cases the imaging of dielectric losses is possible. The probe contains a small electrode placed in close proximity to a conductor, which may be a conducting surface or a base covered by a thin insulator. The two conductors form a capacitor whose capacitance is affected by their separation and by the properties of the material filling the space between them. By scanning, information on locally varying separation, or dielectric constant, or their combination may be obtained. Following this approach, a SCM combined with a STM was developed by Lànyi *et al.* in 1994 [114] [122]. However it requires a more complex tip design and the presence of a preamplifier close to the tip which can be difficult to arrange in a low temperature environment.

A third method is based on measuring the electrostatic force between the sample and a cantilever, to which a DC or AC voltage is connected [123]. This method cannot be performed with AFM in a single scan and would be difficult to operate in superfluid helium due to hydro-dynamic effects on the cantilever.

There are several other methods which can be used to measure capacitance, based

on bridge methods [124] and charge measurement [125]. In a SCM, the modulation in the probe capacitance must be measured both as accurately and as rapidly as possible. Therefore bridge methods, which are more suited to slow, absolute measurements of capacitance, have not been investigated. The charge measurement was proven not to be sensitive enough as described in Bugg's thesis [115].

Since our design is aiming to make a low temperature SCM which is required to work in superfluid helium and compact enough to be contained in the sample space, the tuned filter design by Bugg *et al.* [18] was chosen. This design has been shown to work with a resolution of $10^{-18}$F at a frequency of 80MHz. The noise level was set by the frequency noise in the oscillator. Since a better oscillator and higher modulation frequency were chosen as compared to Bugg and King, it's believed we will have an improved sensitivity.

The technique is illustrated schematically in Figure 7.6. The tuned filter method also has the quite important advantages that, firstly, the amplitude of the RF potential between the probe-tip and the surface can easily be controlled and, secondly, variations in the RF amplitude do not affect the performance of the capacitance sensor to first order as it is operating at the resonant frequency whereas for the slope detection (e.g. DI's capacitance detection) amplitude noise has a large impact on the capacitance signal. This allows us to use low voltages ($\sim$1mV) between the tip and sample to measure the capacitance change.

## 7.4   Overview of the Nottingham SCM System

The block diagram of capacitance detection is shown in Figure 7.7. The frequency of the RF (radio frequency) signal from the RF generator is about 130MHz, in order

Figure 7.6: Schematic diagrams of capacitance sensor of LC tuned filter (from [115])



**Block Diagram of Capacitance Detection**

Figure 7.7: Block Diagram of Capacitance Detection

to avoid the commercial FM band. The signal is split in two, one part is used as the reference signal for the RF mixers, and the other is attenuated to about 10mV and used as the input signal. The reason for using a small RF signal is that on the one hand, only a small voltage can be applied to tip to avoid the danger of damaging the sample, and calculation shows the voltage across the tip-sample junction is related to the input signal times the Q factor of the RF resonance (See Appendix A.4). On the other hand, the RF mixer requires a strong reference signal.

The tip is connected to the capacitance sensor by silver epoxy and a gold ribbon as shown in figure 3.7. The capacitance sensor consists of an LC filter (see Figure 7.6), the tip-sample capacitance and the central inductor make up a LC resonant circuit, the signal is injected into the LC circuit via a mutual inductance. The output of the sensor is coupled to the detector by another mutual inductance. The output signal is pre-amplified and sent to a mixer, which only detects the amplitude of the RF signal at the phase of the reference.

Changes in the tip-sample capacitance, alter the resonant frequency of the LC circuit and so modulate the amplitude of the RF signal. Modulations of the amplitude at the frequency of the tip motion (resonant frequency of the tuning fork) are detected by a low frequency lock-in amplifier which is used as data for SCM images.

## 7.5  Benefits of Using a Tuning Fork Probe

Some commercially available AFMs presently employ a diode laser whose beam is reflected from the back of a micro-machined Si cantilever to monitor the tip-sample interaction. Unfortunately, the use of a diode laser in AFM would introduce a severe difficulty for our experiments. Illumination from the diode laser is detrimental for

many types of measurement. In semiconductors, the laser light can change the sample's electronic properties (e.g. by electron-hole pair formation). The laser light can also heat the sample and probe which is detrimental for low temperature operation. Buh *et al.* give a good example of laser's influences (as shown in figure 7.8) in his paper [126]. The line profiles of $\Delta C/\Delta V$ signal over the *pn* junction, as shown in image (c) in figure 7.8, demonstrated that the illumination of the AFM laser excitated the electrons in the semiconductor and the image contrast is reduced.

## 7.6 Testing the Capacitance Sensor

In order to test the capacitance resolution of the system, a function generator and a circuit replace the capacitance input as shown in Figure 7.9. A varactor diode (whose capacitance can be changed by an applied DC voltage) is used to simulate the 'tip-sample capacitance' (See Figure 7.10 for specifications). The function generator is used to apply a reverse bias across the diode. See Figure 7.11. The $L_1 = L_2 = 100mH$ are RF chokes and the $C_1 = C_2 = 0.22\mu F$ are DC blocks.

In this case, a 1mV change in applied voltage will cause a 210aF change in $C_{diode}$ with bias voltage=9V (Highest voltage may be operated by the function generator, i.e. the smallest change it may reach, refers to the specifications in figure 7.10). However the system is designed to have an aF ($10^{-18}$ F) range detection ability. It would be preferable to have finer control over the capacitance changes.

An alternative way has been found to overcome this limitation. See Figure 7.12. In this circuit, if the upper capacitance equals to C, and let the sum of two capacitors below ($C_d$ and $C_x$) equal to nC (where n is a ratio for simplifying the calculation), the total capacitance will be:

Figure 7.8: (a)Schematic diagram of the structure of sample. (b) SCM images of a *pn* junction under true dark conditions with the AFM laser off (upper image) and under normal SCM measurement conditions (lower image). (c) Line profiles of $\Delta C/\Delta V$ signal along the corresponding paths [the grey lines in (b)] (images from Buh's paper [126]

Figure 7.9: Block Diagram of Capacitance Detection Simulation



Figure 7.10: Capacitance vs reverse bias for a BB833 capacitance diode

Figure 7.11: Schematic diagrams of capacitance detection system



Figure 7.12: Schematic diagrams of serial capacitors ($C_x$ in Figure 7.11 is replaced by this configuration)

Capacitance Simulation

Figure 7.13: Schematic diagrams of detection circuit

$$C_{total} = \cfrac{1}{\cfrac{1}{C} + \cfrac{1}{nC}} = \frac{nC}{n+1}$$

if there is a very tiny change $dC_d$ in $C_d$, the total capacitance change will be:

$$dC = \cfrac{1}{\cfrac{1}{C} + \cfrac{1}{nC + dC_d}} - \frac{nC}{n+1} \simeq \frac{1}{(n+1)^2} \times dC_d$$

The $dC_d$ has been decreased by a factor of $(n+1)^2$ times. In our case, see Figure 7.13, $C_d = 9.5pF$ and $dC_d/dV$=2390aF/mV at bias voltage equals 0.99v. The $(n+1)^2$ equals 3306. Thus the capacitance can be now modulated by 2390/3306=0.72aF/mV.

In Figure 7.14, the data was recorded at a lock-in time constant of 20ms, therefore the noise bandwidth is 5.9875Hz with a filter slope of 24dB/octave. The input

Figure 7.14: Experimental result of the Capacitance Modulation

RF (Radio Frequency) amplitude is $10mV_{rms}$. The function generator is working at 20kHz, 1mV peak to peak, and modulating at 100% depth on bias of 0.99V. The coils' ratio is $L_1 : L_2 : L_3 = 1 : 5.6 : 1$.

The peak to peak of the square wave responses corresponds to dC = 1mV * 100% * 2 * 0.72aF/mV =1.44aF = $1aF_{rms}$. The noise occupies about 1/5 of the signal which is about $0.2aF_{rms}$. By comparing noise levels with and without a preamplifier, we find there is no real benefit in using the preamplifier from the point of view of signal/noise ratio, as the noise has also been amplified. Thus our detection limit is about $0.2aF_{rms}$ at room temperature. However the resolution improves at lower temperature as the noise level is lower.

## 7.7 Design of the Inductances

The inductors play a very important role in the tuned filter method of capacitance measurement. The capacitance sensor's equivalent circuit in the instrument (see

Figure 7.15: Equivalent circuit of the sensor

Figure 7.6) is shown in Figure 7.15. Using the same component symbols as in the figure, the transfer function of this circuit is

$$\frac{V_{out}}{V_{in}} = \frac{-\omega^2 M_{12} M_{23} R_L}{Z_1 Z_3 (Z_2 + Z_c)}$$

(7.7.1)

where

$$Z_1 = R_{1A} + R_{1B} + j\omega L_1,$$

$$Z_3 = R_3 + R_L + j\omega L_3,$$

$$Z_2 = j\omega L_2 + R_2 + \frac{1}{j\omega C_2},$$

$$Z_c = \omega^2 (\frac{M_{12}^2}{Z_1} + \frac{M_{23}^2}{Z_3}),$$

$$M_{12} = k_{12} \sqrt{L_1 L_2},$$

$$M_{23} = k_{23} \sqrt{L_2 L_3}$$

The derivation of these equations is given in the thesis of C. Bugg [115].

The term $Z_c$ describes the effect of the input and output coupling. It has the effect of reducing the Q and shifting the resonant frequency of the circuit and must be kept small to avoid reducing the sensitivity of the sensor [115].

The terms $k_{12}$ and $k_{23}$ are the coupling coefficients, and C$_2$ is the capacitance which we are going to measure.

At resonance, $L_2$ is related to $C_2$ by:

$$\omega^2 = \frac{1}{L_2 C_2} \tag{7.7.2}$$

and in our system

$$R_{1A} = R_L = 50\Omega \quad (50\Omega \text{ impedance})$$

$$R_{1B} \simeq R_2 \simeq R_3 \simeq 0.3\Omega \quad (\text{resistance of the coil})$$

There are only a few components undetermined in this equation: $L_1, L_3, k_{12}, k_{23}$. More calculations show that $L_1 = L_3$ (see appendix A.1) and $k_{12} = k_{23}$(see appendix A.2) will give the best result. For resonant frequency $f_0 = 130MHz$ and C=3 pF, the best amplitude-output ratio for $L_1 : L_2 : L_3$ is about 1:5.6:1 (see Figure A.6).

Figure 7.16 is the final design of the coil-rod. Since the stability of the coils' inductance and stray capacitance is also an important key to optimize the resolution of the SCM, a ceramic rod (Macor) was chosen because of its low thermal expansion coefficient (the rod will be used at 1.5K). Holes were drilled through the rod to make it easy to wind and bind the wire and GE varnish is used to fix the coils after binding. Holes were drilled in perpendicular directions, which helps to avoid the difficulty of drilling parallel holes, therefore gaps between the holes can be as small as possible. This will increase the coupling coefficient $k$. Calculations show that higher $k$ can decrease the voltage across the tip-sample junction (see Appendix A.4). The method used to measure $k$ after winding which is discussed in Appendix A.3.

Two coil-rods were wound: $L_1 : L_2 : L_3$=1:5.6:1 and $L_1 : L_2 : L_3$=2:5.6:1, where $L_2 \simeq 0.8\mu H$, see Table 7.1.

As close as possible

6mm

3.2mm

1.2mm    1.2mm

Ceramic rod for $f_0$ =130MHz

Figure 7.16: Ceramic Rod for $f_0 = 130MHz$

| Rod | Rod1 | Rod2a | Rod2b |
|---|---|---|---|
| In:Mid:Out | 1:5.6:1 | 2:5.6:1 | 1:5.6:2 |
| $f_0$. | 125MHz | 137MHz | 138MHz |
| $V_{In}$ | 10 $mV_{rms}$ | 10 $mV_{rms}$ | 10 $mV_{rms}$ |
| $V_{Out}(f_0)$ | 3.9 $mV_{rms}$ | 1.8 $mV_{rms}$ | 2.8 $mV_{rms}$ |
| $V_{Out}/V_{In}$ | 0.39 | 0.18 | 0.28 |
| Q | 20.5 | 18.3 | 32.9 |

Table 7.1: Experimental Results for Coil-rods

The symmetric rod undoubtedly has the best output. The rod1 in Table 7.1 has a different resonant frequency which is caused by the different middle coils.

## 7.8   SCM at Room Temperature

The room and low temperature SCM work was mostly carried out by Dr. Fergal Callaghan. Analysis of the SCM images obtained has been carried out by myself with the help of Dr. C. J. Mellor. The capacitance measurement (dC/dZ) was modulating at the tuning fork's resonant frequency instead of using the 5kHz dither piezo.

AFM and MFM have been performed with DI's Dimension 3100 on a SCM test sample shown in figure 7.17. The sample consists of an n-type silicon substrate, with p-type regions. Inside the p-type regions, n-type implants have been made to make

Figure 7.17: Scan size=30$\mu$m, AFM and SCM (dC/dV) of the SCM test sample by DI's Dimension 3100 microscope at room temperature. Regions: 1,substrate (Si) 2,implanted areas 3, gate (poly-Si) 4, a lower n-type concentration 5, p-n junction 6, p-type 7, n-type source/drain implants

npn MOS transistors, while p-type implants have been made in the n-type bulk to form pnp MOS transistors. This sample therefore provides both pnp and npn MOS devices in a single scan. The original sample actually had multiple layers of metals and dielectrics on top of it - but they have been removed by a chemical etch. What is left shows 3 levels in the topography. These are: substrate (Si), 2: implanted areas (silicide has been removed and this leaves a somewhat rough surface of Si) and 3: gate (poly-Si) referring to the left image in figure 7.17. In the SCM image, on the right of figure 7.17, the darker areas indicate lower dC/dV values which are N type areas and the lighter areas are P type areas correspondingly. A very clear p-n junction (5) shown in the SCM image can not be seen in the topography. There are smaller n-type areas (7) inside the p-type area (6). These are the n-type source/drain implants. At the edge of those (but only under gates), the contrast is even darker, indicating a lower n-type concentration. This is normal, as the concentration will reduce when moving closer to the channel. The channel is between two adjacent source/drain implants -

Figure 7.18: Scan size=30$\mu$m, (a)AFM and (b)SCM (dC/dz) of the SCM test sample by our microscope at room temperature. 1: p-n junction; 2: poly-Si gate; 3: Insulating debris - low dC/dz; 4: Topographic steps (see image on left)

underneath the gates. The channel observed in SCM images will be smaller than the one in the topography because of under-diffusion of the dopant atoms under the gate.

As shown in figure 7.18 (a) and (b), a comparative scan of AFM and SCM was obtained by our microscope at room temperature. The images are slightly distorted due to the poor linearity of the scan tube for large scan area. A distinct p-n junction (1) can be seen. Apart from similar semiconductor features to the previous image, some dirt (3) took most of the contrast as it has a low dielectric constant. The topographic error signal also reflects on the SCM image (4) due to the sudden change of the tip-sample distance.

Capacitance detection on a conducting sample has also been performed. By applying a bias voltage to a thin gold film sample, force and dC/dz curve against tip/sample distance is plotted in figure 7.19.

Varying the sample bias voltage can distinguish between n-type and p-type semiconductors. A region of the silicon test sample discussed earlier is shown in figure 7.20. It shows that the contrast reversed from (c) to (d) at pn junction caused by the bias voltage reversal. With higher AC gain of $\Delta$C detection applied, the noise level of

Figure 7.19: Capacitance/force - distance curve of conducting surface (gold thin film).



Figure 7.20: Scan size=20$\mu$m, (a)Topograph (b) Topographic error signal (c) sample bias=-21V with AC gain=10dB (d) sample bias=21V with AC gain=10dB (e) sample bias=21V with AC gain=50dB.

Figure 7.21: (left)AFM and (middle)SCM images of the quantum wire sample obtained at room temperature;(right)topography error signal. ($5\mu m$ by $5\mu m$ area, 6 seconds per line)

image (e) was reduced. The poly-silicon gates look narrower in image (d) and (e) (ignore the artifact lines from error signal) which agree with the DI SCM image.

## 7.9   SCM at Helium Temperature

The sample imaged in superfluid helium is a quantum wire sample which was provided by Dr. A. J. Kent. The sample is grown on a GaAs substrate with a 2 micron thick GaAs buffer layer and with 20.2nm GaAlAs bottom layer, 39.1nm n-type GaAlAs layer and 16.8nm GaAlAs top layer (wafer number NU1624). The gaps and mesas are all 500nm wide and the gaps are 100nm deep. The room temperature topography, topography error signal and capacitance images are shown in figure 7.21 with our microscope. These images were obtained with following settings: time constant=20ms, AC gain=50db, scan speed=500nm/s, resonant frequency=128.538MHz and driving amplitude=300mV. For comparison the topography of the sample imaged with a DI Dimension 3100 is shown in figure 7.22.

The capacitance signal increases when the sample is exposed to light by a red LED at 181K. The capacitance signal line profiles with or without LED on, as shown in

Figure 7.22: $5\mu m$ by $5\mu m$ area AFM of the quantum wire sample obtained with DI Dimension 3100 at room temperature



Figure 7.23: Red LED light effects on the capacitance signal of quantum wire

figure 7.23, are compared. When the red LED was turned on, the capacitance signal from the wire is clearly raised above the signal from between the wires. This is a promising result for a working SCM.

SCM images have been obtained in superfluid helium as shown in figure 7.26. Due to the limitation of reduced scan range at 1.7K, only one quantum wire was studied ($1.6\mu m$ by $1.6\mu m$ area). The microscope was running in constant height mode, however the topography still contributed to the capacitance signal. The topographic capacitance background may be due to a similar reason as the hydrodynamic force interactions discussed in the MFM chapter. From the capacitance detection software simulation, it's found that the capacitance changes contributes from the cantilever/sample are bigger than the tip/sample which is due to the cantilever being too close to the sample. However the cantilever averages the capacitances with a much wider area includes hundreds of gaps and mesas. Therefore when the tip follows the topography traces the average distance between the cantilever and sample changes with a same ratio. This will give a negative capacitance background as sketched in figure 7.24. It is clear that the topographic signal caused the sharp edges and buried the capacitance signal between the tip and sample.

It is possible to do a reasonable correction by adding a ratio of topographic signal into the real capacitance signal to extract the capacitance signal between the tip and the sample if most of the extra capacitance signals are due to the topography. As shown in figure 7.25, it seems that the corrected capacitance image has even lower noise than the topographic one.

The same method was applied to the image obtained in superfluid helium. The corrected capacitance signal was fitted with a Gaussian distribution ($f(x) = exp(-x^2)$)

Figure 7.24: A sketch of total capacitance signal caused by topographic signal



Figure 7.25: (left)Topography (middle) capacitance and (right)capacitance after correction images in high contrast color scale at room temperature. (original topography data shown in figure 7.21)

Figure 7.26: upper row: (left)Topographic (middle)capacitance and (right) corrected capacitance images in superfluid helium.($1.2\mu m$ by $0.75\mu m$ area) lower row: line profile of line 80 in corresponding images

as shown in figure 7.27. The full image can be found in figure 7.26. The coefficient of the capacitance correction was determined by comparing the capacitance signal of the sides of the step. A correct image should have no dips at those points. This was done by comparing an array of corrections generated by Matlab. However, due to the greater background signal which is cause by the tuning fork tine and a long distance between the tip and the carriers, the gaussian fit of capacitance signal in figure 7.27 may only be the integration of the capacitance of an area around the tip instead of the distribution of the 2DES.

Figure 7.27: Gaussian distribution fit of the capacitance signal of 1DES in superfluid helium. Zero in $\Delta C$ is taken on trough between wires for the sake of clarity. The dielectric contribution to $\Delta C$ is actually much greater than the electronic contribution shown here.



Figure 7.28: Sketch of depletion depth and carrier of the sample.

## 7.10 Summary

In this chapter, different detection methods of tip-sample capacitance were discussed. The sensitivity of the capacitance detection was calculated and simulated by a varicap diode. Room temperature images were compared with those obtained with the DI Dimension 3100. Finally a quantum wire sample was studied at low temperature and in the superfluid helium. The reason for seeing topographic signals in capacitance images was explained and the corrections have been done with the room and helium temperature images.

A Gaussian curve-fit was used to make a simple model of tip convolution in which the length scale can be easily varied. However it is not intended as a physical model. If wire is in a single lateral quantum state (unlikely due to the temperature) then $n_s(distance) \simeq sin^2(2\pi x/L)$. It is not possible for our microscope to have this resolution since the carriers are about 56nm away from the surface of the sample plus the tip oscillation (about 30nm) and the distance between the tip and the sample. In our case, it is possible that the $n_s$ is uniform but will have a depletion of depth of around 80nm or so. This is consistent with the fit in Figure 7.27.

# Chapter 8

# Conclusions and Suggestions for Further Work

## 8.1   Summary and Conclusions

The construction and operation of a novel Scanning Probe Microscope using a quartz tuning fork as the force sensor, which is able to operate at low temperature and in superfluid helium and in magnetic fields as high as 12T, has been described. The project aimed to build an instrument to study various material properties at various temperatures and in various magnetic fields. An important goal is to investigate those phenomenon which take place at very low temperatures such as the Quantum Hall Effect (QHE).

There are a few aspects of the design make the microscope suitable for work at low temperature:

1. All components of the microscope head were made from non-magnetic materials and with similar thermal expansion coefficients. Thus it is suitable for operations at different temperatures and in high magnetic field.

2. The SPM head is small and compact. The scan tube moves the sample instead

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of the probe which allows us to use a heavier, and perhaps more complex, microscope sensor.

3. The tuning fork was chosen as the force sensor which is a self-sensing low dissipation probe. Commercial available laser cantilever detection methods might change the property of the sample as reported in Chapter 7 and are complex to implement at low temperature.

4. Q control method was developed and mathematically studied to provide an alternative way to change the quality factor of the tuning fork to meet the requirements in different operation conditions. These were described in Chapters 2 and 5.

5. One-tine immobilization method of the tuning fork probe overcame the difficulties of operation in superfluid helium reported by earlier researchers.

6. Lift-mode and phase detection method was developed in order to provide enough sensitivity for MFM using a tuning fork probe.

7. A number of capacitance detection methods were discussed and a simple efficient design inspired by the work of Bugg and King's was chosen in Chapter 7. It is not only compact and simple but also suitable for the work taking place in superfluid helium.

8. A tip etching method was introduced to produce tungsten wire probes for the sensor. However for convenience in the end, commercial micro-machined tips were used. The long tip obtained by the etching method can be useful for later

MFM researches as the influence of hydrodynamic effects in lift-mode MFM operations described in Chapter 5.

9. A comprehensive anti-vibration system was designed for the SPM. It provides a high degree of acoustic isolation from the surroundings. The sources of system noise have been discussed and the noise level has been decreased as described in Chapter 6.

10. Temperature and pressure control in the sample space have been investigated in Chapter 4 to ensure a suitable conditions for different operations.

The capabilities of the microscope are summarized below:

Referring to the AFM image taken in superfluid helium in Chapter 4, a lateral resolution better than 20nm can be achieved in superfluid helium. The scan range reduces from 46.2 micron by 46.2 micron at room temperature to 8.4 micron by 8.4 micron at 4K, and the Z range decreases from 4.477 micron to 0.814 micron correspondingly. The MFM and SCM have a reduced lateral resolution due to the longer-range nature of the forces involved and the required average distance between tip and sample. However a fast scan speed can decrease the lateral resolution due to the time constant of the feedback loop.

Referring to the STM image of gold monatomic steps in Chapter 4 (figure 4.16), an atomic level vertical resolution ($<0.1$nm) can be achieved with this scan tube. The sensitivity of the force/magnetic force sensor is determined by the Q of the tuning fork and also the background noise level. With a typical Q of 5,000 at low temperature (or may be easily achieved by using the Q control circuit at room temperature) and phase detection method, a force gradient sensitivity better than $3 \times 10^{-4}$ N/m or

equivalent magnetic field gradient may be achieved as discussed in Chapter 6. This is determined by comparing the MFM images taken at low temperature with room temperature MFM images of the sample taken by DI Dimension 3100. A capacitance sensitivity of 0.2aF was demonstrated in Chapter 7 with the SCM images and room temperature simulations.

## 8.2   Suggestions for Further Work

There are numbers of areas which can be addressed in future work.

1. For improvement of the microscope:

    - Develop a better mechanical coarse X/Y and Z motion motor as mentioned in Chapter 2.

    - Improve the linearity of the scan tube by software with scan tube calibration data.

    - Build an integrated detection circuit for the tuning fork which may reduce the noise level further. (This has been done)

    - Try other designs of force and capacitance detections for better sensitivity.

    - Apply the long etched tungstun wires as tips to overcome the difficulties such as hydrodynamic effects in Lift-mode and seeing topography in capacitance images.

2. For further magnetic thin film research:

    - Image a bigger area which allows the domain width to be determined more accurately.

- Work with the Southampton group to simulate the domain structure around a hysteresis loop.

- Measure the hysteresis loop of the sample at low temperature.

- Determine the relationships between the temperature, domain width and hysteresis loop.

- Do a similar study on magnetic ordered alloy samples.

3. For further works in SCM:

- Study Quantum Hall effect with SCM of Hall bar in superfluid helium.

- Study location of dopants in GaN.

# Appendix A

# Derivations of the Formulae

## A.1 Calculations to Prove $L_1 = L_3$

In Equation 7.7.1

I presume:

$$V(C_2) = \frac{V_{out}}{V_{in}} \tag{A.1.1}$$

where $C_2 = C_{stray} + C_{tip-sample}$

In order to find out best result of $\frac{dV}{dC}$ at resonant frequency, let's do some approximation:

assume:

$$
\begin{aligned}
k_{12} &= k_{23} = k \\
R_{1B} &= R_2 = R_3 = 0 \quad \text{(real value} \leq 0.3\Omega, \text{ so can be neglected)}
\end{aligned}
$$

to cancel the k term within the equation.

and let:

$$L_1 = aL,$$

$$L_3 = bL,$$

$$L_2 = L,$$

$$C_2 = C$$

where a,b are just coefficients.

so that:

$$Z_1 = R_1 + j\omega L_1 = 50 + j\omega L \times a,$$

$$Z_3 = R_3 + R_L + j\omega L_3 = 50 + j\omega L \times b,$$

$$M_{12} = k_{12}\sqrt{L_1 L_2} = kL\sqrt{a},$$

$$M_{23} = k_{23}\sqrt{L_2 L_3} = kL\sqrt{b},$$

$$Z_c = \omega^2 \left( \frac{M_{12}^2}{Z_1} + \frac{M_{23}^2}{Z_3} \right) = \omega^2 k^2 L^2 \left( \frac{a}{Z_1} + \frac{b}{Z_3} \right)$$

and because of the RF condition in equation 7.7.2

$$\omega L = \frac{1}{\omega C}, \tag{A.1.2}$$

$$Z_2 = j\omega L_2 + R_2 + \frac{1}{j\omega C_2} = j\omega L - \frac{j}{\omega C} = 0 \tag{A.1.3}$$

substitute these conditions into equation A.1.1, we have:

$$V(C) = \frac{-50\omega^2 k^2 L^2 \sqrt{ab}}{Z_1 Z_3 \left( \omega L j + \dfrac{1}{j\omega C} + Z_c \right)}$$

$$= \frac{-50\sqrt{ab}}{a Z_3 + b Z_1}$$

separating into real and imaginary components:

$$V_{Re}(C) = \frac{-625\sqrt{ab}(a+b)}{625(a+b)^2 + \dfrac{a^2b^2}{\omega^2 C^2}} \tag{A.1.4}$$

and:

$$V_{Im}(C) = \frac{25ab\sqrt{ab}}{\omega C\left(625(a+b)^2 + \dfrac{a^2b^2}{\omega^2 C^2}\right)} \tag{A.1.5}$$

Our final purpose is to determine the best ratios of $L_1 : L_2 : L_3$, which can give us the biggest dV/dC at resonant frequency, so that new functions are defined:

$$dV_{Re} = V_{Re}(C + dC) - V_{Re}(C) \tag{A.1.6}$$

and:

$$dV_{Im} = V_{Im}(C + dC) - V_{Im}(C) \tag{A.1.7}$$

$$dV_{abs} = \sqrt{V_{Im}(C+dC)^2 + V_{Re}(C+dC)^2} - \sqrt{V_{Im}(C)^2 + V_{Re}(C)^2} \tag{A.1.8}$$

where:

C is a fixed capacitance (C=~3 pF) which comes mainly from stray capacitance of the circuit and also the tip-sample capacitance. dC is a only fixed number, say $10^{-18}$ (1 aF), to compare the results for different a and b ($L_1 : L_2 : L_3$). Therefore, the max dV will give us the best choice of the coils' ratio.

These are the 3D plot of the functions by maple: Figure A.1 for $dV_{Re}$, Figure A.2 for $dV_{Im}$ and Figure A.3 for $dV_{Abs}$. The vertical coordinate is dV, the rest two is 1/a and 1/b.

From the diagrams, clearly, it shows the symmetry of a and b, and when a=b, it gives the maximum value.

Figure A.1: 3D plot for $dV_{Re}$ (Z=$dV_{Re}$, X=1/a, Y=1/b)



Figure A.2: 3D plot for $dV_{Im}$ (Z=$dV_{Im}$, X=1/a, Y=1/b)



Figure A.3: 3D plot for $dV_{Abs}$ (Z=$dV_{Abs}$, X=1/a, Y=1/b)

## A.2 Calculations to Prove $k_{12} = k_{23}$ Is Optimum Relationship

As before, let's make some approximations and assume that:

assume:

$$k_{12} = mk_{23} = mk \quad (\text{namely, } k_{23} = k)$$

$$R_{1B} = R_2 = R_3 = 0$$

let:

$$L_1 = L_3 = nL$$

$$L_2 = L,$$

$$C_2 = C$$

The m,n are the only 2 variables this time.

Now we have:

$$Z_1 = Z_3 = Z = 50 + j\omega L \times n = 50 + \frac{nj}{\omega C},$$

$$M_{12} = k_{12}\sqrt{L_1 L_2} = mkL\sqrt{n},$$

$$M_{23} = k_{23}\sqrt{L_2 L_3} = kL\sqrt{n},$$

$$Z_c = \omega^2\left(\frac{M_{12}^2}{Z_1} + \frac{M_{23}^2}{Z_3}\right) = \omega^2 k^2 L^2 n\left(\frac{m^2}{Z_1} + \frac{1}{Z_3}\right)$$

ibid, substitute these conditions into equation A.1.1, we have:

$$V(C) = \frac{-50\omega^2 k^2 L^2 mn}{Z_1 Z_3 Z_c}$$

$$= \frac{-50m}{Z(m^2 + 1)}$$

Figure A.4: 3D plot for $dV_{Re}$ (Z=$dV_{Re}$, X=m, Y=1/n)

again, the V(C) is separated to real and imaginary part:

$$V_{Re}(C) = \frac{-2500m}{(m^2+1)(2500 + \dfrac{n^2}{\omega^2 C^2})} \tag{A.2.1}$$

and:

$$V_{Im}(C) = \frac{\dfrac{50mn}{\omega C}}{(m^2+1)(2500 + \dfrac{n^2}{\omega^2 C^2})} \tag{A.2.2}$$

In the same way as before, these functions were plotted by Maple: Figure A.4 for $dV_{Re}$, Figure A.5 for $dV_{Im}$ and Figure A.6 for $dV_{Abs}$. The vertical coordinate is dV, the rest two is m and 1/n.

The answer is what we expected: m=1 ($k_{12} = k_{23}$, when $L_1 = L_3$).

## A.3 The Derivation to Measure k

In the circuit shown in Figure A.7, Kirchhoff's voltage law requires that the equations:

Figure A.5: 3D plot for $dV_{Im}$ (Z=$dV_{Im}$, X=m, Y=1/n)



Figure A.6: 3D plot for $dV_{Abs}$ (Z=$dV_{Abs}$, X=m, Y=1/n)



Figure A.7: Equivalent Circuit for k Detection

$$V_1 \;=\; (R_1 + R_{1B} + j\omega L_1)I_1 + j\omega M * I_2, \qquad\qquad \text{(A.3.1)}$$

$$0 \;=\; (j\omega L_2 + R_2 + R_{2B})I_2 + j\omega M * I_1 \qquad\qquad \text{(A.3.2)}$$

$$M \;=\; k\sqrt{L_1 L_2} \qquad\qquad\qquad\qquad\qquad\qquad \text{(A.3.3)}$$

be satisfied.

Assume:

$$R_{1b} \;=\; R_{2b} = 0, \qquad\qquad\qquad\qquad\qquad \text{(A.3.4)}$$

$$R_1 \;=\; R_2 = R \qquad\qquad\qquad\qquad\qquad\quad \text{(A.3.5)}$$

now:

$$I_1 \;=\; -\frac{I_2 R + j\omega L_2 I_2}{j\omega M},$$

$$V_1 \;=\; (R_1 + j\omega L_1) \times -\frac{I_2 R + j\omega L_2 I_2}{j\omega M} + j\omega M I_2,$$

$$\frac{-V_1}{I_2} \;=\; \frac{R^2 + j\omega R(L_1 + L_2) + \omega^2(M^2 - L_1 L_2)}{j\omega M}$$

$$\frac{-V_1}{V_2} \;=\; \frac{1}{\omega M R}\left[(\omega R(L_1 + L_2) + j\left(R^2 + \omega^2 L_1 L_2(k^2 - 1)\right)\right]$$

$$\left|\frac{V_1}{V_2}\right| \;=\; \frac{1}{\omega M R}\sqrt{\omega^2 R^2 (L_1 + L_2)^2 + (R^2 + \omega^2 L_1 L_2(k^2 - 1))^2}$$

$$\omega^2 R^2 L_1 L_2 k^2 \left|\frac{V_1}{V_2}\right|^2 \;=\; \omega^2 R^2 (L_1 + L_2)^2 + \left(R^2 + \omega^2 L_1 L_2(k^2 - 1)\right)^2$$

with this equation, the k can be evaluated by Maple.

Figure A.8 is the plot for k versus $\dfrac{V_{out}}{V_{in}}$ (f=140MHz), which shows higher k result in better output. k is about 0.2 in our circuit.

Figure A.8: k vs $\dfrac{V_{out}}{V_{in}}$ which shows the larger k the larger output ratio

## A.4 Estimation of $V_c$

As we did before, Kirchhoff's voltage law requires the equations for the circuit shown in Figure 7.15:

$$
\begin{cases}
V_1 = (R_{1A} + R_{1B} + j\omega L_1)I_1 + j\omega M_{12} * I_2, \\[2mm]
0 = (j\omega L_2 + R_2 + \dfrac{1}{j\omega C_2})I_2 + j\omega M_{12} * I_1 + j\omega M_{23} * I_3, \\[2mm]
0 = (R_3 + R_L + j\omega L_3)I_3 + j\omega M_{23} * I_2, \\[2mm]
V_3 = I_3 R_L
\end{cases}
$$

in our circuit:

$$\because \quad R_1 = R_3, L_1 = L_3 = L,$$

$$\therefore \qquad Z_1 = Z_3 = Z$$

$$\because \qquad k_{12} = k_{23},$$

$$\therefore \qquad M_{12} = M_{23} = M,$$

$$\Rightarrow$$

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Figure A.9: k vs $V_c/V_1$, range of k from 0 to 0.25

$$
\begin{cases}
I_1 = \dfrac{V_1 - j\omega M I_2}{Z}, \\[2mm]
0 = R_2 I_2 + j\omega M (I_1 + I_3), \\[2mm]
I_3 = \dfrac{-\,j\omega M I_2}{Z}
\end{cases}
$$

$$
\Rightarrow \qquad \frac{j\omega M V_1}{Z} = \left(\frac{2\omega^2 M^2}{Z} - R_2\right) I_2
$$

$$
\therefore \qquad \frac{j\omega M V_1}{Z} = \left(\frac{2\omega^2 M^2}{Z} - R_2\right)\frac{jV_c}{\omega L}
$$

$$
\therefore \qquad \frac{V_c}{V_1} = \frac{\omega^2 M L}{2\omega^2 M^2 + Z R_2}
$$

$$
\left|\frac{V_c}{V_1}\right| = \frac{\omega^2 M L}{\sqrt{(2\omega^2 M^2 + R_L R_2)^2 + (\omega L_1 R_2)^2}}
$$

it's plotted in Figure A.9 and Figure A.10.

If the $R_2$ is ignored, the equation will be:

$$
\left|\frac{V_c}{V_1}\right| = \frac{1}{k}\sqrt{\frac{L_2}{L_1}} \simeq \frac{1}{0.6k}
$$

Figure A.10: k vs $V_c/V_1$, range of k from 0.25 to 1

# Appendix B

# Schematics of Our Etching Circuit

**Table of Cut-off Time Settings.**

| Marker | Cut-off Time(ms) |
|--------|------------------|
| 0      | 15               |
| 1      | 25               |
| 2      | 40               |
| 3      | 60               |
| 4      | 80               |
| 5      | 100              |
| 6      | 120              |
| 7      | 140              |
| 8      | 160              |
| 9      | 180              |
| 10     | 200              |

Table B.1: Table of Cut-off Time Setting.

Figure B.1: Schematic of Tip Etching Controller

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