DESIGN OF A SELF-ALINGED, HIGH RESOLUTION, LOW TEMPERATURE (30 mK – 300 K) MAGNETIC FORCE MICROSCOPE

KENDİLİĞİNDEN HİZALAMALI, YÜKSEK ÇÖZÜNÜRLÜKLÜ, DÜŞÜK SICAKLIK (30 mK – 300 K) MANYETİK KUVVET MİKROSKOBU TASARIMI

ÖZGÜR KARCI

PROF. DR. ŞADAN ÖZCAN
Supervisor
PROF. DR. AHMET ORAL
Co-Supervisor

Submitted to Institute of Sciences of Hacettepe University
As a Partial Fulfillment to the Requirements
for the Awarded of the Degree of Doctor of Philosophy
in Nanotechnology and Nanomedicine

2015
This work named “Design of a Self-Aligned, High Resolution, Low Temperature (30 mK – 300 K) Magnetic Force Microscope” by ÖZGÜR KARCI has been approved as a thesis for the Degree of DOCTOR OF PHILOSOPHY IN NANO TECHNOLOGY AND NANOMEDICINE by the below mentioned Examining Committee Members.

Prof. Dr. Hüseyin Zafer DURUSOY
Head .................................................................

Prof. Dr. Şadan ÖZCAN
Supervisor ..............................................................

Prof. Dr. M. Recaï ELLİALTİOĞLU
Member .................................................................

Doç. Dr. Hakan Ö zgür Ö ZER
Member .................................................................

Doç. Dr. Alpan BEK
Member .................................................................

This thesis has been approved as a thesis for the Degree of DOCTOR OF PHILOSOPHY IN NANO TECHNOLOGY AND NANOMEDICINE by Board of Directors of the Institute for Graduate Studies in Science and Engineering.

Prof. Dr. Fatma SEVİN DÜZ
Director of the Institute of Graduate Studies in Science
ETHICS

In this thesis study, prepared in accordance with the spelling rules of Institute of Graduate Studies in Science of Hacettepe University,
I declare that

• all the information and documents have been obtained in the base of the academic rules
• all audio-visual and written information and results have been presented according to the rules of scientific ethics
• in case of using others Works, related studies have been cited in accordance with the scientific standards
• all cited studies have been fully referenced
• I did not any distortion in the data set
• and any part of this thesis has not been presented as another thesis study at this or any other university.

13/02/2015

ÖZGÜR KARCI
ABSTRACT

DESIGN OF A SELF-ALINGED, HIGH RESOLUTION, LOW TEMPERATURE (30 mK – 300 K) MAGNETIC FORCE MICROSCOPE

ÖZGÜR KARCI

Doctor of Philosophy, Department of Nanotechnology and Nanomedicine

Supervisor: Prof. Dr. Şadan ÖZCAN
Co-Supervisor: Prof. Dr. Ahmet ORAL

February 2015, 134 pages

The invention of Scanning Tunneling Microscope (STM) in 1981 opened a new avenue in atomic scale world and surface science. STM made it possible to either image atoms or manipulate them as well as measuring the local density of states of electrons on the surface. This invention also leaded invention of the another Scanning Probe Microscope (SPM) method called Atomic Force Microscopy (AFM) soon which measures the interaction forces between a sharp tip attached at the end of a lever and sample surfaces at atomic length scales. AFM could measure various forces between tip-sample like van der Waals, electrostatic, friction or magnetic forces. Measurement of these specified forces was named in the force microscopy with their force name like Friction Force Microscopy, Electrostatic Force Microscopy and Magnetic Force Microscopy (MFM) which is subject of the thesis. While STM measures only conductive specimens, AFM measures both conducting and insulating specimens. Easy sample preparation, easy to use, high resolution and affordable price of these systems have collected great attention over last two decades. Moreover, operations in various environments like low temperatures, high magnetic fields, either in vacuum or ultra-high vacuum have broadened application areas of the AFMs.

Magnetic Force Microscope (MFM) plays a crucial role for magnetic imaging down to 10 nm magnetic resolution in material science and physics. Especially, low temperature AFM/MFM
has the capability of working in extreme physical conditions like down to few hundreds of milliKelvin temperature range or up to few tens of Tesla magnetic field. High density magnetic recording media, superconductivity, spintronics, magnetic phase transitions, spin-glass systems, magnetic nanoparticles, topological insulators are some of the hot research topics in this area in which high resolution MFM is required for investigations.

In low temperature AFM/MFM (LT-AFM/MFM) systems, deflection of the cantilevers is measured by means of a fibre interferometer in which the light is carried into the cooling system utilizing a fibre cable. A special mechanism aligns the fibre end with respect to the cantilever for measuring the deflections. The critical thing is here that this alignment may collapse when the system is cooled down because of both the design and different thermal contractions of the materials. The developed alignment mechanisms also enlarge volume of the microscope which is crucial for fitting inner free sample space of the cooling systems.

In this study, we developed a self-aligned, low temperature atomic force / magnetic force microscope operating between 300 K and 2 K temperature ranges of liquid Helium. The OD of the microscope is less than 25.4 mm and compatible with most of the cryostat systems from various vendors. We used a specially designed alignment mechanism for eliminating tedious and time consuming alignment mechanism which utilizes alignment chip sets from Nanosensors. The alignment-free design makes the life easier for end users of the system for the whole operation ranges of the temperature. Deflection of the cantilever was measured by means of a developed Michelson type fibre interferometer. We obtained unprecedented noise floor for the interferometer that ~25 fm/√Hz at 300 K and ~12 fm/√Hz at 4 K. The shot noise was calculated to be 7.8 fm/√Hz at 4 K. This noise floor enabled us to achieve 10 nm magnetic force microscopy (MFM) resolution on the high density hard disk sample with commercial cantilevers, routinely. We showed Abrikosov vortex lattice in BSCCO(2212) single crystal at 4 K and vortices in YBCO thin film superconductor at 50 K in MFM mode. Atomic steps of both mica and HOPG samples were shown in AFM modes, too.

Liquid Helium is the most common and popular cryogen which boils at 4.2 K and if the vapor pressure is decreased, one can reach ~1.5 K temperature limit for cooling. For lowering the temperature below 1.5 K, $^3$He based rather complicated cryostat systems are used that they reach ~300 mK base temperature level. Furthermore, using a dilution refrigerator system, it is also possible to reach ~5 mK which uses $^4$He/$^3$He mixture as a cryogen in a special way. Operating LT-AFM/MFM at these ultra low temperatures is important for material science and physics for investigating many scientific phenomena. We demonstrated two separate
microscope designs for a $^3$He system and a dilution refrigerator system. The capabilities and potentials of the microscopes are shown both in AFM modes and MFM mode at these ultra low temperatures, successfully.

In the proceeding study, the shot noise limited sensitivity of Michelson fibre interferometer was improved an order of magnitude utilizing fibre Fabry-Pérot interferometer (FFPI) which has $\sim$1 fm/$\sqrt{\text{Hz}}$ noise floor. The inaugural performance of the LT-AFM/MFM using fibre Fabry-Pérot interferometer both in AFM and MFM mode were shown between 300 K and 4 K. FFPI with this ultra noise floor would be a standalone metrology instruments for many research areas, too.

In the last part of study, we describe a novel radiation pressure based cantilever excitation method for imaging in dynamic mode atomic force microscopy (AFM) for the first time. Piezo excitation is the most common method for cantilever excitation, but it may cause spurious resonance peaks. Therefore, direct excitation of the cantilever plays a crucial role in AFM imaging. A single light beam was used both for excitation of the cantilever at the resonance and measuring the deflection of the cantilever. The laser power was modulated at the cantilever’s resonance frequency by a digital Phase Lock Loop (PLL). We typically modulate the laser beam by $\sim$500 $\mu$W and obtained up to 1,418 Å$_{pp}$ oscillation amplitude in moderate vacuum levels between 4 - 300 K. We have demonstrated the performance of the radiation pressure excitation in AFM/MFM by imaging CoPt multilayers between 4-300 K and Abrikosov vortex lattice in BSCCO(2212) single crystal at 4 K for the first time.

**Keywords:** Atomic force microscope, AFM, magnetic force microscope, MFM, low temperature, magnetic field, high resolution imaging, cryostat, milliKelvin, dilution refrigerator, fibre interferometer, fibre Fabry-Pérot interferometer, radiation pressure, direct excitation, spring constant
ÖZET

KENDİLİĞİNDEN HİZALAMALI, YÜKSEK ÇÖZÜN SexyylÜKLÜ, DÜŞÜK SICAKLIK (30 mK – 300 K) MANYETİK KUVVET MİKROSKOBU TASARIMI

ÖZGÜR KARCI
Doktora, Nanoteknoloji ve Nanotıp Bölümü
Tez Danışmanı: Prof. Dr. Şadan ÖZCAN
Eş-Danışman: Prof. Dr. Ahmet ORAL
Şubat 2015, 134 sayfa

Manyetik Kuvvet Mikroskopları (MKM), malzeme bilimi ve fizikte manyetik görüntüleme alanında önemli bir yere sahiptir. Bu tez çalışmaları kapsamında 10 nm gibi yüksek bir manyetik çözünürlük limitine ulaşılmıştır. Düşük sıcaklık AKM/MKM sistemleri ise miliKelvin sıcaklık seviyelerinde ve çok yüksek manyetik alanlar altında çalışabilen özel sistemlerdir. Yüksek yoğunlukta manyetik veri depolama, süperiletkenlik, spin fiziği, manyetik faz geçişleri, manyetik nano parçacıklar veya topolojik yalıtkanlar gibi bilimsel ve teknolojik öneme sahip araştırma alanlarında, yüksek çözünürlükte ve değişken sıcaklık/manyetik alan ortamında, manyetik görüntüleme önemli yere sahiptir.


Tez kapsamında, fiber-yay arasında hizalama gerektirmeyen, kendiliğinden hizalamalı esasına dayalı bir düşük sıcaklık AKM/MKM geliştirerek 300 K - 2 K sıcaklık aralığında başarıyla çalıştırıldı. Geliştirilmişimiz mikroskobun dış çapı 25.4 mm den daha küçük ve bu nedenle bir çok soğutma siteminin içersinde çalışma potansiyeline sahiptir. Kendiliğinden hizalama mekanizaması için izel bir tasarım geliştirildik ve bu tasarım içerisinde Nanosensor firmasının ürettiği hizalama çiplerini kullanarak. Bu mekanizma ile karmaşık hizalama mekanizmalarını gerektirecek ve kolayca kullanılabilir bir düşük sıcaklık mikroskobu ortaya koyduk.

Düşük sıcaklık AKM/MKM sistminde yay ile örnek yüzeyi arasındaki etkileşimleri ölçmek için bir fiber girişim ölcer tasarlayarak çalıştırdık. Geliştirilmişimiz yüksek hassasiyetekici girişim ölcer için 300 K de ~25 fm/√Hz, 4 K de ~12 fm/√Hz gürtülü seviyesi ölçüldü. Photon gürtültüsü ise 4 K de 7.8 fm/√Hz olarak hesaplandığı ve girişim ölcerin gürtülü tabanını kısıtladığı görüldü. Bu yüksek hassasiyetekici girişim ölçer kullanarak yaptığımız MKM çalışmalarında yüksek yoğunlukta sabit disk örneklerinde rutin olarak 10 nm manyetik çözünürlük elde ettik. Bunun yanında, BSCCO(2212) tek kristal ve YBCO ince film
süperiletken örnekler üzerinde oluşan girdapları 4 K ve 50 K sıcaklıklarında başarıyla görüntüledik. Mica ve HOPG örneklerindeki atomik terasları AKM modunda gösterdik.

Sıvı Helyum, kaynama noktasına 4.2 K olan en yaygın soğutucudur. Buhar basıncını düşürlerek, kullanıldığı sistemin sıcaklığını 1.5 K seviyesine indirebilir. Bu sıcaklık aralığı için geliştirilmiş düşük sıcaklık AKM/MKM sistemini, 1.5 K’ın altında hava yoğunluğu düşük programlara inmeye imkan tanıyan $^3$He soğutma sistemine de entegre edecek şekilde tasarlayarak ~300 mK de sistemi çalıştırarak test etmeye başlardık. Bunu bir adım daha ileri görüşerek, $^3$He/$^4$He seyreltilik karışım ile soğutma yaparak sıcaklığı ~5 mK’e indirebilen seyreltilik soğutucu için de yaptığımız tasarım ile mikroskobu ~30 mK seviyelerinde çalıştırık. Bu şekilde mikroskobu, çok geniş bir sıcaklık aralığında (~30 mK -300 K) çalıştırarak uygulama alanlarını genişlettiğini ve potansiyelini artırdık.

Çalışmanın sonraki aşamasında, tasarladığımız girişim ölçerlerin gürültü seviyesini bir derece düşürdüğümüz saflayarak ~1 fm/$\sqrt{Hz}$ gürültü tabanına ulaştığımız fiber Fabry-Pérot girişim ölçerini geliştirdik. Bu çok yüksek hassasiyetli girişim ölçerleri AKM/MKM sisteminde kullanarak 300 K - 4 K sıcaklık aralığındaki AKM ve MKM tarama kiplerinde görüntüler aldık. Gelişirdiğimiz bu sensör aynı zamanda değişik alanlardaki birçok metroji ve kalibrasyon çalışmalarında kullanılmaya da adaydır. İleriki çalışmalarla bu hassas sensör ile MKM çözünürlüğünü 10 nm seviyesinin altında indirmek mümkün olabilecektır.


Anahtar Kelimeler: Görüntüleme, atomik kuvvet mikroskobu, AKM, manyetik kuvvet mikroskobu, MKM, manyetik alan, yüksek çözünürlükte görüntüleme, kriyostat, miliKelvin, seyreltilik soğutucu, fiber girişim ölçer, fiber Fabry-Pérot girişim ölçer, radyasyon basıncı, doğrudan titreşim, yay sabiti
ACKNOWLEDGEMENTS

While writing the thesis, I went to my last 10 years spent in NanoMagnetics Instruments. I did many experiments again and again. I spent nights and weekends on the setups again and again. I travelled around the world for conferences, exhibits, installations, trainings and sales. I did many friendships during my travels and had wonderful times and experiences. I have learned look at the world from their eyes which is colorful, rich and exciting for me all the time. When I go back to August 2005 for a job interview, I’d like to thank to Ahmet for giving a chance to me work with him in NanoMagnetics. I have learned many things during years like doing research, passing the borders, working hard, aiming the best and more. His guiding and great perspectives made it possible to bring the thesis to this position. Thanks a lot for everything and shaping my life and future.

I would like to thank to Prof. Dr. Şadan Özcan for his continuous support and friendship. Without him I could not pass the break point that I lived in the company. His extensive experiences on low temperature physics and cryostat systems helped us to pass the self-aligned design tests in Hacettepe SNTG laboratory over nights and weekends. I achieved many of the first things with his guidance in PPMS.

One of the my committee member, Assoc. Prof. Dr. Hakan Ö zgür Özer came from İstanbul to Ankara many times even at nights or very early mornings. I appreciate his time and his efforts since he just did this as a contribution or duty for academy. His evaluations, suggestions and attributes to science were so valuable and did a great contribution to this thesis.

I would like to thank to Prof. Dr. Hüseyin Zafer Durasoy for his continuous support during committee meetings and encouragements for finalizing the work. The most important thing that I learned from him is being continent. My visits to him or our meetings instructed me much. I will always remember our sushi experiences in Dresden and Ankara.

I would like to thank to Prof. Dr. M. Recai Elliathoğlu and Assoc. Prof. Dr. Aplan Bek for their contribution during my PhD defense. They saved time for the thesis evaluation and provided valuable suggestions.

I had gained very deep understanding of low temperature physics and $^3$He system operation during my work at Rice University, USA. I’d like to thank to Prof. Dr. Rui Rui Du, Dr. Kristjan Stone and Dr. Zhouquan Yuan. Without their contributions, I could not conclude mK experiments at all.
I have learned many things during my installations besides giving training. They were always mutual. The experiments we did in Madrid with Dr. Carmen Manuera on piezoresponse force microscopy and conductive AFM were quite exciting and instructive for me. I’d like to thank to also Prof. Mar Garcia Hernandez and Dr. Norbert Nemes for their valuable contributions and unforgettable hosting in Madrid.

India is the most colorful and rich in cultural diversity country that I have been. I did the first LT-AFM/MFM installation in Indore which was great experiences for me with the promising experimental results. I’d like to thank to Dr. Rajeev Rawat, Dr. Archana Lakhani and Dr. Pallavi Kushwaha for their contribution and friendship.

I have spent peaceful July in Lausanne next to Lake Geneva in 2013. Without Oscar Julian Piatek’s guidance and contributions on dilution refrigerator, I could not conclude fridge integration of the microscope and tests.

The inaugural tests and results of the systems obtained in PPMS system at SNTG laboratory. Dr. Burak Kaynar’s guidance and help about PPMS even at nights was so valuable. We did many discussions with Dr. Telem Şimşek and Assoc. Prof. Dr. Abdullah Ceylan. Their friendship was amazing.

I have marvelous colleagues and friends at NanoMagnetics for the last 10 years. Their contributions are great for the thesis. I’d like to thank to Gizem Durak for her great patience, hard work and contributions. She was an excellent co-worker that I worked together. Without Muharrem Demir, his patience and ability to find quick solutions to the problems, I could not go ahead. The drawings and designs of Serhat Çelik and Bülent Çolak for the microscope parts embodied them. The intuitive understanding and explanations of Ümit Çelik helped me to overcome problems that I met. The friendship of Ramin Abbaszadi and solving software problems that I met was so important for me. And many others that I did not share their name.

In August 16, 2005, I did a meeting with Dr. Munir Dede and he offered me my job and stated if I could start for the next day instead of next Monday. I started the work next day in low temperature group which change my way in the company I believe. I’d like to thank to him for everything that we spent many years together.

I’d like to thank to Yiğit Uysallı from METU for performing reflectivity simulations of the cantilevers which contributed to the radiation pressure section deeply.

At the end, the support of my family and their love during all my lifetime makes this thesis possible. Duru, Nihat, Özgü and Mert, I owe you much…
My grand-grandmother Necat, I put the pen its place…

My grandmother Emine and grandfather Gazi. They always prayed for me and shared what you had. This is one of the key in my life that I learned from them.

My uncle Isa and his family was my second family in Ankara over years. Their love and support were precious. When I was a little child, I always remember my uncle Osman’s laughs. His analytical thinking enriched me all the time. My cousin Fatma was always with me and Necdet and Vicdan.

And my wife and our love, Nehir. I always feel their love that was my only reason to come to this end point.

To Dicle and Nehir
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>ETHICS</th>
<th>i</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>ii</td>
</tr>
<tr>
<td>ÖZET</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>viii</td>
</tr>
<tr>
<td>SYMBOLES AND ABBREVIATIONS</td>
<td>xiv</td>
</tr>
<tr>
<td>CHAPTER 1</td>
<td>1</td>
</tr>
<tr>
<td>1.1 Introduction and Overview</td>
<td>1</td>
</tr>
<tr>
<td>1.2 The Thesis and Work Plan</td>
<td>3</td>
</tr>
<tr>
<td>CHAPTER 2</td>
<td>5</td>
</tr>
<tr>
<td>2.1 Magnetic Force Microscopy (MFM)</td>
<td>5</td>
</tr>
<tr>
<td>2.2 Theory of Operation</td>
<td>6</td>
</tr>
<tr>
<td>2.2.1 Static Mode</td>
<td>6</td>
</tr>
<tr>
<td>2.2.2 Dynamic Mode</td>
<td>7</td>
</tr>
<tr>
<td>2.2.3 Magnetic Interaction between Tip and Sample</td>
<td>10</td>
</tr>
<tr>
<td>2.2.4 Tip-Sample Interaction</td>
<td>11</td>
</tr>
<tr>
<td>CHAPTER 3</td>
<td>14</td>
</tr>
<tr>
<td>3.1 Instrumentation of the Microscope</td>
<td>14</td>
</tr>
<tr>
<td>3.1.1 LT-AFM/MFM Head</td>
<td>14</td>
</tr>
<tr>
<td>3.1.2 LT AFM/MFM Insert</td>
<td>15</td>
</tr>
<tr>
<td>3.1.3 Scanning Mechanism</td>
<td>16</td>
</tr>
<tr>
<td>3.1.4 Coarse Approach Mechanism</td>
<td>19</td>
</tr>
<tr>
<td>3.1.5 XY Sample Positionar</td>
<td>20</td>
</tr>
<tr>
<td>3.1.6 Fine Approach Mechanism</td>
<td>21</td>
</tr>
<tr>
<td>3.2 MFM Alignment Holder</td>
<td>22</td>
</tr>
<tr>
<td>3.2.1 Alignment Holder Design</td>
<td>22</td>
</tr>
<tr>
<td>3.2.2 Fibre Cable</td>
<td>25</td>
</tr>
<tr>
<td>3.2.3 Cantilevers</td>
<td>26</td>
</tr>
<tr>
<td>3.3 Michelson Fibre Interferometer</td>
<td>27</td>
</tr>
<tr>
<td>3.3.1 Operation Principles and Design</td>
<td>27</td>
</tr>
<tr>
<td>3.3.2 Fibre Interferometer Card</td>
<td>30</td>
</tr>
</tbody>
</table>
CHAPTER 4
4.1 Low Temperature Tests of the Self-Aligned Mechanism ........................................... 36
4.2 Noise Analysis and Measurements ............................................................................. 38
  4.2.1 Laser Noise ........................................................................................................... 39
  4.2.2 Shot Noise ............................................................................................................ 40
  4.2.3 Electrical Noise ................................................................................................... 40
  4.2.4 Total Noise .......................................................................................................... 41
  4.2.5 Noise Measurements ......................................................................................... 42
4.3 Images ...................................................................................................................... 44
  4.3.1 AFM Images ....................................................................................................... 44
  4.3.2 MFM Images ....................................................................................................... 48
  4.3.3 Vortex Imaging on Superconductors ................................................................. 55

CHAPTER 5
5.1 Introduction to milliKelvin AFM/MFM (mK-AFM/MFM) .......................................... 61
5.2 Cryogenics and 3He as a Cryogen ............................................................................. 61
  5.2.1 3He Cryostat System ......................................................................................... 62
  5.2.2 3He Insert and Integration of AFM/MFM Head .................................................. 64
  5.2.3 Heat Transfer Through the 3He Insert ............................................................... 65
  5.2.4 Cooling the mK-AFM/MFM .............................................................................. 68
5.3 Images ...................................................................................................................... 69
5.4 Conclusions .............................................................................................................. 71

CHAPTER 6
6.1 Introduction to DR-AFM/MFM .............................................................................. 72
6.2 Dilution Refrigerators and Operations .................................................................... 72
6.3 Cryostat and Vibration Isolation Stage ................................................................... 75
6.4 DR AFM/MFM Design .......................................................................................... 76
6.5 Experimental Results ............................................................................................. 78
  6.5.1 Temperature Stability and Heat Load ............................................................... 78
  6.5.2 Fibre Interferometer Signal ............................................................................... 80
  6.5.3 Cantilever Tune ................................................................................................. 82
6.6 Images ...................................................................................................................... 83
SYMBOLS AND ABBREVIATIONS

Symbols
A      Amplitude
A₀     Amplitude at resonance frequency
Å      Angstrom
Åpp    Angstrom peak to peak
Aₐ     Cross-sectional area
a      Vortex spacing
Bₖₜ    Bandwidth
c      Speed of light
D      Outside diameter of the piezo tube
d      Fibre-cantilever separation
d₃₁    Transverse piezoelectric coefficient
E      Magnetostatic energy
e      Charge (1.6x10⁻¹⁹ C)
ε      Thermal conductivity coefficient
emu    Electromagnetic unit
F      Force
Fₛₜ    Tip-sample interaction force
F₅₉ₜ    Driving force
f₀     Resonance frequency
fm     Femtometer
H      Magnetic stray field
Hₖ     Critical field density
h      Planck constant (6.63x10⁻³⁴ J.s)
i      Photocurrent
i_shot Shot noise current
i₀     Midpoint photocurrent
Jₖ     Critical current density
k      Spring constant
kₜ     Boltzmann constant (1.38x10⁻²³ J/K)
kₓ     Piezo coefficient in x-axis
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k_y$</td>
<td>Piezo coefficient in y-axis</td>
</tr>
<tr>
<td>$k_z$</td>
<td>Piezo coefficient in z-axis</td>
</tr>
<tr>
<td>$L$</td>
<td>Length</td>
</tr>
<tr>
<td>$\vec{M}$</td>
<td>Magnetization</td>
</tr>
<tr>
<td>$N_{Total,,rms}$</td>
<td>Summation of noises</td>
</tr>
<tr>
<td>$m$</td>
<td>Mass</td>
</tr>
<tr>
<td>$m$</td>
<td>Interference slope</td>
</tr>
<tr>
<td>$ms$</td>
<td>Milisecond</td>
</tr>
<tr>
<td>$\bar{m}$</td>
<td>Magnetic moment</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Micro</td>
</tr>
<tr>
<td>$\mu_0$</td>
<td>Permeability of free space $(4\pi \times 10^{-7} , \text{H})$</td>
</tr>
<tr>
<td>$N$</td>
<td>Newton</td>
</tr>
<tr>
<td>$n$</td>
<td>Refractive index</td>
</tr>
<tr>
<td>$n_{\text{rms}}$</td>
<td>Average mean square value of noise</td>
</tr>
<tr>
<td>$n_m$</td>
<td>Number of moles</td>
</tr>
<tr>
<td>$n_p$</td>
<td>Number of photons</td>
</tr>
<tr>
<td>$nF$</td>
<td>Nano Farad</td>
</tr>
<tr>
<td>$ns$</td>
<td>Nanosecond</td>
</tr>
<tr>
<td>$\text{Oe}$</td>
<td>Oersted</td>
</tr>
<tr>
<td>$P$</td>
<td>Optic power</td>
</tr>
<tr>
<td>$p$</td>
<td>Pico</td>
</tr>
<tr>
<td>$P_{RP}$</td>
<td>Momentum of the light</td>
</tr>
<tr>
<td>$Q$</td>
<td>Q-Factor</td>
</tr>
<tr>
<td>$Q_{\text{Total}}$</td>
<td>Total heat</td>
</tr>
<tr>
<td>$\theta$</td>
<td>Half angle of the maximum cone of the light</td>
</tr>
<tr>
<td>$R$</td>
<td>Reflectivity</td>
</tr>
<tr>
<td>$R_F$</td>
<td>Feedback resistor</td>
</tr>
<tr>
<td>$R_{\text{number}}$</td>
<td>Thermal conductivity</td>
</tr>
<tr>
<td>$S$</td>
<td>Spectral noise density</td>
</tr>
<tr>
<td>$s$</td>
<td>Wall thickness of tube piezo</td>
</tr>
<tr>
<td>$S_{PD}$</td>
<td>Responsivity of the photodetector</td>
</tr>
<tr>
<td>$T$</td>
<td>Temperature</td>
</tr>
<tr>
<td>$T$</td>
<td>Tesla</td>
</tr>
<tr>
<td>$T_F$</td>
<td>Fermi temperature</td>
</tr>
</tbody>
</table>
\( t \) Time
\( T_c \) Superconducting transition temperature
\( U \) Applied voltage to the electrodes
\( z \) Position
\( \Delta x \) Lateral piezo displacement
\( \Delta z \) Displacement
\( \Delta f \) Frequency shift
\( \Delta A \) Amplitude shift
\( \Delta \phi \) Phase shift
\( \Delta l \) Length change
\( \Delta T \) Temperature difference
\( \Delta v \) Lindwidth
\( \nabla \) Gradient
\( \lambda \) Wavelength
\( \alpha \) Thermal contraction coefficient
\( \beta \) Fabry-Pérot slope coefficient
\( \tau \) Optical path difference of the cavity gap
\( z_0 \) Initial position, equilibrium position
\( <z^2> \) Mean square amplitude of the cantilever
\( \delta F' \) Derivative of the force
\( \frac{\partial z}{\partial t} \) Derivative of position with respect to time
\( \frac{\partial V}{\partial z} \) Derivative of potential energy with respect to position
\( \delta \) Damping factor
\( \phi \) Phase
\( \Phi_0 \) Flux quantum \( (2.07 \times 10^{-15} \text{Tm}^2) \)
\( V(z) \) Potential
\( V \) Visibility
\( \bar{v} \) Voltage noise
\( v_{\text{shot}} \) Shot noise voltage
\( \omega \) Angular frequency
\( W \) Watt
\( \Omega \) Ohm
# Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>Alternating current</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscope</td>
</tr>
<tr>
<td>BSCCO</td>
<td>Bismuth Strontium Calcium Copper Oxide</td>
</tr>
<tr>
<td>Co</td>
<td>Cobalt</td>
</tr>
<tr>
<td>CoPt</td>
<td>Cobalt platinum</td>
</tr>
<tr>
<td>DC</td>
<td>Direct current</td>
</tr>
<tr>
<td>DOS</td>
<td>Density of states</td>
</tr>
<tr>
<td>DR</td>
<td>Dilution refrigerator</td>
</tr>
<tr>
<td>EFM</td>
<td>Electrostatic Force Microscope</td>
</tr>
<tr>
<td>EI</td>
<td>Experimental insert</td>
</tr>
<tr>
<td>Fe</td>
<td>Iron</td>
</tr>
<tr>
<td>FC/APC</td>
<td>Face centered angled polished contact</td>
</tr>
<tr>
<td>FFPI</td>
<td>Fibre Fabry-Pérot interferometer</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width Half Maximum</td>
</tr>
<tr>
<td>FMR</td>
<td>Frequency Modulation Reflective coating</td>
</tr>
<tr>
<td>GB</td>
<td>Gigabit</td>
</tr>
<tr>
<td>GBP</td>
<td>Gain bandwidth product</td>
</tr>
<tr>
<td>Gbpsi</td>
<td>Giga bit per square inch</td>
</tr>
<tr>
<td>HF</td>
<td>Hydrofluoric acid</td>
</tr>
<tr>
<td>HOPG</td>
<td>Highly Ordered Pyrolytic Graphite</td>
</tr>
<tr>
<td>$^3\text{He}$</td>
<td>Helium three</td>
</tr>
<tr>
<td>ID</td>
<td>Inner diameter</td>
</tr>
<tr>
<td>IR</td>
<td>Infrared</td>
</tr>
<tr>
<td>ITO</td>
<td>Indium tin oxide</td>
</tr>
<tr>
<td>I-V</td>
<td>Current to voltage</td>
</tr>
<tr>
<td>IVC</td>
<td>Inner vacuum jacket</td>
</tr>
<tr>
<td>K</td>
<td>Kelvin</td>
</tr>
<tr>
<td>kHz</td>
<td>Kilo Hertz</td>
</tr>
<tr>
<td>LT</td>
<td>Low Temperature</td>
</tr>
<tr>
<td>LT-AFM/MFM</td>
<td>Low Temperature Atomic Force Microscope/Magnetic Force Microscope</td>
</tr>
<tr>
<td>MC</td>
<td>Mixing chamber</td>
</tr>
<tr>
<td>MFM</td>
<td>Magnetic Force Microscope</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
</tr>
<tr>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>MFMR</td>
<td>Magnetic Force Microscopy Reflective coating</td>
</tr>
<tr>
<td>MHz</td>
<td>Mega Hertz</td>
</tr>
<tr>
<td>mK</td>
<td>milliKelvin</td>
</tr>
<tr>
<td>mbar</td>
<td>milibar</td>
</tr>
<tr>
<td>NA</td>
<td>Numerical aperture</td>
</tr>
<tr>
<td>nm</td>
<td>Nanometer</td>
</tr>
<tr>
<td>Ni</td>
<td>Nickel</td>
</tr>
<tr>
<td>Nb$_2$O$_5$</td>
<td>Niobium pentoxide</td>
</tr>
<tr>
<td>OD</td>
<td>Outside diameter</td>
</tr>
<tr>
<td>OFHC</td>
<td>Oxygen-free high thermal conductivity copper</td>
</tr>
<tr>
<td>OPAMP</td>
<td>Operational amplifier</td>
</tr>
<tr>
<td>PCB</td>
<td>Printed circuit board</td>
</tr>
<tr>
<td>PD</td>
<td>Photodetector</td>
</tr>
<tr>
<td>PhBr</td>
<td>Phosphor bronze</td>
</tr>
<tr>
<td>PID</td>
<td>Proportional Integral Derivative</td>
</tr>
<tr>
<td>PLL</td>
<td>Phase Lock Loop</td>
</tr>
<tr>
<td>PrFM</td>
<td>Piezoresponse Force Microscopy</td>
</tr>
<tr>
<td>RF</td>
<td>Radio frequency</td>
</tr>
<tr>
<td>RuO$_2$</td>
<td>Ruthenium oxide</td>
</tr>
<tr>
<td>SHPM</td>
<td>Scanning Hall Probe Microscopy</td>
</tr>
<tr>
<td>SNR</td>
<td>Signal to noise ratio</td>
</tr>
<tr>
<td>SPM</td>
<td>Scanning Probe Microscope</td>
</tr>
<tr>
<td>ss</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>SSS</td>
<td>Super Sharp Silicon</td>
</tr>
<tr>
<td>STM</td>
<td>Scanning Tunneling Microscope</td>
</tr>
<tr>
<td>SHO</td>
<td>Simple harmonic oscillator</td>
</tr>
<tr>
<td>TL</td>
<td>Top loading</td>
</tr>
<tr>
<td>VCO</td>
<td>Voltage Controlled Oscillator</td>
</tr>
<tr>
<td>VTI</td>
<td>Variable temperature insert</td>
</tr>
<tr>
<td>V$_{PD}$</td>
<td>Signal photodiode voltage</td>
</tr>
<tr>
<td>YBCO</td>
<td>Yttrium Barium Copper Oxide</td>
</tr>
<tr>
<td>ZnO</td>
<td>Zinc oxide</td>
</tr>
</tbody>
</table>
CHAPTER 1

1.1 Introduction and Overview

The invention of scanning tunneling microscopy (STM) in 1981 [1-3] has revolutionized the surface science. STM made it possible to either image or manipulate individual molecules and atoms on the surface. Besides these unique capabilities of STM, local density of states (DOS) on the sample surface could be also measured [4]. A schematic description of STM is given in Figure 1.1. A bias voltage, which is applied between a sharp metallic tip and a conductive sample surface, leads a tunneling current that is an exponential function of the tip-sample distance. The tunneling current read in the system (I) is compared with the set point current (Iset) and the difference is feed back to z piezo for adjusting the tip height. A raster scan on the sample by means of xy piezo explores topography of the sample at atomic scales. This revolutionary invention was awarded with 1986 Nobel Prize in physics to H. Rohrer and G. Binning.

![Figure 1.1: Schematic of scanning tunneling microscope](image)

The theory of STM was constructed between real samples and a modeled tip by Tersoff and Hamann [5] ignoring tip-sample interaction forces. The forces exerted on the tip in the tunneling regime collected attention of Binning and coworkers which was born of the another imaging technique, atomic force microscope (AFM). In 1986, they measured the forces
between a diamond tip attached at the end of gold-foil and a ceramic sample (Al₂O₃) [6]. The deflection of the gold-foil was measured in their setup by means of STM feedback, too. They showed atomic resolution on a graphite sample soon [7]. Whereas STM could image only conducting surfaces, AFM could image both conductive and insulating samples.

Depending on the tip sample distance, either repulsive or attractive forces dominate the interaction. In the repulsive force regime, the tip is in contact with the sample surface that is called contact mode AFM. Both atomic periodicity [8] and lateral frictional forces [9] were studied utilizing contact mode. When the tip-sample distance is increased, attractive forces can be measured. Frequency modulation technique was developed to measure the attractive forces by Albrecht et. al. [10] called non-contact AFM. True atomic resolution was shown on the silicon surface using this technique by Giessibl in 1995 [11]. Another AFM mode, which works in both repulsive and attractive force regime, called tapping mode (or semi-contact) [12] or phase imaging mode [13] AFM, was developed, too. Tapping mode AFM is the most common used mode today and gives additional information besides topography.

AFM could not only image the sample surfaces, it could also measure various force between tips and sample directly. Magnetic, electrostatic, friction or van der Waals forces could be measured using specified tips. For example, with the metal coated tips, electrostatic charge distribution and amount of the sample surface can be imaged when an electric field is applied between tip and sample which is called electrostatic force microscope (EFM) [14]. When the tip is coated with magnetic materials, magnetic images of the samples could be obtained which is called magnetic force microscope (MFM) and the subject of the thesis. The force, which is measured in AFM, gives name of the method. Therefore, AFM is the common name of the family with many members.

Measurement of magnetic forces using force microscopy has opened a new avenue in magnetic imaging [15]. Furthermore, MFM is a relatively simple and easy to use and can be operated in a wide range of environments like vacuum, high magnetic fields [16], low temperatures, etc. in contrast to the other magnetic imaging techniques. Magnetic imaging at variable temperatures and external high magnetic field has special importance in material science and physics like vortex imaging [17-21] or manipulation of vortices [22-24] in superconductors, interface dynamics [25], magnetic phase separation [26-27], domain walls in ferromagnetic thin films [28-29], magnetization reversal [30], and topological insulators [31]. Applications of the low temperature MFM on various material systems were discussed extensively in the literature [32-34], too.
There is relatively limited number of low temperature AFM & MFM (LT-MFM) described in the literature [35-45] at temperatures at and below 4 K. Since the space is quite limited to a few centimeters, measuring deflection of the cantilever in a cryostat is not simple like in the ambient systems. A number of different deflection measurement methods are reported by various groups. Most of the optical methods rely on the fibre interferometer [46-48], however due to thermal contractions the cantilever should be aligned with respect to the fibre. Other methods are self-sensing methods which require no optical alignment. These utilize piezoresistive cantilevers [44, 49, 50] and quartz tuning forks with a magnetic tip glued at the end of one of the prongs [51, 52]. However, the sensitivity of the piezoresistive sensors is limited because of the power dissipation [50] and the spring constant of the quartz tuning is too high, ~1,800 N/m, which limits the magnetic sensitivity and applicability.

1.2 The Thesis and Work Plan

In this thesis, we developed an ultra low noise, ~12 fm/√Hz, self-aligned Michelson fibre interferometer based LT-AFM/MFM operating between ~30 mK to 300 K which does not require any optical alignment between fibre and cantilever, capable of achieving ~10 nm magnetic resolution. We have used an alignment chip from NanoSensors [53] or Applied NanoStructures [54] to make the system alignment free and very easy to operate, compared to other microscopes. The mechanical design of the LT-AFM/MFM was carefully crafted and tuned such that the thermal contractions are cancelled out and minimized during the temperature cycling, enabling us hassle free operation, down to ~30 mK. Therefore, all the tedious alignment procedures and unnecessary positioning mechanisms which would complicate the design were eliminated for a LT-AFM/MFM system.

In Chapter 2, principles of both atomic force microscopy and magnetic force microscopy are discussed with theory of their operation. In the proceeding Chapter 3, the instrumentation of LT-AFM/MFM, scanning mechanism and coarse approach mechanism of the sample and design of self-aligned mechanism are explained extensively. The designs of fibre interferometer as a deflection sensor and noise reduction of the fibre interferometer are discussed. Vibration isolation issue is also discussed in this section.

In chapter 4, both tests and experimental results are given and discussed. First, test results of the self-aligned mechanism between ~30 mK – 300 K are given and explained. Then, noise analysis of the LT-AFM/MFM is studied and noise measurements are discussed between 4 K and 300 K. Finally, images obtained in both AFM modes and MFM are given with
discussions. Both 10 nm MFM resolution on a high density hard disk sample and vortex imaging on BSCCO and YBCO type II superconductors are demonstrated in this section.

In chapter 5, design of a milliKelvin AFM/MFM for a $^3$He cryostat system is discussed. The operation principles of a $^3$He system, which has the base temperature of ~300 mK, are given in this part. Thermal heat transfer analysis with design parameters are discussed, too. At the end, both AFM and MFM results are given and capabilities of the system are demonstrated.

In chapter 6, design of dilution fridge AFM/MFM for a dilution refrigerator system is discussed. The operation principles of a dilution refrigerator system, which has the base temperature of 8 mK, are explained. Integration of the AFM/MFM head into the fridge is given. The cooling results at 20 mK and images at 150 mK are given in this chapter. The initial results are the first results in the literature for a cantilever based MFM at these ultra low temperatures. For future works, design of a low power fibre interferometer with experimental results and noise analysis are discussed, too.

In chapter 7, design of a low temperature fibre Fàbry-Perot interferometer with 1 fm/√Hz noise level is given. Multilayer coating of cleaved end of the fibre, adjustable cavity gap by means of a dedicated fibre slider are discussed in this section. This unprecedented noise floor is crucial for both AFM and MFM resolution which is discussed with results.

In chapter 8, for the first time in the literature, we demonstrated radiation pressure excitation operation of the LT-AFM/MFM. Radiation pressure plays a vital role for direct cantilever excitation instead of standard piezo excitations. We also developed a new method that a single beam source is used for both exciting the cantilever and deflection measurement simultaneously. Performance of the method is shown both in AFM and MFM modes: atomic steps of mica sample is demonstrated at 300 K in tapping mode AFM and vortex lattice structure of BSCCO single crystal is shown at 4 K in MFM mode, successfully.
CHAPTER 2

2.1 Magnetic Force Microscopy (MFM)

In magnetic force microscopy [15], magnetic interaction forces between a magnetic tip and a magnetic sample are measured across the sample and recorded as a magnetic image. For this purpose, the cantilever with magnetically coated tip (Fe, Ni, Co, etc.,) is oscillated at or near the resonance frequency. The scan is composed of two parts: “forward” scan and “backward” scan as seen in Figure 2.1. During forward scan, topography of the sample is recorded by means of measuring short range or van der Waals forces either in tapping mode [12] or non-contact AFM [10] mode. Prior to the backward scan on the same recorded line, the tip is lifted such an amount to get rid of the short range forces. The feedback is switched off and the tip follows the same path acquired during forward scan with a lift-off value which has a typical value between about 10 nm and few hundred nanometers. The long range magnetic forces cause shift in the resonance frequency of the cantilever during backward scan which is recorded as magnetic image. The amplitude of interaction force depends on the density of stray field emanating from the sample, tip-sample distance and magnetization of the tip.

![Diagram of magnetic force microscopy](image)

Figure 2.1: Schematic principles of magnetic force microscopy

Deflection of the cantilever is measured by means of a deflection sensor which is a fibre interferometer in our system. In Figure 2.2, both topography image and magnetic image of the garnet single crystal are given as a typical example obtained from an MFM image. MFM image explores the magnetic domain structure of the crystal as bright and dark texture on the
image, Figure 2.2(b). Bright regions in the texture show repulsive tip-sample interactions whereas dark regions show attractive tip-sample interactions. Overall, the magnetic image shows magnetic contrast between the tip and the sample for magnetic force microscopy with unprecedented lateral resolution down to ~20 nm.

Figure 2.2: MFM image of the garnet single crystal recorded at 300 K. Forward scan (a) is used for gathering topography of the sample and backward scan (b) gives magnetic image. Scan area is 72 µm x 72 µm and lift-off value for the backward scan is 90 nm.

Physics of the both atomic and magnetic force microscopy are discussed in the following extensively.

2.2 Theory of Operation

The interaction between tip and sample is measured in different ways which defines the AFM imaging modes. The modes can be classified into two parts: static mode and dynamic mode. Dynamic mode is also split into two parts: semi-contact mode (also called tapping mode or intermittent contact mode) and non-contact mode. In our microscope, we can use three of these modes for different applications.

2.2.1 Static Mode

When the cantilever is brought into close proximity of the sample surface, the cantilever gives bending response to the force according to Hooke’s law:

$$\Delta z = \frac{F_{ts}}{k}$$  \hspace{1cm} (2.1)
where $\Delta z$ is the displacement, $k$ is the spring constant of the cantilever and $F_{is}$ is the normal interaction force between the cantilever and the sample. If the deflection of the cantilever is measured, the force can be calculated according to the equation. In static mode, the cantilever is in contact with sample and it is not preferred for MFM measurements since its sensitivity is poor compare to the dynamic modes.

### 2.2.2 Dynamic Mode

In dynamic mode, the cantilever is oscillated by an actuator in its resonance frequency. The equation of cantilever motion can be modeled utilizing a simple spring-mass system as seen in Figure 2.3. The motion of a spring-mass system is described by a simple harmonic oscillator (SHO) using Newton’s 2nd law of motion:

$$F = ma = m \frac{\partial^2 z}{\partial t^2} = -k(z - z_0)$$

(2.2)

$$m \frac{\partial^2 z}{\partial t^2} + k(z - z_0) = 0$$

(2.3)

$$f_0 = \sqrt{\frac{k}{m}}$$

(2.4)

where $f_0$, $k$, and $m$ are natural resonance frequency, spring constant and mass of the cantilever, respectively. $z_0$ is equilibrium position of the cantilever in absence of a force field, $z$ is the distance between tip apex and the sample surface.

![Figure 2.3: Schematic analogy between a spring-mass system and a cantilever](image-url)
In presence of a friction force in this kind of oscillatory system, motion of the system is described as damped harmonic oscillator and the equation of motion is given in the following:

\[ m \frac{\partial^2 z}{\partial t^2} + \delta \frac{\partial z}{\partial t} + k(z - z_0) = 0 \]  

(2.5)

where \( \delta \) is damping factor which is described by spring constant, resonance frequency and quality factor, \( Q \), in the following way:

\[ \delta = \frac{k}{f_0 Q} \]  

(2.6)

In a damped oscillatory system, if there is a driving force, \( F_d(t) = F_0 \cos \omega t \), the motion is called harmonic oscillator with damping by driving force and the equation of motion is given by:

\[ m \frac{\partial^2 z}{\partial t^2} + \delta \frac{\partial z}{\partial t} + k(z - z_0) = F_d(t) \]  

(2.7)

The driving force is provided to the cantilever by a piezo actuator in the actual system. If this system is brought into a force field like tip-sample interaction force, \( F_{ts} \), the equation of forced harmonic oscillator with damping in a force field is given by:

\[ m \frac{\partial^2 z}{\partial t^2} + \delta \frac{\partial z}{\partial t} + k(z - z_0) = F_d(t) + F_{ts}(z) \]  

(2.8)

This is a non-linear equation because of the non-linear term of tip-sample interaction force \( F_{ts}(z) \). To solve the equation analytically, \( F_{ts}(z) \) term is expanded into a Taylor series as in the following:

\[ F_{ts}(z) = F_{ts}(z_0) + \frac{\partial F_{ts}(z_0)}{\partial z} (z - z_0) + \cdots \]  

(2.9)

In the case of small interaction force and adequate oscillation amplitude, \( F_{ts}(z) \) can be approximated by the first two terms and then Eq. 9 can be written as:

\[ m \frac{\partial^2 z}{\partial t^2} + \delta \frac{\partial z}{\partial t} + k(z - z_0) = F_d + F_{ts}(z_0) + \frac{\partial F_{ts}(z_0)}{\partial z} \bigg|_{z = z_0} (z - z_0) \]  

(2.10)

\[ m \frac{\partial^2 z}{\partial t^2} + \delta \frac{\partial z}{\partial t} + \left[ k - \frac{\partial F_{ts}(z_0)}{\partial z} \right] \bigg|_{z = z_0} (z - z_0) = F_d + F_{ts}(z_0) \]  

(2.11)
The term \( k - \partial F_{ts}/\partial z \) in Eq. 11 is called effective spring constant, \( k_{eff} \), which will be different than the spring constant in absence of a force field [55]. The solution of the differential equation in Eq. 11 is given by the well-known function:

\[
z(t) = z_0 + A \cos(2\pi f_d t - \phi)
\]  

with these three variables in the function:

\[
f_d = \sqrt{\frac{k}{m} - \frac{1}{m} \frac{\partial F_{ts}}{\partial z}}
\]  

\[
A = \frac{A_0/m}{\sqrt{(f_0^2 - f_d^2)^2 + \left(\frac{F_d f_0}{Q}\right)^2}}
\]  

\[
\phi = \cos^{-1}\left[\frac{f_0^2 - f_d^2}{\sqrt{(f_0^2 - f_d^2)^2 + \left(\frac{F_d f_0}{Q}\right)^2}}\right]
\]

When the cantilever is oscillated at the resonance frequency, the amplitude and phase are reduced to:

\[
A_0 = \frac{F_0 Q}{mf_0^2}
\]

\[
\phi_0 = \frac{\pi}{2}
\]

Assuming that \( k \gg \partial F_{ts}/\partial z \) with \( \sqrt{1 - x} \approx 1 - x/2 \) approximation and differentiating Eq. 2.13:

\[
\Delta f \approx \frac{f_0}{2k} \frac{\partial F_{ts}}{\partial z}
\]

The force gradient can be measured using either the slope detection method [56] or frequency modulation method [10]. We used slope detection method in which, the cantilever is oscillated with amplitude of \( A \) at the frequency where the slope of the amplitude change is maximum, slightly off-resonance. When the cantilever is brought into a force field, the resonance frequency will be shifted and the force gradient can be measured by measuring.
either the chance in amplitude (ΔA) or the change in phase (ΔΦ) as seen in Figure 2.4. They are equal to:

\[
\Delta A = \frac{-2A_0Q \frac{\partial F_{ts}}{\partial z}}{3\sqrt{3}k} \quad (2.19)
\]

\[
\Delta \Phi = \frac{Q F_{ts}}{k} \frac{\partial F_{ts}}{\partial z} \quad (2.20)
\]

The derivation of the measurable quantities above mentioned are discussed extensively in the related articles, too [56-59].

Figure 2.4: Frequency versus oscillation amplitude (A) and frequency versus phase graph (Ω) and measurement of amplitude change and phase change using slope detection method. MFM cantilever is driven at 75 kHz resonance frequency (f₀) and the Quality factor is 200.

### 2.2.3 Magnetic Interaction between Tip and Sample

In the case of both the tip and the sample are magnetic, in Figure 2.1, the total magnetostatic energy of the system is equal to [60]:

\[
\frac{1}{2} \left( M_1 \cdot \frac{\partial M_2}{\partial z} + M_2 \cdot \frac{\partial M_1}{\partial z} \right) 
\]
\[ E = -\frac{\mu_0}{2} \left[ \int \vec{M}_{\text{tip}} \vec{H}_{\text{sample}} dV + \int \vec{H}_{\text{tip}} \vec{M}_{\text{sample}} dV \right] \] (2.21)

where \( M \) and \( H \) are magnetization and emanating stray field, respectively. Magnetization is the volume density of the magnetic moment, \( m \), depicted in Figure 2.1 and \( \vec{m} = \int \vec{M} dV \). Regarding the reciprocity principle [61] two integrals are equal to each other and the equation will be:

\[ E = -\mu_0 \int \vec{M}_{\text{tip}} \vec{H}_{\text{sample}} dV \] (2.22)

Considering both vertical vibration of the cantilever and vertical magnetization, only the vertical component of tip-sample interaction force can be taken into account. Integrating over volume of the magnetic tip, \( F_{ts}(z) \) can be simplified and equal to:

\[ F_{ts}(z) = -\nabla E = \mu_0 \int_{v_{\text{tip}}} \nabla(\vec{M}_{\text{tip}} \vec{H}_{\text{sample}}) dV_{\text{tip}} \] (2.23)

\[ F_{ts}(z) = \mu_0 \int_{v_{\text{tip}}} \vec{M}_{\text{tip}} \frac{\partial \vec{H}_{\text{sample}}}{\partial z} dV_{\text{tip}} \] (2.24)

\[ \frac{\partial F_{ts}(z)}{\partial z} = \mu_0 \int_{v_{\text{tip}}} \vec{M}_{\text{tip}} \frac{\partial^2 \vec{H}_{\text{sample}}}{\partial z^2} dV_{\text{tip}} \] (2.25)

The derivative of the \( F_{ts}(z) \) in Eq. 25 is used relation for forming MFM contrast. When the tip is brought into close proximity of the sample surface, variety of forces experience on the cantilever. At the atomic length scale tip-sample separation, ionic, covalent and metallic forces are effective that they are repulsive forces. At nanometer length scales, Coulombic, Van der Waals or capillary forces dominates the interaction. Electrostatic and magnetostatic forces are long range forces and have influence on the interaction up to hundreds of nanometers [58, 59, 62].

### 2.2.4 Tip-Sample Interaction

For more quantitative analysis, the empirical Lennard-Jones potential between the sample and the tip can be used. The potential between two atoms or non-polar molecules, \( V(z) \), and the force \( F(z) \) are described as:
\[ V(z) = \frac{a}{z^{12}} - \frac{b}{z^{6}} \]  

\[ F(z) = -\frac{\partial V}{\partial z} = 12 \frac{a}{z^{13}} - 6 \frac{b}{z^{7}} \]

where \( z \) is the tip-sample distance and both \( a \) and \( b \) are system dependent coefficients. For large \( z \) values, the second term in the Eq. 2.27 is dominating the force equation and the forces are attractive. When the distance is decreased to few nanometers, the attractive forces go through to the maximum deep value as seen in Figure 2.5.
Figure 2.5: Tip-sample separation versus Lennard-Jones potential graph and force versus distance curve on a sample surface. (Assuming $a=b=1$ in Eq. 26). The shift on the retract motion at bottom part of the graph was caused by the capillary forces between the tip and the sample.

Tapping mode AFM is operated at this force regime of the repulsive and attractive forces. Then with decreasing tip-sample distance, the direction of the forces reverse and the forces are becoming repulsive. Contact mode AFM is operated in this repulsive force regime. In magnetic force microscopy, the magnetic forces, that the magnetic tip is under the influence, are in the regime of about 10 nm to hundreds of nanometers.
CHAPTER 3

3.1 Instrumentation of the Microscope

3.1.1 LT-AFM/MFM Head

The microscope head was designed with 25.4 mm OD and 200 mm length to fit into a 30 mm free ID sample space of standard helium cryostats from various manufacturers. The microscope head is composed of two concentric piezotubes: the inner piezo tube is used for scanning and the outer one is used for the sample positioned, Figure 3.1. The length of the scan piezo is 3" (EBL #2) and has ~18 µm XY scan range and ~1.4 µm Z range at 4 K. The scan piezo is composed of quadrant electrodes and a dither piezo at the end, which is used to dither the cantilever for dynamic mode operation. The MFM alignment holder with cantiever/fibre assembly is mounted at the end of the scanner piezo. Concentric design eliminates most of the thermal drift.

![Figure 3.1: Picture of LT-AFM/MFM head and its components](image)

The length of the sample slider piezo is 1.5" (EBL #2), which also has quadrant electrodes and a glass tube mounted at the end. A sample slider puck is loaded on this glass tube using a leaf spring and two screws, Figure 2. The stick-slip sample approach mechanism is used to move the sample in XYZ directions. Sample slider piezo can move the sample in z direction ~10 mm and in XY directions within Ø3 mm. We have also integrated a capacitive encoder to measure the XY position with ±3 µm accuracy, with no heat dissipation. The motion and step sizes 50-800 nm are controlled by our dedicated LT-AFM/MFM Control Electronics and Software. A protection shield is used for covering over fragile part of the head, Figure 3.2. Both concentric piezo holders and shield are made of PhBr and plated with gold.
3.1.2 LT AFM/MFM Insert

The microscope head seen in Figure 3.1 is detachable using a docking station, comprising low temperature high density miniature connectors, which makes it possible to attach the microscope to many different cryostat systems, Figure 3.3. The length of the insert above the docking station can be arranged to bring the cantilever/sample couple to magnet center of the cryostat. Radiation baffles were placed on the insert up to the neck to eliminate the radiation coming from room temperature.

The insert has a KF 40/50 neck to fit the variable temperature insert (VTI) space of the cryostats. The neck is the bottom part of stainless steel spherical head which contains vacuum seal three connectors (LEMO Inc.) and fibre feedthrough where fibre cable is introduced into the insert, Figure 3.4. All the wirings follow the main stainless steel tubing at the body of the insert. All the components were chosen from non-magnetic materials for compatibility of the high magnetic field.
3.1.3 Scanning Mechanism

Piezoelectric materials are the basic components of the scanning probe microscope systems since they require picometer range positioning for atomic resolution imaging. Piezoelectric effect is originated from anisotropic crystal structure of certain materials. Anisotropy in their unit cell has a non-zero charge which develops an electric polarity. When these materials are subjected to a mechanical stress, the electric polarity strength is changed which is called the ‘direct’ piezoelectric effect. If the piezoelectric material is subjected to an electric field, electric field causes distortion in unit cell hence the materials change their shape which is called ‘transverse’ piezoelectric effect. Using proper architecture and design the change in the shape can be controlled for desired directions with amounts.

Piezo tube made from lead zirconium titanate seen in Figure 3.5 is used for a scanner for LT-AFM/MFM system. It has four electrodes at outside and a single electrode inside walls. If opposite voltages are applied reciprocal quadrature electrodes when the inner electrode is grounded, bending occurs (xy-planes) on the tube as seen in Figure 3.5. This effect is used for scanning the sensor across the sample. When the opposite voltages are applied all quadrature electrodes with respect to the inner electrode, the tube is extended or contracted (z-plane) which is used for keep the distance between the sensor and the sample during scanning. The electric dipoles should stay in polarized state in one direction to see these mechanical effects. During the long term usage, applying access voltage or raising the temperature above Curie
state of the piezo material, the polarization may collapse. Therefore, the piezo element has to be polarized properly prior to operation and calibration has to be checked periodically.

![Figure 3.5: Schematic architecture of a tube piezo scanner: quadrant electrodes with a dither piezo and its bending mechanism (exaggerated). Reciprocal axis and grounded inner electrode from top view.](image)

The vertical displacement, $\Delta l$, of the piezo tube scanner (EBL #2) can be calculated using the equations below [60]:

$$\Delta l = \frac{d_{31} l U_z}{s} \quad (3.1)$$

where $d_{31}$, $l$, $U_z$, $s$ are transverse piezoelectric coefficient, length of the tube, applied voltage to the electrodes and wall thickness of the tube, respectively. The values: $d_{31}$ is $-1.73 \text{ Å/V}$ at 300 K, $l$ is 3”, $U_z$ is $\pm 100 \text{ V}$ and $s$ is 0.5 mm.

The lateral displacement, $\Delta x$, of the piezo tube scanner can be calculated using the equations below [60]:

$$\Delta x = \frac{2\sqrt{2} d_{31} l^2 u_k}{\pi D t} \quad (3.2)$$

where $D$ is OD of the piezo tube which is $6.35 \text{ mm}$. At the end of the quadrants, there is the unique top electrode which is used for dithering the cantilever for dynamic mode AFM operations.
Figure 3.6: Applied voltage to the reciprocal quadrant electrodes of the tube scanner piezo by the high voltage amplifier. The maximum voltage swings between ±100V.

Three different lengths of the piezo tubes were used with different scan limits: 3’’ length, 1’’ length and 1’’ length. In Table 3.1, both the calibrated scan limits and z-ranges of these piezos are given for three different temperature ranges of 300 K, 77 K and 4 K. Micro-fabricated gratings were used for xy calibration and the fibre interferometer were used for z calibration of the tube piezo scanners.

Table 3.1: Typical capacitance values of the tube piezo scanner at different temperatures

<table>
<thead>
<tr>
<th>Type (EBL#2)</th>
<th>300 K</th>
<th>77 K</th>
<th>4 K</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>x, y (µm)</td>
<td>z (µm)</td>
<td>x, y (µm)</td>
</tr>
<tr>
<td>3’’ Scanner</td>
<td>108</td>
<td>7.08</td>
<td>39</td>
</tr>
<tr>
<td>2’’ Scanner</td>
<td>57</td>
<td>4.80</td>
<td>15</td>
</tr>
<tr>
<td>1’’ Scanner</td>
<td>17</td>
<td>2.26</td>
<td>6</td>
</tr>
</tbody>
</table>

When the microscope is cooled down to 4 K from 300 K, the capacitance value of the piezo tube scanners were also decreased with the similar ratio of the decrease in the scan range. Typical capacitance values of the quadrants at three different temperatures are given in Table 3.2. The ratio of the capacitance values between 300 K and 4 K is ~6 which is the same with scan limits at these two temperatures.

Table 3.2: Typical capacitance values of the tube piezo scanner at different temperatures
<table>
<thead>
<tr>
<th>Type (EBL#2)</th>
<th>300 K</th>
<th>77 K</th>
<th>4 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>3” Scanner</td>
<td>~10.6 nF</td>
<td>~3.5 nF</td>
<td>~1.8 nF</td>
</tr>
</tbody>
</table>

A typical temperature versus piezo coefficients for three axis of the tube piezo scanner graph is given in Figure 3.7. As seen in the graph, the piezo coefficient of slow axis (ky) is greater than fast axis (kx) of the scan which is the drawback of the tube piezo scanners. The coefficient of the slow scan axis is ~ 20% greater than the fast scan axis. Therefore, both scan axes were calibrated separately.

![Figure 3.7: Temperature dependent calibration of the 3” tube piezo scanner](image-url)

### 3.1.4 Coarse Approach Mechanism

For coarse approach a piezo tube with quadrant electrodes is used. A special glass tube is mounted at the end of the piezo tube by means of low temperature epoxy where the sample puck slides on it using stick-slip approach mechanism. The sample puck is mounted on the glass tube and the stiffness is adjusted by means of a metal leaf spring and two M1.6 screws.

The principle of the motion is depicted in Figure 3.8. A desired voltage is swept about ~3 ms from A to B exponentially which is called ‘stick’. Then the voltage is decreased to zero in a very short time scale of ~900 ns from B to C which is called ‘slip’. At this step, the inertia of the puck competes with the friction between puck and glass surface and move in vertical. This slider mechanism can carry up to few hundred grams at 4 K, successfully. Step size, which is
approximately between 50 nm to 800 nm, is arranged by peak voltage given to the slider piezo.

![Diagram of Quartz Piezo Puck and Extension Displacement](image)

Figure 3.8: Exponential pulse given to the slider piezo for stick-slip approach mechanism

### 3.1.5 XY Sample Positionar

An orthogonal XY sample positionar enables us to move the sample in Ø3 mm diameter. The sample holder is pin-loaded style with 5 pins: one is for sample bias and 4 for spares. Sample holder stationary part which is seen in Figure 3.9, is made of PhBr and machined in a way that it has two V-shaped grooves at the back side where it sits on the zirconia rails. Zirconia rails are glued on the other PhBr which has V-shaped grooves perpendicular to the first one at the bottom and sits on another zirconia rail couples. All are placed on the one side of the base plate. At the bottom part of this base plate, there is sapphire disk and fixing part, respectively that a spring loaded screw holds these three stages at proper stiffness. The motion of the sample is tracked by a capacitive encoder with ~3 µm resolution.

The sample holder is driven at these orthogonal directions by means of a slider piezo. A high voltage is given to the two neighbor quadrant couples at the same time with respect to the opposite quadrant couples as seen in Figure 3.10.
Figure 3.9: Orthogonal XY sample positionar, capacitive encoder and pin loaded sample holder

Figure 3.10: XY movement mechanism of the slider piezo. Slider pulses given to the AB-CD electrodes move the sample in X-axis and pulses given to the AD-BC electrodes move the sample in Y-axis

3.1.6 Fine Approach Mechanism

To achieve this, both slider and scanner piezo work reciprocally. First, a specified voltage is given to the scanner piezo and retracted away from the sample (~7 µm for 3” scanner). As a second, a pulse is given to the slider piezo and sample slider approach toward to deflection sensor one step. Finally, scanner piezo approaches through the sample, gradually, Figure 3.11. Meantime, feedback works and checks for the interaction between sample and deflection sensor. If there is no interaction, this process repeats up to finding the interaction. When the
interaction is found, scanner piezo stops at a specified applied voltage value and feedback works.

![Graph](image)

Figure 3.11: Motion of the tube piezo scanner in z direction during fine approach

### 3.2 MFM Alignment Holder

#### 3.2.1 Alignment Holder Design

The LT-AFM/MFM head with a self-aligned cantilever holder mechanism was designed which keeps the XYZ position of the cantilever with respect to fibre within ±5 µm accuracy, as the temperature was changed from 300 K to 30 mK, Figure 3.12.

![Diagram](image)

Figure 3.12: Schematic design of the MFM alignment holder
The self-aligned mechanism is achieved by means of two key points. The first one is alignment chips with compatible cantilevers as seen in Figure 3.13 (from NanoSensors or Applied Nanostructures). There are three protrusions on the chip and three matching groves at the back of the cantilevers. When a new cantilever is placed on this alignment chip, ±3 µm repositioning accuracy is obtained. This would be a great advantage for fibre-cantilever alignment if the alignment is not broken when the temperature is lowered from room temperature.

Figure 3.13: Alignment holder chip and cantilever

We tried several prototypes that they failed when the system was cooled down. The alignment was broken and the interference pattern was lost below 200 K. One of the failed designed is given in Figure 3.14 which was composed of a PCB mounted at the end of the tube scanner piezo holder by means of two M1.6 screws, piezo stack with alignment chip and tilt plane and copper tubing guide for the fibre. Weaknesses of this design were that the fibre and cantilever have two different independent planes and material selection like copper tubing.
Figure 3.14: One of the failed preliminary design of deflection sensor. (a) Top view: A PCB mounted at the end of tube scanner piezo of microscope (b) Side view: stack piezo, alignment chip and tilt plane and cantilever assembly were glued to the each other and on the PCB. A cleaved fibre was guided by bended copper tubing. The fibre-cantilever alignment was failed during cooling down at ~200 K.

At the end of several tries, we achieved true design of the alignment holder that the fibre cantilever alignment sustained for the whole operation range of temperatures. In this design, the material selection was revised and tried to use materials with similar thermal contraction coefficients as in Table 3.3. The body is made of titanium and mounted at the end of the tube scanner piezo two M1.6 screws. The cantilever alignment chip is glued on top of a small piezo stack element which is sandwiched between two alumina plates to get rid of high voltage leaks, and this assembly is aligned with respect to the cleaved fibre end under the optical microscope before everything is glued using a low temperature compatible epoxy. The separation between the back of the cantilever and fibre is arranged to be ~30 μm, which is a secure gap distance for cantilever replacement as shown in Figure 3.15. The cantilever is kept in position by a metal spring. The whole fibre-cantilever assembly is tilted 11° with respect to the sample by means of Ti holder.

![Figure 3.15: (a) Picture for alignment of the cantilever with respect to the ferrule tubing and (b) picture for the reflection of cantilever from sample surface](image)

This self-aligned design works very reliably even after many temperature cycles between 30 mK-300 K. Alignment procedures for the users are eliminated and the usability of the microscopes is greatly improved.
Table 3.3: Materials used in the alignment holder design with their dimensions and thermal contraction coefficients (α)

<table>
<thead>
<tr>
<th>Component</th>
<th>Material</th>
<th>Size XY/OD (mm)</th>
<th>Size Z (mm)</th>
<th>α (10^{-6}/K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fibre piezo</td>
<td>Lead Zirconate Titanium</td>
<td>3 x 3</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td>Ceramic</td>
<td>Alumina, Al₂O₃</td>
<td>3 x 4</td>
<td>0.5</td>
<td>8</td>
</tr>
<tr>
<td>Alignment holder</td>
<td>Titanium</td>
<td>11 x 11</td>
<td>6</td>
<td>8.6</td>
</tr>
<tr>
<td>Alignment chip</td>
<td>Silicon, Cr coated</td>
<td>2.9 x 6.7</td>
<td>0.53</td>
<td>6</td>
</tr>
<tr>
<td>Ferrule tubing</td>
<td>Zirconium</td>
<td>11</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>Fibre</td>
<td>Silica, SiO₂</td>
<td>0.125</td>
<td>7</td>
<td>0.75</td>
</tr>
</tbody>
</table>

3.2.2 Fibre Cable

Standard single mode, 1310 nm wavelength compatible, with FC/APC connector telecom fibre cables (TTAF Inc.-Turkey, OZ Optics-Canada) were used for the experimental setups, Figure 3.16.

![Fibre Cable Image](image)

Figure 3.16: Single mode, 1310 nm wavelength, FC/APC connectorized fibre cable

First, the layers above Ø125 µm were stripped and cleaved using fibre cleaver (Fujikura Inc. Japon). Then, placed into the Ø125 µm ID zirconia ferrule tubing (Kientech Inc., USA) that front part of the ferrule is aligned with respect to the fibre prior to this step. End of the fibre was positioned few microns back inside the ferrule to get rid of any damage and reflectivity loss. The fibre was glued from back part of the ferrule tubing by means of a low temperature
epoxy (Staycast, Oxford Instruments, UK) to fix it and follows the main stainless steel (ss) tubing through the fibre feedthrough on the LEMO head. A sealing mechanism developed at feedthrough which fixes the fibre there and works down to high vacuum level safely.

### 3.2.3 Cantilevers

The cantilevers compatible with alignment chip from NanoSensors were used for the experiments given in Table 3.4. Back sides of the cantilevers are Al coated for enhancing the reflectivity of the laser beam. The MFM cantilevers are obtained with hard magnetic coating on the PPP-FMR cantilevers. PPP-MFMR has ~300 Oe coercivity with ~300 emu/cm³ remanence magnetization. SSS-MFMR has ~125 Oe coercivity with ~80 emu/cm³ remanence magnetization. Therefore, magnetic moment of the SSS type MFM cantilevers are less than PPP ones.

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>Dimensions (LxWxT) (μm)</th>
<th>$f_o$ (kHz)</th>
<th>$k$ (N/m)</th>
<th>Tip Radius of Curvature</th>
</tr>
</thead>
<tbody>
<tr>
<td>PPP-FMR</td>
<td>225x28x3</td>
<td>45-115</td>
<td>0.5-9.5</td>
<td>&lt;10 nm</td>
</tr>
<tr>
<td>PPP-MFMR</td>
<td>225x28x3</td>
<td>45-115</td>
<td>0.5-9.5</td>
<td>&lt;30 nm</td>
</tr>
<tr>
<td>SSS-MFMR</td>
<td>225x28x3</td>
<td>45-115</td>
<td>0.5-9.5</td>
<td>&lt;15 nm</td>
</tr>
</tbody>
</table>

The guaranteed tip radiuses of curvature for PPP-MFMR cantilevers are better than 30 nm whereas tip radiuses of curvature for SSS-MFMR are better than 15 nm, Figure 3.17.

Figure 3.17: SEM image of the Super Sharp Silicon MFM cantilever (SSS-MFMR) which is adopted from Nanosensors [53].
The tip is one of the two critical parameters which determine the MFM resolution with noise floor of the deflection sensor. For enhancing typical the best MFM resolution of ~20 nm, various home-made cantilevers were fabricated and produced in the literature. For example, CoFe coating of carbon nanotubes [63], Fe-filled carbon nanotubes [64], Co tips grown by focus electron beam [65], focus ion beam milled CoPt tips [66], Co spike tips by electron beam induced deposition [67] and paramagnetic material coating on the tip [68] were tried for enhancing the MFM resolution.

3.3 Michelson Fibre Interferometer

3.3.1 Operation Principles and Design

A low noise Michelson type fibre interferometer is designed for measuring the cantilever displacement. This is similar to the Rugar design [46-47] with a couple of tricks to improve the sensitivity [69]. A pigtailed, 1310 nm laser diode, Figure 3.17, is operated at constant power up to 5 mW and coupled into a 2x2, single mode, 50% fibre splitter, as shown in Figure 4.

![Figure 3.18: Power versus wavelength spectrum of the IR pigtailed laser diode](Hi-Optel Technology, HLD-3214-7612F)
Figure 3.19: Schematic design of the Michelson fibre interferometer and the cavity

One of the fibre splitter output is connected to the fibre which goes to the end of cantilever into the LT-AFM/MFM system. The other output is connected to the pigtailed reference photodiode which is used for monitoring the laser power. The input of fibre splitter is connected to the pigtailed signal photodiode. The end of fibre is cleaved using fibre cleaver. Typically, 3% of the light is reflected back from at the end of cleaved fibre. The remaining light exits the fibre and hits the cantilever and then part of it is reflected back into the fibre. These two light beams pass through the fibre splitter and reaches to signal photodiode where they interfere. This interference generates a photocurrent which can be written as:

\[ i = i_0 \left[ 1 - V \cos \left( \frac{4\pi d}{\lambda} \right) \right] \quad (3.3) \]

\[ i_0 = \frac{i_{\text{max}} + i_{\text{min}}}{2} \quad (3.4) \]

\[ V = \frac{i_{\text{max}} - i_{\text{min}}}{i_{\text{max}} + i_{\text{min}}} \quad (3.5) \]
\[ R = \frac{V_{\text{Signal}}/R_{F,\text{Signal}}}{V_{\text{Reference}}/R_{F,\text{Reference}}} \]  

where \( i_0 \), \( V \), \( d \), \( R \) and \( \lambda \) are midpoint current, visibility, cantilever-fibre separation, reflectivity and wavelength of the laser, respectively. The interferometer is at the most sensitive position at the quadrature points, \( d = \lambda/8, 3\lambda/8, 5\lambda/8, \ldots \). The slope of the interference is:

\[ m = \frac{\Delta i}{\Delta d} = 4\pi i_0 \frac{V}{\lambda} \]  

where \( m \) and \( \Delta d \) are slope of the interference pattern and cantilever displacement, respectively. The typical value of the slope for our interferometer is ~4 mV/Å. The quadrature point of the fibre interferometer, where the slope is maximum, is determined by measuring the interference pattern by moving the cantilever with the stack piezo beneath the cantilever. The stack piezo is driven between 0-125 V forward and backward direction, with respect to the fibre. The quadrature point is detected by the software and locked into position during the operation with a subroutine in the software.
Figure 3.20: Typical Michelson fibre interferometer interference pattern and slope. Average power at quadrature is 160 µW, fringe visibility is 0.3. The average interference slope is 4 mV/Å.

3.3.2 Fibre Interferometer Card

Circuit diagram of the fibre interferometer card is given in Figure 3.21. The photocurrent is produced at a pigtailed Signal PD (HiOptel Technologies Inc.,) [70] and the current is converted to voltage at the I-V converter which is comprised of operational amplifier and R_F feedback resistor. The gain bandwidth product (GBP) of the OPAMP is 8 MHz and compatible with standard ~75 kHz MFM cantilevers. The converted signal can be multiplied (x1, 2, 4, 8) by the software using PGA gain control. In the last part of the circuit, the signal is multiplied x10 by the last pre-amplifier in the circuit. At the end, the V_PD output is used for as a deflection signal in the system and feeds the Phase Lock Loop (PLL). The card has 3 BNC outputs called Reference, Signal and V_PD. The reference and Signal outputs are used for monitoring.

![Circuit diagram of the fibre interferometer card. The V_PD output signal is used for as a deflection signal.](image)

Picture of the fibre interferometer card is given in Figure 3.22. There is a FC/APC connector at the front panel which is used for plugging a FC/APC single mode fibre cable to carry the light into the LT- AFM/MFM. 3 BNCs on the front panel are called Signal, Output and Reference. The Output BNC is the final signal obtained from fibre interferometer and feeds PLL for measurement.
3.3.3 Voltage Controlled Oscillator and RF Injection

Besides intrinsic noises of the laser diode, there are two major noise sources: optical feedback noise and optical interference noise. Optical feedback noise is originated by reflections and scatterings of the beams inside optical resonator of the laser diode which causes additional oscillation mode. Optical interference noise is originated by the incident of scattered and reflected beams on the PD that they follow unexpected paths and produce additional unstable signal [71].

Figure 3.23: Oscilloscope signal of the fibre interferometer V_PD output signal: (a) RF off and (b) RF on. Timescale is 5 ms.
Injection of RF current into the laser diode about 300 MHz both broadens the linewidth and changes the oscillation mode of the laser diode from single to multimode which suppress these two noises [72, 73]. A voltage controlled oscillator (VCO) is used for RF injection to the circuit. Indeed, RF injection reduces the noise more than an order of magnitude as seen oscilloscope signal in Figure 3.23.

3.4 LT-AFM/MFM Controller and Software

A schematic diagram of control mechanism of the microscope is given in Figure 3.23. A dedicated LT-AFM/MFM controller electronics and software from NanoMagnetics Instruments Ltd [74] were used. The deflection signal is measured by means of the fibre interferometer [74] and given to the digital Phase Lock Loop (PLL) [74] which excites the cantilever at the resonance frequency and measure the frequency shift, phase and amplitude change. Either frequency shift or amplitude change is used for a feedback which is operated by the controller. The scan and coarse approach mechanism is also managed by the controller. All the functions are controlled and adjusted by the C# based software.

![Schematic diagram of the LT-AFM/MFM control mechanism](image)

Figure 3.24: Schematic diagram of the LT-AFM/MFM control mechanism
The LT-AFM/MFM controller contains a very low noise power supply unit for related modular cards. It has four channels of low noise high voltage amplifiers to drive scan piezo to ±200 V swings. This card is driven by 3 channels of XYZ 24Bit digital to analog converters. The feedback signal is digitized with 16 Bits ADC at 250 kHz at the digital feedback controller card. A Digital PID loop is operated at 250 kHz for the feedback, which gives analog 24 Bits signals to drive the Z position and 32 Bits digital output for the software. A sample slider card produces exponential pulses up to 380 V for the stick-slip coarse approach mechanism of the sample slider as well as XY sample positioning with adjustable step size.

![Figure 3.25: LT AFM/MFM controller electronics which is comprising of modular cards](image)

### 3.5 Vibration Isolation

Vibration issue is one of the critical points of SPM measurements. Therefore, all the vibration sources in the laboratory environment have to be considered carefully. For example, the floor of laboratory where the cryostat is located, pumping lines, cryocooler on the cryostat for dry systems, nitrogen jacket for wet systems, connection lines or other instruments which creates vibration in the same place. During the research, we used various cryostat systems of both dry and wet cryostats from various vendors. For wet systems, isolation is rather easy and there is no pumps connected to the cryostat.

Dry systems are popular recently because of the unprecedented increase in liquid helium price in the last decade. A pulse tube cryocooler is integrated onto the cryostat for these systems and liquefy the gas Helium and circulate it continuously. The drawback of this cryostat is that few tens of nanometer of vibration is created by the pulse tube and directly coupling to the microscope during measurement. We designed a vibration isolation stage specially and decoupled the pulse tube vibrations from the microscope to solve this problem, in Figure 3.26. In our design, the microscope sits on the vibration isolation table which is comprised of three air damped isolation legs [75] and a massive stainless steel plate (~300 kg).
The vibration table is connected to the cryostat by an edge welded bellow which is very soft and absorbs the vibration coming through the pulse tube. All the staff of the stage is made of non-magnetic materials for high field applications. In addition to that, the scroll pump connection line to the cryostat, which is used for the circulation of the Helium gas, was passed through a massive concrete block (~125 kg). The concrete block eliminates vibration coming through the pumping line in a good way.

To check the performance of vibration isolation stage, the vibration spectrum was measured by means of an accelerometer (Wilcoxon Research, Meggitt 731A, US) and Rohde Schwarz spectrum analyzer. The stage definitely eliminated the vibrations especially below 300 Hz as seen in Figure 3.27. The stage decreased both the amplitude of specified noises like 47 Hz and 95 Hz and noise floor about two orders of magnitude.

At the end, we obtained better than 4 Å noise on the atomically flat HOPG sample when all the vibration sources were on. This is enough for most of the MFM applications using force microscopy except that atomic resolution imaging or manipulation. For further performance,
the dry systems require further design parameters in engineering part like removing the pulse tube from top of the cryostat dewar and place it apart.

Figure 3.27: Spectral noise density of the vibration measured by means of an accelerometer
CHAPTER 4

4.1 Low Temperature Tests of the Self-Aligned Mechanism

When the microscope was cooled down from 300 K to 300 mK, the thermal contractions in the z-direction could be easily measured, directly by the fibre interferometer. During the cool down process, typically 6 period ($\lambda/2$, $\lambda$ is 1310 nm) shifts were measured in the interference pattern, which corresponds to ~3.93 µm displacements in z-direction. The shift is in opposite direction during cooling and warming up process.

For the XY drift, a simple calculation is shown below to find the geometric spot size of the laser beam. The core diameter of the fibre is 10 µm with a nominal numerical aperture (NA) value of 0.14 and the nominal width of the cantilevers is 28 µm. To calculate the spot size of the fibre, $\theta$ has to be calculated in Figure 4.1 regarding the equation:

$$NA = n \sin \theta$$  \hspace{1cm} (4.1)

where $n$ is the refractive index of the medium, the numerical value is equal to 1 for air and $\theta$ is the half-angle of the maximum cone of the light.

![Figure 4.1: Laser spot size calculation falls on the cantilever](image)

As shown in Figure 4.2, the spot size which falls onto the back of the cantilever is calculated to be 19 µm, for the initial separation of 30 µm. This cavity gap is necessary for the secure cantilever replacement. The typical XY drift was calculated to be less than 5 µm for the entire temperature range of 300 K to 30 mK. In Figure 4.3, two interference pattern recorded at 300 K and 4 K is seen. The maximum power and the average power were similar at these two different temperatures which shows sustainability of the alignment at low temperatures.
Figure 4.2: Spot size diameter versus cavity gap graph which shows increase in spot size diameter with increasing cavity gap.
Figure 4.3: Cantilever-tip alignment sustains for the all operation range of the temperature: (a) 300 K, (b) 4 K

The numbers of patterns seen in Figure 4.3 are different at low temperatures. When the temperature is cooled down from 300 K to 4 K, the capacitance of the piezo is also decreased from ~85 nF to ~15 nF. The capacitance is proportional with the piezo displacement. Therefore, at 4 K the piezo displacement at 125 V$_{DC}$ is about 400 nm as seen in Figure 4.3(b).

![Figure 4.4: Capacitance change of the fibre stack piezo (PI Inc., Germany, PL033.31) via temperature.](image)

### 4.2 Noise Analysis and Measurements

Various noise sources contributing to the fibre interferometer deflection sensor were summarized in Figure 4.5. The noise sources can be divided into the three main categories following way of the light from its source, light to current conversion at the photo detector and current to voltage conversion at the pre-amplifiers which is used for as a deflection signal. Each electronic component contributes such a factor of noise in the system.

The categories are laser noise, shot noise and electrical noise, respectively and discussed in the following.
4.2.1 Laser Noise

Laser diodes have many intrinsic noise sources like mode-hopping, mode-partition, 1/f and spontaneous emission that they can be grouped into the laser intensity noise. Laser intensity noise for a typical laser is described as [76]:

\[
\overline{v^2}_{\text{intensity}} = S_{PD}^2 P^2 R_F^2 \times 10^{-13} \left( \frac{V^2}{Hz} \right) \quad (4.2)
\]

where \( S_{PD} \), \( P \) and \( R_F \) are responsivity of the photo detector, incident optical power and the feedback resistor in the photo detector I-V converter in Figure 3.20.

Laser diode has a finite coherence length that is the propagation distance over which a laser wave maintains a specified degree of coherence. This causes the laser phase noise which is also dependent on the cavity gap between the fibre and cantilever. Laser phase noise can be described as:

\[
\overline{v^2}_{\text{phase}} = S_{PD}^2 P^2 R_F^2 4\pi \Delta v \tau^2 \times 10^{-13} \left( \frac{V^2}{Hz} \right) \quad (4.3)
\]

where \( \Delta v \) is the linewidth of the laser diode and \( \tau \) is the optical path length difference of the cavity gap and divided by the speed of light.
4.2.2 Shot Noise

Laser shot noise is a kind of electronic noise that occurs when the finite number of photons that carry energy is small enough to give rise to detectable statistical fluctuations in a measurement. When photons strike on the photo diode, a photo current, \(i\), is generated on the photo diode which is proportional to the optic power, \(P\), fall onto it:

\[ i = S_{PD}P \]  

(4.4)

The average root-mean squared variation of the current noise is given by:

\[ \bar{i}_{\text{shot}} = \sqrt{2eS_{PD}P} \left( \frac{A}{\sqrt{\text{Hz}}} \right) \]  

(4.5)

where \(e\) is the electronic charge, \(1.6\times10^{-19}\) C and \(S_{PD}\) is the responsivity of the photo detector, \(~1\ A/W\). If this current passes through a resistor, \(R_F\), in the I-V converter circuit the average root-mean squared variation of the voltage noise is given by:

\[ \bar{v}_{\text{shot}} = \sqrt{2eS_{PD}PR_F} \left( \frac{V}{\sqrt{\text{Hz}}} \right) \]  

(4.6)

4.2.3 Electrical Noise

There are three components of the electrical noise in the circuit described in Figure 4.5. They are discussed in the following:

4.2.3.1 Johnson Noise

Johnson noise is resulted from thermal interaction between free electrons and vibrating ions in the resistor, \(R_F\). The noise is described to the Gaussian distribution statistics and average mean-squared variation of the equation is given by:

\[ \bar{v}_{\text{Johnson}} = \sqrt{4k_BT R_F} \left( \frac{V}{\sqrt{\text{Hz}}} \right) \]  

(4.7)

where \(k_B\) is Boltzmann’s constant, \(1.38\times10^{-23}\) J/K and \(T\) is the temperature. \(R_F\) is the feedback resistance in I-V converter circuit where photocurrent passes through on it.

4.2.3.2 Voltage & Current Noise

Voltage and current noises are associated with intrinsic properties of the operational amplifiers that used in the circuit of the fibre interferometer. The operational amplifiers we
used in the circuit and nominal noise values are given in the datasheet. Average root-mean squared variation value of voltage noise and current noises are $3 \times 10^{-9}$ nV/√Hz and $4 \times 10^{-13}$ A/√Hz, respectively.

### 4.2.4 Total Noise

The numerical contributions of these noises discussed above mentioned are calculated as seen in Table 4.1. Summation of the noises is calculated using square root of the sum of the average mean squared values of the individual sources. If the individual sources are $n_{1,\text{rms}}$, $n_{2,\text{rms}}$, $n_{3,\text{rms}}$, ..., the total noise, $N_{\text{Total, rms}}$, is calculated using the equation:

$$N_{\text{Total, rms}} = \sqrt{n_{1,\text{rms}}^2 + n_{2,\text{rms}}^2 + n_{3,\text{rms}}^2 + \cdots}$$

(4.8)

**Table 4.1:** Numerical values of the noise components. The interference slope value is 5.6 mV/Å and the average incident power is 229 µW at 4 K.

<table>
<thead>
<tr>
<th>Noise Source</th>
<th>Noise (V/√Hz)</th>
<th>Noise (fm/√Hz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrical Noise</td>
<td>$2.9 \times 10^{-7}$</td>
<td>5.2</td>
</tr>
<tr>
<td>Shot Noise</td>
<td>$4.4 \times 10^{-7}$</td>
<td>7.8</td>
</tr>
<tr>
<td>Laser Noise</td>
<td>$2.4 \times 10^{-7}$</td>
<td>4.3</td>
</tr>
<tr>
<td><strong>Calculated Total Noise</strong></td>
<td>$5.8 \times 10^{-7}$</td>
<td>10.4</td>
</tr>
<tr>
<td><strong>Measured Total Noise</strong></td>
<td>$\sim 6.1 \times 10^{-7}$</td>
<td>$\sim 11.0$</td>
</tr>
<tr>
<td>Measured/Calculated Deviation</td>
<td></td>
<td>$\sim 5%$</td>
</tr>
</tbody>
</table>

The total noise calculated to be $10.4 \ \text{fm/√Hz}$ and the shot noise is dominating the total noise as seen in Figure 4.6. Therefore, sensitivity of the deflection sensor is limited by the shot noise which is calculated to be $7.8 \ \text{fm/√Hz}$. The shot noise of the interferometer can be decreased further, if the incident power on the photo detector is increased which is another research topic handled in the thesis. There is about 5% deviation between calculated and measured noise values and compatible each other.
4.2.5 Noise Measurements

The spectral noise density of deflection sensor, which is the thermal noise of the cantilever including the other noise sources discussed above, is measured between 300 K and 4 K. The noise is measured utilizing a Rohde-Schwarz spectrum analyzer when the fibre interferometer was at quadrature point and both the dither and the scanner piezo were grounded. The noise measured at both 300 K and 4 K is given in Figure 4.7 and Figure 4.8, respectively. They were measured to be ~20 fm/√Hz at 300 K and ~11 fm/√Hz at 4 K.

The spectral noise density, $S$, is also calculated according the equation and fitted to the experimental data [77]:

$$S = \sqrt{\frac{2k_B T}{\pi f_0 Q} \left( \frac{1}{\left(1 - \frac{f}{f_0}\right)^2 + \left(\frac{f}{f_0 Q}\right)^2} \right)}$$

(4.9)

where $f_0$, $f$, $T$, $Q$, $k$, and $k_B$ are resonance frequency, frequency, temperature, Q-factor, spring constant of the cantilever and Boltzmann constant, respectively. The cantilevers were MFM cantilevers from NanoSensors Inc. (PPP-MFMR) with nominal spring constant of 1.9 N/m. In theoretical fits, spring constant of the cantilever were calculated to be 1.5 N/m at both temperature of 300 K and 4 K. Spring constant calculation using thermal noise spectrum can
be used as an alternative method which is much accurate than the most common used Sader method [78].

Figure 4.7: Spectral noise density of the fibre interferometer at 300 K. Theoretical calculation was also fit into the graph where \( f_0 \) is 60,975 Hz, Q-factor is 300 and \( k \) is 1.5 N/m
Figure 4.8: Spectral noise density of the fibre interferometer at 4 K. Theoretical calculation was also fit into the graph where $f_0$ is 61,590 Hz, Q-factor is 1,000 and $k$ is 1.5 N/m. The shot noise is calculated to be 7.8 fm/$\sqrt{\text{Hz}}$.

Noise of the deflection sensor was also analyzed with respect to the laser power as seen in Figure 4.9. When the laser power was decreased, the noise level of the interferometer was increased dramatically an order of magnitude because of increase in the shot noise. The optimum operation power was determined at or above 3 mW that we used this value for the all noise measurements and operations.

![Image of Laser Power vs Noise Graph]

Figure 4.9: Laser power versus noise graph recorded at 77 K. The tendency of noise is decreasing with the increasing laser power.

4.3 Images

4.3.1 AFM Images

4.3.1.1 Contact Mode AFM

When the tip is brought into the repulsive force regime as seen in Lennard-Jones potential graph in Figure 2.5, the cantilever is bended by means of repulsive short range forces. The bending of the cantilever is measured by the fibre interferometer as a normal force which is used for feedback, too. A force versus distance curve is given in Figure 4.10 in which pink line represents approach to the sample and blue one represents retract back. When the tip was beyond 40 nm piezo displacement position, there was no interaction between tip and sample.
Below 40 nm piezo position, the attractive forces appear first and signal shows deflection in positive direction. Then, the tip was continued to approach toward the sample, the direction of the signal was shifted in reverse direction by means of the repulsive forces. The set point of the feedback is chosen from on this graph for contact mode AFM in the linear region. If the spring constant of cantilever is calculated prior to this measurement, the exerted force can also be calculated.

![Graph](image)

**Figure 4.10**: Force versus distance curve on the sample for deciding set point of the normal force for feedback. Pink line shows the tip motion towards the sample and blue line shows retract back.

In Figure 4.11, a contact mode image of the bluray disk sample is given which was obtained at 4.2 K in liquid Helium dewar at the end of manual cool down process. Figure 4.11(a) shows data tracks that they are produced by the bluray laser burn process on the polymer film. Figure 4.11(b) shows error signal in the feedback, that’s normal force measured. Bluray disks are also good calibration sample since they have 320 nm standard distances between two adjacent bit lines.

Contact mode is one of the crucial modes of the microscope since it is not only the basic mode of topography imaging; it is also used for piezo response force microscope (PrFM) and conductive AFM measurements. We have also developed these two modes for increasing capability of the microscope.
Figure 4.11: Contact mode AFM image of the bluray disk sample at 4.2 K. (a) Topography image and (b) normal force (feedback) image. Scan area was 4 µm x 4 µm with scan speed of 0.5 µm/s. Set normal force was 0.5 V for feedback.

4.3.1.2 Tapping Mode AFM

Tapping mode [12] is the most common and used AFM mode in practical that theory of the operation was described in the operation principles. Besides the topography image, tapping mode AFM provides phase image which gives information about material composition of the specimen, hardness, either charge or magnetic field distribution in specified measurements [13]. In this mode, the cantilever is oscillated by the dither piezo in its resonance frequency with constant excitation. Oscillation amplitude of the cantilever is used for as the feedback signal. Approximately, half of the free oscillation amplitude is used for feedback set point, which gives good results for imaging most of the time. Tapping mode is also the basic mode of the MFM measurements in which during forward scan topography is imaged in the same principle and for backward scan phase shift gives magnetic image.

A couple of examples are given about tapping mode images in the following. First, a mica sample was imaged to observe the atomic terraces and to demonstrate the performance of the microscope as seen in Figure 4.12(a). Prior to the experiment, the mica sample was cleaved using scotch tape and etched in 48% HF solution (Merck, Germany) for four hours. The sample was then washed with deionised water and dried with dry nitrogen. Atomic terraces of mica were imaged (~10 Å) at various temperatures in tapping mode AFM as shown in Figure 4.12(b).
Figure 4.12: (a) Tapping mode AFM image on HF etched mica sample recorded at 77 K. (b) Cross-section on the image shows atomic steps. Scan size is 3 µm x 1.75 µm with scan speed of 1 µm/s.

In the second example, Figure 4.13, tapping mode AFM image of ZnO sputtered thin film sample is given which is used for solar cell applications. AFM explores topography structure of the sample, grain size and average roughness that they are crucial parameters for thin film researches. In Figure 4.13(b) shows the phase image of the sample recorded at 77 K.
Figure 4.13: Tapping mode AFM image of the sputtered ZnO thin film at 77 K: (a) topography image and (b) phase image. Grains structure of the film shows sub-50 nm grain sizes. Scan area is 1 µm x 1 µm with scan speed of 0.2 µm/s.

4.3.2 MFM Images

4.3.2.1 Lift-Height Adjustment

Adjusting lift-height parameter during backward scan plays a crucial role obtaining a good MFM image. If the lift-amount is not enough as seen Figure 4.14(a), the topographic futures can be seen in the magnetic image since the tip is still under the influence of the short range forces besides the long range magnetic forces. Therefore, the lift amount was not enough and has to be increased to get rid of the mixture of topography and magnetic image.

In Figure 4.14(b), there are no topographic artifacts in the image and lift-off value seems ideal for this scan. In addition, the resolution of bit series in the image is good, distinguishable and clear. When the lift amount was gradually increased, the resolution went to worse that some of the bit sequences are indistinguishable since magnetic stray field strength decreases with $1/r^3$. Moreover, with the increasing lift-height, the interaction force is also decreasing as expected that the phase shift was decreased about 70° to 8° from image a to d.

Average roughness of the sample topography plays a key role for deciding the optimum lift-off amount. The rough sample topographies sometimes set for decreasing lift-off amount has a drawback to get high resolution MFM images.
Figure 4.14: MFM image of Samsung 80 GB/platter hard disk at 77 K and adjusting lift-height parameter which was increased gradually: (a) 35 nm, (b) 50 nm, (c) 80 nm and (d) 100 nm

4.3.2.2 Tip Magnetization Direction

In MFM, a magnetic contrast between a tip and a sample is recorded as an image. If the magnetization direction of the tip (M) is known, direction of the stray field emanating from the sample can also be determined. For example, as seen in Figure 4.15, the contrast mechanism is vice versa following from the topographic artifact on the sample surface that green arrow indicate as a reference point on the sample surface. The contrast of the bit along the yellow arrow is vice versa. In Figure 4.15(a), the tip was magnetized along +z direction
with the saturation field of 450 Oe at 300 K and the force was attractive. In Figure 4.15(b),
the tip was magnetized along $-z$ direction by 450 Oe and the force was repulsive. 
Demagnetization field of the sample is several kOe and field direction of the sample did not 
change during this experiment.

Figure 4.15: MFM image of Maxtor 81750A4 Hard Disk at 300 K and demonstration of tip 
magnetization direction. (a) Tip is magnetized with $+450$ Oe (b) Tip is magnetized with $-450$ 
Oe. The green arrow indicates the topographic spot and the yellow arrow sits on the bit that 
the direction is vice versa. Scan area is 15 µm x 15 µm with scan speed of 10 µm/s. Lift-off 
amount is 90 nm.

In Figure 4.16, a similar experiment was performed on single image at 300 K. During scan, 
the field was swept opposite directions hence the magnetization direction of the tip was 
changed. Therefore, the contrast mechanism was changed as seen in clearly in rectangle. The
stray field of the sample is the same but the tip different. Therefore, the contrast is different. This step is crucial for MFM experiments for making comments on the sample magnetization regarding the applied magnetic field.

Figure 4.16: Changing the tip magnetization direction during scan at 300 K on a hard disk sample (80 GB/platter). Scan area is 15 µm x 15 µm with scan speed of 1 Hz. Lift-height amount is 160 nm.

4.3.2.3 Tip Magnetization and Saturation Field

Magnetic coating properties of a tip like soft coating, hard coating or coating thickness are also important for applications and the magnetic properties of the sample has to be taken into account which will be investigated. For example, a hard coating and high magnetic moment of a tip may change the soft sample magnetization. The room temperature nominal magnetization or saturation fields of the cantilevers are provided by vendors. The magnetization of the cantilevers may diminish in time regarding environmental conditions. Therefore, a cantilever has to be magnetized properly prior to experiment. For low temperature systems, it is rather easy since the superconducting magnet can do this function properly. In Figure 4.17, a typical example was recorded at 4 K is given. The experiment was started with an unused of the selves cantilever and at bottom unmagnetized section of the image the magnetization of the tip was so poor and the contrast was very low. Hence, the interaction force was very low. When the field was swept to 400 Oe, the contrast was getting better but the tip was not magnetized completely. Then, the field was raised to 750 Oe, the contrast was much better and the tip was magnetized fully.
Figure 4.17: (a) Magnetization of PPP-MFMR cantilever and role in MFM contrast mechanism at 4 K. Scan area is 9 μm x 9 μm with scan speed of 1 Hz. Lift-height amount is 140 nm. (b) Cross-section on the image which shows phase shift variation at each step.

### 4.3.2.4 Erasing a Hard Disk in a High Field

The magnetization starts changing with few hundred Oe for hard PPP-MFMR and 400 Oe is enough to saturate the tip magnetization at 300 K. At low temperatures, this value is getting higher as seen in Figure 4.18 since the spins are much more stable at low temperature.
Figure 4.18: Erasing of a hard disk by an external magnetic field at 300 K. The field values were: (a) –1 kOe, (b) –2.5 kOe, (c) –4 kOe, (d) –5 kOe, (e) –6 kOe, (f) –7.5 kOe. Scan area was 15 µm x 15 µm with scan speed of 10 µm/s. Lift-off amount was 90 nm.

When the field is continued to increase above saturation field of the tip, the magnetization of the sample starts changing. In Figure 4.14, the field was swept from zero to 7.5 kOe gradually and the hard disk sample was imaged in MFM mode at each step. At the field of 5 kOe, the magnetization was started to change and direction of the spins initiated to changing randomly. At the field of 7.5 kOe, all spins shows random orientation and the pre-magnetization was collapsed as seen from magnetic images.

4.3.2.5 High Resolution Magnetic Imaging

The ultimate performance of the LT-MFM was demonstrated on the high density perpendicular recording media, Seagate Momentus 5400.6 hard disk with 398 Gbpsi as shown in Figure 4.19(a) at 77 K in Helium exchange gas environment. Super sharp MFM cantilevers were used (SSS-MFMR, NanoSensors Inc.) for MFM imaging. Typical oscillation amplitudes between 10 to 15 nm were employed with lift-off amount of 15-30 nm to achieve this resolution. The cross-section on the bit sequence is shown in Figure 4.19(b) which routinely gives 10 nm MFM resolution.
Figure 4.19: (a) MFM image of a perpendicular recording medium, Seagate Momentus 5400.6 hard disk with 398 Gbpsi density, at 77 K and (b) a cross-section on the image with full width half maximum (FWHM) analysis

4.3.3 Vortex Imaging on Superconductors

After liquefying Helium, Heike Kamerlingh Onnes measured the resistance of solid mercury in 1911 [79]. He observed that at the temperature 4.2 K, the resistance of solid mercury abruptly disappeared. It was the discovery of superconductivity and he got the Nobel Prize in 1913. Below certain temperature level, which is called ‘critical temperature, T_c’, the resistance of some materials disappears. In the following years, the resistivities of many materials were measured via temperature to see their critical temperatures.

In 1933, Meissner and Ochsenfeld discovered other unique property of superconductors. When they cooled down a superconductor material below critical temperature in the presence of applied external magnetic field, they observed that these materials cancelled all magnetic fields inside [80]. Superconducting materials are perfect diamagnetic materials and expelling magnetic field, Figure 4.20. A screening current is produced on the superconducting surface for cancelling the field inside.

Superconducting materials are divided into two categories: Type I and Type II. Type I superconductors are elemental materials and their T_c very is low and close to the boiling temperature of helium. Both the critical current, J_c, and the critical field, H_c, amounts are very limited that destroy superconductivity, Figure 4.21. Both restrict their technological applications.
Type II superconductors are compound based materials and have higher critical temperature with higher critical current and magnetic field. Their discovery has a breakthrough in superconductivity [81]. They have two states: Meissner state like Type I and vortex or mixed state, Figure 14. In vortex state, magnetic field can penetrate through the superconductor at some regions and remaining parts are still superconductor and expel the magnetic fields. For Type II superconductors, both the critical current and field are very high which enable these materials to be used for technological applications.

Figure 4.21: Temperature versus magnetic field relation for two types of the superconductors.

Type II superconductors have vortex (or mixed) state.

Penetration of the external magnetic field through the superconductor is seen in Figure 4.22. They are a vortex of supercurrent in a Type II superconductor. The theoretical prediction of
these vortices was done by A. A. Abrikosov, in 1957 [82] and called Abrikosov vortices. The first time, this prediction was shown by U. Essmann and H. Trauble experimentally using Bitter decoration technique on lead indium rod at 1.1 K [83]. Moser et. al. also imaged these vortices on YBCO thin film using a home-made LT-MFM system [17].

Exploration of the vortex structures on superconductor has great importance in physics. LT-MFM is one of the powerful tools used for this investigation. Therefore, we imaged the vortices on two high temperature superconductor samples to show the applicability of our instrument on this research topic.

Figure 4.22: Type II superconductor (SC) and penetration of magnetic field through the superconductor. Schematic view (a) from top and (b) from side

4.3.3.1 BSCCO Single Crystal

Abrikosov vortex lattice in BSCCO(2212) single crystal [84] were also imaged at 4.5 K. Prior to the experiment, the sample was cleaved using scotch tape as seen in Figure 4.23 and the cantilever (PPP-MFM, NanoSensors Inc.) was magnetized in the cryostat at 300 K. The sample was cooled down to 4.5 K under the magnetic field of +40 Oe that field direction was the same with tip magnetization direction. BSCCO is type-II superconductor and the field was trapped in the vortices as seen in Figure 4.24(a).
Figure 4.23: Pictures of the cleaved BSCCO single crystal glued on the sample holder using silver epoxy

The vortices form perfect hexagonal honeycomb structures in the sample and each vortex carries a single flux quantum, $\Phi_0$, which has a value of:

$$\Phi_0 = \frac{h}{2e} = 2.07 \times 10^{-15} \, (Tm^2)$$  \hspace{1cm} (4.10)

where $h$ and $e$ are Planck constant ($6.63 \times 10^{-34} \, J.s$) and electron charge ($1.6 \times 10^{-19} \, C$), respectively. The vortex spacing between two adjacent cores was measured $\sim 765$ nm and the phase difference was measured to be $\sim 2^\circ$ as seen in Figure 4.24(b). The vortex spacing, $a$, can be calculated for the triangular unit cell according to the equation below:

$$a = \frac{2\Phi_0}{\sqrt{3}H}$$  \hspace{1cm} (4.11)

where $H$ is applied external magnetic field for cool down. For our case of 40 Oe applied magnetic field, the vortex spacing calculated to be 773 nm which was compatible with measured value. When the field is increased, the vortices are getting closer and the vortex density is increased. In the next step, the sample was cooled down in an opposite field of $-160$ Oe with respect to the tip. The vortex cores appear as bright spots as shown in Figure 4.24(c) and getting closer as expected. The average vortex spacing was measured to be $\sim 375$ nm, Figure 4.24(d) which was also close to calculated value of 387 nm. The slight discrepancy in vortex spacing could be caused by both remnant field in the superconducting magnet and error in the scanner calibration. In Figure 4.24(c), another part of the sample was imaged and it was seen that the vortices were pinned along the atomic terrace of the sample since surface...
defects behave as pinning centers. The images were obtained in constant height mode with 65 nm lift height.

Figure 4.24: MFM images of Abrikosov vortices in BSCCO single crystal at 4.5 K. (a) The sample was cooled down under +40 Oe external magnetic field. Scan size is 15 µm x 15 µm with scan speed of 10 µm/s. (b) Cross-section on the image a, (c) the sample was cooled down under –160 Oe external magnetic field. Scan size is 6.7 µm x 6.7 µm with scan speed of 4 µm/s and (d) cross-section on the image c. Tip was magnetized in the positive field direction at 300 K.

4.3.3.2 YBCO Thin Film

Vortex imaging and superconductivity of YBCO films have been studied extensively by several research groups using homemade LT-MFM systems in literature [17, 21, 24]. We also studied on YBCO films [85]. The topography of the YBCO film was obtained in semi-contact
mode AFM at ambient conditions, Figure 4.25. The average roughness on the sample is 67 nm which shows poor quality of the film.

![Topography image of the YBCO film at 300 K](image)

Figure 4.25: Topography image of the YBCO film at 300 K which shows granular structure of the film. Scan area is 9 µm x 9 µm with the scan speed of 1 Hz.

The explored granular structure in topography may pins the vortices. They are called pancake style vortices with weak interlayer coupling [4]. Even if on this very rough film, we could achieve to see some pancake vortex bundles on different regions at the end of many attempts.

![MFM image and vortices in YBCO HTS film at 50 K](image)

Figure 4.26: MFM image and vortices in YBCO HTS film at 50 K. The sample is cooled down with the field +9 Oe with respect to the magnetization direction of the tip. 3D of a single vortex is also shown. Lift-off amount was 150 nm.
CHAPTER 5

5.1 Introduction to milliKelvin AFM/MFM (mK-AFM/MFM)

Operating a microscope system below liquid Helium limits (<1.5 K) carries critical importance for physics and material science in which there are many interesting problems below 1 K like heavy Fermion superconductivity, magnetic phase transition and spin-glass structures. Therefore, a milliKelvin AFM/MFM system would be very useful for understanding and exploring concepts of these scientific topics.

The cryostat systems, which cools below liquid He limits and reaches ~300 mK, are called $^3$He cryostats discussed in the following sections. The work done in this section was performed with collaboration with Prof. Rui Rui Du from Rice University, Department of Astronomy and Physics. We duplicate one of the $^3$He insert from there and integrated LT-AFM/MFM head onto it here and performed the tests at Rice University.

The results that we obtained in this chapter were published in Review of Scientific Instruments [86].

5.2 Cryogenics and $^3$He as a Cryogen

Various cryogens were invented and have been using in the cryogenic history of science. In Figure 5.1, different cryogens with their average minimum temperature levels one can reach for cooling are shown.
Figure 5.1: Cryogens with minimum temperature levels that can be reached

Liquid nitrogen, which has 77 K boiling temperature, is the most abundant cryogen since it is obtained from compressed air. The temperature level which can be succeeded using liquid nitrogen is around 60 K if the vapor pressure is decreased in a closed system using a pump. Helium ($^4\text{He}$) is the most exiting cryogen with boiling temperature of 4.2 K and shows interesting physical properties. When the vapor pressure is decreased by pumping, one can reach to ~1.5 K base temperature level using liquid $^4\text{He}$. An isotope of helium, $^3\text{He}$, which has one neutron missing, boils at 3.19 K. If vapor pressure of the liquid $^3\text{He}$ is decreased, one can reach the temperature level of 300 mK.

To cool a sample down to 300 mK temperature range, rather a complicated cryostat systems called $^3\text{He}$ cryostats have to be used. They used two cryogens liquid Helium and $^3\text{He}$ in a sequence. First, the cryostat system uses liquid Helium to cool the sample to ~1.5 K, than; $^3\text{He}$ gas is introduced into the system which liquefies at 3.19 K. In high vacuum space of the cryostat, the temperature drops below 300 mK at the end of this sequence.

5.2.1 $^3\text{He}$ Cryostat System

A $^3\text{He}$ cryostat system from Oxford Instruments (Heliox TL) is used for this work. The cooling power of the cryostat is 0.5 mW. The schematic design of the cryostat system is seen in Figure 5.2. This is a top loading (TL) cryostat and the $^3\text{He}$ dump is placed outside of the $^3\text{He}$ insert as a separate unit. 9 Tesla superconducting magnet is integrated into the cryostat.

The variable temperature insert (VTI) of the cryostat is composed of two sections: 4 K pot and 1 K pot. These pots are named regarding the read temperature values when the sample space reaches 300 mK. At 4 K pot, there is a spring loaded heat transfer unit which is in a physical contact when the mK-MFM insert is put inside the VTI and cools down that part of the system to ~4 K using cooling power of the helium. 1 K pot is placed beneath 4 K pot which is in a conical shape and this part is in a physical contact with the 1 K pot of the mK-MFM insert, too. A sorption pump, which is made of charcoal, is placed next to 4 K pot. Therefore, the temperature is similar to 4 K pot. This charcoal either absorbs or releases $^3\text{He}$ regarding its temperature. If its temperature is lowered, it absorbs $^3\text{He}$ atoms and holds them inside. If the temperature is raised up by means of an integrated heater into the charcoal, it releases $^3\text{He}$ atoms through outside. At the bottom of the VTI below 1 K pot, sample space is placed at the center of the magnet. The inner diameter of the sample space 27 mm at most.
A gate valve is put at the top of the VTI to isolate the system from atmosphere all the time because of the existence of $^3$He atoms in VTI all the time. Prior to placing insert into the VTI, the insert is pumped out down to $\sim 4 \times 10^{-5}$ mbar vacuum level. Then, gate valve is opened and mK-MFM is sliding down by means of Wilson seal which is integrated to the mK-MFM insert, Figure 5.3(a).

Figure 5.2: Schematic design of the $^3$He cryostat
$^3$He is used for as an exchange gas to cool down the microscope. The microscope is lowered inside the VTI gradually since we obey 3 K/min cooling rate to get rid of quartz tubing cracks on the head. At the end, both 4 K and 1 K pots of the insert sit the pots in VTI. At this step liquid Helium is used for to cool down the insert so far. At the equilibrium state, the charcoal temperature is raised up 70 K and $^3$He is introduced to the system. First, $^3$He atoms pass 4 K pot and cooled. Then cold atoms reach 1 K pot and condensation starts there. Condensed $^3$He is accumulated at the bottom of the sample space and the temperature is lowered down to 300 mK.

The cryostat is placed on an air cushion table to eliminate vibrations. The pumping lines was extended and put inside a sand box to get rid of vibrations coming from mechanical pumping station. The dewar has a nitrogen jacket to increase the hold time of the liquid helium. The boiling of liquid nitrogen also causes some vibration but this is not so vital for our MFM experiments.

5.2.2 $^3$He Insert and Integration of AFM/MFM Head

The insert for the $^3$He cryostat is designed to minimize heat load to the microscope as shown in Figure 5.3(a). mK-MFM insert is composed of the units from top to the bottom: lemo head, stainless steel tubing body, housing, 4 K pot, docking station, radiation shields, 1 K pot and mK-MFM head, Figure 2.

Lemo head has three Lemo connectors and a fibre feed through. A lemo head is connected to the 4 K pot by means of stainless steel tubing. Outside of this tubing, there is stainless steel (ss) housing. Tubing moves up and down inside housing utilizing a Wilson seal. The housing sits onto the gate valve of the cryostat to start the operation.

The insert has two main stages: 4 K and 1 K pot. They are made of high purity copper and are in a physical contact with the variable temperature insert (VTI) of the cryostat. Constantan ribbon cable was chosen to minimize the heat load. The ribbon cables are tightly wound on the copper posts at the both 4K and 1K pots for thermally anchoring the heat load as shown in Figure 5.3(b). The microscope docking station is attached just beneath 4 K pot. The microscope head attached to the 1 K pot by using a thin walled stainless steel tubing and G10 Helium displacer to minimize the heat load into the $^3$He liquid. To fit 27 mm free sample space of the VTI, the head designed with the OD of 23.6 mm. The head is detachable by means of a docking station which is put neat to the 4 K pot between 4 K pot and 1 K pot. The reason for putting the docking here is that it has three miniature sockets and conducting well.
To minimize the heat transfer, it is placed at a point above 1 K pot which is the most critical point for heat transfer issue.

Figure 5.3: AFM/MFM head (a) schematic design of the head, (b) photograph of the head

5.2.3 Heat Transfer Through the $^3$He Insert

Heat transfer from the top to the bottom is the key factor for designing the insert and selecting the materials used in the design. All the design issues are focused on how the heat transfers could be minimized from the top to the bottom of the insert.

At the beginning of the work, a docking station was built below 1 K pot without any heat sinks and the temperature never went down below 1 K. However, the docking station was vital for handling and carrying the microscope from one place to another place for us.
In Figure 5.3(a), the design of mK-MFM insert is seen. In this design, cooling efficiency is tried to be maximized and heat transfer from top to the bottom is minimized. Two cooling points 4 K and 1 K pot is made from good conductors like brass and phosphor bronze, respectively. They are in a good thermal contact with VTI. The connection between 4 K pot and 1 K pot is done utilizing ss tubing and docking station which is made of G10 and lemo connectors. Docking station is put beneath 4 K pot since it has large cooling capacity if it is compared with connector pins. The mK-MFM head is connected to 1 K pot utilizing ss tubing, too. A G10 shield put on ss tubing to minimize dead volume inside the sample space.

Figure 5.4: Heat sink applications at (a) 4 K pot and (b) 1 K pot. Constantan loom cables and coax cable are wounded on the copper rods which are screwed to the pots

The actors of heat transfer were also calculated from 300 mK region to 1 K pot to see to the numerical values. It is supposed that there is 8 cm³ liquefied $^3$He at the bottom of the sample space which covers the phosphor bronze shield of the microscope up to the cantilever level. Therefore, the whole shield, which is made of phosphor bronze, should be in thermal equilibrium. The heat transfer was modeled between these two regions as seen in Figure 5.5.

Table 5.1: Heat transfer parameters between the bottom (~300 mK) and 1 K pot

<table>
<thead>
<tr>
<th>Part</th>
<th>Notation</th>
<th>$\varepsilon \left( \frac{W}{K.m} \right)$</th>
<th>$A_d \left( m^2 \right)$</th>
<th>$L \left( m \right)$</th>
<th>Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS Tubing</td>
<td>R₁</td>
<td>0.272</td>
<td>7.28E-06</td>
<td>0.145</td>
<td>1</td>
</tr>
<tr>
<td>G10 body</td>
<td>R₂</td>
<td>0.072</td>
<td>4.30E-04</td>
<td>0.121</td>
<td>1</td>
</tr>
<tr>
<td>Constantan loom cable</td>
<td>R₃</td>
<td>1.500</td>
<td>7.85E-09</td>
<td>0.170</td>
<td>40</td>
</tr>
<tr>
<td>SS Coax cable</td>
<td>R₄</td>
<td>0.272</td>
<td>5.81E-07</td>
<td>0.200</td>
<td>1</td>
</tr>
</tbody>
</table>
Both the notations of the resistances and physical properties of the parts are given in Table 5.1.

Figure 5.5: Modeling of the heat transfer between 1 K pot and liquid $^3$He region

Thermal conductivity of a material, $R\left(\frac{K}{W}\right)$ is calculated utilizing the equation:

$$ R = \frac{L}{\varepsilon A_d} $$

(5.1)

where $L$, $A_d$ and $\varepsilon$ are length (m), cross-sectional area (m$^2$) and thermal conductivity coefficient ($\frac{W}{K.m}$), respectively. The transferred heat, $Q$ (W), is calculated by the equation:

$$ Q = \frac{\Delta T}{R_{Total}} $$

(5.2)

where $\Delta T$ is temperature difference between two points where the transfer is calculated.

$$ R_{Total} = \frac{1}{\frac{1}{R_1} + \frac{1}{R_2} + \frac{1}{R_3} + \frac{1}{R_4}} $$

(5.3)

$$ Q_{Total} = \frac{\Delta T}{R_{Total}} = \frac{T_{1Kpot} - T_{300mK}}{R_{Total}} $$

(5.4)
~32 µW heat transfer between 300 mK region and 1 K pot was calculated regarding the calculation above. This is well below the 500 µW nominal cooling power of the cryostat. We expected to reach 300 mK referring this calculation omitting the electrical powers when the controller is switched on and laser power of the fibre interferometer.

5.2.4 Cooling the mK-AFM/MFM

When the microscope was cooled down below 1 K and it reached 325 mK and stayed there. During this cool down, the controller was switched off just to see thermal performance of the design. The temperature sensor had been placed at the end of the sample slider which was supposed to be in liquid $^3$He. No temperature fluctuation was observed which expressed that heat load is much less than cooling power of the cryostat. When the controller was switched on without operating laser diode, the temperature was still stable at the same level of 325 mK. Finally, laser diode was operated with low power level of 0.5 mW to see the interferometer signals. The fibre interferometer was sustained well. Then, the laser power was increased gradually from 0.5 mW to 4.0 mW and the temperature was started to increase with the power level of 1.5 mW. When the laser power was increased to 4.0 mW level, the temperature was jump to the 1 K level. At the level of 1.0 mW, a stable temperature level below 400 mK was obtained and the scans were performed there. In Figure 5.6, an interference pattern is seen that measured at 325 mK.

![Graph](image)

Figure 5.6: Fibre interferometer signal at 325 mK. The laser power was hold at 1.0 mW. Operating power on the pattern is 45 µW, visibility is 0.6 and the slope is ~1 mV/Å.

In Figure 5.7, the measured resonance frequency and phase scan are seen for MFM cantilever. The resonance frequency was ~74,470 Hz with the quality factor of ~10,000 which was
decreased order of magnitude during time since the boil of $^3$He by the laser power changed the pressure inside the sample space pretty much. We could manage the feedback in semi-contact mode and run the MFM in standard constant height mode.

![Amplitude vs Frequency Plot](image1)

**Figure 5.7:** Cantilever tune and phase scan at 350 mK. Q factor is 9,930 in high vacuum condition of the $^3$He sample space. Cantilever was PPP-MFMR.

### 5.3 Images

Initially, the microscope was cooled down to 1.5 K in the $^3$He cryostat for checking functionality of the both insert and microscope. The temperature read values at both 4 K and 1 K pots were compatible with standard insert which gave clue about wiring functionality and heat load of the insert. Then, Blu-ray disk sample was imaged in tapping mode of AFM, successfully, that confirmed functionality of the system, too. Typically, ~50 nm oscillation amplitude of the cantilever was used for imaging, in Figure 5.8. The cantilever was used for this experiment was PPP-FMR. These initial results had unveiled the functionality of the mK-AFM/MFM insert in a good manner.
Figure 0.1: Tapping mode AFM image of blu-ray disk at 1.5 K. (a) Topography, (b) Amplitude (Error) and (c) Phase images. Scan area was 4 µm x 4 µm with scan speed of 1 µm/s.

For MFM tests, CoPt multilayer sample was imaged at between 350 mK and 300 K. The constant-height mode was used for imaging with typical lift-off value of 50-100 nm. In Figure 5.7, MFM image of CoPt multilayer at 1.5 K is seen prior to $^3$He condensation.

In Figure 5.8, MFM images recorded at 350 mK is given. The cross-sections give better than 60 nm MFM resolution on the CoPt multilayer sample.
Figure 0.2: MFM image of CoPt multilayer at 1.5 K prior to $^3$He condensation into the system. (a) Topography and (b) MFM image in constant height mode. Scan area is 3 µm x 3 µm with scan speed 1 µm/s. The lift-off amount was 75 nm.

Figure 0.3: MFM image shows magnetic domains of the CoPt multilayers at 350 mK. Scan size is 1.6 µm x 1.4 µm with scan speed of 1 µm/s. The lift-off amount was 75 nm.

5.4 Conclusions

In this chapter of the thesis, we developed a self-aligned milli-Kelvin MFM and tested between ~300 mK and 300 K, successfully. The self-aligned design has sustained down to 300 mK. These results have confirmed our heat transfer analysis and our expectations. In addition, heat load of the microscope is low enough to have enough hold time at ~300 mK for imaging. We used very low level of the laser power for the fibre interferometer and could get successful results even though we have comparably low signal to noise ratio.
CHAPTER 6

6.1 Introduction to DR-AFM/MFM

There are few reports about interferometer based atomic force microscope for fridges in the literature. The first one was reported by D. Rugar group that described only the sensitivity measurement of the soft cantilevers at 110 mK [87]. Others were reported by D. V. Pelekhov about AFM on the samples at 25 mK [88-89]. There is no more report on MFM or any other AFM applications so far.

In this chapter, we described the design and operation of the DR-AFM/MFM both in AFM and MFM modes at these ultra low temperatures. The work was performed and achieved with the collaboration with Prof. Henrik M. Ronnow from École Polytechnique Fédérale de Lausanne (EPFL), Switzerland.

The results partially obtained in this section were published in Review of Scientific Instruments [90].

6.2 Dilution Refrigerators and Operations

To lower the temperature below 300 mK, rather complicated and expensive cryostat systems are used that is called ‘dilution refrigerator’ or simply fridge. Dilution refrigerators use $^4\text{He}/^3\text{He}$ mixture as a cryogen and can reach ~5 mK base temperature level. The theoretical works of $^4\text{He}/^3\text{He}$ mixture were done by Heinz London in the early 1950s and the behavior was experimentally shown in 1964 at Leiden University of Kamerlingh Onnes Laboratory [79]. The operation principles of a fridge system are discussed in the following section extensively.

The entire dilution refrigerator is immersed into a liquid helium bath as shown in Figure 6.1. The fridge is shielded by two main radiation shields and composed of five cooling stages that we call as ‘pot’. The first shield is vacuum tight at 4 K pot (Stage 1) and all the pots are inside this shielded region. The fridge is pumped to $<10^{-5}$ mbar prior to cooling to get rid of any contamination in the fridge. The second shield is located at cold plate pot (Stage 4) and used for blocking blackbody radiation coming from 4 K regions.
Some of the liquid helium is siphoned off into 1 K pot (Stage 2) that is pump out for decreasing the vapor pressure inside the pot. Pumping decreases the temperature of this pot down to 1.5 K. A precisely measured mixture of $^3$He/$^4$He is introduced into the fridge. The mixture gas reaches 1 K pot passing through 77 K cold trap which is located outside of the cryostat and 4 K trap inside of the cryostat to hold possible nitrogen and oxygen impurities, respectively and cooled down to 4.2 K. The mixture is cooled down at 1.5 K pot, too and starts condensing. Then, the mixture passes through first impedance which is capillary tubing with a large flow resistance and reaches still heat exchanger. The temperature of the still (Stage 3) is ~800 mK and cools the liquid down. The liquid drips down through 2nd impedance and sintered silver heat exchangers which cool the mixture down further. Finally, the liquid enters the mixing chamber (Stage 5) which is coldest part of the fridge where the cooling power is produced. The sample is attached to mixing chamber for measurements.
In the mixing chamber, below 870 mK, the mixture goes spontaneous phase separation to form lighter $^3$He rich fraction floating on $^4$He rich fraction. The top $^3$He rich fraction is called ‘the concentrated phase’ and the bottom $^3$He poor phase is called ‘the dilute phase’. At very low temperatures, the concentrated phase is completely composed of $^3$He and the dilute phase is composed of approximately 6.6% $^3$He and 93.4% $^4$He as shown in Figure 6.2.

![Spontaneous phase separation diagram of $^3$He/$^4$He mixture at saturated vapor pressure. $T_F$ is the Fermi temperature of $^3$He component [91].](image)

All the helium atoms are attracted to each other by means of van der Walls forces. However, $^3$He is less dense than $^4$He and much more zero point motion. $^3$He are much ordered state in concentrated state than dilute phase. Therefore, the natural tendency of $^3$He atoms is crossing the phase boundary from the concentrated phase to the dilute phase. For this reason, $^3$He atoms need energy to cross phase boundary which is gathered around in the mixing chamber. This is source of cooling power in the mixing chamber and fridge. At the dilute phase, $^3$He flows through superfluid $^4$He, which is stable and rest, and follows the way through the still. On the way, it takes the heat from condensing atoms coming from condenser at the heat exchangers. In the still, there is liquid/gas interface and the gas is composed of $^3$He. A powerful pumping unit is connected to the still for $^3$He circulation in the system. An attached heater on the still helps the evaporation rate of $^3$He for proper cooling power. The cooling
power of the DR is proportional to the amount of $^3$He crossing the phase boundary and expressed as [91]:

$$Q(T) = 84n_mT^2$$  \hspace{1cm} (6.1)

where $Q(T)$ is temperature-dependent heat transfer and $n_m$ is the number of moles $^3$He passing through the phase boundary in one second. Then, the pumped $^3$He atoms are directed to the condenser and follow the way through to the mixing chamber leaving their energy at each stage continuously.

### 6.3 Cryostat and Vibration Isolation Stage

An Oxford Instruments cryostat with a 9 Tesla superconducting magnet which is capable of accepting either a dilution refrigerator inserts (Kelvinox MX–400) or a variable temperature inserts (VTI) was used for the test and the experiments.

![Figure 6.3: Picture of the cryostat system hanging on the vibration isolation stage and pumping lines were fixed into the massive concrete block](image)

The microscope head was designed such that it can be used in both of the inserts. The dilution fridge (DR) has 400 µW cooling power at 100 mK and it has the minimum base temperature level of 8 mK at mixing chamber base plate without any thermal load. The fridge has a detachable experimental insert onto which the sample holder and related wiring for the MFM
are mounted. The cryostat is mounted on a 5 cm thick triangular aluminum plate with a hole to accept the cryostat, which was floated by three air stabilizer legs [75] with 1.5 Hz nominal cut-off frequency and auto weight-balance mechanism as seen in Figure 6.3. The floor where the vibration isolation stage sits was isolated from remaining laboratory floor where all the pumps and controller units were located. The vacuum tubes, Helium circulation pipes and the electrical leads attached to the fridge and the MFM are connected through a 60 kg concrete block in order to minimize vibration coupling to the microscope.

6.4 DR AFM/MFM Design

The microscope head was integrated on the fridge by means of a docking station as seen in Figure 6.4(b). A detachable docking station was designed and mounted at the end of the experimental insert. The docking station comprises low temperature high density miniature connectors and has a large physical contact area with the mixing chamber to ensure good thermal conductivity. The microscope head, which has 25.4 mm OD and 200 mm length, is attached to the other side of the docking. The length of the microscope was designed so that the sample/sensor couples sit at the center of the magnetic field. Both the microscope and docking were made of oxygen-free high thermal conductivity copper (OFHC) for enhancing the thermal conductivity from the mixing chamber through the sample. The whole body of the microscope and docking station are electroplated with gold. The sample holder was thermally anchored using a copper loom through the microscope body and the temperature is directly monitored by a RuO$_2$ thermometer placed on the microscope body.

Table 6.1: Cables used for wiring of the microscope for fridge

<table>
<thead>
<tr>
<th>Wire Type</th>
<th>Purpose</th>
<th>OD of wire (mm)</th>
<th># of Wire</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS Coax</td>
<td>Tip connection</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Constantan Loom</td>
<td>Piezo, Spares</td>
<td>0.1</td>
<td>24</td>
</tr>
<tr>
<td>Si/Cu Ball Coated</td>
<td>Fibre</td>
<td>0.175</td>
<td>1</td>
</tr>
</tbody>
</table>

The wiring of the microscope from ambient to the mixing chamber was optimized in order minimize the heat transfer. A single soft stainless steel coaxial cable for carrying the tunneling signal and 24 constantan loom pairs for three piezo motors and spare connections were used, Table 6.1. The cables were wrapped around 45 mm length copper rods for thermal
anchoring at the inner vacuum can (IVC) roof, 1 K pot, still, cold plate and mixing chamber stages from top the bottom on the experimental insert, respectively, Figure 6.4(a).

Figure 6.4: (a) Dilution Refrigerator (DR) insert, OD of the MC base plate is 100 mm. The approximate temperatures at 1 K pot is ~1.5 K, at still is ~600 mK, at cold plate is ~50 mK and at mixing chamber is ~5 mK. (b) Integrated AFM/MFM head onto the mixing chamber, OD of the microscope shield is 25.4 mm.
6.5 Experimental Results

6.5.1 Temperature Stability and Heat Load

During the measurement procedure, the temperature stability has been measured using the thermometer on the microscope body as shown in Figure 6.6. The base temperature was measured to be 19 mK when both the controller was on and off. That’s, there was no power dissipation on the microscope. The image shows the temperature variations during several approaches to the surface, two scans and retracting from the surface for removal. The vertical dashed lines indicate the starting and stopping times when the sample has been moved using
stick-slip motion. This reveals that the coarse motion of the sample does produce a very large amount of heat on the sample due to the friction loaded sample puck. The microscope body heats up by over 100 mK, depending on the step rate during stick-slip motion, and takes up to 15 minutes to thermalize back to base temperature. The scans are started and stopped at the positions marked by solid lines and show gradual heating of up to 10 mK throughout the scan. This indicates that while coarse repositioning sample cannot be done while staying cold, the actual measurements do not significantly heat the sample.

Figure 6.6: Image showing the temperature stability of the microscope during stick-slip motion (vertical dashed lines) and scans (vertical solid lines)

When the laser power was introduced into the fridge, a sudden jump was observed as seen in Figure 6.7. 1 mW laser power drives 500 μW power into the fridge which was also scattered by the cantilever into the fridge and raised the temperature up to 150 mK and leave 250 μW cooling power aside. If the laser power in the fibre was increased to 750 μW, the temperature was raised to 175 mK without no cooling power aside. The microscope was operated between these two temperature windows, initially.

Obviously, the fridge required very low power operation of the fibre interferometer. For this reason, we modified fibre interferometer card to run it below 100 μW operation power which is discussed in the following sections for the future works.
6.5.2 Fibre Interferometer Signal

The fibre interferometer signal, that’s the fibre/cantilever alignment, sustained the whole operation range of the temperature of 300 K - 150 mK, successfully. At 300 K, the laser was operated at standard driving power of 3 mW, the slope was 4 mV/Å and the visibility of the interference pattern was 0.388. At 150 mK, for 1 mW driving laser power, the slope was 1 mV/Å and the visibility was measured to be 0.499. The average power on the signal photodetector was 18 µW that this was very far below 250 µW operation power of 300 K.
Figure 6.8: Interference pattern measured at 300 K for laser power of 3.0 mW. The average slope at quadrature point is 4 mV/Å.
Figure 6.9: Interference pattern measured at 150 mK for laser power of 1.0 mW. The average slope at quadrature point was less than 1 mV/Å.

6.5.3 Cantilever Tune

The sample/cantilever was in ~10⁻⁵ mbar vacuum at 300 K in the fridge. All the cooling power from mixing chamber to the sample is transferred by properly designed copper looms by conductively. When the temperature is dropped, the vacuum has to be reached high vacuum level that the damping effect of the gas inside on the cantilever is minimized. Hence, the Q-factor of the cantilever was increased, dramatically as seen in Figure 6.10. Moreover, very small amount of dither excitation would be enough to get proper oscillation amplitudes for the scanning. At 175 mK, the Q-Factor was increased to 43,119 for MFM cantilever which has 0.5 to 9 N/m nominal spring constant. This type of spring constant is rather soft for high vacuum operations and the cantilever was snapped into the sample surface when the cantilever was approached to the sample for dynamic mode operations. Otherwise, vacuum decreases the spectral noise density. Moreover, Q-factor increases the minimum detectable force gradient, \( \delta F'_{\text{min}} \), in dynamic mode of AFM operation [10-92]:

\[
\delta F'_{\text{min}} = \frac{2k k_B T B_W}{\sqrt{\langle z^2 \rangle}}
\]

where \( B_W \) is the measurement bandwidth and \( \langle z^2 \rangle \) mean square amplitude of the cantilever oscillation.

Figure 6.10: Cantilever (PPP-MFMR) tunes (a) at 77 K (b) at 175 mK and Comparison of the two frequency scan results at two different vacuum levels are given in Table 6.2.

Table 6.2: Tune parameters comparison at two different temperatures
<table>
<thead>
<tr>
<th>Parameter</th>
<th>77 K</th>
<th>175 mK</th>
<th>75,447</th>
<th>75,458</th>
<th>Hz</th>
<th>1,973</th>
<th>43,119</th>
<th>mV&lt;sub&gt;pp&lt;/sub&gt;</th>
<th>mV&lt;sub&gt;RMS&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>$f_0$</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Q-Factor</td>
<td>1,973</td>
<td></td>
<td>43,119</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Excitation Voltage</td>
<td>300</td>
<td>10</td>
<td>mV&lt;sub&gt;pp&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oscillation Amplitude</td>
<td>2,300</td>
<td>750</td>
<td>mV&lt;sub&gt;RMS&lt;/sub&gt;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

6.6 Images

6.6.1 AFM Images in Contact Mode

The scans were initiated at 250 mK with 3 mW driving laser power in contact mode to get rid of snapped into the sample problem. The driving laser power was gradually decreased to half of the initial value and imaged on the calibration grating at each step as seen in Figure 6.11.
Figure 6.11: Contact mode AFM, topography images of the calibration grating (1 µm pitch size) for different operating powers of the laser at 250 mK: (a) 3.0 mW and (b) 1.5 mW. Images at 175 mK with 1.5 mW laser power: (c) Topography and (d) Normal force (feedback). Qualities of the images were conserved during these processes. Scan parameters were like that scan area was 10 µm x 10 µm, image size was 256 pixels x 256 pixels and scan speed was 1.5 µm/s. The set normal force value was -1.0 V for feedback. Afterwards the temperature was cooled down to 175 mK and got the images with 1.5 mW laser power with the same scan parameters.

6.6.2 AFM Images in Tapping Mode

In the next step, tapping mode AFM was employed utilizing the PLL and could get the images in Figure 6.12. Between these two topography images of contact mode and tapping mode, there is a clear difference that in contact mode the spheres were not ideal which is caused by the tip shape we guess. Otherwise, they were as expected in tapping mode image. The oscillation amplitude of the cantilever was 139 nm for this scan.
Figure 6.12: Tapping mode AFM images at 175 mK with 1.5 mW laser power: (a) topography, (b) amplitude (feedback) and (c) phase images. Scan area was 7 µm x 10 µm, scan speed was 2 µm/s.

6.6.3 MFM Image

In the second experiment, the microscope was run in MFM mode and the base temperature of the 150 mK was reached with 1.0 mW laser driving power. CoPt multilayer sample was imaged to see the magnetic domains on the sample as seen in Figure 6.13.

![MFM Image](image)

Figure 6.13: MFM image of garnet single crystal at 150 mK with 1.0 mW laser power. Scan area was 12 µm x 12 µm, image size was 256 pixels x 256 pixels and scan speed was 4 µm/s. Lift-off amount was 150 nm.

6.7 Low Power Fibre Interferometer

Driving the laser diode at/above 1 mW limited the base temperature of the fridge at 150 mK. Therefore, the operation power of the fibre interferometer has to be decreased. The power is proportional to signal at photodetector but the shot noise is proportional to $\sqrt{P}$. Therefore, decreasing the power increases the shot noise in the fibre interferometer.

Spectral noise densities of the fibre interferometer for different laser power between 390 µW and 24 µW are seen in Figure 6.14. Half of this power is sent into the fridge and the remaining half is sent to the reference photodetector ($V_{ref}$) for reading. For 195 µW power in the fibre, the noise level was measured to be less than 150 fm/$\sqrt{\text{Hz}}$. When the power is decreased to 12 µW, the noise level was jumped to the 800 fm/$\sqrt{\text{Hz}}$. 12 µW power level in the
fibre is $1/41$ of the initial lowest value of the driving power in the fibre. Decreasing the driving power decreases the power in the signal photodetector, too. Hence the $R_F$, which is the gain factor, of the OPAMP at signal photodetector circuit has to be increased from 5.1 kΩ to 2 MΩ, in Figure 6.15. Indeed, signal to noise ratio (SNR) of the fibre interferometer will be proportional to square root of the $R_F$ in the signal photodetector circuit:

$$SNR \sim \sqrt{R_F}$$  \hfill (6.3)

Figure 6.14: Spectral noise density of the fibre interferometer versus laser power in the fibre graph

Figure 6.15: Circuit diagram of the signal photodetector and output signal
The average power on the signal photodetector and slope change versus power in the fibre graph is plotted as seen in Figure 6.16. Both average power and slope were proportional to the drive laser power. However, the shot noise is inversely proportional to drive laser power as seen in calculation Table 6.3. We could get 1.2 µW power signals from interferometer as the lowest level using 2 MΩ $R_F$ feedback gain in the circuit but the shot noise level exceeded 496 fm/√Hz.

Figure 6.16: Power on the signal PD and interference slope versus laser power in the fibre graph

Table 6.3: Shot noise calculation for different laser powers in the fibre. $P_{Signal}$ is the average power in the PD. Shot Noise Current and $V_{Shot @ PD}$ are the shot noise in related units.

<table>
<thead>
<tr>
<th>$P_{Signal}$ ($\mu W$)</th>
<th>$i_{shot}$ (A)</th>
<th>$V_{shot at PD}$ (V)</th>
<th>Shot Noise (fm/√Hz)</th>
<th>Slope (mV/Å)</th>
<th>$R_F$ (Ω)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.3</td>
<td>6.5E-13</td>
<td>3.3E-06</td>
<td>193</td>
<td>1.7</td>
<td>5.1E+5</td>
</tr>
<tr>
<td>2.2</td>
<td>8.4E-13</td>
<td>4.3E-06</td>
<td>186</td>
<td>2.3</td>
<td>5.1E+5</td>
</tr>
<tr>
<td>4.6</td>
<td>1.2E-12</td>
<td>6.2E-06</td>
<td>132</td>
<td>4.7</td>
<td>5.1E+5</td>
</tr>
<tr>
<td>9.5</td>
<td>1.7E-12</td>
<td>8.9E-06</td>
<td>129</td>
<td>6.9</td>
<td>5.1E+5</td>
</tr>
<tr>
<td>18.0</td>
<td>2.4E-12</td>
<td>1.2E-05</td>
<td>102</td>
<td>12.0</td>
<td>5.1E+5</td>
</tr>
<tr>
<td>1.2</td>
<td>6.2E-13</td>
<td>1.2E-05</td>
<td>496</td>
<td>2.5</td>
<td>2.0E+6</td>
</tr>
</tbody>
</table>
The interference pattern was given at the end of new design is seen in Figure 6.17. The laser was driven just by 13 µW laser power. Compare to the high power operation, the signal was so noisy since the shot noise was increased from 7.8 to 496 fm/√Hz. However, we could obtain pretty good results on the calibration sample using low power.

<table>
<thead>
<tr>
<th>Laser Power</th>
<th>13</th>
<th>µW</th>
</tr>
</thead>
<tbody>
<tr>
<td>V_{Reference}</td>
<td>6.5</td>
<td>µW</td>
</tr>
<tr>
<td>V_{Signal}</td>
<td>1.2</td>
<td>µW</td>
</tr>
<tr>
<td>Slope</td>
<td>2.5</td>
<td>mV/Å</td>
</tr>
<tr>
<td>Visibility</td>
<td>0.142</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6.17: Interference pattern for low power fibre interferometer and related parameters in the table
Figure 6.18: Tapping mode AFM image of the calibration grating (a) topography and (b) amplitude images. The data are raw and there is no image process. Scan area was 20 µm x 20 µm, image size was 180 pixels x 180 pixels and scan speed was 8 µm/s.

6.8 Conclusions

We have designed and constructed an atomic force microscope / magnetic force microscope for a dilution refrigerator and operated at 150 mK, successfully. To decrease the minimum operation temperature, a low power fibre interferometer was designed and operated below 100 µW laser power for future works. The microscope is also a good platform for standalone STM, SHPM or any other scanning probe microscopy applications at this ultra low temperature.
CHAPTER 7

7.1 Introduction to Fibre Fabry-Pérot Interferometer

We achieved ~12 fm/√Hz noise floor for our deflection sensor using Michelson fibre interferometer with 7.8 fm/√Hz laser shot noise which limits sensitivity of the sensor. Laser shot noise is a kind of electronic noise that occurs when the finite number of photons that carry energy is small enough to give rise to detectable statistical fluctuations in a measurement. When photons strike on the photo diode, a photo current, \( i \), is generated on the photo diode which is proportional to the optic power, \( P \), fall onto it:

\[
i = S_{PD}P
\]  

(7.1)

The average root-mean squared variation of the current noise is given by:

\[
\bar{\ell}_{\text{shot}} = \sqrt{2eS_{PD}P \left( \frac{A}{\sqrt{Hz}} \right)}
\]

(7.2)

where \( e \) is the electronic charge, 1.6x10\(^{-16}\) C. If this current passes through a resistor, \( R_F \), the average root-mean squared variation of the voltage noise is given by:

\[
\bar{v}_{\text{shot}} = \sqrt{2eS_{PD}PR_F \left( \frac{V}{\sqrt{Hz}} \right)}
\]

(7.3)

If the optic power falls onto the photo detector is increased, the signal sensitivity is increased in the same ratio, too. However, the shot noise is just increased as the ratio of square root of the signal. Indeed, the noise could be decreased with increasing optical power.

This is the motivation point for decreasing the noise floor to improve a fibre Fabry-Perot interferometer (FFPI) for our deflection sensor. FFPI requires two reflecting surfaces (cantilever’s back side and fibre-air interface surface) and multiple reflections occur in the cavity. These multiple reflections produce the interference pattern in the photo detector this time in a much sensitive way. For this purpose, the cleaved end part of the fibre has to be coated with Si/Au for achieving multiple reflections.

FFPI has been developed by few groups [69, 93-97] in the literature for obtaining high sensitivity deflection sensor for atomic resolution imaging in liquid at ambient conditions. They reported ~2 fm/√Hz noise level at 1 MHz bandwidth using modified ambient AFMs. The cavity gap between the cantilever and the fibre is adjusted using 5-axis positioners critical the help of optical microscope.

The critical points are raised for designing a FFPI, in Figure 7.1:
- Coating the fibre end for increasing reflectivity of the reference beam and creating a parallel mirror system
- Adjusting the gap between the fibre and the cantilever
- Increasing the optical power falls on the photo detector as much as possible

Figure 7.1: Schematic design of a fibre Fabry-Perot interferometer (FFPI). Coating the fibre end increases ratio of the internal reflection and provides multiple reflections between two parallel mirrors.

These three critical points helped to us for embodying a FFPI and we established our new interferometer on these roots. For this purpose:

1. The cleaved end of the fibre is coated
2. A low temperature stick-slip approach mechanism is designed for adjusting the gap between the fibre and the cantilever
3. The 2x2 50% coupler is replaced with fibre optic circulator in the interferometer

7.2 Fibre Coating

Coating the fibre end with multilayers has two major roles from optical view. The first one is that the coating process increases the bare reflectivity from 3% to 30-70% at the cleaved fibre end. The second one is that the coated end face and cantilever occurs two parallel mirrors state which enables multiple reflections between these plates. Interference pattern is produced by at the end of these multiple reflections which amplifies the signal gain considerably. The
Michelson interferometer is changed to Fabry-Perot interferometer and one can get high finesse, visibility and slope values with this interferometer.

We used commercially coated fibre cables (Oz Optics, Canada), with the variable reflectivity value between 30-50%. The architecture of the coating material is Si/Au multilayers. When the bare fibre is coated with silicon and gold, the reflectivity of the fibre is changed typically as seen in Figure 7.2. The critical thing is thickness of the films coated. From our research experiences, typically, 90 nm of Si followed by 20 nm Au coating gives 55% reflectivity [98].

![Figure 7.2: Typical reflectivity values used for the coated fibre](image)

**7.3 Low Temperature Fibre Slider (2 K – 300 K)**

Adjusting the distance between the fibre and the cantilever is crucial for obtaining high interference slope values. For the Michelson interferometer design, the distance is fixed about ~30 µm, which gives Ø19 µm laser spot size, for secure tip replacements. If the distance could be adjustable, it would be possible to get smaller spot size equal to the fibre core and the signal gain would be much in the interferometer. In addition, the drawbacks of the misalignments between the fibre and the cantilever caused by fabrication would be fewer effects on the interferometer signal.

**7.3.1 Low Temperature Fibre Slider Design**

For this purpose, we improved a new design for the MFM alignment holder in which the fibre assembly is sliding. The parts of the new low temperature fibre slider are seen in Figure 7.3.
Figure 7.3: Low temperature fibre slider parts: V-shaped mfm holder, zirconia ferrule tubing, bare fibre with Ø125 µm and PhBr leaf spring for the tubing.

In this design, the zirconia tubing with Ø1 mm diameter is placed on a V-shaped titanium holder and pressed by a leaf spring. The polished zirconia surface has three contact surfaces and slides between two titanium surfaces and a PhBr leaf spring surface. The stiffness of the leaf spring is adjusted by two M1.6 screws which are mounted on the titanium body. The alignment of the cantilever with respect to fibre is done under the optical microscope as described previously for the minimum cavity gap and the parts are glued to each other using stycast epoxy. When the zirconia tubing is retracted to back, which has 0.5 mm z-range, and approached to the cantilever again, the alignment is conserved and the same interferometer signal is obtained for repetitive motions.

Figure 7.4: (a) V-shaped MFM holder mounted at the end of tube piezo scanner and sliding mechanism. (b) Picture of the fibre slider.
The motion of the zirconia tubing is provided by the scanner piezo of microscope using stick-slip approach mechanism. A brass weight (0.505 g) was mounted to the end of zirconia tubing (0.050 g) to increase the sliding weight an order of magnitude. Hence, momentum of the sliding part was also increased in the same ratio. Without this additional weight, the ferrule itself did not overcome the friction below 125 K and the motion was stopped. The pps holder was also redesigned in a way that it houses the whole massive ferrule assembly and limits the retract motion for the security reasons, Figure 7.4. The approach limit was also restricted when the brass weight touches the leaf spring, the motion is terminated.

### 7.3.2 Fibre Slider Drive Mechanism

The exponential slider pulses produced by the slider card are applied to the scanner piezo for approaching and retracting the fibre assembly with respect to cantilever, Figure 7.5.

**Figure 7.5**: Slider pulses for stick-slip approach mechanism of the fibre assembly (a) normal pulse for approaching and (b) time reverse pulse for retracting. Inset (a) shows falling time for slip motion is 900 ns.

The scanner piezo of microscope is used for this special duty besides scanning. The voltages between 40-350 V are applied all quadratures of the scanner (named as S, N, E, W) at the same time when the inner electrode is grounded.

In Figure 7.6, motion of the ferrule tubing is seen with respect to the cantilever. The motion is proceed up to touching to the cantilever, in which the signal is broken, and then retracted back in a way where the interference slope is maximum by means of the fibre slider.
Figure 7.6: Observing the ferrule tubing motion by the fibre slider stick-slip mechanism under the optical microscope

This sliding mechanism is tested and optimized for all operation range of the temperature for 2-300 K, successfully. The applied voltage has to be increased up to 350 V with decreasing temperature since the capacitance of the piezo is also decreased. The fibre slider produces traceable steps which enables us to adjust optimum cavity gap to give maximum slope for all temperature range. The step sizes are tunable adjusting slider pulse height by the software, easily, as seen in Figure 7.7 in a typical example. The pattern was moved toward right by two approach steps which gave 130 nm step size with 245 V slider pulse height at 77 K.
Figure 7.7: Traceable fibre steps were measured on the interference pattern at 77 K. 245 V height slider pulse was applied for approaching that produces 130 nm step size.

When the approach is completed, the fibre crashes the cantilever that the interference pattern is disturbed. Even after few more steps towards cantilever after initial crash, the cantilever still survives. We usually, retracted few steps back for operation and try to maximize the interference slope using slider pulse height with approach/retract steps. In Figure 7.8, both retracted and approached steps by stick-slip motion are given at 4 K.
Figure 7.8: Stick-slip motion of the fibre slider at 4 K. The motion is followed and measured by change in the interference pattern: (a) retracted away and (b) approached and one step retracted after cantilever touch down.

7.3.3 Fibre Optic Circulator

Our standard Michelson fibre interferometer uses 2x2 50% coupler for dividing the laser beam that one is used for reference photo diode for monitoring the power and the second one is used for deflection measurement. Indeed, half of the beam is not used for measurement. For increasing the optic power falls onto the signal photo diode, it would better to use most part of the laser beam for deflection measurement.

Fibre optic circulators can be used for this purpose that uses the power 100%. It is a three-port non-reciprocal optical device that allows light to travel in only one direction as seen in Figure 7.9.
A laser beam entering to Port 1 will exit Port 2, while a signal entering Port 2 will exit Port 3. Utilizing the fibre optic circulator [99] in the interferometer, the optical power falls onto the signal photo detector is increased four times, theoretically. Hence, the slope is expected to be increased in the same ratio. In Table 7.1, comparisons of two fibre optic components are given for 3 mW drive laser power.

Table 7.1: Comparison of the 2x2 fibre optic coupler with the fibre optic circulator

<table>
<thead>
<tr>
<th></th>
<th>Power in the Fibre (mW)</th>
<th>Typical Average Power at Signal PD (µW)</th>
<th>Slope (mV/Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2x2 50% Coupler</td>
<td>1.5</td>
<td>200</td>
<td>30</td>
</tr>
<tr>
<td>Circulator</td>
<td>3.0</td>
<td>800</td>
<td>120</td>
</tr>
</tbody>
</table>

7.4 Fibre Fabry-Pérot Interferometer

The pigtailed laser diode is coupled into a fibre optic circulator in the standard fibre interferometer as shown in Figure 7.9. A laser beam entering to Port 1 (a pigtailed LD is connected) will exit Port 2 (a pigtailed fibre goes to the LT AFM/MFM) with minimal loss, while a signal entering Port 2 will exit Port 3 (a pigtailed signal PD is connected) with minimal loss. Cleaved coated end of the single mode fibre in the LT AFM/MFM system reflects typically 40-50% of the light back. The remaining beam exits the fibre and hits the cantilever end surface and reflected back. Multiple reflections occur between these two
parallel mirrors which enhances the optical power falls on the signal photodetector with the increased internal reflectivity.

In the case of multiple reflections, the photocurrent generated by interference of the beams in the signal PD which is written as:

\[ i = i_0 \left[ 1 - V F \left( \frac{4\pi d}{\lambda} \right) \right] \quad (7.4) \]

where \( V \), \( d \), and \( \lambda \) are visibility, cantilever-fibre separation, wavelength of the laser, respectively. Cosine function in the Michelson fibre interferometer is replaced with a much complicated function, \( F \) of the distance, \( d \) in this case. The most sensitive position produced at the quadrature points are \( d = \lambda / 8, 3\lambda / 8, 5\lambda / 8, \ldots \). The slope of this new function, \( \alpha = F'(d) \), is much greater than the Michelson one at the quadrature point and can be written as:

\[ \frac{\Delta i}{\Delta d} = \left( 4\pi i_0 \frac{V}{\lambda} \right) \beta \quad (7.5) \]

where the value of \( \beta \) is \( \approx 30 \) and \( V \) is about 0.9. Typical modeled interference signals of the both interferometer types are shown in Figure 7.10. Fibre Fabry-Pérot interferometer has higher slope value that means higher sensitivity for the deflection sensor.

Figure 7.10: Modeling Michelson and Fabry-Pérot interferometer signals for 1,310 nm wavelength
7.5 Experimental Results

7.5.1 Fibre Fabry-Pérot Interferometer Signals

Fibre Fabry-Pérot interferometer signal with fibre optic circulator at ideal tip-sample distance is given in Figure 7.11. The fibre optic circulator has increased the power at signal PD as expected about x4 times. For 3 mW drive laser power, the average maximum power at Michelson interferometer was 500 µW whereas this value is about 2,000 µW for the same laser drive power. The average power on the Michelson interferometer was 200 µW whereas this value is about 800 µW for Fabry-Pérot one. The relation of the average power or power at quadrature point at signal PD is given in Figure 7.12. There is a linear relationship between drive laser power and the power at quadrature point.

The maximum slope that we obtained with 2x2 fibre optic couple is \( \sim 30 \text{ mV/Å} \) in Figure 7.8(b) for Fabry-Pérot interferometer. However, the slope is increased to \( \sim 120 \text{ mV/Å} \) for fibre optic circulator. The increase in the slope is x4 which is proportional with the signal power. The average slope for Michelson interferometer was \( \sim 4 \text{ mV/Å} \). Overall, the gain in slope is about x30 with both fibre optic circulator and Fabry-Pérot interferometer design.

Figure 7.11: Fibre Fabry-Perot signal for different drive laser power. The circulator increased the signal power about x4.
Figure 7.12: Average power at quadrature point versus drive laser power graph which shows linear relationship.

Figure 7.13: Fibre Fabry-Perot interference slope versus drive laser power graph.

Similar Fabry-Perot interference signals were obtained at 5 K after adjusting the fibre cantilever gap properly. In Figure 7.14-15, both Fabry-Perot interference signal and interference slope measurements are given recorded at 5 K for different drive laser powers.
Comparison of two different interferometers are given in Table 7.2. The interference slope hence the sensitivity has increased x30 with the new design of fibre Fabry-Pérot interferometer.
<table>
<thead>
<tr>
<th></th>
<th>Power in the Fibre (mW)</th>
<th>Slope (mV/Å)</th>
<th>Visibility</th>
<th>Finesse</th>
</tr>
</thead>
<tbody>
<tr>
<td>Michelson</td>
<td>1.5</td>
<td>4</td>
<td>0.3</td>
<td>1.0</td>
</tr>
<tr>
<td>Fabry-Perot</td>
<td>3.0</td>
<td>120</td>
<td>0.9</td>
<td>6.5</td>
</tr>
</tbody>
</table>

### 7.5.2 Fabry-Pérot Interferometer Noise Measurements

When the spectral noise density of the sensor was measured at 300 K, the noise level of \(\sim 2\) fm/√Hz was obtained at 1 MHz bandwidth as seen in Figure 7.16. The shot noise is calculated to be 0.84 fm/√Hz at 300 K. The theoretical fit was also done in the graph with the parameters: resonance frequency is 69,050 Hz, Q Factor is 25 and spring constant is 15 N/m for PPP-FMR sensor. The average slope value at 300 K was about 120 mV/Å for 3 mW laser drive power, Figure 7.17.

At 77 K, the noise level was measured to be below 1 fm/√Hz at 1 MHz bandwidth as seen in Figure 7.17. The shot noise is calculated to be 0.66 fm/√Hz at 77 K. The theoretical fit parameters were in the following: resonance frequency is 68,790 Hz, Q Factor is 300 and spring constant is 25 N/m for PPP-MFMR sensor. The similar slope value of 120 mV/ Å was obtained at 77 K for 3 mW laser drive power, Figure 7.19.

![Figure 7.16: Spectral noise density of the fibre Fabry-Perot interferometer at 300 K. The shot noise is calculated to be 0.84 fm/√Hz. The slope value for this measurement was ~120 mV/Å.](image)
Figure 7.17: Fabry-Perot interferometer signals at 300 K

Figure 7.18: Spectral noise density of the fibre Fabry-Perot interferometer at 77 K. The shot noise is calculated to be 0.66 fm/√Hz. The slope value for this measurement was 122 mV/Å.
7.5.3 AFM Images

Inaugural test of the Fabry-Perot interferometer based LT-AFM/MFM was done on the calibration sample and various thin film samples for checking functionalities. In Figure 7.20, tapping mode AFM image of the calibration grating is given recorded at 300 K. Typical employed oscillation amplitude was 8 nm for this application. The interference pattern given in Figure 7.11 with slope value of ~120 mV/Å was used for this experiment.

In Figure 7.21, tapping mode AFM image of the calibration grating is given recorded at 4 K. The interference pattern given in Figure 7.14 with slope value of ~120 mV/Å was used for this experiment.

In Figure 7.22, topographies of the two thin film samples are given that they show surface morphology of the surfaces and film qualities recorded at 300 K. These application results confirmed us about the functionality of the microscope in AFM mode.
Figure 7.20: Tapping mode AFM image the calibration grating at 300 K. (a) Topography, (b) Amplitude (Feedback) and, (c) Phase images. Scan Area was 20 µm x 20 µm with scan speed of 1 Hz. Sample periodicity is 2 µm.

Figure 7.21: Tapping mode AFM image the calibration grating at 4 K. (a) Topography, (b) Amplitude (Feedback). Scan Area was 15 µm x 15 µm with scan speed of 1 Hz.
Figure 7.22: Tapping mode AFM images of (a) Nb$_2$O$_5$ thin film and (b) ITO film at 300 K

7.5.4 MFM Images

We also tested the microscope in MFM mode either at 300 K or low temperatures. For a typical MFM application, we imaged a 80 GB/Platter hard disk sample at 15 K which is seen in Figure 7.23.

Figure 7.23: MFM image of the 80 GB/Platter hard disk sample at 15 K: (a) Topography and (b) Magnetic image. Scan area was 10 µm x 9 µm with scan speed of 5 µm/s. Lift-off amount was 90 nm.

For further performance check, Type-II superconducting BSCCO(2212) single crystal sample was imaged to see Abrikosov vortex lattice in the crystal. The microscope was cooled down to 4 K under the small amount of magnetic field of +14 Oe. The tip was also magnetized in
the same field direction prior to cool down process. The sample was cleaved prior to the experiment. The result is given in Figure 7.24.

![MFM image of the BSCCO(2212) single crystal at 4 K. Scan area was 10 µm x 10 µm, image size was 128 pixels x 128 pixels and scan speed was 5 µm/s. Lift-off value was 65 nm.](image)

**Figure 7.24**: MFM image of the BSCCO(2212) single crystal at 4 K. Scan area was 10 µm x 10 µm, image size was 128 pixels x 128 pixels and scan speed was 5 µm/s. Lift-off value was 65 nm.

### 7.6 Conclusions

We designed and constructed a low temperature fibre Fabry-Perot interferometer with the noise level of ~1 fm/√Hz at 77 K. So far there was no report such kind of interferometer design for a low temperature AFM setup in the literature. A low temperature stick-slip fibre slider is another breakthrough for our low temperature AFM/MFM setup that this is operational between 300 K and 2 K.
CHAPTER 8

8.1 Introduction to Direct Excitation of the Cantilevers and Radiation Pressure

In this section, we showed direct excitation of the cantilevers instead of piezo one by means of radiation pressure effect of the laser beam. We developed a new method for radiation pressure excitation of the cantilevers and deflection measurement. For the first time in the literature, we employed this technique for AFM/MFM imaging either at room temperature or low temperature, successfully.

Piezo excitation is the most common indirect way to excite cantilevers that we also used in our design. However, it has some drawbacks that sometimes spurious resonance peaks and non-ideal Lorentzian curves can be observed especially at low temperatures as seen in Figure 8.1. The instabilities are originated by thermal contraction of mechanical parts pressing the cantilever. The problem is also valid for imaging in liquid environments that acoustic modes of the liquid and the setups disturb the resonance peaks. These created disturbances in the cantilever resonance frequency prevent users to get either a meaningful or good images most of the time. Therefore, direct excitation of the cantilevers plays a crucial role for dynamic modes of atomic force microscopes [12] both imaging in liquids or low temperatures environments.
Figure 8.1: (a) Piezo excitation and spurious resonance peaks and (b) corresponding phase response. Red curves indicate theoretical calculations [108].

To get rid of this problem, couples of methods were developed in the literature to excite the cantilevers in a direct way. Magnetic modulations [100] of the cantilevers or thermal excitation utilizing resistive heating [101] were the developed methods. These two direct techniques have also brought disadvantages that magnetic modulation requires magnetic tips which limit the applications and thermal excitation requires special cantilever design with undesired thermal fluctuations on both the sample and the cantilever.

The third method was photothermal excitation of the cantilevers which was raised as the most elegant alternative method at this point. In this method, two laser sources are used in which one is focused in front of the cantilever for deflection measurement and the second one is focused at near the base part of the cantilever for excitation [102-103]. The cantilever is vibrated by the optical absorption of this second laser beam whose intensity is modulated at the resonance frequency of the cantilever. For retrofitting absorption of the laser beam, the cantilever surface, where the laser beam falls onto it, is blackened regarding the used wavelength [104] to be absorbed the light by the surface. The absorption of the light causes a thermal gradient between two sides of the cantilever and this bimetal effect excites the cantilever photothermally [105]. Potential of this technique was demonstrated in the early experiments that a lever made of a foil was vibrated photothermally and could be obtained enough oscillation amplitude for atomic force microscopy imaging [104]. Photothermal excitation of the cantilevers in liquid [103,106], mass detection on a cantilever in the air [107], imaging in a low Q environments like liquid [102, 108-110], fast AFM spectroscopy [111], force versus distance measurement [112] and high speed AFM imaging [113] were also demonstrated for possible applications of this technique.

When the laser beam hits to the cantilever end, regarding the absorption and the reflection coefficients of the surface with respect to wavelength of the laser beam, photothermal effect competes with radiation pressure effect [112-113]. The force exerted by radiation pressure of the light will be dominant if most fraction of the light is reflected from the cantilever surface. The amount of the force will be enough to get proper oscillation amplitude for driving AFM cantilevers [114]. The reports on the radiation pressure for AFM are so limited in the literature with surpassing thermal noise of the very soft cantilever utilizing radiation pressure [115], calibration of micro structures using sub-μN forces [116] and demonstrating the radiation pressure excitations on the custom-made soft cantilevers [114,117]. Otherwise, there
is no more report on radiation pressure excitation for neither the cantilever dynamics nor imaging in the literature.

Two laser beams were used for radiation pressure excitation of the cantilevers that one for the excitation and the other one for the deflection measurement similar to the photothermal excitations. However, this kind of optical design is impossible for low temperature AFM/MFM systems since there is really limited space to make a complicated optical design for radiation pressure excitation.

The questions arise that if a single laser beam would be enough for both exciting the cantilever and measuring the deflection of the cantilever in a simple way and the radiation pressure excitation could be used for AFM imaging. This is the emerging point of idea worked in this section that we developed a new method to use a single light beam for both excitation and deflection measurement of the cantilever, successfully. Using this new technique, we imaged the samples in both AFM and MFM mode between 300 K and 4 K, for the first time. In addition, the spring constant of the cantilever could be calculated directly as a non-destructive way and an alternative method.

8.2 Radiation Pressure

The question whether falling light onto a material exerts a pressure was a very popular question in the history of science. In 1619, Kepler explained the tails of comets by means of solar radiation which vaporizes particles on the surface of comets and creates the tails in the same direction of the light coming from the sun in addition to the orbital tail of comets [118].

The most famous portraits of science like Newton, Maxwell, and Einstein had also engaged with this question. The theoretical works was done by Maxwell and he stated that [119]:

“"In a medium in which waves are propagated there is a pressure in the direction normal to the waves .... rays falling on a thin metallic disk, delicately suspended in a vacuum, might perhaps produce an observable mechanical effect. However, the electromagnetic light pressure was very small and quite insufficient to account..."

Scientist had tried to both measure and observe radiation pressure effect of the light. Evolution of the works on this subject in the history was studied in a good manner in the reference [120]. The developments in the micro and nano technology for the last three decades have enabled to produce very small and soft micromechanical systems like cantilevers which makes possible to both measure and observe the radiation pressure effect.
When light beam hits to the end of cantilever and reflected back, the momentum of the light, $P_{RP}$, is transferred to the cantilever which is equal to:

$$P_{RP} = 2\frac{h}{\lambda}\cos \theta$$

(8.1)

where $h$, $\lambda$ and $\theta$ are the Planck constant, wavelength of the light beam and angle of the incident beam with respect to the normal direction. The momentum transferred by absorbed photons is half of this value and the transmitted photons do not transfer momentum. The number of photons, $n_p$, incident on the surface per unit time can be calculated from optical power, $P$, is equal to:

$$P = \frac{n_p \hbar c}{\lambda}$$

(8.2)

where $c$ is speed of the light. The force exerted by the radiation pressure, $F_{RP}$, to the cantilever is equal to [121]:

$$F_{RP} = [2R + (1 - R)]n_pP_{RP} = (1 + R)\frac{P}{c}\cos \theta$$

(8.3)

where $(1-R)$ is the absorbed fraction of the incident beam. The radiation pressure force depends on two resultant parameters as seen in Eqn. 8.3: reflectivity of the cantilever surface and the incident light power. The cantilever is well aligned with respect to the fibre and the incident beam is normal to the cantilever surface. Therefore, numerical value of the force can be calculated precisely which opens a new door for cantilever spring constant calculation as an alternative method discussed in the next sections.

### 8.3 Radiation Pressure Excitation of the Cantilever

#### 8.3.1 The Circuit Diagram and Operation Principles

A single laser beam was used for both radiation pressure excitation and deflection measurement of the cantilever, simultaneously. We used our Michelson fibre interferometer with some modifications for this purpose as seen in Figure 8.2. The laser was driven by a DC current to give 3 mW laser power that half of this value hits to the cantilever to measure deflection of the cantilever as usual. At the same time, an AC modulation signal is injected into the laser diode by the PLL at the cantilever actual resonance frequency, $f_0$. The cantilever is oscillated around this DC offset level by means of the modulation power. Digital PLL excitation output also feed into a phase shifter to get rid of the excitation effect on the signal.
photodiode. Signal photodiode output and phase shifted excitation signals are subtracted from each other by an operational amplifier and used as an output signal for the measurement.

Figure 8.2: Schematic design of the fibre interferometer circuit diagram for the radiation pressure experiment (PD: photo diode, VCO: voltage controlled oscillator, PLL: phase locked loop)

Figure 8.3: Typical interference pattern obtained by Michelson fibre interferometer at 300 K
The measurements were performed utilizing the LT AFM/MFM system. A typical interference pattern obtained by the fibre interferometer at 300 K is given in Figure 8.3. Using the theoretical model described by Wilkinson and Pratt [122] the interference pattern was simulated using MATLAB and fitted to the experimental interference pattern. The simulations showed that there is a 1.2° angular mismatch between the fibre and the cantilever. Moreover, the simulation gives numeric loss at FC/APC connectors in the fibre interferometer. Total loss of the light at the connectors for a round trip is 49%. All the power calculations incident on the cantilever is revised regarding the model developed.

8.3.2 Cantilever Excitation

The technique was employed to the dynamic mode commercial cantilevers (PPP-FMR and PPP-MFMR) with nominal spring constants of 0.5 – 10 N/m at moderate vacuum level of $10^{-1} – 10^{-4}$ mbar between 4–300 K temperature range. In atmosphere conditions, damping of the air is dominant and the radiation pressure effect cannot be measured on the cantilever. Hence, we could not observe the resonance frequency during the frequency scan by the PLL. When the microscope space was initiated pumping, the peak frequency started appearing soon as seen in Figure 8.4(a). Then, the pumping was continued, the ideal Lorenzian curve was obtained as seen in Figure 8.4(b). The technique that we offered was successful and can excite the cantilever and measure the deflection simultaneously.

![Figure 8.4: Radiation pressure excitation in two different vacuum levels: (a) pumping was just initiated and (b) pressure was 2.5x10^-4 mbar. Cantilever was PPP-FMR, DC laser power was 1.071 mW and AC modulation amplitude was 506 µWpp.](image)

The commercial cantilevers have 30 nm Al coating at the back side and the reflectivity was measured to be ~95% at 1,310 nm wavelength. Total laser power that passing across the cantilever was measured less than 5%, which is negligible, using an IR photodiode setup on the table. The laser spot, which was focused at the free end of the cantilever, do not cause any...
thermal gradient except that focused territory and no effects on the cantilever vibrational modes [102] that radiation pressure effect dominates photothermal effect at this territory [123]. Up to 506 µW (peak to peak) modulation power, which is equal to 3.29 pN force exerted by radiation pressure, was employed to excite the cantilevers in addition to the 1.071 mW DC power in the fibre. The modulation at the resonance frequency, amplifies the oscillation amplitude of the cantilever as much as Q-factor.

![Graph](image)

Figure 8.5: (a) Resonance frequency measurement for different modulation power and (b) the linear relationship between oscillation amplitude of the cantilever and the modulation power.

The measurements were recorded at 4 K with PPP-MFMR cantilever.
Resonance frequency of the cantilever for different modulation powers were measured as seen in Figure 8.5(a). The oscillation amplitude is proportional with the modulation power as expected. Figure 8.5(b) shows linear relationship between oscillation amplitude of the cantilever and the modulation power.

We obtained up to 111 Å\textsubscript{pp} oscillation amplitude at 300 K with Q-factor of 5,950 at \( \sim 2.5 \times 10^{-4} \) mbar pressure. When the temperature was cooled down to 4 K, up to 1,418 Å\textsubscript{pp} oscillation amplitude was obtained with 1,125 Q-factor in the vacuum level of \( \sim 1 \times 10^{-2} \) mbar. The oscillation amplitude was increased more than an order of magnitude from 300 K to 4 K even if lower vacuum levels. The microscope is cooled down in Helium exchange gas of 760 mbar and the pressure inside sample space is decreased to few tens of mbar at 4 K. Without special vacuum treatment at 4 K, we could still observe resonance frequency which was usable for imaging.

### 8.3.3 Spring Constant Calculation

The spring constant, of the cantilever can also calculated utilizing basic Hooke’s law since the exerted force to the cantilever and the displacement of the cantilever has already been known. The spring constant, \( k \), is equal to [117]:

\[
k = \frac{(1+R)PQ}{cA}
\]

where \( A \) is the peak-peak amplitude of the cantilever. In the equation, the numerical values of the parameters: \( R \) is 0.95, \( P \) is 506 µW, Q-factor is 5,950, \( c \) is \( 3 \times 10^8 \) m/s and \( A \) is 111 Å. Then, \( k \) was calculated to be 1.06 N/m within the nominal range.

The result was compared with the spring constant calculation from thermal noise measurement, too. Therefore, spectral noise density, \( S \), of the cantilever was measured at 300 K and theoretical calculation was fit into the measurement, Figure 8.6. The spring constant from fitting was calculated to be 0.55 N/m. There is about a factor of 2 between two results which requires more analysis and samples.

In conclusion, radiation pressure can be used for spring constant calculation as an alternative method for AFM cantilevers that measuring the spring constant is always a difficult task. Moreover, this method gives a direct result in a non-destructive way compare to the spring constant calculation from force vs distance curve. Compare to the other alternative and most common used Sader method [78], this would be much accurate.
8.4 Imaging Using Radiation Pressure Excitation

The method was employed to a low temperature atomic force / magnetic force microscope (LT-AFM/MFM) for the first time and various samples were image in both AFM and MFM modes between 300 K and 4 K to demonstrate performance of the method. A calibration grating and HOPG sample were imaged in AFM tapping mode and a hard disk sample, a multilayer film and BSCCO single crystal sample were imaged in MFM mode. They are the first results in literature obtained using radiation pressure excitation method.

8.4.1 Tapping Mode AFM Images

The inaugural promising results were obtained in AFM tapping mode on the calibration sample at 300 K, Figure 8.7. 506 µW modulation power was employed to the PPP-FMR cantilever which corresponds to 3.29 pN radiation pressure force and ~11 nm oscillation amplitude. The vacuum level and Q-factor were $2.5 \times 10^{-4}$ mbar and ~6,000, respectively. Atomic terraces of the freshly cleaved HOPG sample were imaged with the similar parameters above to show the z-resolution performance of the method, Figure 8.8(a). The cross-section in Figure 8.8(b) shows 3.4 Å atomic layers on the sample.

Figure 8.6: Spectral noise density of the PPP-FMR cantilever at 300 K and theoretical fit into the graph. The fit parameters are $f_0=79,744$, Q-factor$= 5,950$ and $k=0.55$ N/m. The shot noise is calculated to be 10.1 fm/√Hz.
Figure 8.7: Tapping mode AFM image of the calibration grating at 300 K. The periodicity of the sample is 2 µm.
Figure 8.8: (a) Topography image of the HOPG shows atomic steps on the sample obtained in tapping mode at 300 K. Scan area was 4.5 µm x 2.8µm with scan speed 5 µm/s. (b) cross-section on the topography image.

8.4.2 MFM Images

The method was also employed to MFM mode of the microscope. The initial results were obtained on a hard disk sample (80 GB/platter) at 300 K, 77 K and 4 K as seen in Figure 8.9. 298 µW modulation power was employed to the PPP-MFMR cantilever which corresponds to 1.93 pN radiation pressure force. The Q-factors were 10,800 at 300 K, 4,060 at 77 K and 6,930 at 4 K. The employed oscillation amplitudes of cantilevers were 14 nm at 300 K, 11 nm at 77 K and 51 nm at 4K. The lift-off value between 90 and 150 nm were employed regarding the oscillation amplitudes used for MFM images. Regarding the measured Q-factors for these types of commercial cantilevers, vacuum is necessary especially at room temperature.

Figure 8.9: MFM image of the hard disk sample at (a) 300 K, (b) 77 K and (c) 4 K
The magnetic domains of the CoPt multilayer sample were also imaged at 4 K in MFM constant height mode with 35 nm lift-off value, Figure 8.10(a). The modulation power of 263 µW was employed which corresponds radiation pressure force of 1.71 pN and caused ~69 nm oscillation amplitude. The similar force and modulation power with 50 nm lift-off value were employed for imaging Abrikosov vortex Lattice in BSCCO(2212) single crystal at 4 K, Figure 8.10(b). The sample was cooled down under the external magnetic field of −120 Oe. The tip was magnetized positive field direction, prior to cool down. The average vortex spacing between adjacent vortices was measured to be ~444 nm as seen in Figure 8.10(c) which was compatible with theoretical calculation value of 446 nm. For 4 K operations, the microscope was at the vacuum level of ~1x10^{-2} mbar and Q-factor was ~1,200.

Figure 8.10: MFM images using radiation pressure excitation at 4 K: (a) Magnetic domains of CoPt multilayer. Scan area was 2µm x 2µm with scan speed 4 µm/s and lift-off value was 35 nm. (b) Vortex lattice in BSCCO(2212) single crystal. Scan Area was 2.3 µm x 2.3 µm with
scan speed 10 μm/s and lift-off value was 50 nm. (c) The cross-section shows the distance between two adjacent vortices.

8.5 Conclusions

In conclusion, we described a new method for radiation pressure excitation of the cantilevers. A single light source was used for both exciting the cantilever and measuring the deflections which eliminates complicated optical designs. The radiation pressure excitations would be very useful alternative method for vacuum or low temperature environments of the force microscopy for direct excitation of AFM cantilevers.

We employed the radiation pressure excitation technique for the first time for imaging both in room temperatures as well as low temperatures, successfully.

We also showed cantilever spring constant calculation by means of radiation pressure in a non-destructive and direct way as an alternative method.
CONCLUSIONS AND DISCUSSIONS

In this study, we demonstrated a self-aligned, alignment-free low temperature atomic force / magnetic force (LT-AFM/MFM) operating between ~30 mK - 300 K. Alignment-free design eliminated time consuming, tedious fibre-cantilever alignment procedures and complicated alignment setups. In this way, we could shrink OD of the microscope below 25.4 mm that provides compatibility with various cryostat systems, easily.

For measuring deflection of the cantilever, a Michelson type fibre interferometer was developed and obtained unprecedented sensitivity. The noise floor was measured to be ~25 fm/√Hz at 300 K and ~12 fm/√Hz at 4 K. Using this sensitive deflection sensor, we demonstrated 10 nm MFM resolution with commercial cantilevers on the high density magnetic recording media. In addition, further performance of the microscope was shown on BSCCO(2212) single crystal that we imaged Abrikosov vortex lattice at 4 K under various external magnetic field cool down.

We also designed and integrated our AFM/MFM head onto the $^3$He insert from Oxford Instruments and showed capability of the microscope down to 300 mK in both AFM and MFM modes. In the next step, for decreasing base temperature about 30 mK that we can operate the microscope, we designed and integrated or AFM/MFM head onto the dilution refrigerator from Oxford Instruments, successfully. These two standalone AFM/MFM systems are good candidate for exploring and imaging scientific phenomena at these ultra low temperatures. Moreover, they are good platforms for applications of many AFM modes as well as STM.

The shot noise limited Michelson fibre interferometer noise floor enhanced down to ~1 fm/√Hz which is in the same range with a proton radius. We achieved this with developing a fibre Fabry-Pérot interferometer. Such kind of an ultra-low noise interferometer is a good standalone metrology platform and would be applicable to many research areas. Initially, we employed this for LT-AFM/MFM system successfully and demonstrated inaugural results both in AFM and MFM modes. As a future perspective, utilizing this ultra low noise deflection sensor we believe that it would be possible to improve the MFM resolution about 5 nm.

In the last part of study, we demonstrated radiation pressure excitation of commercial cantilevers for LT-AFM/MFM between 300 K – 4 K. Radiation pressure is an alternative method for direct excitation of the cantilevers. We developed a new novel method for using
single laser beam which is used for both exciting the cantilever and measuring the deflection of it. We obtained up to 111 Å oscillation amplitudes at 300 K at the vacuum of $2.5 \times 10^{-4}$ mbar with 500 µW modulation power which is quite enough for dynamic for AFM applications. At 4 K, we obtained up to 1,418 Å oscillation amplitudes. For the first time in literature, we used this technique for imaging both in AFM and MFM modes. We imaged atomic steps of HOPG samples in tapping mode AFM mode at 300 K and Abrikosov vortex lattice in BSCCO(2212) single crystal superconducting sample at 4 K, successfully. These results show that radiation pressure technique can be applied for a LT-AFM/MFM system as a routine method to get rid of resonance frequency anomalies. Radiation pressure could be also used for cantilever calibration as a non-destructive way as an alternative method. We could not have hundred percent matches with spring constant calculation from thermal noise but further studies can explores the discrepancy and develop a novel way.

As a last sentence, we believe that the instruments and methods that we developed in this study can help to solve many problems stated in physics and material science.
REFERENCES


125


Nanosensors Inc., Rue Jaquet- Droz 1, Case Postale 216, CH-2002, Neuchatel, Switzerland.

Applied NanoStructures Inc., 415 Clyde Ave, Suite 102, Mountain View, CA 94043, U.S.A.


[70] HiOptel Technologies Inc., China, Part Number: HPD-701SF.


[74] NanoMagnetics Instruments Ltd., Suite 290, 266 Banbury Road, Oxford OX2 7DL, United Kingdom. www.nanomagnetics-inst.com

[75] Newport Corp., S2000 Stabilizer Series.1791 Deere Avenue, Irvine, CA 92606, United States.


[84] The samples are the courtesy of Prof. Kazuo Kadowaki, Tsukuba University, Japon.

[85] The samples are the courtesy of Prof. Mehmet Ertuğrul and Dr. Mustafa Tolga Yurtcan, Atatürk University, Erzurum, Turkey.


[99] AFW Technologies, Victoria 3803, Australia. Part number is CIR-3-13-L-1-0.


CURRICULUM VITAE

Credentials
Name, Surname : Özgür KARCI
Place of Birth : Bozdoğan, Aydın
Marital Status : Married
E-mail : ozgurkarci@gmail.com
Address : 100. Yıl İşçi Blokları, 1538. Sokak, 25/3, Çankaya, Ankara

Education
High School : Bozdoğan Lisesi, 1994-1997
BSc : Middle East Technical University, MSc Physics Education, 1998-2004
MSc : -
PhD : Hacettepe University, Nanotechnology and Nanomedicine, 2008-2015

Foreign Languages
English (Good)

Work Experiences
August 2005 – Continued : NanoMagne
ics Instruments Ltd. Ankara, Physicist

Areas of Experiences
Low Temperature Atomic Force Microscope / Magnetic Force Microscope
Scanning Probe Microscopes (STM, SHPM, PrFM, SSRM, etc.,)
Fibre Interferometers (both Michelson & Fabry-Pérot)
Wet/Dry cryostat systems (2 K – 300 K)
\(^3\)He cryostat systems (300 mK- 300 K)
Dilution Refrigerators (5 mK -300 K)
Low temperature stick-slip slider mechanisms
Superconductivity

Projects and Budgets
Publications


Oral and Poster Presentation


3. Ö. Karcı, M. Dede and A. Oral, *Alignment-free, low temperature magnetic force microscope (LT-MFM) design for 300mK-300K temperature range*, INTERMAG Conference, Sacramento, California, USA, May 4-8, 2009. (Poster)
