

T.R.
GEBZE TECHNICAL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**DEVELOPMENT of TOOLS and Ti/Pt/Co REFERENCE SAMPLES
for TRANSFER FUNCTION BASED QUANTITATIVE STRAY FIELD
MEASUREMENTS**

BAHA SAKAR
A THESIS SUBMITTED FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY
PHYSICS DEPARTMENT

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SUMMARY

Reliable and quantitative magnetic stray field measurements with high spatial resolution are of great importance for research and industrial quality control. However, most magnetic force microscopes (MFM) only deliver uncalibrated qualitative stray field images. Transfer function based MFM calibration to obtain quantitative MFM is a well-developed technique for such measurements, however, the number of validated calibration samples is limited, and the samples are scarcely available. Furthermore, the classical tip calibration approach has the limitations of the reference field calculability and accessible spatial frequencies.

In this dissertation, we present a Ti/Pt/Co multilayer stack with a stripe domain pattern as a potential calibration sample for transfer function based quantitative MFM measurements. The requirements for reference samples are discussed and a validation process based on micromagnetic simulations combined with different characterizations is established. The Ti/Pt/Co is shown to be a well calculable reference sample for calibrations on a 100nm to 10 μm scale. Its moderate magnetic field strength makes this sample a good candidate for calibrations also of low moment tips and the well-controlled fabrication process makes it highly available. Furthermore, we report a new approach to calibrate the MFM by an atomic quantum sensor allowing quantum calibrated MFM measurements (QuMFM). To do so we measure the MFM tip's stray field distribution by a nitrogen vacancy (NV) center in diamond. We further demonstrate the quantum calibration of an MFM reference sample thereby opening the road towards quantum traceable calibration of virtually any MFM.

Key Words: Magnetic Force Microscopy, Quantum Sensing, Micromagnetism, Metrology for Magnetism, Nitrogen-Vacancy Centers.

ÖZET

Yüksek çözünürlüklü, kantitatif manyetik alan ölçümleri bilimsel araştırma ve endüstriyel kalite kontrolü için büyük önem taşımaktadır. Bununla birlikte, çoğu manyetik kuvvet mikroskobu (MFM) ölçümleri yalnızca kalibre edilmemiş kalitatif manyetik alan dağılımlarını verir. Kantitatif MFM (qMFM) ölçümleri elde etmek için, tip kalibrasyonuna dayalı transfer fonksiyonu yaklaşımı, ölçümler için iyi geliştirilmiş bir tekniktir. Ancak, test edilip onaylanmış kalibrasyon numunelerinin sayısı oldukça azdır. Ayrıca, klasik yaklaşım ile yapılan kalibrasyonlar, referans numunesinin teoriye uygunluğu ve domenlerin uzaysal özellikleri ile sınırlıdır.

Bu tez çalışmasında, transfer fonksiyonuna dayalı qMFM ölçümleri için potansiyel bir kalibrasyon numunesi olarak, şerit manyetik domen yapısına sahip Ti/Pt/Co çok katmanlı bir film hazırlanmıştır. Yeni referans numunesinin, kalibrasyon için gereklilikleri tartışılıp, farklı karakterizasyon teknikleri ile desteklenen mikromanyetik simülasyon çalışmaları ile incelenerek geçerliliği kanıtlanmıştır. Ti/Pt/Co numunesinin, referans alanı hesaplanabilir ve 100 nm 10 µm aralığındaki nanoyapıların kalibrasyonu için uygun bir numune olduğu gösterilmiştir. Orta büyüklükteki manyetik alan şiddeti, bu numuneyi düşük zorlayıcı alana sahip MFM tiplerinin kalibrasyonu için uygun, iyi tanımlı ve yüksek kontrollü örnek hazırlama prosedürü ise ulaşılabilir/tekrarlanabilir bir referans numunesi yapar. Bunların yanı sıra, çalışma sonucu olarak, kuantum kalibrasyonlu MFM ölçümlerine (QuMFM) fırsat yaratan, tipin atomik bir kuantum sensörü kullanarak kalibre edildiği yeni bir yaklaşım önerilmiştir. Bunun için, MFM tipinin manyetik alan dağılımı elmas içindeki bir nitrojen-boşluk merkezi (NV-center) kullanılarak ölçülmüştür. Ayrıca, bir MFM referans numunesinin kuantum kalibrasyonu gerçekleştirilerek, teorik olarak her MFM sisteminin kuantum kalibrasyonunun yapılabileceği gösterilmiştir.

Anahtar Kelimeler: Manyetik Kuvvet Mikroskobu, Kuantum Algılama, Micromanyetizma, Manyetizma için Metroloji, Nitrojen-Boşluk Merkezleri.

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LIST of ABBREVIATIONS and ACRONYMS

<u>Abbreviations and Acronyms</u>	<u>Explanations</u>
MFM	: Magnetic Force Microscopy
qMFM	: Quantitative Magnetic Force Microscopy
TF	: Transfer Function
TTF	: Tip Transfer Function
ICF	: Instrument Calibration Function
LCD	: Lever Correction Function
Pt	: Platinum
Co	: Cobalt
Ti	: Titanium
Si	: Silicon
Q	: Quality Factor
c	: Cantilever Stiffness
σ_{eff}	: Effective surface charge density
H	: Magnetic Field
B	: Magnetic flux density
M_s	: Saturation Magnetization
M	: Magnetization
m	: Reduced Magnetization
XPS	: X-ray Photoelectron Spectroscopy
XRD	: X-ray Diffraction
GI	: Grazing Incidence
DC	: Direct Current
RF	: Radio Frequency
QCM	: Quartz Crystal Microbalance
MOKE	: Magneto Optical Kerr Effect

VSM	:	Vibrating Sample Magnetometer
CPS	:	Count per Second
PTB	:	Physikalische Technische Bundesanstalt
IFW	:	Leibniz-Institut für Festkörper- und Werkstoffforschung
LLG	:	Landau-Lifshitz-Gilbert Equation
RKKY	:	Ruderman–Kittel–Kasuya–Yosida exchange
cal	:	Calculated
dec	:	Deconvoluted
sim	:	Simulated
SPAD	:	Single Photon Avalanche Detector
NV	:	Nitrogen Vacancy
ODMR	:	Optically Detected Magnetic Resonance
H_z	:	z-component of stray field
$H_{ }$	:	stray field projected on NV-axis

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1. INTRODUCTION

Magnetism and magnetic materials lie in the heart of most technological breakthroughs. With the technological capabilities developed in the last century, magnetism and magnetic interactions have been better understood, and their technological applications have spread to many wide areas such as electronics, medicine and automotive. Especially with the progress of nano-technology, magnetic thin-films and multilayers attract a lot of attention. The increase for higher and higher data storage with each passing day unique aspects of sensors based on magnetic nanostructures [1], breakthroughs in cancer treatments with magnetic nanoparticles [2], and many other applications attract the interest of the scientific and technological communities towards nanomagnetism.

The quantitative stray field measurements of magnetic nanostructures are of great importance for both industry and research. One of the most common technique for such measurements is quantitative magnetic force microscopy (qMFM). MFM is a cheap, fast, robust magnetic imaging technique. However, due to the unknown stray field distribution of its tip, it is unfortunately qualitative. The quantitative MFM is a technique proposed by Hans Joseph Hug in 1998 where this unknown tip stray field distribution was obtained by a calibration from a well-defined reference sample [3]. The accuracy of the qMFM strongly depends on the calculability and the spatial frequencies of the reference sample. Therefore, the number of reference samples that can be used for calibration is limited. Furthermore, the calibration and the quantitative field measurement was never been validated by using an independent measurement technique. This dissertation was aimed to further develop quantitative field measurements by focusing on the aforementioned limitations. A new reference sample was introduced to the literature with analyzing it in detail with magnetic, structural characterization techniques and micromagnetic simulations. The qMFM calibration and stray field measurement were validated by measuring the stray field of the magnetic field of the magnetic tip with a

single Nitrogen-Vacancy center. Furthermore, a new approach was proposed to calibrate the MFM tip without any limitations on a reference sample and assumptions on the tip.

1.1. Magnetic Properties of Thin Films

The magnetic properties of thin films differ greatly from the bulk properties. Surface sciences study the properties of the interface between two phases i.e., solid vacuum, or two different materials i.e., Pt/Co. The different physical properties at these interfaces usually arise due to the non-trivial neighborhoods of the molecules or atoms [4]. These attributes are called surface effects. Although surface effects are present in bulk materials, their relative contribution is very low and therefore undetectable. Surface effects are dominant in thin films ($< \mu m$) and ultra-thin films ($< nm$), as they are almost composed of the surface. Surface effects can be also increased by stacking thin/ultra-thin films in multilayers. The large number of interfaces found in multi-layered samples cause the structure to appear as if it were composed of only surfaces. In this way, the surface effects can be observed predominantly in thick samples.

Materials that have a spontaneous magnetization even in the absence of any magnetic field are called ferromagnetic materials. The energy of ferromagnet can be described by;

$$E_{tot} = E_{ani} + E_{ex} + E_{ext} + E_{demag} + E_{me} + E_{magstr} \quad (1.1)$$

where the indices *tot*, *ex*, *ext*, *demag*, *me*, and *magstr* are the total, anisotropy, exchange coupling, Zeeman, magnetostatic, magnetoelastic and magnetostrictive self energies [5]. Magnetoelastic and magnetostrictive energy terms are related to the deformation of the magnetic body.

- Exchange Energy

The energy of ferromagnetic materials strongly depends on the electron spin orientation of the relative to the neighboring electrons. This energy is called exchange energy and derived from the quantum mechanical nature of magnetism. The exchange energy between two local spins can be described by

$$E_{ex_{ij}} = -2J_{ij}\mathbf{S}_i \cdot \mathbf{S}_j = -2J_{ij}S^2 \cos(\varphi_{ij}) \quad (1.2)$$

where the J_{ij} is the exchange integral and φ_{ij} is the angle between the spin $\mathbf{S}_i$ and $\mathbf{S}_j$. The exchange energy can be written in the form of the Heisenberg model;

$$E_{ex} = H_{ex} = \sum_{i,j} J_{ij} \mathbf{S}_i \cdot \mathbf{S}_j = \sum_{i,j} J_{ij} \cos(\varphi_{ij}) \quad (1.3)$$

In the special case of collinear alignment, the energy gets minimum when spins are parallel ($\varphi = 0$) and $J < 0$. This type of material is called ferromagnetic. The other minimum energy is obtained for samples with $J > 0$ when the spins are anti-parallel ($\varphi = \pi$). These materials are called anti-ferromagnetic.

In micromagnetic model, the magnetization vector $\mathbf{m}(x)$ is introduced instead of the local spins where the angle between the spins (φ_{ij}) is considered too small. The expansion of the $\mathbf{m}$ over the whole sample in continuum limit is described by [5];

$$E_{ex} = \int_V \frac{JS^2n}{a} [(\nabla m_x)^2 + (\nabla m_y)^2 + (\nabla m_z)^2] dV \quad (1.4)$$

where the exchange constant is defined as $A_{ex} = \frac{JS^2n}{a}$.

- Magnetostatics

The magnetic field produced by the magnet itself due to its magnetization is called demagnetizing field. In the micromagnetic approach, this field can be treated statically as long as the eddy currents are neglected. In this case, there is no time-dependent quantity (magnetostatics) and the magnetic field $\mathbf{H}$ can be written as;

$$\mathbf{H} = \mathbf{H}_{ext} + \mathbf{H}_{demag} \quad (1.5)$$

where the $\mathbf{H}_{ext}$ is the external field and the $\mathbf{H}_{demag}$ is the demagnetizing field. In magnetostatics for stationary magnetic current, the Maxwell equations can be written as;

$$\nabla \times \mathbf{H} = \mathbf{j} \quad (1.6)$$

$$\nabla \cdot \mathbf{B} = 0 \quad (1.7)$$

where $\mathbf{j}$ is the current density and $\mathbf{B}$ is the magnetic flux density. Using the definitions of the $\nabla \cdot \mathbf{H}_{ext} = 0$ and $\nabla \times \mathbf{H}_{demag} = 0$, the Ampere law (1.6) can be rearranged as;

$$\nabla \times \mathbf{H}_{ext} = \mathbf{j} \quad (1.9)$$

The relation between the magnetic flux density and the magnetic field is described by;

$$\mathbf{B} = \mu \mathbf{H} \quad (1.10)$$

where μ is the permeability of the material. This relation can be used to solve magnetostatic fields for designing magnetic circuits where μ describes the response of the material to the external field by itself. However, in Micromagnetism, the local

distribution of the magnetization, thus the response of the system is calculated as a function of the external magnetic field.

$$\mathbf{B} = \mu_0(\mathbf{H} + \mathbf{M}) \quad (1.11)$$

In summary, magnetostatic energy is the energy of the magnet inside a demagnetizing field $\mathbf{H}_{demag}$. The magnetostatic energies are long-range dipolar interactions inside the material. Each moment creates a dipole field and exposed to the fields created by the other dipoles in the system. Therefore, the magnetostatic energy cannot be calculated by using the volume integral as it strongly depends on the local quantities.

- Zeeman Energy

The Zeeman energy is the energy of the magnet inside an external field $\mathbf{H}_{ext}$. The energy of a magnetic dipole moment μ_i inside and external flux density can be written as;

$$E_{ext}^i = -\mu_0 \mathbf{B}_{ext} = -\mu_0 \mu_i \mathbf{H}_{ext} \quad (1.12)$$

Therefore, the sum of the local magnetic moments energy over the ferromagnetic material is;

$$E_{ext} = -\mu_0 \sum_i \mu_i \mathbf{H}_{ext} \quad (1.13)$$

In the continuum model for Micromagnetism, the Zeeman energy is obtained by replacing the sum with an integral, using the magnetization $\mathbf{M} = N\mu$ and defining the volume per atom as $V_i = 1/N$, where N is the number of atoms;

$$E_{ext} = -\mu_0 \int_V (\mathbf{M} \cdot \mathbf{H}_{ext}) dV \quad (1.14)$$

The magnetization can be replaced by the unit vectors $\mathbf{M} = M_s \mathbf{m}$ of magnetization. This unit vector $\mathbf{m}$ is also called reduced magnetization.

$$E_{ext} = - \int_V \mu_0 M_s (\mathbf{m} \cdot \mathbf{H}_{ext}) dV \quad (1.15)$$

- Anisotropy Energy

The energy of a ferromagnet depends on the magnetization direction relative to the crystal axis. This relation is the result of the spin-orbit interactions and called anisotropy energy [6]. Therefore, the anisotropy energy of the sample is directly related to the crystal structure of the ferromagnet. The direction where the anisotropy energy is minimum is called the easy axis and the direction where the anisotropy is maximum is called the hard axis. Therefore, anisotropy energy can be described as the work done by the external field to move the magnetization vector away from the easy axis direction. The energy density of the anisotropy, $e_{ani}(\mathbf{m})$ can be expanded to the power series by using the direction cosines of the magnetization. The energy density for the cubic anisotropy can be written by;

$$e_{K_c} = K_{c1}[(a \cdot m)^2(b \cdot m)^2 + (b \cdot m)^2(c \cdot m)^2 + (c \cdot m)^2(a \cdot m)^2] \\ + K_{c2}[(a \cdot m)^2(b \cdot m)^2(c \cdot m)^2] + \dots \quad (1.16)$$

$$e_{K_c} = K_{c1}(m_a^2 m_b^2 + m_a^2 m_c^2 + m_b^2 m_c^2) + K_{c2} m_a^2 m_b^2 m_c^2 + \dots \quad (1.17)$$

where a, b, c are the unit vectors along the cubic axes and m_i are the magnetization components along the cubic axes. The higher-order terms are mostly neglected. The

direction of the anisotropy either in $\langle 100 \rangle$ or $\langle 111 \rangle$ direction is determined by the sign of the K_{c1} . The uniaxial anisotropy is attributed to the hexagonal and tetragonal crystals. The energy density of the uniaxial anisotropy can be written as;

$$e_{K_u} = K_{u1} \sin^2 \vartheta + K_{u2} \sin^4 \vartheta + \dots \quad (1.18)$$

where the ϑ is the angle between the magnetization direction and the anisotropy axis. The orthorhombic anisotropy of the orthorhombic crystals is described by;

$$e_{K_o} = K_{o1} m_a^2 + K_{o2} m_b^2 + K_{o3} m_c^2 \quad (1.19)$$

The surface anisotropy introduced by Néel [7] applies to interfaces and surface magnetizations. The surface anisotropy term can be expressed by;

$$e_{K_s} = K_s [1 - (\mathbf{m} \cdot \mathbf{n})^2] \quad (1.20)$$

where $\mathbf{n}$ is the surface normal. This term is minimum when the magnetization is parallel to the surface normal ($\mathbf{m} \parallel \mathbf{n}$). Therefore, surface anisotropy prefers magnetization perpendicular to the sample surface. Although the surface anisotropy term is quite large, its contribution is very weak in bulk samples due to the multiplication of the other anisotropy terms with the thickness of the sample. On the other hand, as the thickness of the ferromagnet decreases, the effect of surface anisotropy increases and becomes dominant at sub-nm thicknesses.

- Hysteresis of an Ideal Ferromagnet

The hysteresis loop or the magnetization curve describes the average magnetization of a sample as a function of the external field [8]. The $M(H)$ loop of the ferromagnetic material depends on the external field's direction relative to the easy axis. If an external magnetic field is applied in the direction of the easy axis, enough to rotate all the magnetic dipoles in the same direction (saturation magnetization), since the

dipoles will be in the minimum energy state, they will tend to maintain their orientation even if the external magnetic field is reduced to zero, and a net remnant magnetization will be observed in the sample. However, when the external magnetic field is applied in the direction of hard axis, the dipoles will be in the high energy, and as the external magnetic field is decreased the dipoles lose their orientation and the magnetization of the sample decreases. The magnetization curve $M(H)$ of a ferromagnet strongly depends on the domain structure of the sample. Therefore, an ideal single-domain ferromagnet can be considered to understand the fundamentals of the hysteresis loop.

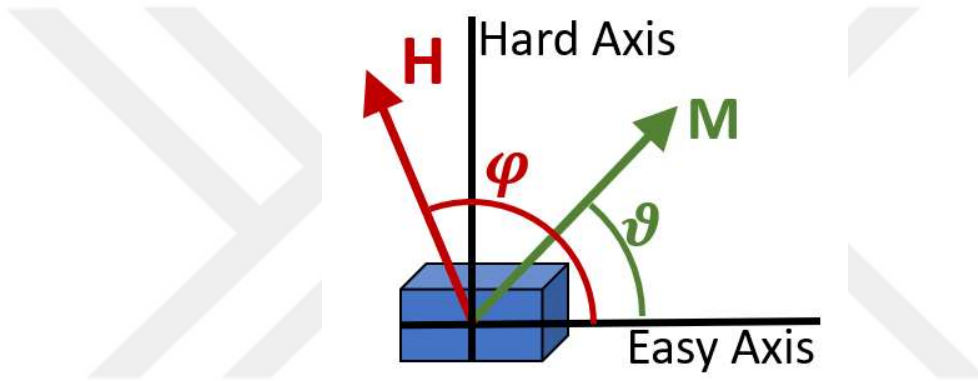


Figure 1.1. The schematic drawing of a ferromagnet inside an external field. M is the net magnetization vector, H is the external magnetic field, ϑ is the angle between magnetization and easy axis, φ is the angle between the external field and easy axis.

The energy of a ferromagnet small enough to be formed of a single domain with uniaxial anisotropy will be dominated by the anisotropy energy given in the equation (1.18) $E = E_a \approx K_{u1} \sin^2 \vartheta$ where ϑ is the angle between the magnetization and the easy axis. When the external field is applied, the Zeeman energy from the equation (1.15) will be added to the energy;

$$E_{ext} = \mu_0 H M_s \cos(\varphi - \vartheta) \quad (1.21)$$

where $H = H_{ext}$ is the external field strength and φ is the angle between the applied field and the easy axis (Figure 1.1). In this case, the total energy is;

$$E = E_a + E_{ext} = K_{u1} \sin^2 \vartheta + \mu_0 H M_s \cos(\varphi - \vartheta) \quad (1.22)$$

The solutions of this equation where the external field is perpendicular to the easy axis ($\varphi = \frac{\pi}{2}$), gives the hard axis magnetization and where the external field is parallel to the easy axis ($\varphi = 0$), gives the easy axis magnetization. For the hard axis magnetization, the equation (1.22) becomes;

$$E = K_{u1} \sin^2 \vartheta + \mu_0 H M_s \cos\left(\frac{\pi}{2} - \vartheta\right) = K_{u1} \sin^2 \vartheta + \mu_0 H M_s \sin \vartheta \quad (1.23)$$

The ϑ values minimize the E can be derived by its first and second derivatives;

$$\frac{dE}{d\vartheta} = (2K_{u1} \sin \vartheta + \mu_0 H M_s) \cos \vartheta = 0, \quad \vartheta = \pm \frac{\pi}{2} \quad (1.24)$$

The $\pm \frac{\pi}{2}$ are $\mathbf{M} \uparrow \uparrow \mathbf{H}$ and $\mathbf{M} \uparrow \downarrow \mathbf{H}$. the second derivative is;

$$\frac{d^2 E}{d\vartheta^2} = -2K_{u1} \sin^2 \vartheta - \mu_0 H M_s \sin \vartheta + 2K_{u1} \cos^2 \vartheta > 0 \quad (1.25)$$

for $\vartheta = \pm \frac{\pi}{2}$, the solution gives;

$$\vartheta = \frac{\pi}{2} \rightarrow \mu_0 H < -\frac{2K_{u1}}{M_s}, \quad \vartheta = -\frac{\pi}{2} \rightarrow \mu_0 H > \frac{2K_{u1}}{M_s} \quad (1.26)$$

This condition can only be satisfied if;

$$\mu_0 H_K = \frac{2K_{u1}}{M_s}, \quad K_{u1} = \frac{\mu_0 H_K M_s}{2} \quad (1.27)$$

where H_K is the anisotropy field. This is a critical field where the magnetization is saturated.

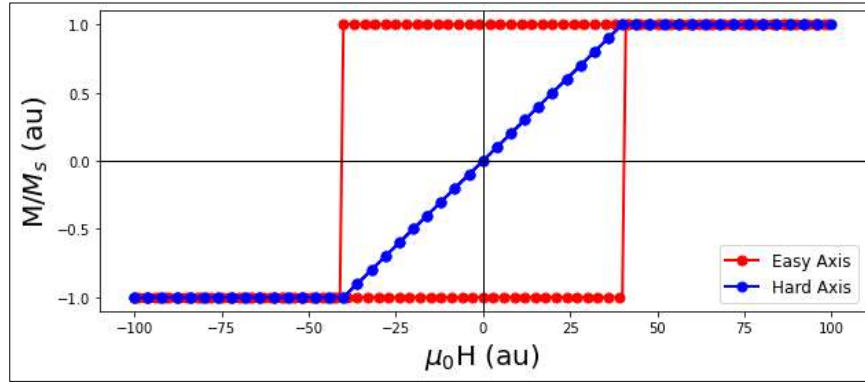


Figure 1.2. Illustration of an ideal hysteresis loop. The ideal hysteresis loop with normalized magnetization measured in easy (red) and hard (blue) axis for a perfect single domain ferromagnet.

This equation can be used to derive the uniaxial anisotropy constant (K_{u1}) of a sample from the hard axis magnetization [9]. If the external field is below the H_K , the first derivate can be simplified by;

$$2K_{u1} \sin \vartheta = -\mu_0 H M_s \quad (1.28)$$

The magnetization components parallel to the applied field, then $\sin \vartheta = -\frac{M}{M_s}$, therefore the equation becomes;

$$2K_{u1} \frac{M}{M_s} = \frac{2\mu_0 H_K M_s}{2} \cdot \frac{M}{M_s} = \mu_0 H M_s, \quad M = M_s \frac{H}{H_K} \quad (1.29)$$

The solution gives a linear dependence of the M with the external field H below between $\pm H_K$ (Figure 1.2). The energy equation (1.22) for the easy axis ($\varphi = 0$) magnetization is;

$$E = K_{u1} \sin^2 \vartheta + \mu_0 H M_s \cos \vartheta \quad (1.30)$$

and the solutions for the minimum energy are;

$$\frac{dE}{d\vartheta} = (2K_{u1} \cos \vartheta + \mu_0 H M_s) \sin \vartheta = 0, \quad \vartheta = 0, n\pi \quad (1.31)$$

$$\frac{d^2E}{d\vartheta^2} = 2K_{u1} \cos^2 \vartheta - \mu_0 H M_s \cos \vartheta - K_{u1} \sin^2 \vartheta > 0 \quad (1.32)$$

for $\vartheta = 0, -\pi$, the 2nd derivative gives;

$$\vartheta = 0 \rightarrow \mu_0 H < \frac{2K_{u1}}{M_s}, \quad \vartheta = -\pi \rightarrow \mu_0 H > -\frac{2K_{u1}}{M_s} \quad (1.33)$$

The solution indicates that the energy will be minimized for all the fields between $\pm \frac{2K_{u1}}{M_s}$. This limit field is called the coercive field $H_c = \frac{2K_{u1}}{M_s}$. For an ideal case, this will result in a rectangular-shaped hysteresis and H_c will be equal to H_K .

2. QUANTITATIVE MAGNETIC FORCE MICROSCOPY

Magnetic force microscopy (MFM) is an imaging technique for magnetic nanostructures [10] which could be performed by most of the commercially available scanning probe microscopes (SPM). Recently, 10 nm resolutions limits have been achieved in MFM measurements [11-13]. High availability of these devices, possibility to measure in ambient condition makes MFM a versatile tool for nanomagnetic researches. The technique relies upon measuring the magnetic stray field gradient of the sample by using a magnetically coated tip. In lift mode MFM measurements, two different measurements are performed in each slow axis line scan; In the 1st pass, the topography of the surface is recorded by intermittent contact mode. In the second pass, the tip is lifted to a certain height and follows the recorded topography. This allows eliminating the force gradient acting on the tip due to the Van Der Waals interactions. Therefore, in the 2nd pass, the free-oscillation of the tip only interacts with the magnetic force gradient acting on the tip (Figure 2.1). This also shows that MFM is solely sensitive to the changing magnetic field and cannot be used to monitor a uniformly magnetized structure.

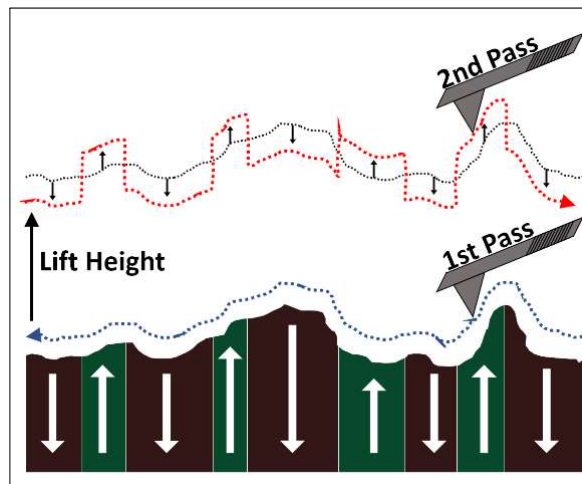


Figure 2.1. The schematic drawing of a lift mode MFM measurement. The blue path indicates the trajectory of the 1st pass-height measurement. The red path indicates the 2nd pass-phase shift measurement. The black path indicates the height measurement trajectory with the lift height offset added.

2.1. Principles of Quantitative Magnetic Force Microscopy

The signal measured in an MFM is a result of the interaction of the magnetic tip's stray field with the magnetization of the sample [14]. As the magnetic properties of the tip are unknown, the information obtained from the measurement is qualitative. However, there are methods to obtain quantitative information from MFM measurements by calibrating the tip. In literature, there are two different approaches to calibrate the tips. The first one is the point probe model. This model describes the tip as a point like [15,16] or extended [17] magnetic dipole and/or monopole. There are various types of reference samples for point-probe model-based calibrations which have been proposed recently. The most common reference samples used are hard disks [18,19]. However, microcurrent rings [20] and parallel wires [21] were also used for calibration. Recently, Di Giorgio et.al. performed calibrations by using superconducting flux quanta [22]. The disadvantage of the point-probe model is the strong dependence on the feature size. The second approach to calibrate is the tip transfer function which overcomes this disadvantage. In the transfer function (TF) approach, the non-local structure of the tip is fully considered [3,23]. The high-resolution quantitative field imaging can be achieved by using the TF approach which corrects the smearing caused by the extended tip. Recently, local Dzyaloshinskii-Moriya Interaction (DMI) values of the skyrmion systems were determined by using qMFM calibrated by the TF approach [24]. Furthermore, TF calibrated qMFM also used to measure the stray fields of the asymmetric Néel walls [25] and determine the coexistence of distinct skyrmion phases in hybrid Ferro/Ferrimagnetic systems [26]. In 2018, using the TF approach, Xiukun Hu et.al. measured the stray fields of nanostructures on a long-range metrological SPM with scanning ranges in the order of millimetres. In the TF approach, a reference measurement is used to calculate an instrument calibration function (ICF) in Fourier space. That is why the TF based calibrations require a specific type of reference sample that has a similar spatial size spectrum with the sample wanted to be measured quantitatively (target sample). Furthermore, the reference sample should be calculable which requires a high

knowledge of the magnetic properties of it, i.e., thickness-dependent magnetization distribution, saturation magnetization (M_s), domain wall width/type, etc... Because of this challenge, only a handful of reference sample was discussed in the literature. The first set of reference samples was introduced by Hug et.al. soon after they firstly proposed the TF based qMFM approach. The samples were different stacks of $\text{Co}_x\text{Ni}_{1-x}$ and Pt layers [27]. Later, Cu/Ni/Cu [23] and Pt/Co multilayers [28,29] were proposed as reference samples. There are also studies where the pattern written on a hard disk was used for the TF calibration [30].

2.2. Phase Shift of the Cantilever Oscillation

The signal in MFM measurement is measured as a phase shift in the tip's oscillation. This signal can be described as a convolution of two elements; one from the tip and the other from the sample. Theoretically, if one of the inputs of the data, which is the convolution of the two inputs, is known, the other can be calculated by deconvolution. Transfer function approach relies on this approach.

First of all, the equation which gives the deconvolution of tip and sample must be described. The force acting on an MFM tip inside a stray field caused by the sample's magnetization can be described by [31];

$$\hat{\mathbf{F}}(\mathbf{k}, z) = \mu_0 \int \begin{pmatrix} -ik_x \\ -ik_y \\ \partial/\partial z \end{pmatrix} \cdot \hat{\mathbf{M}}_{tip}^*(\mathbf{k}, z') e^{-kz'} dz' \hat{\mathbf{H}}^{spl}(\mathbf{k}, z) \quad (2.1)$$

where $k = \sqrt{k_x^2 + k_y^2}$ and the $\hat{\mathbf{H}}^{spl}(\mathbf{k}, z)$ is the Fourier transform of the stray field of the sample at a certain height z' . This equation can be simplified with;

$$\hat{\mathbf{H}}(\mathbf{k}, z) = -\frac{1}{k} \nabla_k \cdot \hat{\mathbf{H}}_z(\mathbf{k}, z) \quad (2.2)$$

$$\hat{\mathbf{F}}(\mathbf{k}, z) = -\mu_0 \sigma_{tip}^*(\mathbf{k}) \frac{1}{k} \hat{\mathbf{v}} \hat{H}_z(\mathbf{k}, z) \quad (2.3)$$

The $\sigma_{tip}(\mathbf{k})$ is the 2D magnetic effective surface charge density of the tip on the plane at tip apex and describes the magnetic properties of the tip. In most SPMs, the cantilever is tilted relative to the sample surface normal. Together with this angle, the amplitude of the cantilever oscillation should be taken into consideration when calculating the frequency shift of the tip caused by such a force. The frequency shift for finite amplitudes $A > 0$ can be given by [31];

$$\Delta\phi^{A>0}(\mathbf{k}, z) = -TF(\mathbf{k}) * \frac{2}{A} LCF(\mathbf{k}, \theta) \hat{H}_z(\mathbf{k}, z) \cdot \frac{1}{\pi A} \int_{-A}^A e^{nq\hat{\mathbf{v}}} \frac{q}{\sqrt{A^2 - q^2}} dq \quad (2.4)$$

where the $LCF(\mathbf{k}, \theta) = -\frac{1}{k} \mathbf{n} \hat{\mathbf{v}}$ is the lever canting function and $\mathbf{n} = (0, \sin(\theta), \cos(\theta))$ is the normal vector of the cantilever plane tilted with angle θ relative to the surface normal. $TF(\mathbf{k})$ is the tip-transfer function and can be described by the magnetic and mechanical parameters of the tip.

$$TF(\mathbf{k}) \sim \frac{\mu_0 Q}{c} \sigma_{tip}^*(\mathbf{k}) \quad (2.5)$$

where the μ_0 is the vacuum permeability, Q is the quality factor of the cantilever oscillation and c is the spring constant of the cantilever (or cantilever stiffness). Q factor describes the energy loss of the cantilever during oscillation and determines the minimum limit of the measurable force. With a higher quality factor, a lower signal to noise ratio (SNR) thus a higher resolution can be achieved. The calculation of the Q factor from the resonance spectrum will be shown later. The Integral in equation (2.4) can be described as a Bessel function 1st order 1st of its kind $I_1(\zeta)$;

$$I_1(\varsigma) = \frac{1}{\pi A} \int_{-A}^A e^{nq\widehat{V}} \frac{q}{\sqrt{A^2 - q^2}} dq \approx -\frac{1}{\pi} \int_0^\pi e^{\varsigma \cdot \cos(\phi)} \cos(\phi) d\phi \quad (2.6)$$

where $\phi = \frac{q}{A}$ and $\varsigma = An\widehat{V}$, The series expansion of 1st order and 1st of its kind Bessel function is given by;

$$I_v(x) = \sum_{j=0}^{\infty} \frac{(x/2)^{v+2j}}{j! \Gamma(v+j+1)} \quad , \quad I_1(x) = \sum_{j=0}^{\infty} \frac{(x/2)^{1+2j}}{j! (1+j)!} \quad (2.7)$$

Using this expansion, $I_1(\varsigma = An\widehat{V})$ can be calculated as;

$$\begin{aligned} I_1(\varsigma = An\widehat{V}) &= \left\{ \frac{An\widehat{V}}{2} + \frac{1}{1!2!} \left(\frac{An\widehat{V}}{2} \right)^3 + \frac{1}{2!3!} \left(\frac{An\widehat{V}}{2} \right)^5 + \frac{1}{3!4!} \left(\frac{An\widehat{V}}{2} \right)^7 + \frac{1}{4!5!} \left(\frac{An\widehat{V}}{2} \right)^9 + \dots \right\} \\ &= \frac{An\widehat{V}}{2} \left\{ 1 + \frac{1}{1!2!} \left(\frac{An\widehat{V}}{2} \right)^2 + \frac{1}{2!3!} \left(\frac{An\widehat{V}}{2} \right)^4 + \frac{1}{3!4!} \left(\frac{An\widehat{V}}{2} \right)^6 + \frac{1}{4!5!} \left(\frac{An\widehat{V}}{2} \right)^8 + \dots \right\} \\ &= \frac{An\widehat{V}}{2} \sum_{j=0}^{\infty} \frac{\left(\frac{An\widehat{V}}{2} \right)^{2j}}{j! (1+j)!} = I_1 = \frac{An\widehat{V}}{2} I_1^{modified} \end{aligned} \quad (2.8)$$

The $LCF(\mathbf{k}, \theta)$ can be written as;

$$\begin{aligned} LCF(\mathbf{k}, \theta) &= -\frac{1}{k} n\widehat{V} \approx \cos(\theta) - i \frac{k_x}{k} \sin(\theta) \\ n\widehat{V} &= -k \cos(\theta) + i k_x \sin(\theta) \end{aligned} \quad (2.9)$$

$$LCF = \frac{n\widehat{V}}{-k}$$

The equation (2.4) can be rewritten by using (2.8) and (2.9);

$$\begin{aligned}
\Delta\phi^{A>0}(\mathbf{k}, z) &= TF(\mathbf{k}) \cdot \frac{2}{A} \cdot \frac{n\hat{V}}{-k} I_1(An\hat{V}) \hat{H}_z(\mathbf{k}, z) \\
&= TF(\mathbf{k}) \cdot \frac{2}{A} \cdot \frac{n\hat{V}}{-k} I_1(An\hat{V}) \cdot \frac{1}{-k} \frac{d\hat{H}_z(\mathbf{k}, z)}{dz} \\
&= TF(\mathbf{k}) \left(\frac{n\hat{V}}{-k} \right)^2 \cdot \left\{ \sum_{j=0} \frac{(An\hat{V}/2)^{2j}}{j! (1+j)!} \right\} \frac{d\hat{H}_z(\mathbf{k}, z)}{dz}
\end{aligned} \tag{2.10}$$

$$= TF(\mathbf{k}) \cdot LCF^2(\mathbf{k}, \theta) \cdot I_1^{modified}(\mathbf{k}, \theta, A) \cdot \frac{d\hat{H}_z(\mathbf{k}, z)}{dz}$$

where the $I_1^{modified}$ is the modified Bessel function, 1st order and 1st of its kind as given in equation (2.8). The phase shift for zero amplitude can be calculated from (2.10);

$$\Delta\phi^{A \rightarrow 0}(\mathbf{k}, z) = TF(\mathbf{k}) \cdot LCF^2(\mathbf{k}, \theta) \cdot \frac{d\hat{H}_z^r(\mathbf{k}, z)}{dz} \tag{2.11}$$

The Transfer function with the canting and oscillation corrections forms the instrumental calibration function $ICF(\mathbf{k}, \theta, A)$.

$$\Delta\phi^{A \rightarrow 0}(\mathbf{k}, z) = ICF(\mathbf{k}, \theta, A) \cdot \frac{d\hat{H}_z^r(\mathbf{k}, z)}{dz} \tag{2.12}$$

Consequently, equation (2.12) shows that the $\Delta\phi$ depends on the z-component of the tip and sample's magnetic field and can be described as a convolution of these two fields/gradients. If the H_z^s can be calculated or estimated, $TF(\mathbf{k})$ can be deconvoluted from the MFM data ($\Delta\phi$).

2.3. qMFM by Transfer Function Approach

2.3.1. Convolution and Regularized Deconvolution

Convolution and deconvolution mathematical operations are used frequently in image and signal processing. In mathematics, convolution is an integral operation that defines a new signal by blending two other signals;

$$G = T * F \quad (2.13)$$

In image processing, convolution and deconvolution are used frequently as the final image can be described as a convolution of the real image and the imaging source (probe). For point-like imaging sources, point spread function (PSF) is a common approach to define the source's properties thus reconstruct the real image. It is widely used to correcting the blurring in optical systems due to the source and enhancing the resolution [32-34]. PSF is used in various electrostatic force-based microscopies such as Kelvin probe microscopy and scanning near field optical microscopy frequently. In quantitative magnetic force microscopy, the transfer function (TF) approach is similar to the PSF.

The phase shift data of the MFM (G) is the convolution of the instrumental calibration function ICF (T) with the stray field of the sample (F). However, neither of these are known priori.

As it was described in equations (2.11) and (2.12), ICF is the combined function of lever correction LCF and transfer TF functions. If either T or F is known, the other can be deconvolved from the G . One of the common tools for such deconvolution is a regularized pseudo-Wiener deconvolution. For the real measurements, the noise term should be added to the equation (2.13). This noise problem is solved by using regularized Wiener filtered deconvolution. Wiener filtering operates in the frequency domain and is therefore suitable for quantitative MFM deconvolutions. For a system given as;

$$\hat{G}(\mathbf{k}, q) = (\hat{T} * \hat{F})(\mathbf{k}, q) + \eta \quad (2.14)$$

where η is the noise of the system, $T(\mathbf{k}, z)$ can be deconvoluted by;

$$\hat{T}(\mathbf{k}, q) = \hat{G}(\mathbf{k}, q) \frac{\hat{F}^*(\mathbf{k}, q)}{|\hat{F}(\mathbf{k}, q)|^2 + \alpha} \quad (2.15)$$

where α is the power spectrum of the noise process and called the regularization parameter. The α_k spectrum can be obtained by calculating the Fourier transform of autocorrelation of the noise spectrum. Furthermore, other techniques can be used to define frequency-dependent α_k . The wiener deconvolution cannot reconstruct the frequency components which have been degraded with the noise. This makes regularization a critical step in deconvolution. If the α is selected higher than the actual noise of the data, information will be cut with the filtering. Furthermore, filtered data with low α values would have artefacts that result in a falsely high measured field strength. A common approach to optimize the α is L-curve regularization [35] and proven to be suitable for qMFM techniques [36,37].

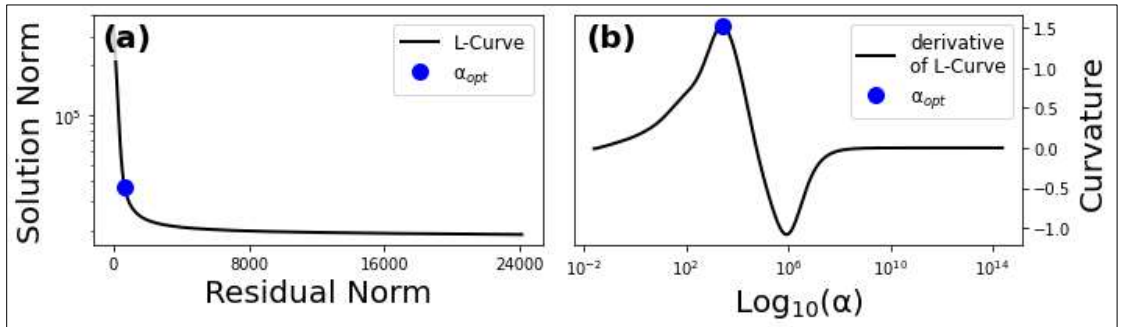


Figure 2.2. L-Curve Regularization **a.** The norm of the deconvolution as a function of norm residue. **b.** The curvature of plot (a) as a function of different α values. The blue dot marks the optimized α .

L-Curve is the plot of the norm of the deconvolution result versus the norm of the residue for varying α . The general shape of this curve looks like the letter “L” and the

maximum curvature of this curve will be the corner of the “L-Shape” (Figure 2.2a). This maximum can be derived by checking the curvature of the plot against α (Figure 2.2b). Briefly, using the L-Curve regularized Wiener deconvolution, F (or T) can be deconvolved accurately from the G . However, for a standard MFM measurement none of the convolution inputs, T and G is known priori. In the next section, the concept of the calibration will be discussed and the calculation of the effective surface charge of a known sample will be explained which will be used to reconstruct the TF of the MFM tip by using Wiener deconvolution.

2.3.2. Conceptualization of the Calibration

The transfer function $TF(\mathbf{k})$ strongly depends on the accuracy of the mechanical properties of the cantilever $-Q$ and c - and the knowledge of the reference sample. However, quantitative MFM is a technique combining at least two separate and sequential measurements from two different samples; a reference measurement for calibration of the tip and a quantitative field/charge density measurement. While the sequential measurements performed without changing the measurement setup, the mechanical properties cancel out as they enter both $TF(\mathbf{k})$ and sample charge/field deconvolutions. Therefore, accurate knowledge of Q and c is not mandatory as long as they remain unchanged. However, the importance of higher Q values stand still as it is directly related to the resolution of the measurement (see Chapter 2.2).

In general, the calibration measurement is performed on a well-known reference sample. Two main parameters define the suitability of the reference sample for tip calibration. The first and most important one the micromagnetic and macroscopic properties of the reference sample. The second one is the common spatial frequency range of the magnetic features present in the samples. In another word, both samples, reference and target samples, should have similar spatial frequency spectra to have an accurate measurement of the stray field. The latter one will be discussed in chapter 5.4 where the new reference sample was introduced and chapter 6.4.2. where the validation

of the qMFM approach was studied by MFM simulations using the NV-center measured tip stray field profile

In the previous section, it was discussed in detail that to reconstruct the $TF(\mathbf{k})$, the effective charge density of the sample must be estimated and the accuracy of this estimate strongly depends on the precision of the measurement. There are various methods to reconstruct the $TF(\mathbf{k})$. Some examples are using the effective surface charge of the sample $\sigma_{eff}^r(\mathbf{k})$, sample's stray field distribution at a certain height $(\widehat{H}_z^r(\mathbf{k}, z))$ or the sample's stray field gradient at a certain height $\frac{d\widehat{H}_z^r(\mathbf{k}, z)}{dz}$. This “certain height” is usually the tip apex ($z = \text{Tip surface distance}$) or the sample surface ($z = 0$). However, any height can be used mathematically. The most important point is one should be aware that the reconstructed $TF(\mathbf{k})$ will change according to the selection of this input. In the cases for the given examples, $TF(\mathbf{k})$ will be related to the stray field gradient of the tip for the first two and the stray field for the third one. The fields and field gradients can be transformed to each other in discrete (or partial) Fourier space by a simple linear operation using the wave vector matrix $(\mathbf{k})$ without loss of information. The field gradient is calculated by multiplying field with $\mathbf{k}$ —matrix;

$$\frac{d\widehat{H}_z(\mathbf{k}, z)}{dz} = \mathbf{k} \cdot \widehat{H}_z(\mathbf{k}, z) \quad (2.16)$$

In the next chapter, the calculation of the effective surface charge then the stray field/field gradient of the reference sample for $TF(\mathbf{k})$ will be explained in detail.

2.3.3. Calculation of Effective Magnetic Surface Charge Density

The reference sample for transfer function-based calibration should be calculable therefore it requires a deep understanding of its micromagnetic properties. For a well-known magnetic structure, the effective magnetic surface charges can be calculated or simulated. The effective surface charge for a sample with uniform magnetization over the thickness is described by;

$$\sigma_{eff}^s = m(\mathbf{k}) \cdot M_s^r \cdot (1 - e^{-kd}) \quad (2.17)$$

where M_s^r is the saturation magnetization of the sample and d is the thickness of the effective magnetic layer thickness and $m(\mathbf{k})$ is the reduced magnetization. To use this approach, the sample should not have any volume charge and should be strictly magnetized out of plane direction inside the domains [23]. It also requires a well-defined reduced magnetization of the sample $m(\mathbf{z})$ (or $m(\mathbf{k})$ in Fourier space). Reduced magnetization can be calculated by using the recorded phase shift of the MFM data. The up and down magnetized domains of the sample can be estimated by discriminating the raw (or lever corrected) MFM data into a binary matrix (1, -1) (Figure 2.3).

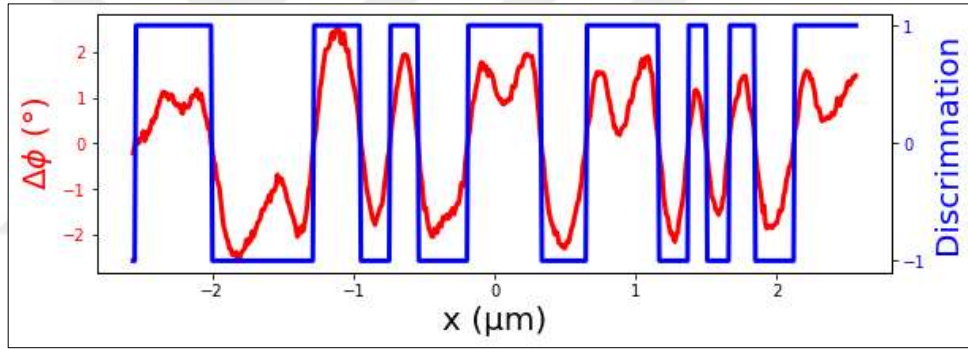


Figure 2.3. The domain guessing from MFM data. The MFM data (red) is discriminated (blue) to ± 1 to define the rough distribution of the dipole orientations over the measurement area.

However, the obtained domain distribution by this method ignores the domain walls and consider one-pixel sized domain transitions. To calculate the reduced magnetization with smooth domain transitions, the domain wall function should be convoluted with the discriminated data thus the samples domain width should be well-defined [36,38]. Domain walls are the regions that mark the transition of two different magnetic domains continuously. This general definition leads to various models for domain walls. The classical definition is shown by Lilley [39] as $\pi\sqrt{A_{ex}/K_{u1}}$. The parameters to calculate the domain wall width (δ_{DW}) can be either measured or simulated by using

micromagnetic or molecular simulations. The micromagnetic simulations can be also used to verify the measured parameters. The domain wall kernel to be convoluted with the discriminated data is defined by the z-component of the magnetization in a domain wall region;

$$m_z^{DW}(r) = \tanh\left(\frac{\pi r}{\delta_{DW}}\right) \quad (2.18)$$

where the kernel can be calculated by;

$$f^{DW}(x, y) = \text{sech}^2\left(\frac{\pi\sqrt{x^2 + y^2}}{\delta_{DW}}\right) \quad (2.19)$$

where δ_{DW} is the width of the 180° Bloch domain wall. In Figure 2.3 the convolution of the domain function with a discriminated domain for magnetization calculation is given.

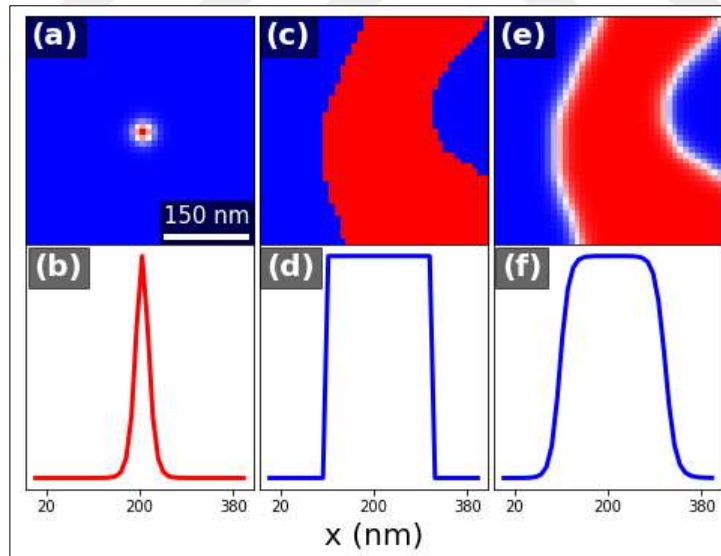


Figure 2.4. Domain wall convolution. **a** The domain wall function defined by the $\tanh()$ function. **c** The discriminated domain structure to ± 1 for up and down dipole orientation. **e** The reduced magnetization m_z calculated by the convolution of **(a)** and **(c)**. **b, d, f.** The line plots from **(a), (c), (e)** respectively.

In Figure 2.4a,b the domain wall kernel calculated by using the equation (2.19) is shown. A large, dummy domain wall width of 50 nm was selected randomly but high enough to emphasize the effect of the domain wall convolution (Figure 2.4c-f). It is obvious that the resolution of the domain wall kernel is low, however, one should take into consideration that a typical Pt/Co sample have domain wall width in the order of tens of nanometers and the resolution of the MFM is also around 10 nm. This results in a domain wall kernel formed by just a handful of pixels. However, the accuracy of the domain wall convolution is confirmed by using micromagnetic simulations and discussed in chapter 4.2. The effective surface charge σ_{eff} can be calculated by using the parameters discussed above ($m(\mathbf{k})$, M_s , d) and used to deconvolve the $TF(\mathbf{k})$ from the MFM data. Furthermore, the perpendicular stray field due to the magnetization of the sample can also be calculated by multiplying σ_{eff} with an exponential decay function;

$$H_z^r(\mathbf{k}, z') = \frac{1}{2} \sigma_{eff}^r(\mathbf{k}) \cdot e^{-kz'} \quad (2.19)$$

where z is the height from which the stray field is calculated. In Figure 2.5 an example of calculated σ_{eff} and H_z is given and it can be seen that the sharp signals at domain edges are decayed too fast with the increasing height. It is due to the nature of the exponential decay function which depends on both z and $\mathbf{k} \rightarrow (x, y)$. This emphasizes that the smaller structures will be decayed faster with increasing height. This is also a problem in the accuracy of the qMFM deconvolution for measuring the effective surface charge. The contributions from the narrow domain walls are sharp structures thus their wave vectors are located in the high-frequency regime of the Fourier spectrum. As was highlighted in chapter 2.3.1, the deconvolution cannot reconstruct the frequencies decayed by the noise. Sharp peaks are due to the narrow domain wall width and they usually filtered out during the deconvolution as their spatial size is similar to the noise. That is why the effective surface charge density measured by qMFM is accurate in terms of amplitude and width but have limited resolution for structures below 20-30 nm.

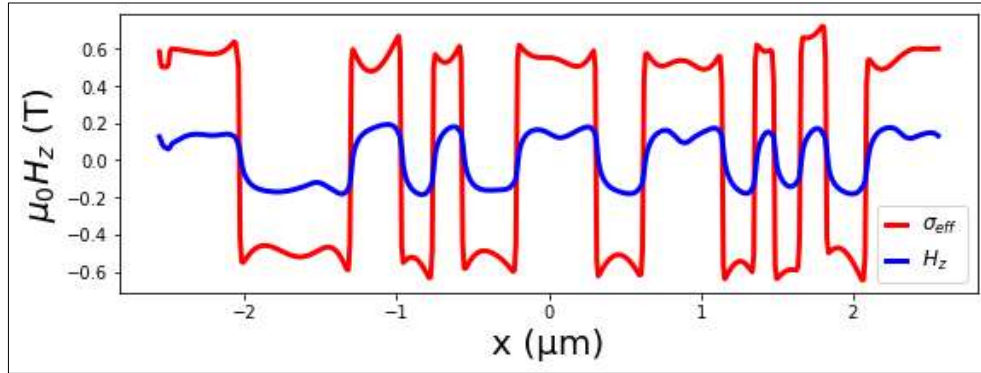


Figure 2.5. Calculation of effective surface charge density (red) and stray field (blue) of a well-known reference sample. The stray field is calculated at $z=40$ nm away from the surface.

In summary, while the micromagnetic and macroscopic properties of the reference sample are well-defined its effective surface charge density or the perpendicular stray field distribution can be calculated and used to reconstruct the transfer function.

2.3.4. Deconvolution of the Transfer Function

Using the equation (2.15) the deconvolution of the transfer function can be calculated where the $\hat{G}(\mathbf{k}, z)$ is the MFM data of the reference sample in phase shift units and $\hat{F}(\mathbf{k}, q)$ is the effective surface charge or field/field gradient of the reference sample in A/m or A/m² units.

In Figure 2.6a, transfer functions reconstructed for different α values have been demonstrated by marking their relevant position on the L-Curve to emphasize the importance of the regularization. In Figure 2.6b the Fourier spectra of these transfer functions are plotted against the spectral frequency. The low-frequency regime of the spectra is related to relatively larger magnetic features. The largest magnetic feature in MFM data can be the full scanning range and the smallest feature can be one pixel. The measurement used in Figure 2.6 has been performed over a 5.12 to 5.12 μm scan range with a pixel size of 10 nm. Therefore, the minimum and maximum $\mathbf{k}$ found in the spectra are 0. 6 μm^{-1} and 314 μm^{-1} . While the minimum magnetic feature to be measured in the

system can be defined priori, this knowledge can be used to check the quality of the regularization.

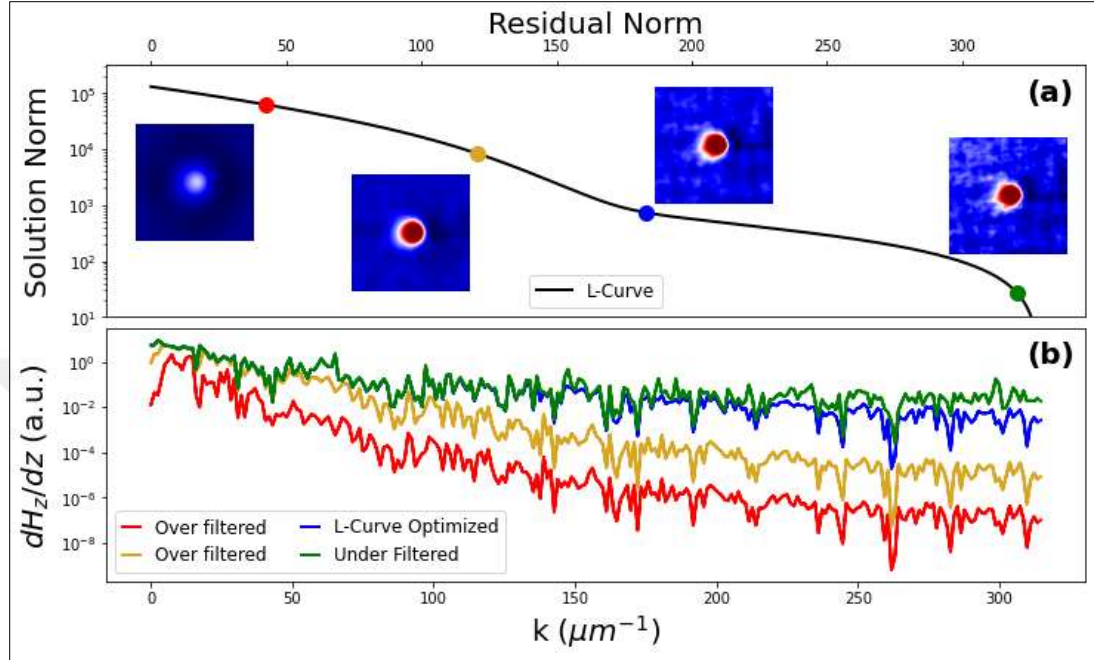


Figure 2.6. Regularization of the transfer function deconvolution. **a.** The L-curve of the Transfer Function deconvolution by MFM data and effective surface charge. The inset figures are the transfer functions of the MFM tip deconvolved with different α values marked on the L-curve with colors red, yellow, blue and green. **b.** The Fourier spectra of the deconvolved transfer functions shown as an inset to **a.** The red and yellow marked transfer functions are over-filtered, the green is under-filtered. The transfer function deconvolved with L-curve optimized α is marked with blue.

The comparison of red and yellow plots with the green plot in Figure 2.6b gives an idea about how the filtering works. The green curve is a nearly zero-filtered image and the red plot is a high-over filtered image. The yellow plot is an over-filtered image but not as dramatic as the red marked one. In all three Fourier spectra (blue, yellow, red), the high-frequency regime is filtered out compared to the green spectra. However, for blue spectra, the frequencies around $150 \mu\text{m}^{-1}$ are started to be cut-off and the lower frequencies are retained. This threshold frequency is related to the structures around 40 nm sizes. Even though the structures can be either related to magnetic features or noise, this threshold frequency is accurate to reconstruct the field from the data. However, for

yellow and red spectra, the low frequencies are also filtered out together with the high frequencies related to the noise spectrum. Moreover, for the red spectrum, even the largest features are cut off. The effect of the over filtering can be seen in the insets of Figure 2.6a. The intensity is significantly lower for the over-filtered TF . (more than a factor of 5). Furthermore, the circle around the peak is often considered a sign of excessive filtering. While the transfer function of the optimum-regularized (blue) deconvolution has considerably lower noise than under-filtered deconvolution (green) its intensity did not change significantly. This is a good example of an accurately regularized deconvolution. The over-filtered transfer function marked by the yellow can be mistaken as an accurate regularization due to its smooth distribution.

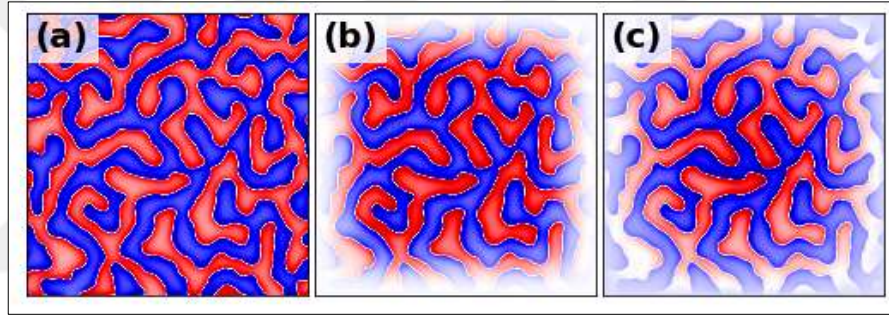


Figure 2.7. The windowing of the deconvolution inputs. a. The non-windowed σ_{eff} . **b,** **c** The windowed image of the σ_{eff} (a) by using Tukey and Welch functions respectively.

This is a common mistake in regularization but can be solved by checking the Fourier spectrum of the regularization. In some cases, due to the sizes of the magnetic features targeted to be measured, the resolution and signal to noise ratio (SNR) of the instrument, L-curve does not work properly. That is why it is important to always mind the Fourier spectrum of the regularization and be sure that the optimized α_{opt} is valid.

Another important problem in deconvolution is the discontinuity of the image boundaries. As the magnetic domains usually expand larger than the measured scan area, they have a discontinuity in the image borders. Due to these discontinuities, the deconvolution in partial Fourier space results in cross-shaped artefacts in the transfer function. To solve this problem, the inputs of the deconvolution is smoothened by using

windowing functions. Two common windowing functions used for smoothing the deconvolution inputs are Tukey [40,41] and Welch [41] functions. In Figure 2.7 an effective surface charge distributions are shown without any windowing (a), windowed with Tukey (b) and Welch (c) functions. In the study of Necas et.al., the best windowing function was found to be the Welch [37].

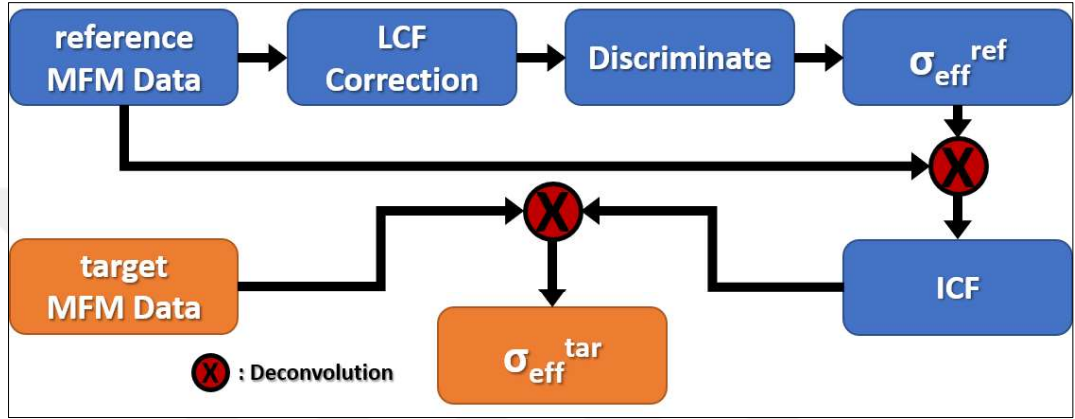


Figure 2.8. The schematic diagram of the qMFM. The blue boxes are related to the tip calibration and the orange boxes are related to the target sample.

2.3.5. Quantitative Field Measurement

The reconstructed transfer function can be used for measuring the stray field of a target sample. The target sample is the sample that targeted to be quantitatively measured. In Figure 2.8 the summary of the basic steps of a qMFM is shown. The blue boxes are related to the reference sample measurement/calculations and the orange boxes are related to the target sample measurement/calculations.

The diagram given in the figure is for calibration on the sample surface due to the σ_{eff} used in the calibration of the tip. Therefore, the ICF is related to the tip's 2D stray field gradient profile on the sample surface plane. The 2nd deconvolution by using this ICF gives the σ_{eff} of the target sample. The stray field at any desired height can be calculated by using σ_{eff}^{tar} result without losing any information. The same result could be obtained by calibrating the tip with H_z of the reference sample at $z = \text{tip apex}$. In this

case, the ICF would be the tip's stray field gradient on the plane at the tip apex. However, the calibrations at the tip apex usually result in higher noise due to the amplified artefacts in the Fourier spectrum. In the calibrations on the sample surface, these artefacts are decayed and the result is much smoother. That is why most of the calibrations and deconvolutions in this study will be performed on a sample surface or another distance that is not close to the tip apex.



3. Ti/Pt/Co MAGNETIC MULTILAYERS

In literature various Pt/Co multilayers are widely used due to their high magnetic anisotropy and tunable magnetic properties. The strength of the perpendicular magnetic anisotropy strongly depends on interface roughness [44,45], layer thickness [46-48], surface roughness [49,50] and strain in the interface [51-53]. Therefore, the growth parameters have a high impact on the PMA [54-57]. The sharpness of the interface holds great importance for PMA. One of the most common methods to control the interface quality is growing a seed layer below the multilayer. Most common seed layers used Ta, Pt and TaO_x [46,51,55-57]. Titanium (Ti) is known for its adhesive behavior and used in PZT applications as a seed layer. In these studies, it has been observed that Ti helps Lead-zirconate-titanate (PZT) film to be grown in fcc(111) structure [58,59]. Only a few studies are using Ti as a seed layer for NM/FM multilayers [60,61] However, its effect was not studied in details

In this part of the study, a sub-nm Ti seed layer was used to promote the PMA in single stack Pt/Co/Pt magnetic multilayers and investigate its effect on structural and magnetic parameters. Afterwards, various repetitions of [Pt/Co/Pt] multilayers were deposited by inserting a Ti buffer layer to control the structure of the sample and tailor the magnetic properties such as magnetic domain size and the strength of the magnetization. After the magnetic characterizations, one of these multilayer stacks was selected to be a candidate as a reference sample for MFM calibrations which was the main purpose of the dissertation. In the later chapters, the micromagnetic properties of that sample investigated by simulations will be reported and its validity as a reference sample will be cross-validated by using a well-known and widely used Co/Pt reference sample.

3.1. Experimental techniques

The samples were prepared by magnetron sputter deposition in a UHV cluster. The UHV cluster has several sections for different purposes; Loading section, deposition chamber, Analysis chamber. The system allows to load up to ten sample holders at once. The complex UHV system allows preparation and analysis of the samples sequentially in-situ without exposing them to atmospheric conditions. This allows to characterize the samples following the depositions of each layer and uniquely define the X-ray photoemission spectrum of each type of sample. The unique “fingerprint” spectra of the samples ensure high reproducibility.

The magnetic characterizations of the samples were performed ex-situ. The magnetization curves of the samples were measured by using magneto-optical Kerr effect (MOKE) and vibrating sample magnetometry (VSM). The domain structures of the samples in remanence magnetization were imaged by using MFM. In the next section, the experimental techniques used in the study will be explained in detail.

3.1.1. Magnetron Sputter Deposition

Sputter deposition is a physical vapor deposition technique based on controlled plasma etching of target material onto a substrate. In magnetron sputtering the plasma is trapped near to target surface by using a magnetic field. This trapped plasma etches the surface of the target and ejects target material. Unlike e-beam deposition or molecular beam epitaxy (MBE), the minimum size of the deposited material is not molecular but in form of clusters. The size of the cluster depends on the intrinsic properties of the target material, and the momentum of the ions. Thus, the cluster size can be controlled by tuning the momentum of the ions. According to the Bragg equation, increasing sputtering power increases the momentum of the ions so the cluster size of the ejected particles [62]. Another method is to control the gas pressure and type. Ar^+ , He^+ , Xe^+ are the most common ions used in sputter deposition. However, in reactive

depositions, other gas sources such as O₂, H₂ and/or N₂ can be also used as a sole or partial ion source.

During deposition, a power source is used to apply a voltage between cathode and anode to ionize the gas and control the plasma. In magnetron sputtering target is used as cathode. Ions accelerate toward the cathode and strike the surface so the target clusters are sputtered. In most cases, the substrate is used as an anode but it is not mandatory. For the sputtering system used in this study, substrates were grounded.

The magnetron sputtering usually classified under three sections according to the power source used in depositions; DC, pulsed-DC and RF. Usually, different sources are used for different types of targets or depositions. DC voltage can accumulate charge on the target surface if it's not conductive enough. Due to the accumulated charge, plasma intensity can decrease gradually and lost eventually. RF source overcomes this charge accumulation problem thus used commonly for reactive depositions, insulator targets and ferromagnets. However, RF source has other disadvantages such as low sputtering rate and power reflections. Pulsed DC is an alternative method combining the characteristics of DC and RF. In pDC depositions, DC voltage is applied by pulses of 10 to 100 kHz. Pt, Co and Ti layers were deposited by using DC, RF and pDC power sources respectively. The thicknesses of the films were measured during deposition by a quartz crystal microbalance (QCM) monitor. The QCM was calibrated by XPS for each element priori. The details of the calibration and deposition conditions will be discussed in the results section (chapter 3.2).

3.1.2. X-Ray Photoelectron Spectroscopy

Surface electron spectroscopies are analytical tools used to study the electronic and chemical state of the atoms and molecules on/near to the surface [63,64]. The x-ray photoelectron spectroscopy is a special type of surface electron spectroscopies where the probe for the surface electron emission is an x-ray source. Some of the other types of electron spectroscopies are Auger electron spectroscopy (AES), electron energy loss spectroscopy (EELS) and ultra-violet photoelectron spectroscopy (UPS).

XPS was introduced by Siegbahn during the 1960s with the name of “Electron Spectroscopy for Chemical Analysis (ESCA)” and based on Einstein’s photoelectric effect [60]. The technique became commercial around the 1970s and Siegbahn awarded with Nobel prize in 1981 for developing XPS.

The electrons emitted due to photon radiation are called photoelectrons and their energy is defined by Einstein’s equation of photoelectric effect [58];

$$E_K = h\nu - E_B - \omega \quad (3.1)$$

where the E_K , E_B , $h\nu$ and ω are kinetic energy of the photoelectron, its binding energy to the corresponding energy level, the energy of the excitation source and work function respectively. As the binding energies are unique for different elements, the XPS spectrum can be used to define the elemental composition by spectroscopic measurements of the E_B . The photon energies used in XPS are usually soft x-ray (<2keV). In laboratory type XPS systems Mg and Al anodes are used for their $K\alpha$ radiation with the 1253.6 eV and 1486.6 eV energies. The photons with this energy can penetrate depths in the order of micrometers. However, the photoelectron cannot travel that much distances without losing most of its energy. Thus, the signal in XPS (after background correction due to the inelastic scatterings) is usually from depths around 5-10 nm. This property makes XPS a highly surface sensitive characterization technique.

Table 3.1. The electron population on subshells and its effect on the peak area in XPS

Subshell	j	Area ratio
s	1/2	-
p	1/2, 3/2	1:2
d	3/2, 5/2	2:3
f	5/2, 7/2	3:4

An XPS spectrum can be classified into two sections; primary and secondary features. The primary features are the core energy levels, valance level and auger series. The secondary features are the variations in the primary features such as tails and

satellite peaks. The core energy levels are observed in two different types depending on their subshells; s-levels are singlets and non-s-levels are doublets. The doublet structure is the result of spin-orbit coupling. The electron populations of the subshells define the area ratio between the two peaks of the doublets. This ratio of the electron population for different subshells are given in Table 3.1.

3.1.3. Magneto-Optical Kerr Effect

Kerr effect is one special type of magneto-optical effect observed by John Kerr in 1876. It is similar to the Faraday effect which describes the change in the polarization of a light beam passing through a magnetized medium [65]. In the magneto-optical Kerr effect (MOKE), the ellipticity and the angle of the polarization of an s- or p polarized light beam changes when it's reflected from a magnetized material [66]. In 1884, Kundt developed the MOKE technique based on Kerr's study by measuring the change in the polarization in the reflected light beam from Fe, Co and Ni films grown on a glass substrate [67]. Today, MOKE is used to measure the magnetization curves [68,69], imaging of magnetic domains [70,71] and terra Hertz (TH) measurements of magnetization dynamics [72]. Even though there are various other techniques to measure the magnetic hysteresis curves of ferromagnetic materials (i.e., VSM, SQUID) MOKE has the surface sensitivity advantage which makes it possible to measure very thin magnetic layers faster with high precision [73-75].

The interaction between the magnetization of material and the electromagnetic wave can be given by the generalized dielectric law;

$$\mathbf{D} = \varepsilon'(\mathbf{E} + iQ_V \mathbf{m} \times \mathbf{E}) \quad (3.2)$$

where the $\mathbf{m}$, ε' and Q_V are the magnetization of the sample, generalized dielectric tensor and the Voigt constant of the material respectively.

The MOKE measurements have three different geometries defined by the orientation of the magnetization with respect to the reflection plane (Figure 3.1). These

geometries are called polar, for the magnetization perpendicular to the sample surface, longitudinal, for the magnetization parallel to the sample surface and lying in the reflection plane, and transverse for magnetization parallel to the sample surface but perpendicular to the reflection plane.

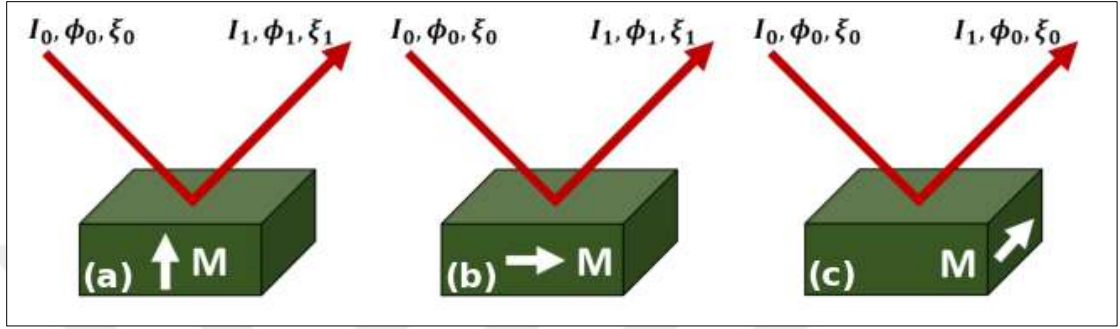


Figure 3.1. Different geometries of MOKE, for Polar (a), Longitudinal (b) and Transverse (c). The rotation ϕ_i , ellipticity ξ_i of the polarization and the intensity I_i of the p-polarized incident ($i = 0$) and reflected ($i = 1$) beam is shown on the diagram where the $\phi_{0=1}$ and $\xi_{0=1}$ did not change in transverse geometry.

The reflection plane is the plane defined by the incident and reflected light beam. This plane is perpendicular to the sample surface. For the case with a magnetization perpendicular to the surface (Polar), the linearly polarized incident light beam will induce an oscillation parallel to the reflection plane. Therefore, the reflected light will be polarized in the same plane with the incident light ($\mathbf{R}_N$). Furthermore, the Lorentz force induces a secondary vibrational motion perpendicular to the magnetization and primary motion ($\mathbf{m} \times \mathbf{E}$). This secondary motion will generate a Faraday amplitude ($\mathbf{R}_F$) for transmission and Kerr amplitude ($\mathbf{R}_K$). The superposition of the secondary amplitudes with the primary amplitude ($\mathbf{R}_N$) will generate a polarization rotation depending on the magnetization for transmission (Faraday Effect; $\mathbf{R}_N$ with $\mathbf{R}_F$) or for reflection (Kerr Effect; $\mathbf{R}_N$ with $\mathbf{R}_K$). For the case with a magnetization parallel to both sample surface and reflection plane (Longitudinal), it will generate a polarization rotation both parallel to this plane and perpendicular to the incident beam light [76]. For the transverse case, the magnetization is perpendicular to the reflection plane and parallel to the sample

surface. In this special case, no magneto-optical effects occur during transmission due to the cross product ($\mathbf{m} \times \mathbf{E}$). However, for the reflection, the parallel polarization will generate a Kerr amplitude but no rotation of polarization or change in the ellipticity (Figure 3.1b) [76,77].

3.1.4. Vibrating Sample Magnetometer

A vibrating sample magnetometer (VSM) is a magnetic measurement technique based on measuring the induction in a pick-up coil which is proportional to the magnetization by the vibration of a magnetic body. The vibration of the sample will create an alternative magnetic field proportional to its magnetization. According to Faraday's law of induction, this time-dependent magnetic field will induce an electric field in the coils which will be read as the signal of VSM. The signal from the pickup coils is compared with the signal from the reference coils with permeant magnets. In VSM the magnetization of the sample is static so eddy current effects can be neglected [78].

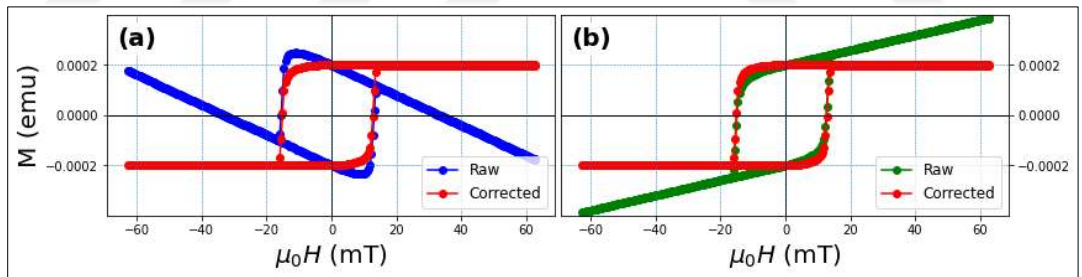


Figure 3.2. Correction of VSM data. The corrected VSM data (red) for diamagnetic contribution (a-blue) and for paramagnetic contributions (b-green).

The data measured in VSM will be the total magnetic signal coming from the whole sample including the substrate or holder. Therefore, in hysteresis measurements of ferromagnetic systems, a correction is usually required for diamagnetic and paramagnetic contributions (Figure 3.2).

3.2. Results

3.2.1. Deposition Specifications

The samples were deposited in UHV conditions. The base pressure of the deposition chamber was $3 \times 10^{-9} \text{ mbar}$. The argon used in depositions has the initial purity of 99.99% as reported by the supplier and further filtered. The substrate cleaning was performed in two steps. Before the loading, substrates were cleaned with distilled water and pure ethanol. The in-situ cleaning was performed by thermal annealing at 450°C for 30 min to remove the residual contaminations on the surface. The purity of the substrates was confirmed by using X-ray photoelectron spectroscopy. The argon flow was controlled by a flow meter during the deposition. The pressure of the deposition chamber during depositions was set to $2.5 \times 10^{-3} \text{ mbar}$. The Ti, Pt and Co films were deposited by using pulsed DC, DC and RF power sources. The cluster size of the sputtered particles is proportional to the deposition power and this directly affects the roughness of the film. As the interface smoothness is one of the most important parameters to obtain high perpendicular anisotropy in NM/Co multilayers, the lowest possible deposition powers were selected by checking the stability of the plasma. The deposition powers densities for Ti, Pt and Co were 55 mW/cm^2 , 11 mW/cm^2 and 82 mW/cm^2 respectively. The deposition rates for each of these power densities specific to the target material were calibrated by using XPS. The calibration is explained in the following chapter. The stability of the deposition was monitored simultaneously by using a quartz crystal microbalance thickness monitor (Maxtech Inc., 6 kHz reference crystal). The QCM was calibrated by using the deposition rates calculated by XPS.

3.2.2. Calibration of Deposition

The XPS calibration of deposition rates was performed by using the thickness-dependent change in the photoelectron intensity. The deposition rate depends on

various parameters such as Ar pressure, the distance between substrate and target, and sputtering power. Therefore, all the parameters were kept constant throughout the calibration.

The intensity of the photoelectron transmitting through a medium is described by modified Beer-Lambert equation [79];

$$I_1^A = I_0^A \cdot e^{-\lambda/d} \quad (3.3)$$

where I_1 and I_0 are the photoelectron intensity before and after the transmission, d is the thickness of the medium and λ is the inelastic mean path of the photoelectron passing through the medium. Using this approach, the thickness of sample B can be calculated by measuring the photoelectron intensity change of sample A due to the deposition of sample B. The inelastic mean free path $\lambda(E)$ is the mean distance the electron can travel without losing energy with inelastic scatterings. It is specific for each medium and depends on the electron's energy.

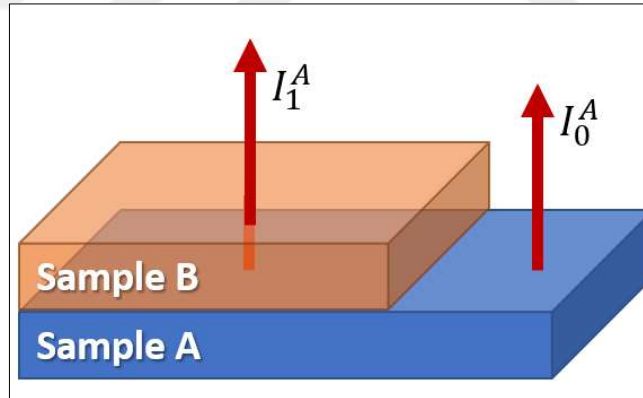


Figure 3.3. Photoemission signal transmitted through a film layer. The red arrows indicate the XPS signal from element A measured from its surface (I_0) and through the film B (I_1).

Sample A can be either a film or substrate. The initial intensity I_0^A was measured from the top of sample A. Afterwards, additional XPS measurements were performed sequentially to the sample B depositions. The intensity I_i^A is the intensity of the signal

from sample A (Figure 3.3) after the i^{th} deposition of B. By plotting the thicknesses calculated with equation (3.3) as a function of deposition time, the rate can be calculated. Furthermore, there will be a thickness limit where the I^A vanish and I^B saturates. However, the approach to saturation and vanish is not imminent, instead, it is slow. Therefore, the calibration should be performed in the linear regime.

The substrates used in the calibration were ultra-pure gold and silver foils. The Au4f energy level was used for Co calibrations. The change in the Ag3d level was used to calibrate the Pt and Ti depositions (Figure 3.4a-c). The substrates were mechanically polished then loaded to the UHV system. The substrates purity was further ensured by sequential Ar plasma etchings and XPS scans.

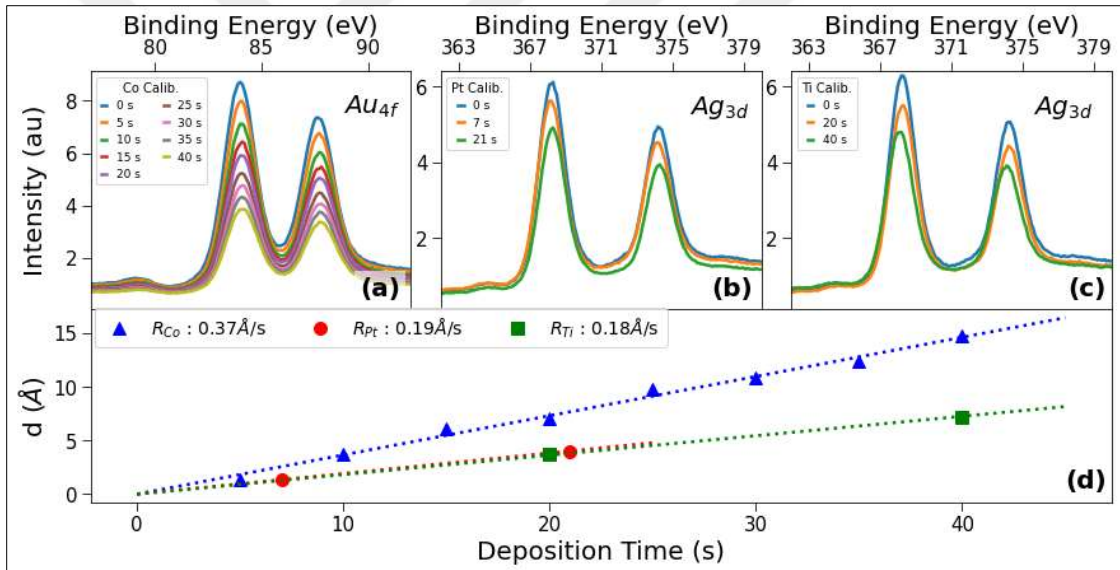


Figure 3.4. X-ray Photoemission spectra for calibration of deposition. a. Au4f, b., c. Ag3d energy levels used for Co, Pt and Ti calibrations respectively. The spectra are recorded after a certain deposition time sequentially. d. The thickness of the depositions as a function of deposition time. Linear fitting of the thicknesses gives the deposition rate for the related element.

The inelastic mean-free paths of the Au4f and Ag3d passing through Co, Pt and Ti are obtained from the NIST database-Tanuma model; $\lambda_{Ti}(1118.47 \text{ eV}) = 25.27 \text{ Å}$, $\lambda_{Pt}(1118.47 \text{ eV}) = 14.17 \text{ Å}$, $\lambda_{Co}(1169.55 \text{ eV}) = 17.38 \text{ Å}$. In Figure 3.4d the calibrated

deposition rates are given. The thickness plot as a function of deposition time is linearly fitted with zero crossings. The slope of the fit gives the deposition rate.

Table 3.2. Magnetron sputter deposition parameters for Titanium, Platinum and Cobalt.

Target	P. Source	P. Density	Distance	IMFP	Dep. Rate
Titanium (Ti)	pDC	55 mW/cm^2	100 mm	25.27 Å	0.18 Å/sec
Platinum (Pt)	DC	11 mW/cm^2	70 mm	14.17 Å	0.19 Å/sec
Cobalt (Co)	RF	82 mW/cm^2	70 mm	17.38 Å	0.37 Å/sec

In table 3.2 the specifications such as power source, power density, deposition rate and sample-target distances are given for Titanium, Platinum and Cobalt depositions.

3.2.3. Single-Stack Samples

In this first part of the dissertation, the effect of Ti was investigated by growing single stack Pt(x)/Co/Pt multilayers with and without Ti seed layer. The samples with/without Ti seed layer will be named tpcp and pcpc throughout the dissertation. The thickness of the Ti, Co and top Pt layers kept constant and were 0.3 nm, 0.5 nm and 1 nm respectively while the thickness of the bottom Pt layer varied. The hysteresis loops of the tpcp and pcpc samples with different bottom Pt layer thicknesses were compared in Figure 3.5. The thickness of the bottom Pt layer was 0.5 nm (blue), 0.8 nm (green) and 1 nm (orange).

Figure 3.5 shows the P-MOKE measurements of the tpcp (a) and pcpc (b) samples performed in MOKE laboratory in GTU, Physics Department by using a HeNe red-laser (632 nm). The pcpc measurements show a hard axis magnetization that indicates an in-plane anisotropy in the samples. On the contrary, tpcp samples with 0.8 and 1 nm bottom Pt thicknesses had perpendicular magnetic anisotropy and showed a remnant magnetization of $M_R/M_S=1$ with a near-perfect square shape. This indicates the magnetization switch as a single domain. However, for 0.5 nm bottom Pt thickness, the easy axis of the tpcp sample was in the plane. These results showed that even 0.3 nm Ti was enough to promote a perpendicular uniaxial magnetic anisotropy in Pt/Co samples.

This thickness is one of the thinnest seed layers ever reported [80,81]. However, the thickness of the bottom Pt layer should be thick enough to produce a sharp interface which was derived ($> 0.5 \text{ nm}$). To understand the effect of the Ti seed layer, the pcp and tpcp samples with 1 nm Pt thicknesses were selected to study further. These specific samples were selected due to two reasons. The first one is the P-MOKE (polar MOKE) measurement of pcp samples; the sample with 0.8 thickness has a small hysteretic behaviour in the near-zero field region. This indicates a possible dual phase in the sample. To have a pure PMA sample, the Pt thickness selected as 1 nm. The second reason is the symmetrical layer architecture of the Pt(1 nm)/Co(0.5 nm)/Pt(1 nm) sample relative to Co. Even though the recent studies in the literature are indicating uneven spin-orbit contributions from top and bottom interfaces, the difference is small [82]. Therefore, to reduce the possible DMI contribution (or at least reducing) on the perpendicular uniaxial anisotropy, the symmetric interface was selected to study the effect of Ti.

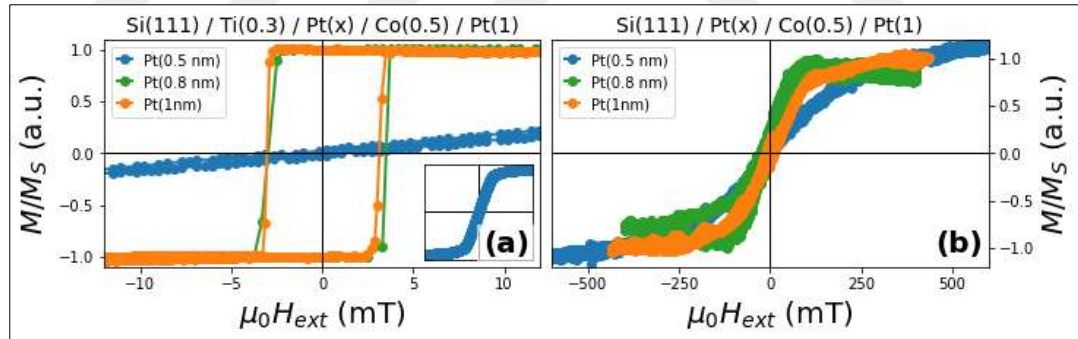


Figure 3.5. Hysteresis loops of the tpcp and pcp samples measured by P-MOKE. Normalized hysteresis loops of tpcp (a) and pcp (b) samples with bottom Pt layer thickness of 0.5 nm (blue), 0.8 nm (green) and 1 nm (orange).

The tpcp and pcp samples were investigated with XPS (in-situ) and XRD (ex-situ). The X-ray diffraction (XRD) measurements were performed at the Center of X-Ray Analytics, EMPA- Swiss Federal Laboratories for Materials Science and Technology by Dr Zoltan Balogh-Michels and Prof. Dr Antonia Neels. The samples were measured in a Bruker D8 Discover Da-Vinci diffractometer in two different modes; Grazing incidence

(GI) out of plane scan and in-plane sector scan. GI-XRD is a suitable and well-known method to investigate thin polycrystalline films. The in-plane sector scan is a set of radial in-plane scans. Between each radial scan, there is an angle of rotation. In this study, the sample is investigated in a 80° wide arc with 1° rotation between each scan. The advantage of this method is obtaining both symmetry and phase information [83]. Figures 3.14 and 3.15 are used with the permission of Dr Balogh-Michels and Dr Neels.

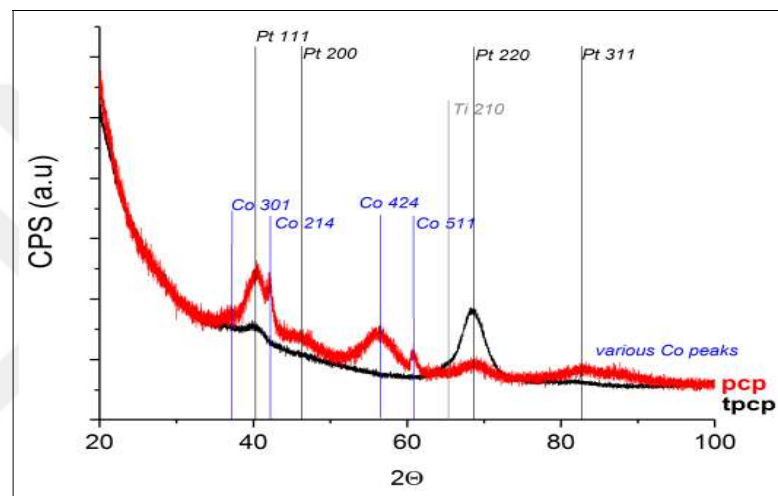


Figure 3.6. Grazing incidence XRD spectra of tpcp and pcp samples. The tpcp (black) and pcp (red) samples 2θ measurement with $< 1^\circ$ incidence angle. The blue horizontal lines mark the Co reflections. Black horizontal lines mark the Pt reflections. The substrate signal is not detected due to the low incidence angle. Co peaks are present in the pcp spectrum but absent in the tpcp spectrum. Pt has various reflections in pcp. However, the Pt (220) reflection dominates the tpcp spectra indicating a fibre textured structure.

In Figure 3.6 the GI-XRD of tpcp (black) and pcp (red) is given. Almost all possible Pt peaks and some Co peaks were observed in the spectrum of the pcp sample. However, the tpcp sample spectrum is dominated by a Pt (220) reflection. Co peaks were not detected in the tpcp measurement most probably due to the small sizes of crystallites. In the in-plane sector scan of tpcp, a Pt (220) is dominant which is perpendicular to $(1\bar{1}1)$ (Figure 3.7b). The single isotropic ring observed in the 2D sector scan indicates that Pt maintains the same angle of reflection for this specific orientation at each

different azimuthal angle measurement (Figure 3.7a). This result shows that the Pt in the tcp sample is in fibre textured structure where all of the crystallites have $(1\bar{1}1)$ orientation in out-of-plane direction but randomly oriented for in-plane direction. On the other hand, there are multiple rings observed in pc sample in-plane sector scan (Figure 3.7c, d). This shows that the pc sample is a random-oriented nano-crystalline film. The single and separate peaks observed in 2D scans are the reflections from the single crystal substrate.

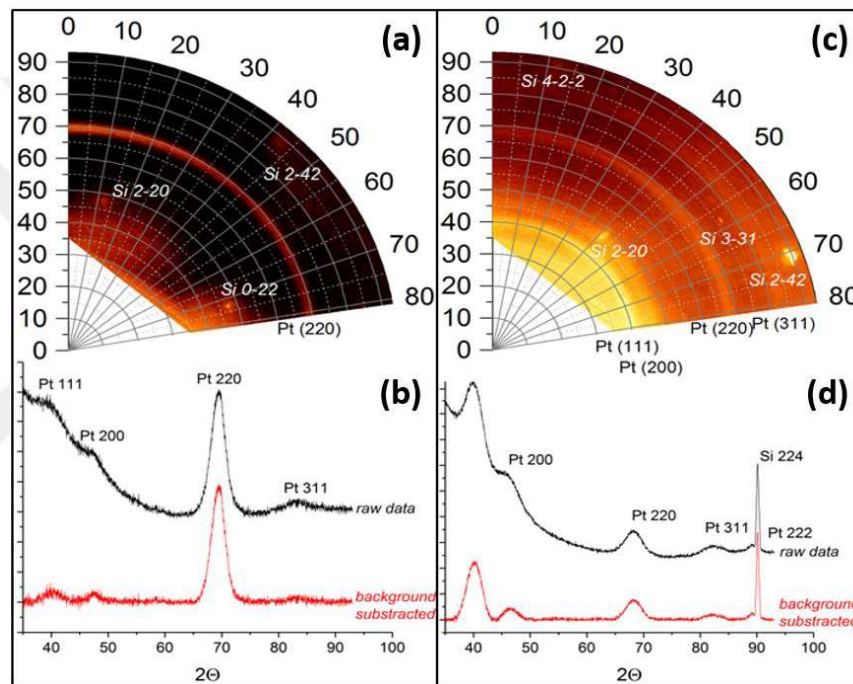


Figure 3.7. In-plane sector scan of tcp and pc samples. 80° wide arc in-plane scan with 1° rotation between each 2θ measurement for **a.** tcp and **b.** pc sample. **(b)** and **(d)** are the line scans for tcp and pc respectively. The sector scan of tcp is dominated with a single Pt (220) peak indicating a fibre-textured structure. The sector scan of pc shows various rings indicating a random-oriented nano-crystalline structure.

Both XRD measurements confirmed that the tcp samples are strongly textured and the pc samples are randomly oriented. This significant structural difference was achieved by a small amount of Ti presence which was not even enough to cover the whole surface of the substrate. In addition, the behaviour of Co reflections in the tcp

and pcp spectra shows that it grows in clusters too small to generate XRD reflections on the fibre-textured Pt. On the contrary, when the Pt grows in randomly oriented crystalline clusters, Co also forms crystalline clusters with enough size to be detected in XRD scans.

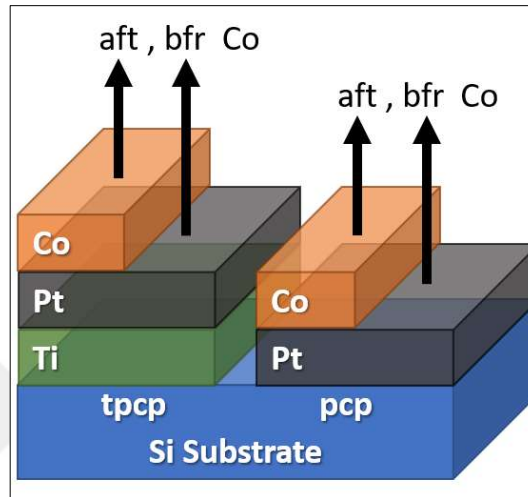


Figure 3.8. The XPS measurement scheme of the tpcp and pcp samples. The black arrows indicate the surface from where the XPS measurement is performed. Initial XPS spectra are recorded from the top of the bottom Pt surface before Co deposition onto it (bfr Co). The final XPS spectra are recorded from the top of the Co surface after the Co deposition (aft Co). The Pt4f, Si2s, Ti2p, O1s and Co2p energy levels are measured with high resolution.

The XPS measurements are performed in-situ and sequential to the depositions as is shown in Figure 3.8. The analysis carried out with a monochromatic Mg K α (1253,6 eV) light source and a SPECS-PHOIBOS 150 hemispherical charged particle analyzer (HCA) with 12 channeltron multipliers. The energy calibration of the system is periodically confirmed by using a pure gold foil. The base pressure of the analysis chamber is 1×10^{-10} mbar. The wide range survey scans are performed with 1 eV resolution to define the surface elemental composition relatively roughly. Afterwards, high-resolution (0.1 eV) window spectra of the core levels are measured.

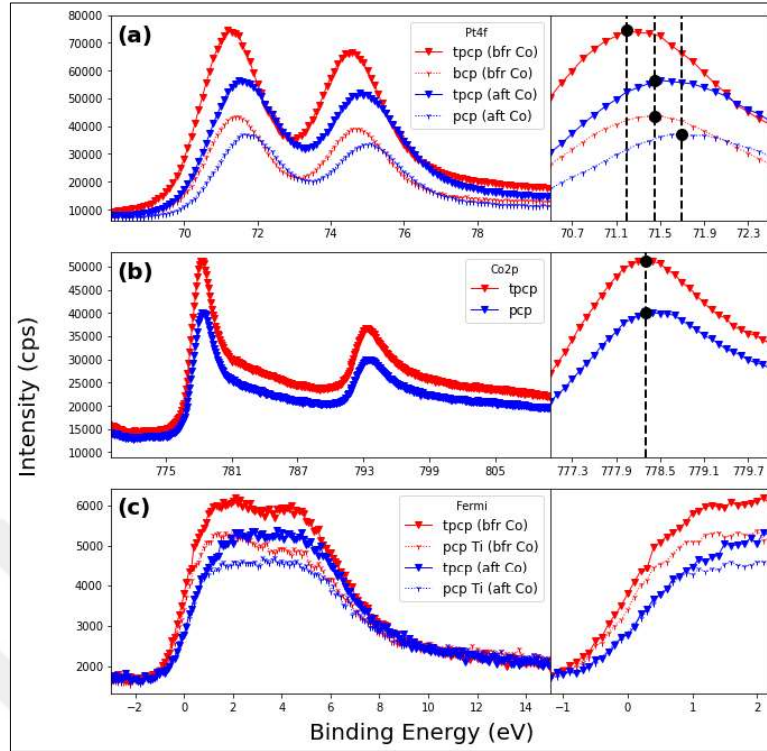


Figure 3.9. The photoemission spectra of tpcp and pcpc samples. a. The Pt4f spectra, **b.** The Co2p spectra and **c.** The Fermi region of tpcp (red) and pcpc (blue) samples before and after Co depositions. The inset plots next to the main figures are zoomed-ins to the near-max region of the spectra. The dashed lines mark the maximum intensities and used to define the energy shift.

The XPS spectra of Pt4f and the fermi region are measured before and after Co depositions. The changes in the intensity for the “before Co” deposition (bfr Co) and “after Co deposition” (aft Co) are related to the loss of intensity due to the thickness (see equation 3.3). The Fermi region is the spectra near 0 eV binding energy (BE). This region gives information about the valance band structure of the samples. However, $BE = 0 \text{ eV}$ means that the electrons measured in this region have high kinetic energy (KE) ($KE \approx h\nu$). Therefore, the Fermi region measured with an x-ray source usually results in a highly noisy spectrum. In Figure 3.9a, the 4f level of Pt is shown. In this spectrum, the bottom Pt layer’s XPS signal is compared for tpcp and pcpc and for prior and after Co deposition (bfr / aft Co) states. After Co deposition, a shift in the Pt4f peak has been observed in both tpcp and pcpc samples. Furthermore, the magnitude of the shift was

$\Delta E = 0.2 \text{ eV}$ and was the same for both types of samples. The shift is towards a higher BE regime. A similar shift is observed in Fermi region spectra (Figure 3.9c). Observing the same type of shifts and intensity changes in both tpcp and pcp measurements indicate that they are not related to the Ti layer, thus the PMA (or textured structure) observed in the tpcp sample. This shift can be interpreted as a shift toward a “less” conductive sample due to the Co deposition compared to a pure Pt surface.

However, the Pt4f signals are not identical for both tpcp and pcp samples. The average BE of the Pt4f spectrum is 71.3 eV and 71.6 eV for tpcp and pcp samples respectively. The Ti seed layer shifted the binding energy of the bottom Pt layer 0.3 eV towards the lower BE region. In literature, the XPS spectrum of Au was studied for different cluster sizes and the shift in the spectra was explained by two different peaks in XPS fits; Bulk and Surface [84].

In Figure 3.10, Pt4f spectra were fitted in the same manner. The background was calculated by using the Shirley approximation [85,86]. The peaks were fitted by Voigt functions with the shape of 0.75. The equation of a Voigt function is given by;

$$V(x) = s \cdot G(x) + (1 - s) \cdot L(x) \quad (3.4)$$

where s is the ratio of Gaussian to Lorentzian functions. The Gaussian and Lorentzian parts are described by;

$$G(x) = e^{-\frac{(x-x_0)^2}{2w^2}}, \quad L(x) = \frac{(\Gamma/2)^2}{(x - x_0) + (\Gamma/2)^2} \quad (3.5)$$

Γ and w are the width parameters for Lorentzian and Gaussian parts and the relation between them is defined by;

$$w = \frac{\Gamma}{2\sqrt{2 \ln(2)}} \quad (3.6)$$

The full width half maximum (FWHM) of the peaks were found 2.03 eV and 2.08 eV for surface and bulk peaks respectively. The width is important to define the accuracy of the fit as it cannot be below the defined resolution of the system which depends on the ion-relaxation time, the bandwidth of the detector and the bandwidth of the excitation source. Selecting wider peaks may indicate an ill-fitting which overestimate other possible phases and interactions.

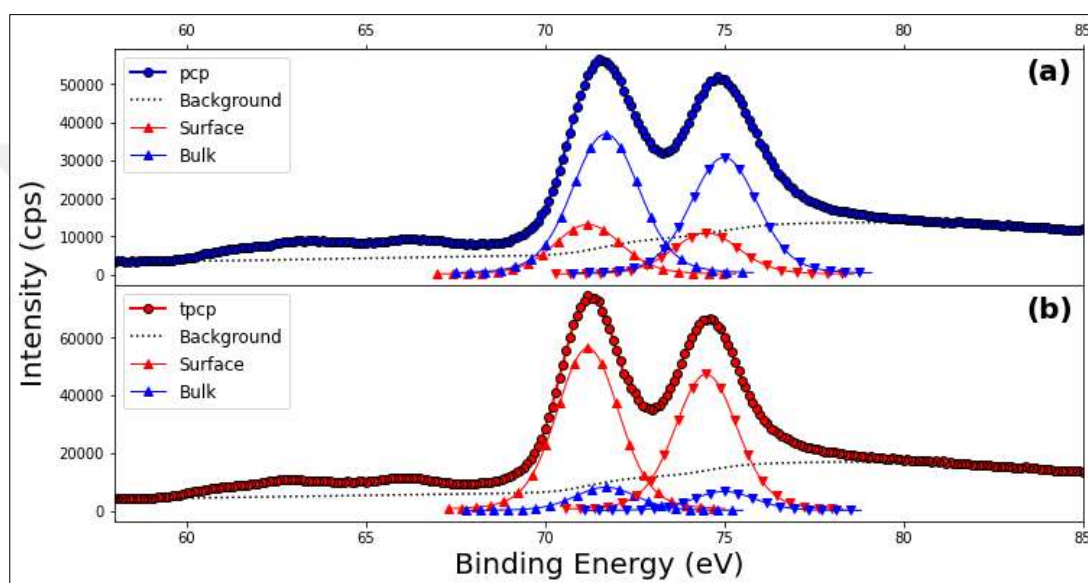


Figure 3.10. The peak fitting of Pt4f photoemission spectra. a. The pcp and **b.** The tpcp samples. A Voigt function with a 0.75 Gaussian/Lorentzian ratio was used in peak fitting. The blue and red peaks are related to the bulk and surface phases in the Pt layer.

The fitting parameters of the XPS curve fittings are selected according to the guidelines [87]. The fit results clearly show that the contributions from bulk and surface phase present in both tpcp and pcp samples. However, the relative intensity of the contributions, are different depending on the presence of the Ti seed layer. The spectrum of the tpcp sample is nearly dominated by surface peak while the pcp spectrum has a strong bulk contribution. The surface to bulk ratio were calculated by the area of the related peaks and found to be 88% to 12% for tpcp and 26% to 73% for the pcp sample. The fitting parameters and results are given in Table 3.3

Table 3.3. Fitting parameters for Pt4f of tpcp and pcp samples.

<i>Sample</i>	Peak	shape	BE (eV)	FWHM (eV)	Area x 10³	Ratio
<i>tpcp</i>	surface	0.75	71.2	2.03	1648	87%
	bulk	0.75	71.7	2.08	236	13%
<i>pcp</i>	surface	0.75	71.2	2.03	384	26%
	bulk	0.75	71.7	2.08	1081	74%

The different surface/bulk ratios in tpcp and pcp can be explained by the size of the clusters. In the pcp sample, the clusters are not big enough to dominate the XPS spectrum with bulk phase but not small enough to form a sharp interface with Pt. Therefore, a significant surface phase can be also observed in the spectrum. There are also additional STM studies performed by Melek Türksöy Öcal where the change in the surface roughness and cluster formations were studied and explained in detail [83].

On the other hand, the Co spectra for both tpcp and pcp sample does not show any sign of energy shift (Figure 3.9b). There is a change in the relative intensity of the Co however, this change is not only observed for the Co spectra but the whole spectrum. The Pt intensity is 24% lower for the pcp sample compared to the tpcp sample. It is well known that the intensity of the peaks in photoemission spectroscopies are highly sensitive to the surface roughness [88]. Briefly, Co does not show any sign of electronic interaction with the Ti layer. This indicates that the PMA is solely related to the structural changes of the Pt layer caused by the Ti seed layer. The structural changes in the Pt layer led to changes in the Pt/Co spin-orbit interaction which result in perpendicular magnetic anisotropy.

In summary, the effect of the Ti seed layer on the magnetic properties is explained by structural characterizations. Even a tiny amount of Ti could promote the fibre-textured growth on the Pt while in its absence, Pt forms relatively big, randomly oriented crystals. The large sizes of the clusters result in an increased roughness of the surface which decreases the intensity of the photoemission signal. Co clusters, on the other hand, grow as a thin layer and with a sharp interface on a well-defined fibre-texture Pt

layer, while they form relatively large clusters on the pcpc surface. This interpretation of XPS results is supported by XRD measurements where Co reflections were observed in pcpc measurements but not in tpcpc measurements. In addition, the minimum Pt thickness required to obtain sufficient Pt/Co interface and thus to obtain perpendicular magnetic anisotropy was found to be 0.5 nm.

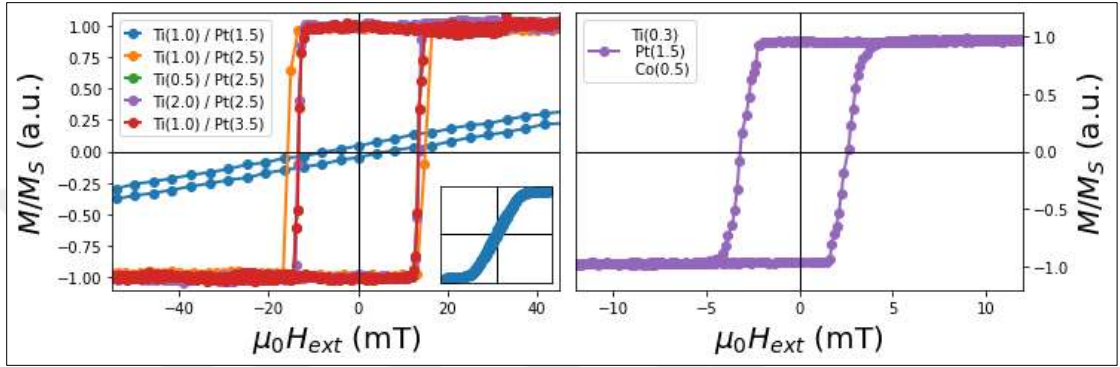


Figure 3.11. Hysteresis loops of the Ti(y)/Pt(x)/Co(z)/Pt(x) samples measured by P-MOKE. Normalized hysteresis loops of the samples with Co thickness **a.** 1.0 nm and **b.** 0.5 nm for different Pt and Ti layer thicknesses. All of the samples have PMA but the one with 1.5 nm Pt and 1 nm Co thickness.

The next set of samples was Ti(y)/Pt(x)/Co(z)/Pt(x) samples with different x, y, z values but symmetric Pt/Co-Co/Pt interfaces. In the first sample, the thickness of Pt layers increased up to 1.5 nm while the Co and Ti layers kept as 0.5 nm and 0.3 nm respectively. The sample had similar magnetic properties in terms of the shape of the magnetic hysteresis curve and the coercive field (Figure 3.11b). However, the trace tilt observed in the hysteresis curve can be interpreted as increasing magnetostatic energy due to Pt thickness. Additionally, other samples were grown with different Pt and Ti thicknesses while the Co thickness kept at 1 nm. The thickness of the Co layer $d_{Co} = 1 \text{ nm}$ is near the limits for Pt/Co multilayers with perpendicular magnetic anisotropy [89].

Table 3.4. Sample architecture and magnetic properties of single-stack Ti(y)/Pt(x)/Co(z)/Pt(x) samples

Thickness (nm)				Magnetic		
Ti	Pt ¹	Co	Pt ²	Easy Axis	$\mu_0 H_c$ (mT)	M_R/M_S
0.0	0.5	0.5	1.0	In-Plane	-	-
0.3	0.5	0.5	1.0	In-Plane	-	-
0.3	0.8	0.5	1.0	Out of Plane	3.2	1
0.0	0.8	0.5	1.0	In-Plane	-	-
0.3	1.0	0.5	1.0	Out of Plane	5.1	1
0.0	1.0	0.5	1.0	In-Plane	-	-
0.3	1.5	0.5	1.5	Out of Plane	2.9	1
1.0	1.5	1.0	1.5	In-plane	-	-
1.0	2.5	1.0	2.5	Out of Plane	16.2	1
0.5	2.5	1.0	2.5	Out of Plane	13.4	1
2.0	2.5	1.0	2.5	Out of Plane	13.5	1
1.0	3.5	1.0	3.5	Out of Plane	14.8	1

^{1,2} are the bottom and top Pt layers respectively

In Figure 3.10a, it can be seen that the Ti thickness does not have any effect on the magnetic properties of a single stack multilayer. Furthermore, the increase in Pt thickness above 2.5 nm did not change the hysteresis curve while for 1.5 nm Pt thickness, the sample showed in-plane magnetitic anisotropy. The samples with 1 nm Co layers showed a higher coercive field as expected. The relatively high coercive with a high squareness ratio of hysteresis for a Co such thickness indicates a sharp and well-defined Co/Pt interface. It is important to emphasize that the sample architecture has a single ferromagnetic layer and the contribution of Ti and Pt layers could/should be different in multiple stacks as they would affect the interlayer exchange interactions. The sample list prepared with single-stack multilayers given in table 3.4.

3.2.4. Multi-Stack Samples

The effect of the Ti is studied in terms of structural contribution by using XPS and XRD in detail. In literature, the domain size of the $[\text{Pt}/\text{Co}]_R$ multilayers is tuned by changing the repetition number R . The domain size is usually defined as a competition between domain wall energy and magnetostatic energy.

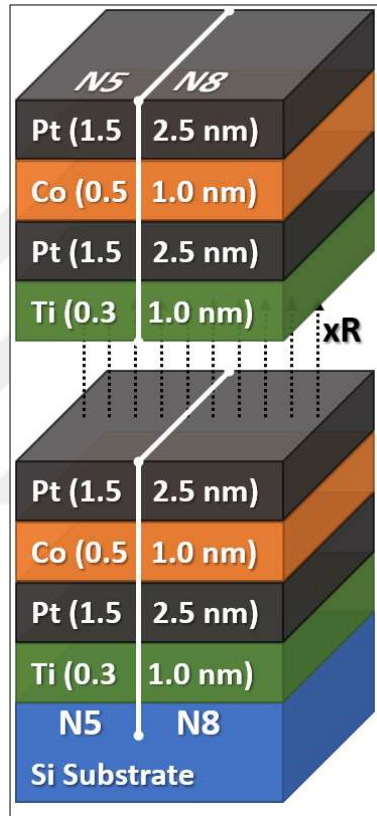


Figure 3.12. Layer architecture of N5 and N8 multi-stack samples. The repetitions of the samples include whole Ti/Pt/Co/Pt stacks. The repetition number is given by $R=1, 5, 10, 20$.

Increasing the number of magnetic layers -so the repetition number- increase the interlayer magnetostatic energy [90]. This increase in energy can be compensated by increasing the total contribution from the domain walls. Therefore, the system compensates this extra magnetostatic energy by forming more domains [91]. More domain formation in a constant area means smaller domains and a longer total length of

the domain walls. This property gives control over the domain size of the sample by tuning the repetition number. However, the number of repetitions usually comes with an increase in the total magnetic moment. Therefore, in our study, we used the Ti layer between the repetition to tune the saturation magnetization M_S alongside the average domain size $\langle D_w \rangle$. With this control, a suitable calibration sample for qMFM applications can be prepared with desired domain size and field strength.

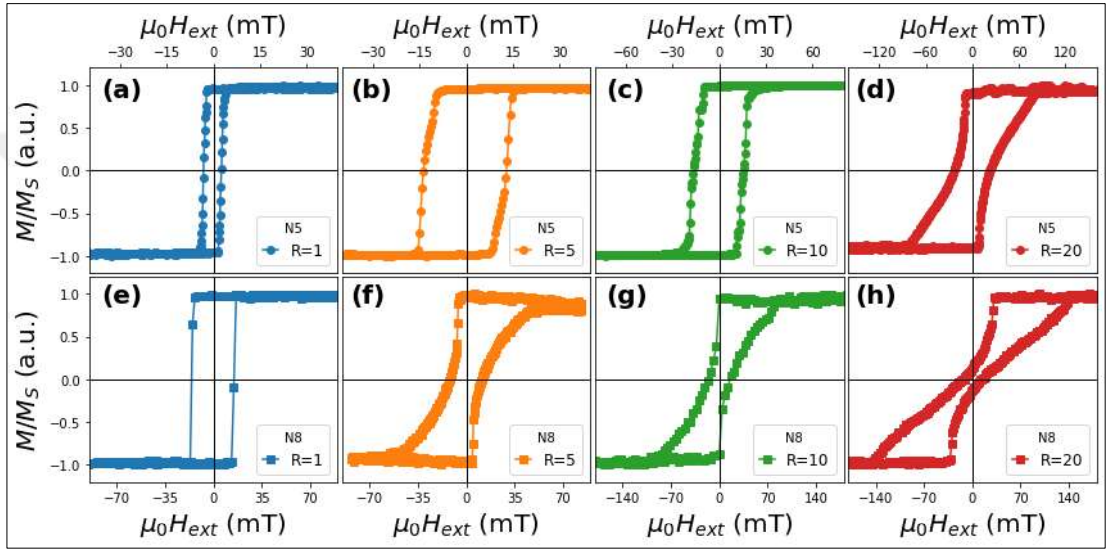


Figure 3.13. Hysteresis loops of N5RX and N8RX multi-stacks measured by P-MOKE. a-d. Normalized hysteresis loops of the N5Rx. e-h. The N8Rx multi-stacks. X indicates the value of R=1, 5, 10, 20 for blue, orange, green and red respectively.

In the following step of the study, two samples from Table 3.4 were selected for growing in multiple stacks. The samples are Ti (0.3nm) / Pt (1.5nm) / Co (0.5nm) / Pt (1.5nm) and Ti (1.0nm) / Pt (2.5nm) / Co (1.5nm) / Pt (1.5nm). These samples will be named N5RX and N8RX in the rest of the study where X indicates the value of R, the repetition number (Figure 3.12).

The P-MOKE measurements of the N5 sample set with 1, 5, 10 and 20 repetitions are given in Figure 3.13a-d). The hysteresis shape slowly changes over increasing the repetition number. The P-MOKE measurements of the N8 sample set are given in Figure

3.13e-h. Similar to the N5 set, the shape of the hysteresis loose squareness over increasing repetition number R. However, in this sample set, the deformation is faster.

In Figure 3.14 the coercivity (H_c) and the switching field (H_{sw}) of the N5 (a) and N8 (b) samples are plotted as a function of repetition number. The coercivity of both samples sets increases over R as expected. The change in the switching field can be attributed to the slow approach to saturation.

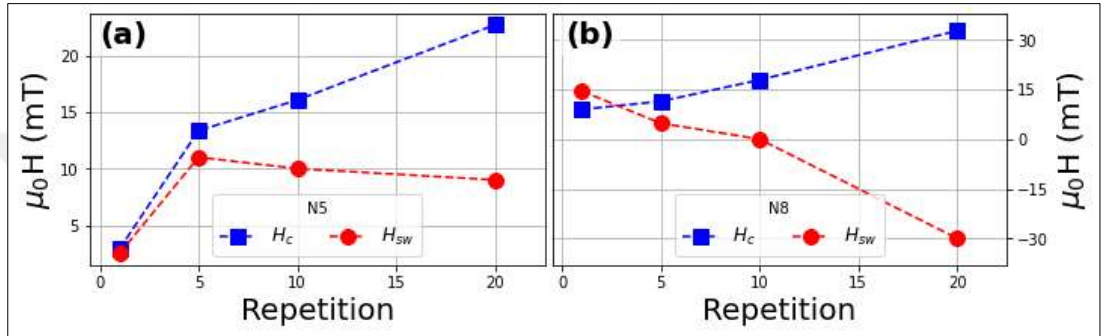


Figure 3.14. Coercive field H_c and switching field H_{sw} of N5 and N8 samples. The H_c (blue) and H_{sw} (red) are plotted as a function of repetition number R for N5 (a) and N8 (b) multi-stack samples. The sign of the fields indicates the direction of the external magnetic field relative to the magnetization. The (-) sign indicates that the rotation starts before the field even drops to zero while $H_{ext} \parallel M$, and the (+) sign indicates that the switching begins after a certain field in the opposite direction to the magnetization $H_{ext} \parallel -M$.

In Figure 3.13d, f-h the hysteresis is shaped like a “bowknot”. The switching of the dipoles starts fast at a certain external field but slowly saturates. This is usually explained by the formation of smaller domains. As it was explained above, with the increasing number of repetitions in NM/FM multilayers, the magnetostatic energy increase and this increase is compensated by decreasing the size of the domains. The dipoles are easy to rotate as long as the two domain walls are not facing each other. Therefore, the domains tend to expand or shrink. However, the domain walls are more stable structures therefore they are harder to annihilate. This is the reason why a single domain structure has a square-shaped hysteresis. For the structures with larger domains and low domain numbers (e.g., single domain MRAM cells), the switching of the dipoles will start from a

nucleation point (e.g., pinned domain wall, structural defect etc..) and expand rapidly until the whole structure is saturated. The number of domain-to-domain encounters will be minuscule. Therefore, switching does not require any extra energy other than domain expansion. However, for the samples with smaller domain sizes and a large number of domains, after the dipolar stabilization of the domains (expansion of domains), the domain-to-domain encounters will require extra external energy to be annihilated. This annihilation will be stepwise with the increasing field [92]. This is observed as a slow approach to saturation and a “bowknot” hysteresis loop. The samples with narrow stripe domains, bubble domains and skyrmion phases exhibit this kind of hysteresis loop.

The thickness of the Co layer increases the magnetostatic energy further. However, it also increases the total magnetic moments in the sample. In Figure 3.17 the saturation magnetization (M_s) and the 1st uniaxial anisotropy constants (K_{u1}) are given. The saturation magnetizations were measured by a vibrating sample magnetometer (VSM, Quantum Design MPMS3). The hysteresis loops measured in both perpendicular ($\perp$) and parallel ($\parallel$) external magnetic fields relative to the sample surface at 295 K. The samples are cleaved into smaller pieces before the measurements. The measured moment in VSM is the sum of the signal from sample, substrate and holder. The holder is measured without any sample and observed no signal. The contribution from the substrate is subtracted by linearly fitting the M-H curve.

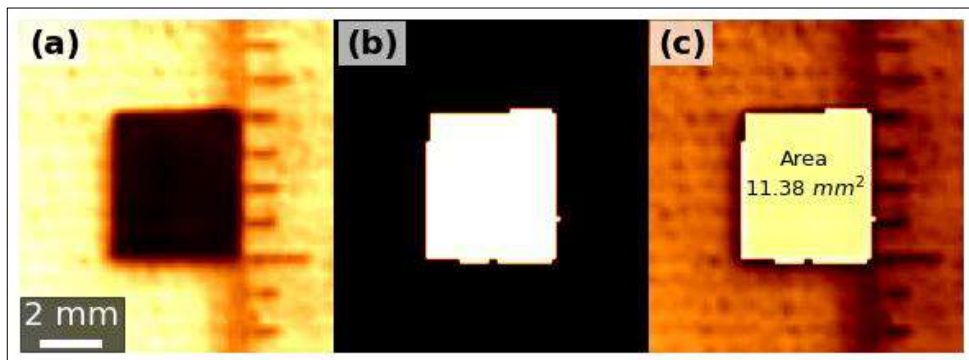


Figure 3.15. The area measurement of the samples for M_s calculations. **a.** The photographed image of the sample by a scale next to it. **b.** The mask calculated by using the color-histogram of the image **a.** **c.** The calculation of area by combining **(a)** and **(b)**.

The remaining signal is the total moment of the sample. To obtain the magnetization per volume, the thickness and the area of the measured film has to be known. The thickness of the film is well-known due to the fine calibration of the deposition thicknesses low-surface roughness confirmed by AFM measurements. The area of the measured sample was performed by image processing. The picture of the samples was taken by a camera next to a scale (Figure 3.15a). Using this scale, the size of one pixel was measured from the picture. Afterwards, the sample area was masked by using a height threshold (Figure 3.15b). The area of the mask was confirmed by merging it with the initial image thus the sample area can be measured by counting the pixels. In Figure 3.14 an example of this process is given step by step.

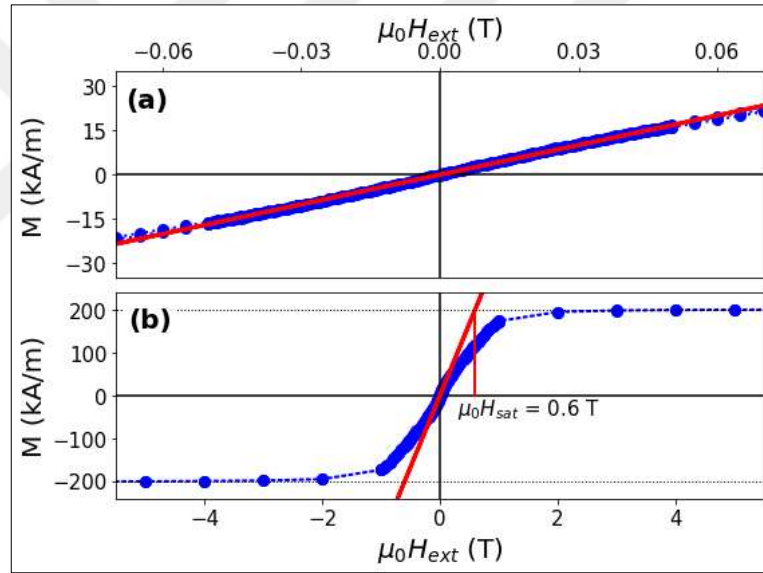


Figure 3.16. Anisotropy constant calculation from VSM measurement. a. The zoomed-in version to the low-field area of the (b). The red line shows the fit to the low-field region and the horizontal line indicates the saturation field H_{sat} which derived by the intersection of the fitting curve with the saturation magnetization (black dotted line).

The conversion of the signals at VSM to emu depends on the sample geometry. The magnetometer used in the measurement is calibrated for a cylindrical standard sample. Therefore, the signal of the VSM should be corrected by using the published report of the manufacturer [93]. The correction coefficients depend on the shape and

size of the sample, vibration amplitude and measurement orientation. The VSM data is corrected by using the corresponding factors and the saturation magnetizations are defined (Figure 3.17a). In the calculation of M_s values, the total thickness of the sample has been used.

The hard axis magnetization on anisotropy constants was discussed in chapter 1.5. by equation (1.27). However, in thin-film systems, the Stoner and Wohlfarth equation [9] is written as;

$$K_{u1} = \frac{\mu_0 H_K M_s}{2} \quad (3.7)$$

where the anisotropy field (H_K) is given by [64];

$$H_K = H_{sat} + M_s \quad (3.8)$$

The H_{sat} is the saturation field. Calculation of H_{sat} is performed by linearly fitting the low-field area of the hard axis measurement (Figure 3.16a). The linearity of low-field and wide range of the M-H curve was different in some of the samples due to the domain structure. Therefore, the saturation field is defined by extrapolating the linear fit to find the intersection with $M = M_s$ which is a dotted horizontal line in Figure 3.16b. Using the M_s and the H_{sat} from the VSM data, the K_{u1} for each sample was calculated by using the equation (3.7). The K_{u1} for each sample is plotted as a function of repetition in Figure 3.17b.

The saturation magnetization of the samples has a linear dependence on the repetition number. The M_s of the N5 sample varied from 120 kA/m up to 200 kA/m for different repetitions. This value was between 185 kA/m and 375 kA/m for the N8 sample where the Co thickness was 1 nm. The repetition number dependent on K_{u1} was similar for both N5 and N8 samples and was fitted exponentially. The H_{sat} values for both N5 and N8 tended to saturate at 5 repetitions. Therefore, the K_{u1} depends only on the change in the M_s after $R = 5$. The magnetic parameters of the N5 and N8 samples are given in table 3.5.

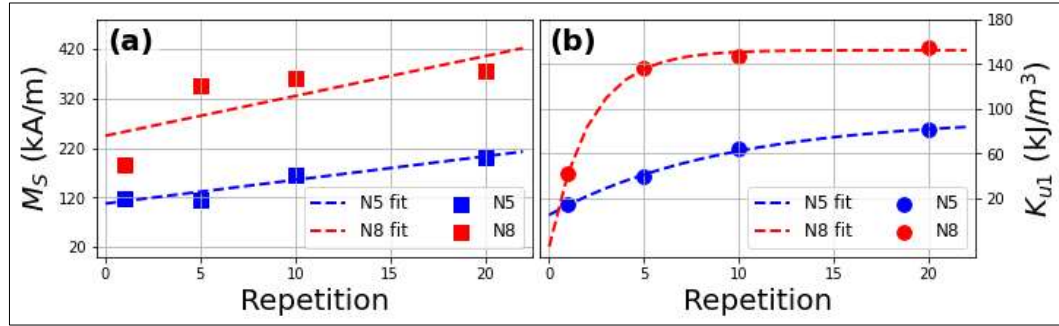


Figure 3.17. M_s and K_{u1} of the N5 and N8 samples. a. The saturation magnetization M_s . **b.** The uniaxial anisotropy constant K_{u1} of the N5 (blue) and N8 (red) samples as a function of repetition number.

In Figure 3.18 the VSM and MFM measurements of the N5R10 (a, b), N5R20 (d, e), and N8R20 (g, h) is shown. The VSM measurements were performed in both perpendicular ($\perp$) and parallel ($\parallel$) geometries. The saturation magnetizations (M_s) from hard axis measurements ($\parallel$) and easy axis measurements ($\perp$) are perfectly in. The zoomed-in hysteresis from easy axis VSM measurements can be seen in the insets of Figure 3.17a, d, e. The shapes of the hysteresis loops, switching fields and coercivity obtained from easy axis VSM measurements are the same as the ones from P-MOKE measurements.

Table 3.5. The magnetic parameters of the N5 and N8 samples

Type	R	H_{sat} (mT)	H_{sw} (mT)	H_c (mT)	M_s (kAm ⁻¹)	K_{u1} (kJm ⁻³)
N5	R1	110	2.5	2.9	119	14.72
	R5	580	11	13.35	115	39.74
	R10	600	10	16.06	166	64.07
	R20	600	9	22.70	201	81.45
N8	R1	250	14.5	8.97	185	42.66
	R5	400	4.8	11.40	344	136.48
	R10	400	0	17.89	362	147.03
	R20	400	-30	32.75	375	155.43

The N5R10, N5R20 and N8R20 samples domain structures are imaged by using MFM in ambient conditions and without any external field (Figure 3.18b, e, h). The zero-

remnant state of the samples is excited by demagnetizing the samples inside an AC magnetic field with damped oscillation. The maximum amplitude of the AC field started higher than the saturation field of the samples and was reduced to zero in 0.5 mT steps.

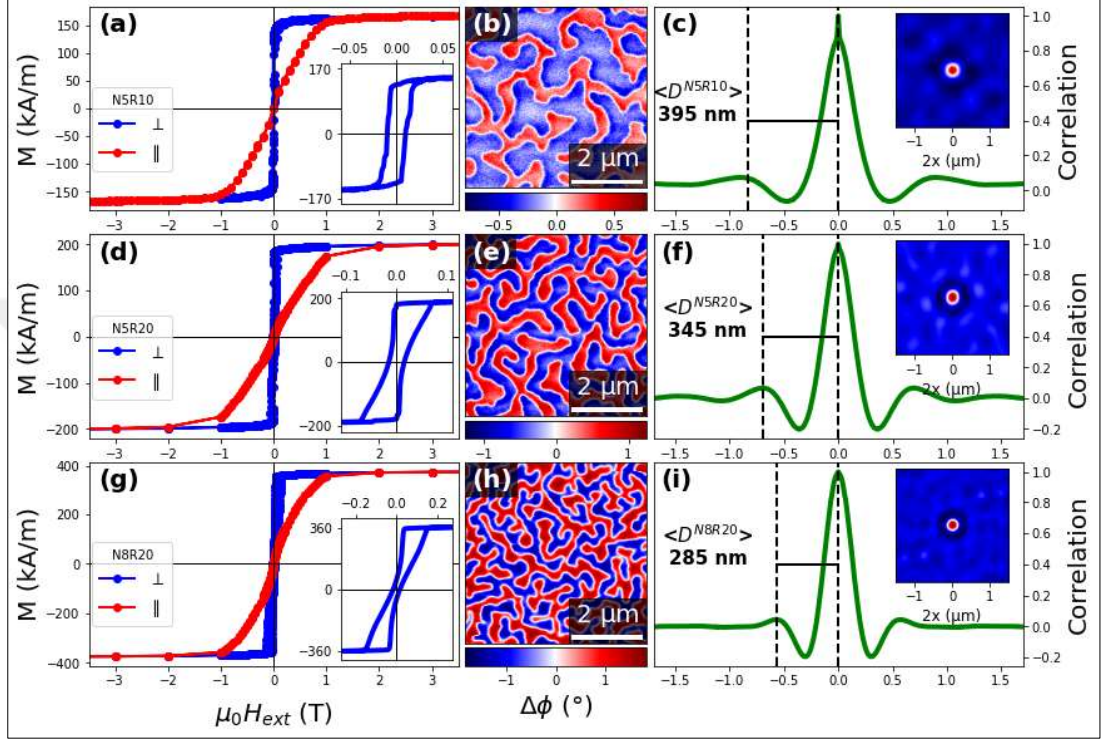


Figure 3.18. VSM and MFM measurements and average domain size calculations of N5R10, N5R20 and N8R20 samples. a, d, g. The VSM measured M-H curves in external fields parallel (red) and perpendicular (blue) to the sample surface. The inset hysteresis loops are the zoomed-in of the perpendicular measurements to emphasize the shape of the hysteresis. b, e, h. The MFM data measured in a $5.12 \times 5.12 \mu\text{m}^2$ scan range and from 40 nm lift height. c, f, i. The autocorrelation spectra of the samples. The inset figures are the 2D of the autocorrelation maps while the main figures are the line scans through the maxima. The distance between the 1st and the 2nd maximum is twice the average domain size. The top, middle and bottom rows are N5R10, N5R20 and N8R20 samples respectively.

The MFM measurement was performed by using Bruker Nanoscope IIIa with a Dimension head using a commercial NT-MDT Low Moment MFM tip. The lift height was fixed to 40 nm. The total height (Tip-Surface distance in phase shift measurement) will be discussed in the quantitative field measurement chapter by calibrating the cantilever

oscillations. The measurements performed with a pixel size of $\delta_A \approx 10 \times 10 \text{ nm}^2$ from a 512×512 pixel grid. The MFM data of all three samples show a stripe domain structure. The domain shapes of the samples are not uniform. However, the shapes of the domains are similar for different samples but their average size is different. Furthermore, the area of total domains in positive dipole orientation is not equal to the total domains in negative magnetization direction. This could be an indication that there is a net magnetization in the sample and means that the sample could not be demagnetized effectively. This prediction can be derived from the hysteresis of the sample (Figure 3.18a). As can be seen from the relatively square hysteresis curve and high switching field of the sample, the sample has larger domain structures and these domains can expand even in very low fields (such as exhibited by the magnetic tip), causing a net magnetization. Therefore, the zero-remnant state cannot be excited effectively.

The average domain widths of the samples are calculated by using the autocorrelation technique (Figure 3.18c, f, i). The distance between the first and second maximum is twice the average domain size [94]. The average domain sizes of the samples are found $\langle D_w^{N5R10} \rangle = 395 \text{ nm}$, $\langle D_w^{N5R20} \rangle = 345 \text{ nm}$ and $\langle D_w^{N8R20} \rangle = 285 \text{ nm}$. The effective magnetic surface charge density and stray field of these samples are measured by transfer function based quantitative magnetic force microscopy.

3.2.5. Quantitative Field Measurements of Multi-Stack Samples

The principles of qMFM, the calibration of the magnetic tip and field deconvolution of the target sample were explained in detail. This technique consists of two different measurements performed using the same tip. In order to eliminate the systematic errors due to the instrumental parameters such as Q factor or cantilever stiffness c , not demount/mount the tip between the reference and the target measurements is crucial. Therefore, the calibration measurements and the N5R10, N5R20 and N8R20 measurements (target) are performed sequentially and without changing any configuration in the SPM.

3.2.5.1. Calibration of the MFM Tip

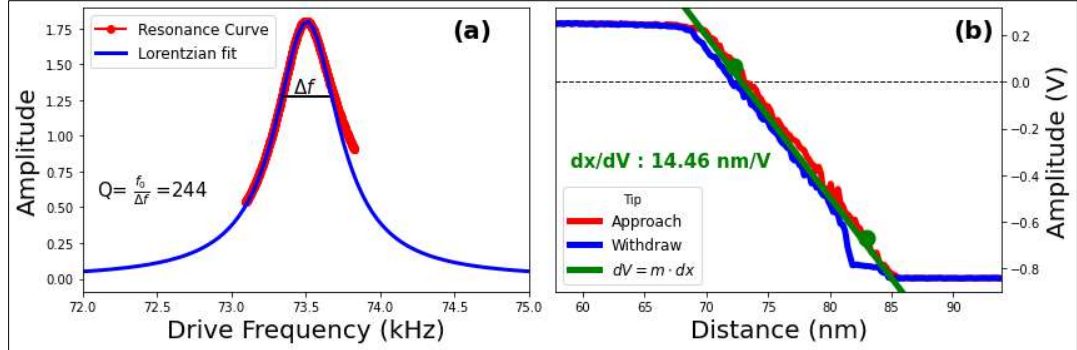


Figure 3.19. Q factor and Force calibration. **a.** The resonance curves of the cantilever oscillation as a function of driving force frequency. $Q = 244$ factor is calculated by fitting the amplitude response. **b.** The force measurement of the cantilever. Linear fit of the tip approach (red) and withdraw (blue) from the surface gives the distance equivalence of the oscillation voltage.

The calibration measurement was performed from a $[\text{Pt}/\text{Co}]_{100}$ multilayer prepared by Dr. Volker Neu from Leibniz-Institut für Festkörper und Werkstofforschung (IFW) Dresden. This sample is a well-known reference sample and used in many qMFM studies in the literature. This sample will be named “ref” or “pre-existing reference sample” for the rest of the thesis. The layer architecture of ref sample is Si / Ta (5 nm) / Pt (5 nm) / $[\text{Pt} (0.9 \text{ nm}) / \text{Co} (0.4 \text{ nm})]_{100} / \text{Pt} (2 \text{ nm})$. The average domain size of the sample is found $\langle D_w^{\text{ref}} \rangle = 235 \text{ nm}$ by using the autocorrelation approach.

During the height scan of the MFM measurement, the cantilever oscillation is damped due to the tapping mode scan. This damped oscillation is defined by the amplitude setpoint voltage (ASP) in Nanoscope. This amplitude is the distance between the centre of oscillation (therefore the average position of the tip) and the sample surface. Furthermore, during the phase scan of the MFM measurement, the cantilever does a free oscillation at lift height. The free oscillation is defined by the drive amplitude (dA) in Nanoscope. The distance equivalence of the ASP and dA is required to be known to calculate the real measurement height and the lever canting function. The real measurement height is the sum of the offset lift height and the ASP .

The voltage distance equivalence is calculated by the force curve calibrations (Figure 3.19b). The force curve is usually performed on a Si substrate. In the force measurements, the deflection force of the approaching and retracting cantilever is given in terms of voltage. The linear fit of the force curve (green line in Figure 3.19b) will give the deflection voltage equivalence in nanometers which will be used to calculate the total height z and the free oscillation amplitude dA .

The Q factor was described in chapter 2.2. as the energy loss of the cantilever during oscillation and determines the minimum limit of the measurable force. Q factor is calculated from the resonance curve of the cantilever (Figure 3.19a). The resonance curve is fitted with a Lorentzian function and its full width at 0.707*maximum is measured. The ratio of this width to the resonance frequency gives the Q quality factor of the cantilever. The quality factor was measured $Q = 244$ from the resonance curve (Figure 3.19a) and the cantilever stiffness $c = 3 \text{ N/m}$ was provided by the tip manufacturer.

The calibration of qMFM requires the calculation of the σ_{eff} of the reference sample from the reduced magnetization of the sample by using the equation (2.17). The steps of the σ_{eff} calculation is explained in chapter 2.3. In Figure 3.20 these steps for the reference sample are given. The MFM data (b) is recorded at 40 nm lift height. The amplitude setpoint ASP of the tapping mode measurement was $0.85 \text{ V} = 24 \text{ nm}$ thus the total measurement height is calculated as $z = 40 \text{ nm} + 24 \text{ nm} = 64 \text{ nm}$.

The cantilever corrected phase shift data is given in Figure 3.20c calculated by using the equation;

$$\text{Lever Correction} = LCF^2(\mathbf{k}, \theta) \cdot I_1^{modified}(\mathbf{k}, \theta, A) \quad (3.9)$$

The $LCF(\mathbf{k}, \theta)$ and the $I_1^{modified}(\mathbf{k}, \theta, A)$ are the correction functions related to the orientation of the cantilever and its finite oscillation amplitude respectively. The details of the functions are discussed in chapter 2.2. The cantilever was parallel to the slow scan axis and the canting angle was $\theta = 10^\circ$. The drive amplitude dA was $0.9 \text{ V} =$

25 nm. The cantilever corrected data is discriminated to the ± 1 binary 2D matrix (Figure 3.20e). The discriminated data is convoluted with the domain wall kernel. The kernel is calculated by using the domain wall width of the reference sample known priori. Domain wall kernel convoluted discriminated domain guess gives the perpendicular component of the reduced magnetization $m_z(x, y)$ or $m(\mathbf{k})$ (Figure 3.20f).

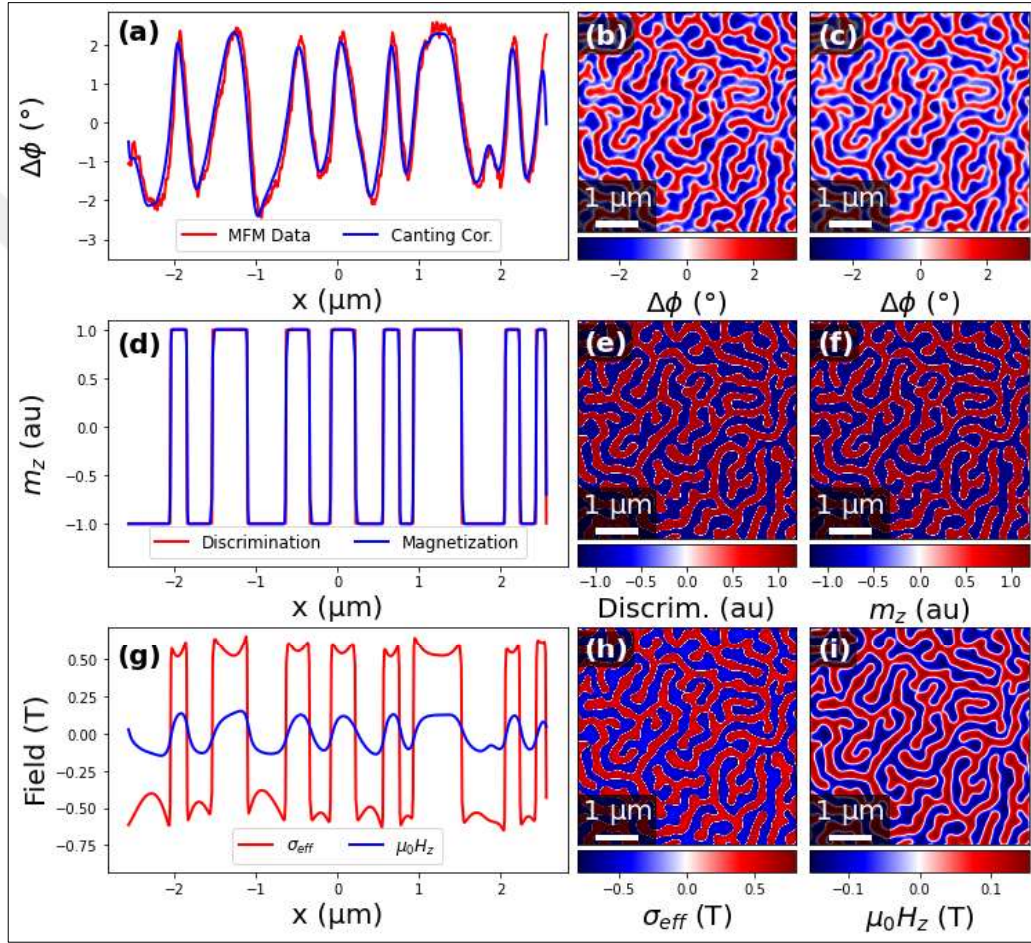


Figure 3.20. Charge density and stray field calculation of ref sample. The MFM data (b) and the cantilever corrected MFM data (c) of the ref sample. e. Discriminated image. f. perpendicular component of reduced magnetization m_z . The z component of h. effective magnetic surface charge and i. stray field at $z=64$ nm. a, d, e. The plotlines through (b), (e), (h) (red) and (c), (f), (i) (blue).

The reference sample has a domain width of $\delta_{DW}^{ref} = 16$ nm which was reported previously. The domain wall convolution is calculated at partial Fourier space. The spatial

components are transferred to Fourier space, $(x, y) \rightarrow (k_x, k_y) = \mathbf{k}$ where $\mathbf{k} = \sqrt{k_x^2 + k_y^2}$. The z component is retained [23,31]. The k_x, k_y are calculated by;

$$k_{x,y} = \frac{2\pi}{\delta_{x,y}} \quad (3.10)$$

where the $\delta_{x,y} = 10 \text{ nm}$ is the pixel size in x or y direction.

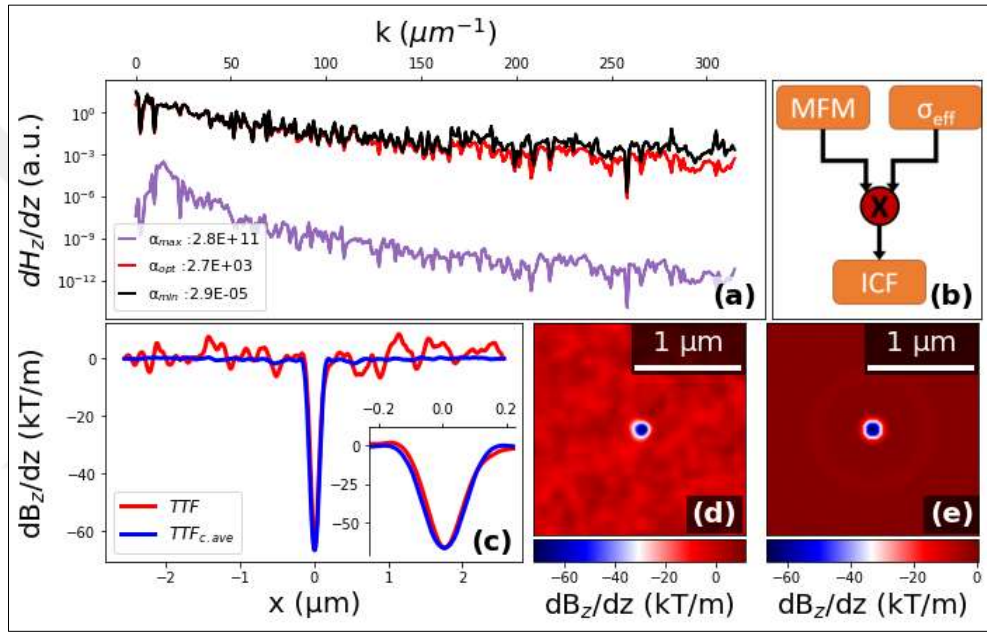


Figure 3.21. Deconvolution of ICF from the reference sample. **a.** The Fourier spectra of the deconvolved ICF by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme. The MFM data measured from the reference sample and the σ_{eff}^{ref} calculated from the reference sample deconvolved to obtain the ICF. **d.** The deconvolved ICF. **e.** The circular average of the ICF. **c.** The plotlines through (d) and (e). The inset figure is the zoomed-in of the (c).

In Figure 3.20h, the effective magnetic surface charge distribution of the reference sample σ_{eff}^{ref} calculated by the equation (2.17) is given. The saturation magnetization and the effective magnetic layer thickness of the sample was reported $M_s = 500 \text{ kA/m}$ and $t = 100 \times 1.3 \text{ nm} = 130 \text{ nm}$. In the calculations, the whole thickness of the multilayer

was considered as an effective magnetic layer. The maximum stray field strength at the sample surface was found 0.3 T. The effective charge density image is used to calculate the stray field of the sample at $z' = 64\text{nm}$ which is the distance between the surface and the tip apex. The maximum stray field of the sample at this height is calculated 150 mT. The deconvolution of the *ICF* was calculated by using the σ_{eff}^{ref} and the phase shift data of the reference sample (Figure 3.21b). The *TTF* deconvolved by the σ_{eff} gives the dB_z/dz of the tip on the sample surface (Figure 3.21d). The regularization parameter α is optimized by using the L-Curve criterion (see chapter 2.3.4). In optimization, α was scanned between 2.9×10^{-5} and 2.8×10^{11} . The optimum $\alpha = \alpha_{opt}$ was found as 2.7×10^3 . In the Figure 3.21a, it can be seen that the cut-off frequency for α_{opt} was around $100 \mu\text{m}^{-1}$.

The lower frequencies are not filtered out thus accurate filtering was performed. The *ICF* deconvolved by this regularized α have noise-like artefacts in real space (Figure 3.21c, d). These artefacts are usually due to the mathematical operations in partial Fourier space or sometimes real space artefacts due to the physical state of the tip and/or sample. During the quantitative measurement of the stray field, these artefacts will affect the accuracy and the quality of the deconvolution. Therefore, the circular average of the *ICF*, is calculated to be used in the further deconvolutions. The circular average is calculated by averaging the 2D profile of the *ICF* around the origin where the intensity is maximum. This suppresses the artefacts and results in a smooth *ICF* In (Figure 3.21e). In the line plot comparison of the *ICF* and $ICF_{c,ave}$ a slight difference in the intensities can be observed (Figure 3.21d). This difference is due to the circular averaging; however, it does not have a significant impact on the results. The calibration of the MFM tip ends here. In the following part, the deconvolution of the stray field for N5R10, N5R20 and N8R20 samples will be discussed.

3.2.5.2. Quantitative MFM of N5R10 Sample

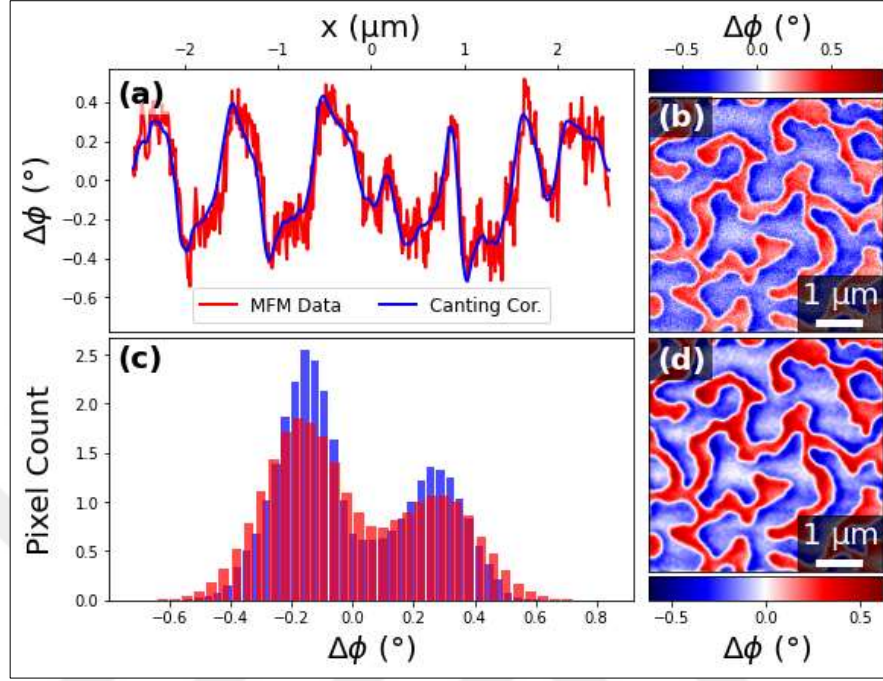


Figure 3.22. MFM data and cantilever correction of N5R10. The raw MFM data (b) and the cantilever corrected data (d) of the N5R10 with the plotlines (a). c. the histogram of the (b) and (d).

The MFM data of the $[\text{Ti}(0.3)/\text{Pt}(1.5)/\text{Co}(0.5)/\text{Pt}(1.5)]_{10}$ (N5R10) taken with the calibrated tip is given in the Figure 3.22b. The sample exhibits stripe domains with an average width of $\langle D_w^{N5R10} \rangle = 395 \text{ nm}$. The cantilever correction is calculated by using the instrumental parameters defined at the calibration step (Figure 3.22d). In the corrected data, the noise is suppressed by the modified first order first of its kind Bessel function I_1^{modified} which corrects the data for finite oscillation amplitudes (Figure 3.22a). In Figure 3.22c, the histogram of the (b) and (c) is given. In the histogram, the area in negatively magnetized area is larger than the area positively magnetized. This indicates that the sample has a net magnetization in the negative direction. It was observed that the average field values in the histogram did not change due to the cantilever correction, but the amplitude has increased. This is due to suppressed noise level by the Bessel

function. The reason why cantilever correction has so much impact on the data is that the sample signal is low and therefore the signal-to-noise ratio (SNR) is relatively high.

In Figure 3.23, calibrated qMFM measurement of the N5R10 sample is shown. The σ_{eff} is calculated by deconvolving the MFM data with circularly averaged ICF (Figure 3.21e) calibrated from reference measurement. The regularization parameter scanned between $\alpha_{min} = 5.5 \times 10^{-11}$ and $\alpha_{max} = 5.2 \times 10^5$ and optimized by L-curve criterion ($\alpha = \alpha_{opt} = 8 \times 10^{-2}$). In Figure 3.23a, the Fourier spectra of the deconvolved σ_{eff} is plotted with the L-curve upper and lower limits. The optimum regularization parameter α_{opt} filter out the frequencies above $40 \mu m^{-1}$.

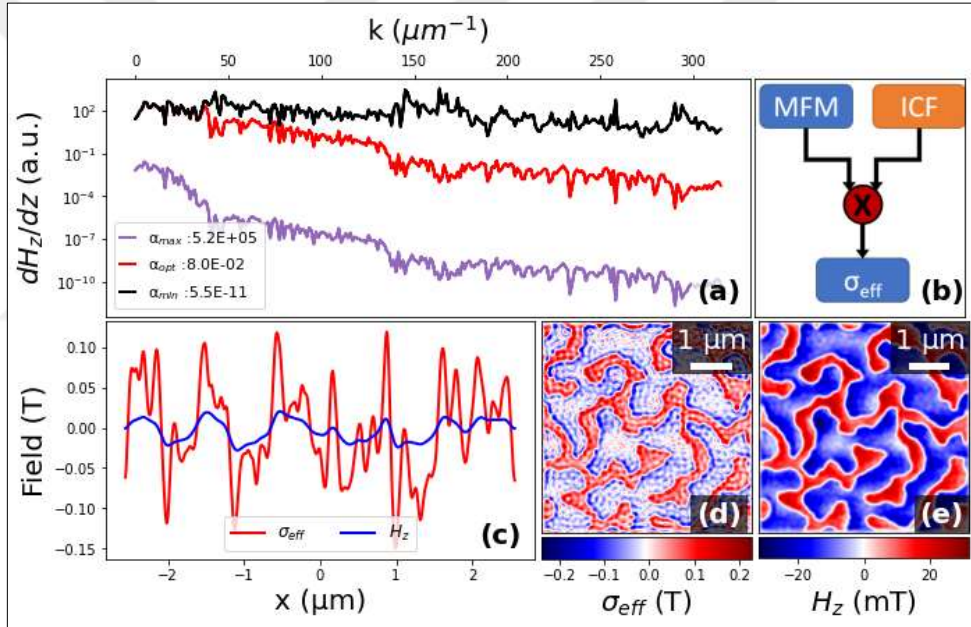


Figure 3.23. Field deconvolution of the N5R10 sample. **a.** The Fourier spectra of the deconvolved σ_{eff} by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data measured from N5R10 sample and the ICF is calibrated from reference sample deconvolved to obtain the σ_{eff}^{N5R10} . **d.** The deconvolved σ_{eff}^{N5R10} . **e.** The stray field of the N5R10 sample H_z^{N5R10} at tip apex ($z' = 64 \text{ nm}$). **c.** The plotlines through (d) and (e).

The sample may have magnetic features with these spatial frequencies; thus, some information of the stray field may be filtered out. The filtered-out information has

relatively higher frequencies compared to the rest of the features. The higher frequency regime in the partial Fourier spectrum is related to spatially smaller magnetic features. The small magnetic features will be decayed faster with increasing height. Therefore, the stray field measurement of the sample at the tip apex will have significantly higher accuracy. The out-of-plane component of the stray field of the N5R10 sample is given in Figure 3.23e which was calculated from the deconvolution result, σ_{eff}^{N5R10} (d) by using the equation (2.19). The maximum field strength was measured 110 mT at the sample surface ($z' = 0$ nm) and 30 mT at the tip apex ($z' = 64$ nm).

3.2.5.3. Quantitative MFM of N5R20 Sample

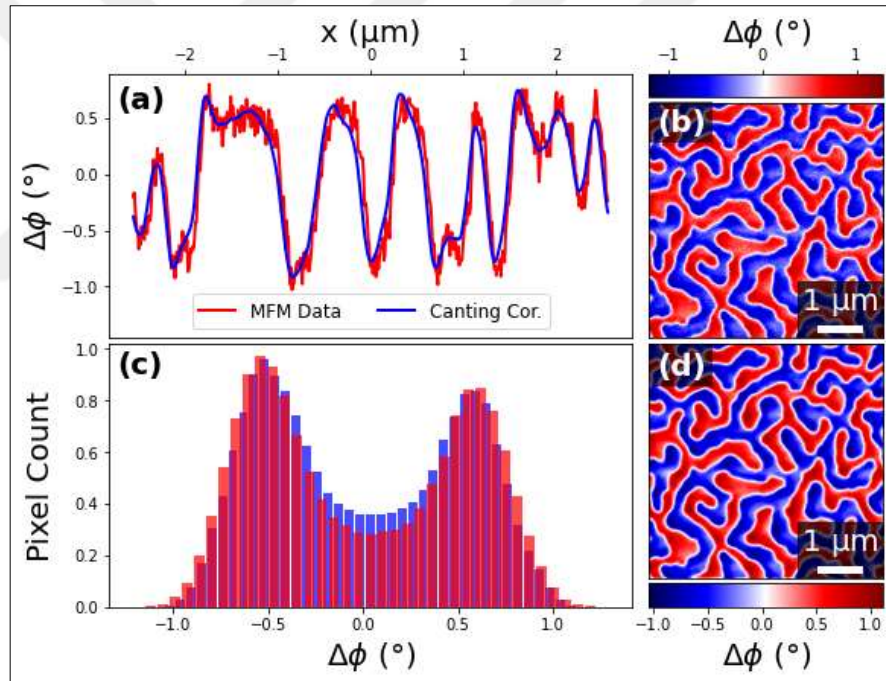


Figure 3.24. MFM data and cantilever correction of N5R20. The raw MFM data (b) and the cantilever corrected data (d) of the N5R20 with the plotlines (a). c. The histogram of the (b) and (d).

The MFM data of the $[\text{Ti}(0.3)/\text{Pt}(1.5)/\text{Co}(0.5)/\text{Pt}(1.5)]_{20}$ sample (N5R20) measured by the calibrated tip is given in Figure 3.24. The stripe domains of the sample have an average width of $\langle D_w^{N5R20} \rangle = 345$ nm. The cantilever correction is calculated by using

the instrumental parameters defined at the calibration step (Figure 3.24d). In the corrected data, the noise suppression is not as strong as in the N5R10 (Figure 3.24a). This is due to the stronger signal of the sample thus the lower SNR. In Figure 3.24c, the histogram of the (b) and (c) is given. The area of the positively and negatively magnetized domains are nearly equal. This indicates that the sample does not have a net magnetization and is in a zero-remnant state. The cantilever correction does not have a significant influence on the histogram.

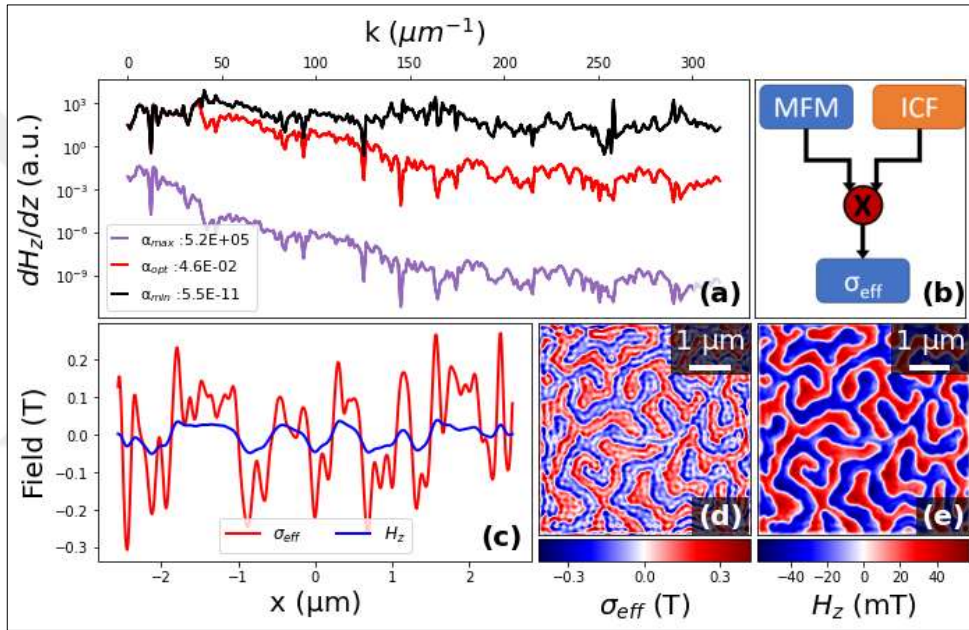


Figure 3.25. Field deconvolution of the N5R20 sample. **a.** The Fourier spectra of the deconvolved σ_{eff} by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data measured from the N5R20 sample and the ICF is calibrated from reference sample deconvolved to obtain the σ_{eff}^{N5R20} . **d.** The deconvolved σ_{eff}^{N5R20} . **e.** The stray field of the N5R20 sample H_z^{N5R20} at tip apex ($z' = 64$ nm). **c.** The plotlines through (d) and (e).

The qMFM calibrated field measurement of the N5R20 sample is given in Figure 3.25. The field deconvolution is calculated by the MFM data of the N5R20 sample and the ICF calibrated from the reference sample, therefore, thereof output is the effective surface magnetic charge density σ_{eff} of the N5R20 sample. The L-curve for

regularization is scanned between $\alpha_{min} = 5.5 \times 10^{-11}$ and $\alpha_{max} = 5.2 \times 10^5$ (Figure 3.25a). The optimum regularization parameter is $\alpha = \alpha_{opt} = 4.62 \times 10^{-2}$. The Fourier spectra of the deconvolution with upper/lower limits and optimized regularization is similar to the deconvolution of N5R10. Even the cut-off frequency is around $40 \mu m^{-1}$. This can be again interpreted as slightly over filtering but in an acceptable range. Furthermore, the N5R20 sample has narrower magnetic domains compared to the N5R10 which means that it has more information in the high-frequency region of the Fourier spectrum. Therefore, the information filtered out is slightly higher in this deconvolution. The artefacts in the σ_{eff} is also present for this sample. To have a more valid comparison, the z component of the stray field distribution on the plane at the tip apex ($z' = 64 \text{ nm}$) is calculated by using the equation (2.19). The maximum stray field of the sample is 282 mT at the sample surface ($z' = 0 \text{ nm}$) and 60 mT at the tip apex ($z' = 64 \text{ nm}$).

3.2.5.4. Quantitative MFM of N8R20 Sample

The MFM data of the $[\text{Ti}(1.0)/\text{Pt}(2.5)/\text{Co}(1.0)/\text{Pt}(2.5)]_{20}$ sample (N8R20) measured by the calibrated tip is given in Figure 3.26. This sample has the narrowest stripe domain width $\langle D_w^{N8R20} \rangle = 285 \text{ nm}$ among the three multilayer-stacks. The cantilever correction is calculated by using the instrumental parameters defined at the calibration step (Figure 3.26d). The noise suppression due to the Bessel function is the lowest in this sample. Because the N8R20 sample has the highest phase shift signal thus the lowest SNR. The histogram of the MFM data shows an equal area for both positively and negatively magnetized domains. This ensures that the sample does not have net magnetization and it is in the zero-remnant state. The qMFM calibrated field measurement of the N8R20 sample is given in Figure 3.27. The field deconvolution is calculated by the MFM data of the N8R20 sample and the ICF calibrated from a reference sample, therefore, thereof output is the effective surface magnetic charge density σ_{eff} of the N5R20 sample. The L-curve for regularization is scanned between $\alpha_{min} = 5.5 \times 10^{-11}$ and $\alpha_{max} = 5.2 \times 10^5$ (Figure 3.27a). The optimum regularization parameter is $\alpha = \alpha_{opt} =$

3.3×10^{-2} . The cut-off frequency is around $40 \mu m^{-1}$. This can be also interpreted as again a slightly over filtering in the limits of the acceptable range. The N8R20 sample has the smallest spatial magnetic features among the three multilayer-stacks. Therefore, the spatially high-frequency information is highest in this sample thus the information filtered out is the highest for this deconvolution.

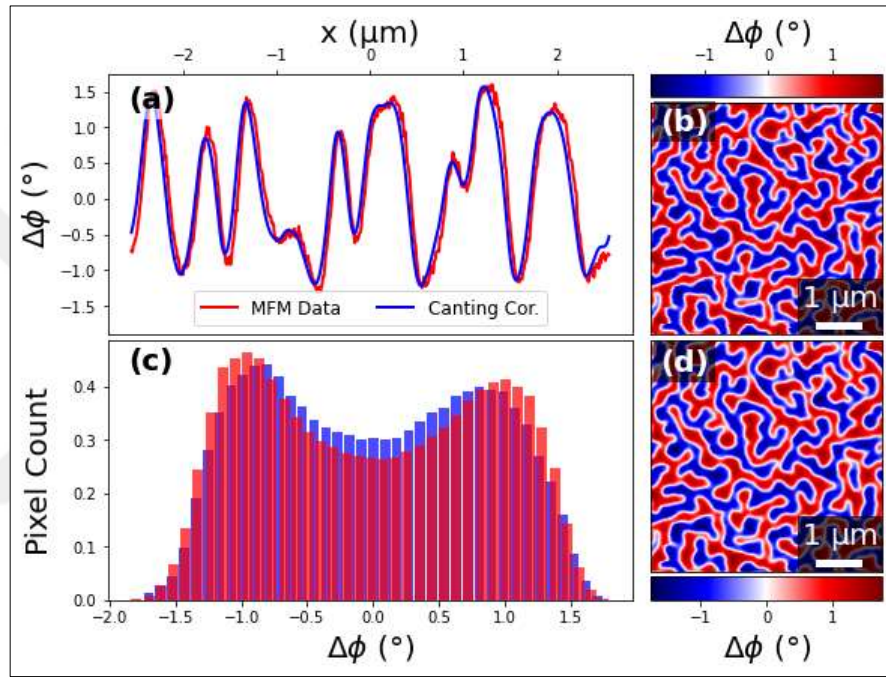


Figure 3.26. MFM data and cantilever correction of N8R20. The raw MFM data (b) and the cantilever corrected data (d) of the N8R20 with the plotlines (a). c. The histogram of the (b) and (d).

The artefacts in the σ_{eff} is also highest for this sample. However, the lost information and undistinguishable artefacts in the effective charge density image are small magnetic features that would already decay at a certain height thus a comparison of the fields calculated at this height will be valid. The perpendicular component of the stray field distribution on the plane at the tip apex ($z' = 64 \text{ nm}$) is calculated by using the equation (2.19). The maximum stray field of the sample is 428 mT at the sample surface ($z' = 0 \text{ nm}$) and 91 mT at the tip apex ($z' = 64 \text{ nm}$).

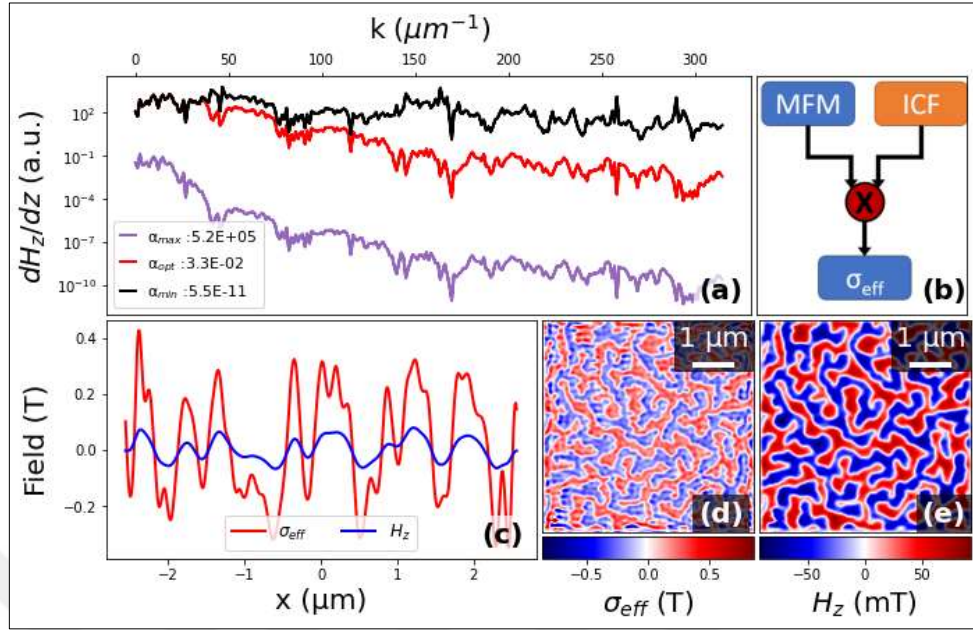


Figure 3.27. Field deconvolution of the N8R20 sample. **a.** The Fourier spectra of the deconvolved σ_{eff} by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data measured from N8R20 sample and the ICF is calibrated from reference sample deconvolved to obtain the σ_{eff}^{N8R20} . **d.** The deconvolved σ_{eff}^{N8R20} . **e.** The stray field of the N8R20 sample H_z^{N8R20} at tip apex ($z' = 64$ nm). **c.** The plotlines through (d) and (e).

3.3. Discussion

In this chapter “3. Ti/Pt/Co Magnetic Multilayers” the Pt/Co/Pt multilayers stacks with a Ti seed layer and buffer layer were studied. The effect of Ti layer was investigated in structural terms.

The Pt/Co/Pt type of samples with various layer thicknesses were grown on naturally oxidized Si(111) substrate at Room Temperature. The orientation of the easy axis was found directly related to the presence of the Ti seed layer. No significant contribution to magnetic properties depending on the thickness of Ti seed layer was observed for single-stack multilayer samples. However, the Pt thickness was observed as an important parameter on the magnetic properties. According to the experimental results, the minimum thickness was derived to promote the perpendicular magnetic

anisotropy. The Co thickness impact on the magnetic properties is the highest. The thickness of the Co is also important to derive the minimum Pt thickness threshold for PMA. While the 0.5 nm Co layer requires at least > 0.5 nm thick Pt, 1 nm Co layer requires > 1.5 nm Pt to promote the PMA. The Coercivity of the samples was found to be dependent on the thickness of the Co layer as expected. The saturation magnetization of the samples was also higher in the multilayers with a thick Co layer.

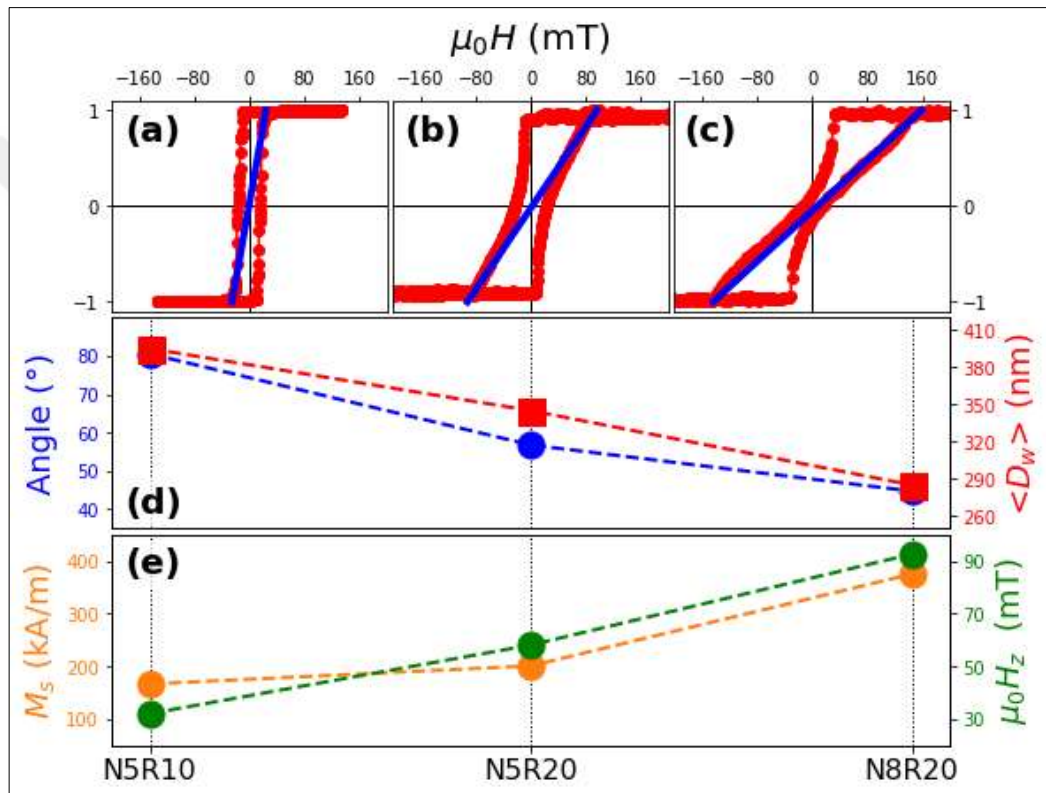


Figure 3.28. The summary of the multi-stack study. Normalized hysteresis loops of N5R10 (a), N5R20 (b) and N8R20 (c) measured by P-MOKE. The blue lines between the positive and negative saturation fields of the easy axis hysteresis loops are used to define the so-called hysteresis angle. The angle of the hysteresis and the average domain size relation of the samples are shown in d. The relation between saturation magnetization and the stray field maximum at 64 nm above the surface for the samples is shown in (e).

The structural analysis of the tpcp and pcpc samples showed that the presence of Ti as seed layer changes the structural phase of the Pt grown onto it. The XPS fitting study

showed that Pt peak is dominated by the surface phase for the tpcp sample while the pcpc sample has slightly mixed bulk and surface phases with a ratio of 3 to 1. Furthermore, the XRD studies showed that the Ti promotes fibre-textured growth of the Pt in (111) direction. Even the grazing incidence XRD cannot detect the Co layer growing on the (111) textured Pt layer which indicates small clusters. On the other hand, for the pcpc sample, the Co reflections are detected in the XRD study due to their relatively larger clusters. XPS and XRD studies together with the magnetic characterizations show that even a trace amount of Ti seed layer between the substrate and the Pt/Co/Pt promotes a sharp and well-defined Pt/Co interface. These results are further supported by STM analysis [83]. Two type of samples with symmetric Pt/Co, Co/Pt interfaces were selected to be grown in multi-stacks; $[\text{Ti}(0.3)/\text{Pt}(1.5)/\text{Co}(0.5)/\text{Pt}(1.5)]_R$ (N5RX) and $[\text{Ti}(1.0)/\text{Pt}(2.5)/\text{Co}(1.0)/\text{Pt}(2.5)]_R$ (N8RX) for $R=1, 5, 10, 20$ (The thicknesses are in nm units). Unlike most of the studies in the literature, the seed layer Ti was used as a buffer layer between the Pt/Co/Pt magnetic stacks. The $M(H)$ curves of the multi-stacks were investigated by using P-MOKE and VSM. The increase in the stack repetition number was accompanied by a significant increase in the coercive field and a decrease in the switching field. The hysteresis becomes tilted due to the increase in magnetostatic energy. Due to the increased thicknesses thus the higher magnetostatic energy in the N8 sample compared to the N5, the tilting is faster in terms of repetition number R .

The N5 type of sample with 10 and 20 repetitions and the N8 type of sample with 20 repetitions is further investigated with MFM. The relation between the shape of the hysteresis and the domain size is emphasized in Figure 3.28d. In the results, It was stated that the increase in the magnetostatic energy is compensated by an increase in the total domain wall area which reduces the average domain size. Therefore, the hysteresis saturates slowly due to the increased energy required to annihilate the domain walls. This relation between the hysteresis and domain structure is clearly shown in Figure 3.28d where both the angle of the hysteresis and the average domain size calculated by autocorrelation of the MFM image decreased altogether. The angle of the hysteresis is defined by linearly fitting the positive and negative saturation fields of the easy axis

hysteresis loops. The slope of the fit gives the so-called “*angle of hysteresis*”. The saturation magnetizations M_s of the stacks are proportional to the maximum field strength of the saturated domains 64 nm above from the surface (Figure 3.28e). The increase in H_z due to the saturation magnetization is somehow compensated by the decrease of the domain sizes thus the relation between them remained more or less linear. These results also showed that the Ti buffer layer between the Pt/Co/Pt stacks can be used to tune the field strength and domain size of the samples effectively.

The qMFM studies showed that N5R20 and N8R20 samples could be a good candidate as a new reference sample for tip calibrations. Even though the domain shapes and most probably the domain wall width of N8R20 sample is different than the pre-existing reference sample (IFW), their average domain widths are close to each other. Furthermore, the saturation magnetization values are also close ($M_s^{ref} = 500 \text{ kA/m}$, $M_s^{N8R20} = 375 \text{ kA/m}$). These parameters would result in a similar field strength to be used in the calibrations. The only advantage would be the spatially non-uniform magnetic features of the N8R20 sample which would have a wider frequency band for the calibrations (The impact of the accessible spatial frequencies on the accuracy of the calibrations will be discussed in the chapter 5.4 and 6.4.2). However, with its slightly larger domains, spatially non-uniform domain structure and considerably lower field strength, N5R20 is a good alternative for MFM tip calibrations. Therefore, the N5R20 sample was chosen to be studied further and the following chapter will focus on its micromagnetic properties and simulations.

4. MICROMAGNETIC SIMULATIONS

4.1. Continuum Theory of Micromagnetism

Micromagnetism is a continuum theory of magnetization for structures with sub-micrometre length scales. The resolution of Micromagnetism is limited in two directions. The length scale should be small enough to investigate the domain walls which marks the upper limit for the micromagnetic simulations. On the other hand, Micromagnetism is a continuum theory that replaces the discrete atomic spins with a continuous magnetization vector. Therefore, the length scale should be large enough to satisfy this assumption. These upper limits and lower limits depend on the magnetic parameters of the sample and can vary from nanometers up to micrometres. Even the minimum length scales are too big for DFT calculations and the billions of atomic spins are replaced by millions of finite elements.

The Micromagnetism, the dynamics of the magnetization is described by the time-dependent Landau-Lifshitz-Gilbert (LLG) equation [91];

$$\dot{\mathbf{m}} = -\gamma' \mathbf{m} \times \mathbf{H}_{eff} + \alpha' \mathbf{m} \times (\mathbf{m} \times \mathbf{H}_{eff}) \quad (4.1)$$

$$\gamma' = \frac{\gamma}{1 + \alpha^2}, \quad \alpha' = \frac{\gamma}{1 + \alpha^2} \alpha \quad (4.2)$$

where $\gamma = \mu_0 g e / 2m_e = g \cdot 1.105 \times 10^5 \text{ m/As}$ is the gyromagnetic ratio and α is the damping constant. Landé factor g and damping constant α are material dependent parameters the g is close to 2 for most of the ferromagnetic materials [6]. $\mathbf{m} = \mathbf{M}/M_s$ is the reduced magnetization (or normalized magnetization vector). The effective field $\mathbf{H}_{eff}$ has contributions from exchange interaction, anisotropy energy, demagnetization field, external fields. The LLG equation describes the precession of the magnetization vector around the effective field, namely the dynamics of magnetization of

micromagnetic processes. Furthermore, the damping term in γ' allows turning the magnetization towards $\mathbf{H}_{eff}$ in static conditions.

The micromagnetic simulations can be classified into two methods; dynamic and static simulations. The dynamic simulations are used to obtain the magnetization as a function of time. This method is widely used to study, domain wall movements, ferromagnetic resonance and spin waves. The static simulations solve the LLG for the equilibrium state. This method is extremely useful to simulate the hysteresis curves and the domain structures of the samples. In this dissertation, static simulations were used to simulate the zero-remnant domain structure of the N5R20 sample.

4.2. Simulations of N5R20 Sample

The micromagnetic simulations of the N5R20 sample were calculated by using an open-source GPU accelerated software, MuMax³ [95]. The magnetic parameters of the samples obtained from the measurements were the saturation magnetization $M_s = 201 \text{ kAm}^{-1}$, and the uniaxial anisotropy constant $K_{u1} = 81 \text{ kJm}^{-3}$. The damping constant α was selected high to calculate the equilibrium state faster as the simulations were static.

The initial simulations were used to define the missing magnetic parameters of the sample such as interlayer exchange and the exchange stiffness A_{ex} and also ensuring the uniaxial anisotropy constant as $K_{u1} = 81 \text{ kJm}^{-3}$. According to the hard axis VSM measurements, 81 kJm^{-3} is obtained by fitting in the low-field region and extrapolating it until it intersects with the saturation magnetization (Figure 3.15). However, this fit could be done by fitting the wide field. In this case, the fit would be unsatisfactory in the low-field region but would be acceptable for the wide range. Therefore, one could claim that the uniaxial anisotropy constant should be $K_{u1} = 150 \text{ kJm}^{-3}$. The simulations ran between these possible values calculated by fitting concerning the low-field region or the wide-range region to check the validity of the selected parameter.

The initial simulations ran over a $(640 \times 640 \times 20)$ cell grid with a cell size of $(5 \times 5 \times 3.8) \text{ nm}^3$. The cell size is smaller than the Bloch length. The minimum Bloch length for the parameters used in simulations is $\lambda_{Bloch} = \sqrt{A_{ex}/K_{u1}} = \sqrt{4 \text{ pJ/m}^{-1}/150 \text{ kJ/m}^{-3}} = 6.32 \text{ nm}$. This indicates that the simulations of the domain walls will have enough resolution to define the transition correctly. The z component of the cell size is equal to the total thickness of the single stack, thus each cell plane represents one Ti(0.3)/Pt(1.5)/Co(0.5)/Pt(1.5) stack.

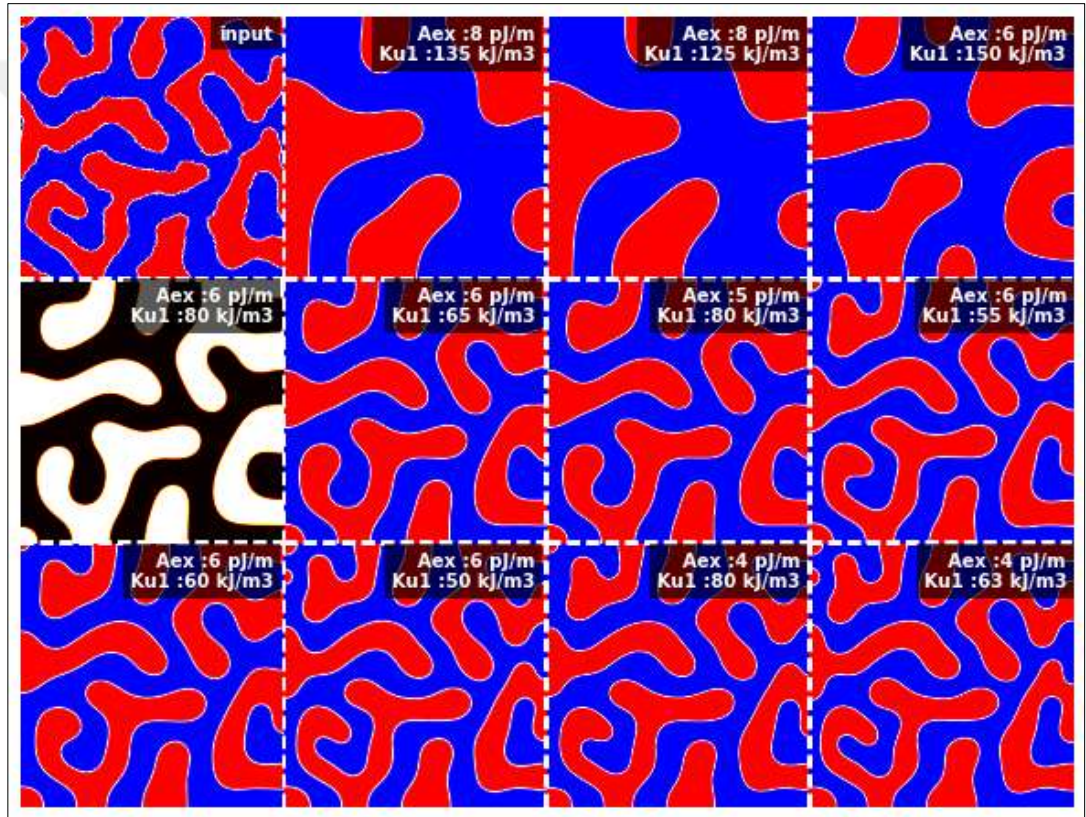


Figure 4.1. Micromagnetic simulations to define/verify magnetic parameters. The simulations are relaxed from a predefined input magnetization which was calculated from the MFM data of the N5R20 sample. The simulation with different colours is the one with the verified parameters.

The simulations to define/verify magnetic parameters were performed by using a predefined magnetization distribution input. The input magnetization is calculated by using the following steps. The MFM data is discriminated. However, the domain wall

function was not convoluted with the discriminated data to have a sharp domain transition (1-pixel width). This approach allowed to initiate a non-equilibrium state to prevent false-positive results. Afterwards, the discriminated data is cropped to $3.2 \times 3.2 \mu m^2$ image. The pixel size of the MFM data was $\delta_A = 10 \times 10 nm^2$. However, the defined spatial component of the simulation cell was $5 \times 5 nm^2$. Therefore, the discriminated image was interpolated to a resolution of (640×640) from (320×320) . The smaller size of the scanning range ($3.2 \times 3.2 \mu m^2$) was selected to be simulated due to the simulation capacity of the used graphic processing unit (GPU).

The initial magnetization interpolated from the discriminated real MFM data was used as an input to the preliminary simulations by using different A_{ex} and K_{u1} input parameters discussed above (Figure 4.1). The results pointed out that the anisotropy constants above $80 kJm^{-3}$ are inaccurate. This K_{u1} calculation by fitting the low-field region of VSM hard axis data was accurate. The simulations with relaxed domains closest to the initial magnetization derived from the experimental data were the ones with the lowest A_{ex} and K_{u1} inputs. However, the K_{u1} cannot be lower than the $80 kJm^{-3}$ due to the VSM data. Furthermore, in the literature the A_{ex} of similar Pt/Co structures were reported between $5 pJm^{-1}$ and $15 pJm^{-1}$. Therefore, the optimum exchange constant and uniaxial anisotropy constant were $A_{ex} = 6 pJm^{-1}$ and $K_{u1} = 80 kJm^{-3}$. These parameters were used in the later simulations.

The exchange interaction between the Co layers over the Ti/Pt structures can be explained by the Ruderman–Kittel–Kasuya–Yosida (RKKY) exchange coupling. RKKY explains the coupling of the magnetic layers by the interaction through the conduction electrons. The RKKY coupling was implemented into the simulations as an effective RKKY exchange field, J_{RKKY} by scaling the exchange coupling between each layer [96]. The scaling factor was defined by the $\Delta S = \frac{(J_{RKKY} \cdot \delta_{c_z})}{(2\langle A_{ex} \rangle)}$ where the $\langle A_{ex} \rangle$ is the average of the exchange stiffness over the coupled layers and the $\delta_{c_z} = 3.8 nm$ is the thickness of the single simulation cell. Similar to the exchange stiffness optimization, the J_{RKKY} was varied throughout the simulations. The optimum J_{RKKY} was found $0.07 mJm^{-2}$.

This coupling energy is used to scale the couplings of each layer in the following simulations.

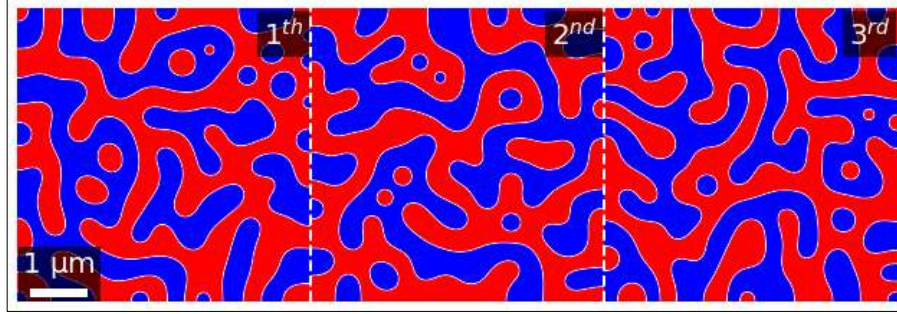


Figure 4.2. Micromagnetic simulations relaxed from random magnetization state. Simulations relaxed from three different random seeds as initial magnetization. The magnetic parameters used in the simulation were obtained from the experimental results and the previous magnetization (Figure 4.1)

The following simulations were relaxed the domain structure from a random magnetization distribution. The magnetization vector of each cell was initially randomly oriented. The spatial grid size of the simulations was (1024×1024) with $(5 \times 5) \text{ nm}^2$ cell size. The simulation size $(5.12 \times 5.12) \mu\text{m}^2$ was the same with the experimental MFM measurements. Three different random seed was used for verification of domain shapes and widths. The simulations reflect a stripe domain structure as the stable equilibrium magnetization pattern which is similar to the MFM measurements shown in Figure 3.17e. The average domain sizes of the simulations calculated from the autocorrelations were compared with the experimental results. However, the experimental domain sizes were calculated from the phase shift data. Therefore, the micromagnetic simulation results were used to forward simulate MFM data. The steps of the forward simulations of MFM images are given in Figure 4.3.

The forward simulations of the MFM are calculated with a tip-surface distance of 64 nm to be in sync with the experiments. The phase shift ($\Delta\phi$) of MFM data is the convolution of the sample magnetization with the tip's magnetization as was explained in chapter 2.2. To eliminate the contribution of the tip, the forward simulation was calculated by using the experimentally derived TF from the reference sample. The

TTF^{exp} is the 2D stray field gradient profile of the tip at 64 nm away from the tip apex (on sample surface). Therefore, the convolution of ICF^{exp} with σ_{eff}^{sim} will result in simulated phase shift data for an MFM measurement performed at 64 nm away from the surface. Added white noise to the convolution to create realistic conditions.

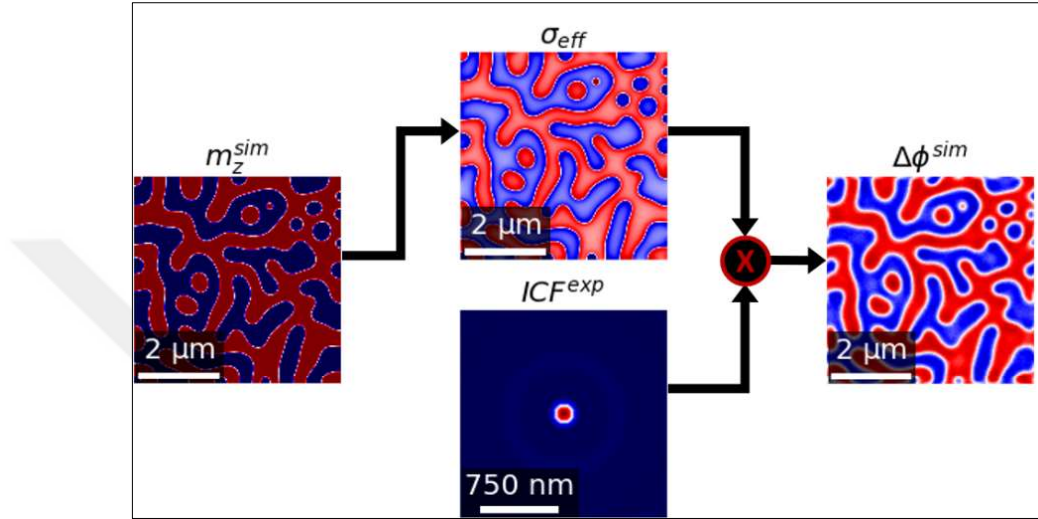


Figure 4.3. Forward simulations diagram of MFM images. The simulated reduced magnetization m_z^{sim} used to calculate the effective surface charge σ_{eff} . The σ_{eff} is convoluted with the ICF deconvolved from the experimental data then the noise is added to forward simulate MFM image.

The average domain widths of the simulations are calculated from the autocorrelations of the forward simulations and found to be the same (with small discrepancies) for different random seeds (Figure 4.4). The slight difference can be due to many parameters; the autocorrelation sensitivity, the bubble domains in the simulations, and simulation borders. The average domain width from simulations $\langle D^{sim} \rangle = 365 \text{ nm}$ is slightly higher than the average width found from the MFM data $\langle D^{MFM} \rangle = 345 \text{ nm}$. Even the widths differ slightly, the stripe domain pattern is well reproduced and the simulations are considered credible.

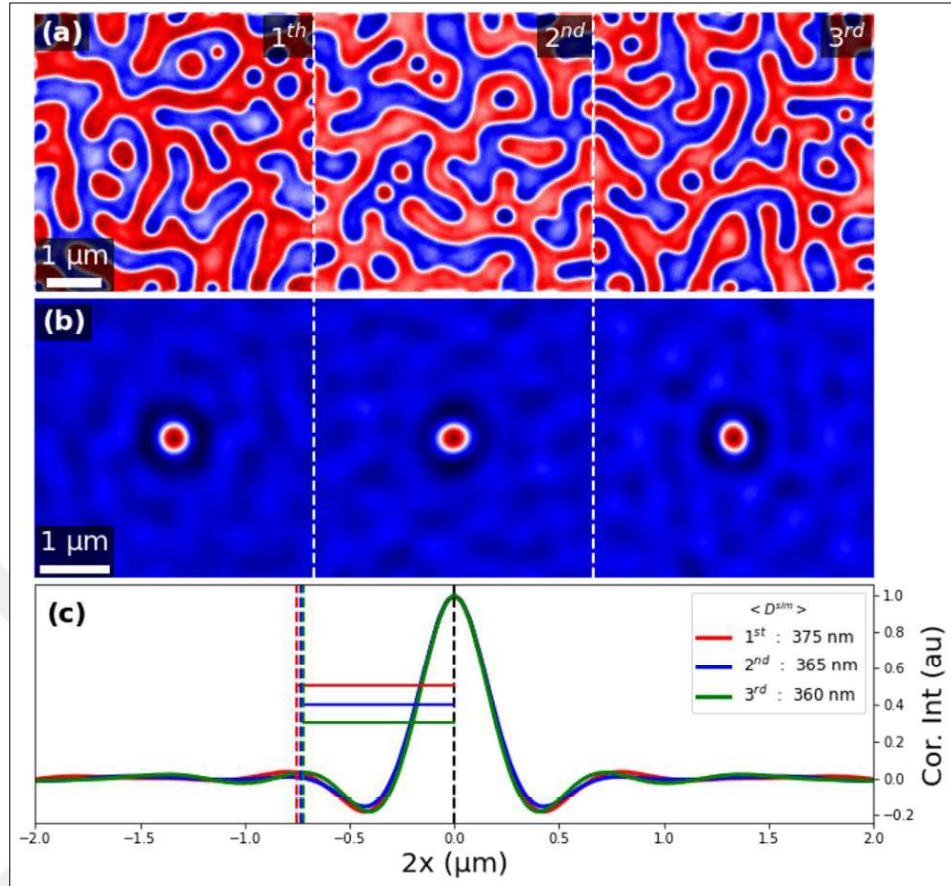


Figure 4.4. Average domain size of micromagnetic simulations. **a.** The forward simulated MFM data by using the micromagnetic simulation output and ICF calibrated from reference. **b.** The 2D autocorrelation images. **c.** The plotlines through (b).

In Figure 4.5, simulation with the 3rd random seed is investigated further. The cross-section of a cut-out of the magnetization shows each 20 layers of the simulation each represent a Ti/Pt/Co/Pt stack. The cross-section is cut out from a vertical line plot marked by the shadow in the zoomed-in view of Figure 4.5a. The spatial component of the domain is selected to be perpendicular to the line plot to ensure an accurate domain transition display.

Figure 4.5c shows a homogeneous magnetization throughout the layers and independent of the thickness. This property was a prerequisite for a reference sample to calculate the effective surface charge of the sample by using the equation (2.17). The

domain transitions show a slight layer dependence, hence the accuracy of the domain wall convolution method presented in section 2.3.3 needs to be validated.

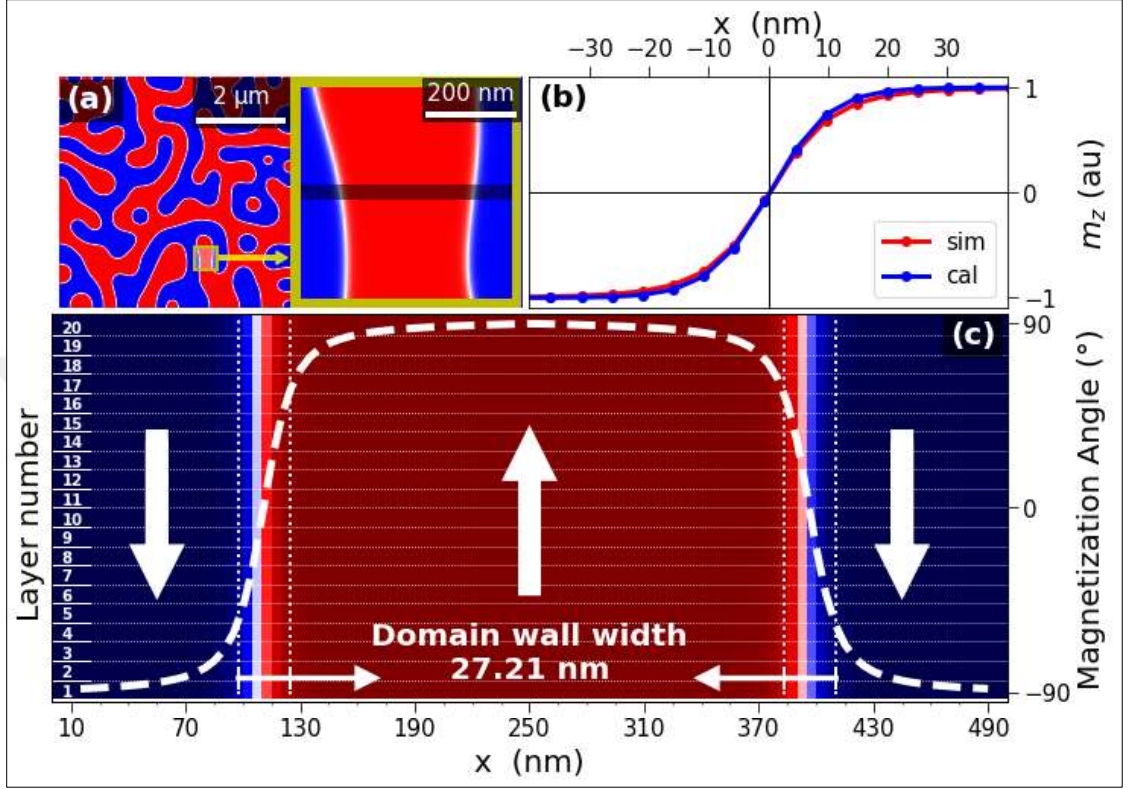


Figure 4.5. Results of the MuMax³ micromagnetic simulations of the N5R20 sample. **a.** The relaxed magnetization pattern averaged over the stack thickness together with a zoomed in view. **b.** Perpendicular magnetization component of the top layer in a transition between two domains together with a calculated transition using the standard Bloch wall model. **c.** Cross section of a cutout of the magnetization of the N5R20 sample showing all 20 layers. The magnetization in the domains is homogeneous and independent of the layer number. The overall magnetization of the domains is depicted by the arrows. The dashed line shows the angle of the magnetization that follows a Bloch-like course of the domain wall transition. The dotted lines mark the domain wall width calculated using the Lilley formula.

The width of Bloch type domains walls is described by Lilley [39] with the following equation;

$$\delta_{DW} = \pi \sqrt{\frac{A_{ex}}{K_{u1}}} \quad (4.2)$$

Domain wall width for the N5R20 sample is calculated $\delta_{DW} = 27 \text{ nm}$ by equation (4.2). In Figure 4.5c, the dotted vertical lines mark the width of the domain walls calculated above and the dashed line shows the angle of the magnetization which follows a Bloch-like domain transition. Therefore, the simulation results can be described with a standard 180° Bloch domain wall model. In Figure 4.5b the model is compared with the simulations. The domain wall shown in the blue line is calculated by the standard approach described in chapter 2.3.3; The average magnetization of each layer $\frac{1}{20} \sum m_z^i$ is discriminated then convoluted with the domain wall kernel described in equation (2.19) by using the domain wall width $\delta_{DW}^{N5R20} = 27 \text{ nm}$. The red curve is the left domain transition shown in Figure 4.5c. The calculated domain transition is in excellent agreement with the simulation which also verifies the domain wall kernel approach used in qMFM. Furthermore, it also indicates that the slight thickness dependence in the domain transitions does not have any influence on the results.

To compare the stray field map of the N5R20 sample measured by qMFM (chapter 3.2.5.3), a simulation that relaxed from a predefined initial magnetization which was calculated from the MFM data was done. This simulation was similar to the simulations in Figure 4.1 but its size was the same as the experimental measurements. The spatial size of the simulation was $(5.12 \times 5.12) \mu\text{m}^2$ over a grid size of (1024×1024) . The micromagnetic simulations of the reduced magnetization are used to calculate the σ_{eff}^{sim} and the H_z^{sim} of the N5R20 sample. The calculations and the comparisons will be discussed in the following chapter.

In summary, micromagnetic simulations verified that the sample does not have any volume charge and its magnetization is homogeneous over the thickness. Therefore, it can be described as only surface charges. Furthermore, all parameters required to calculate the reference effective surface magnetic charge density and/or stray field from

the discriminated MFM image are now available. In the following section, the N5R20 sample will be discussed as a new reference sample. The deconvolved, simulated and calculated stray field of the sample will be compared.



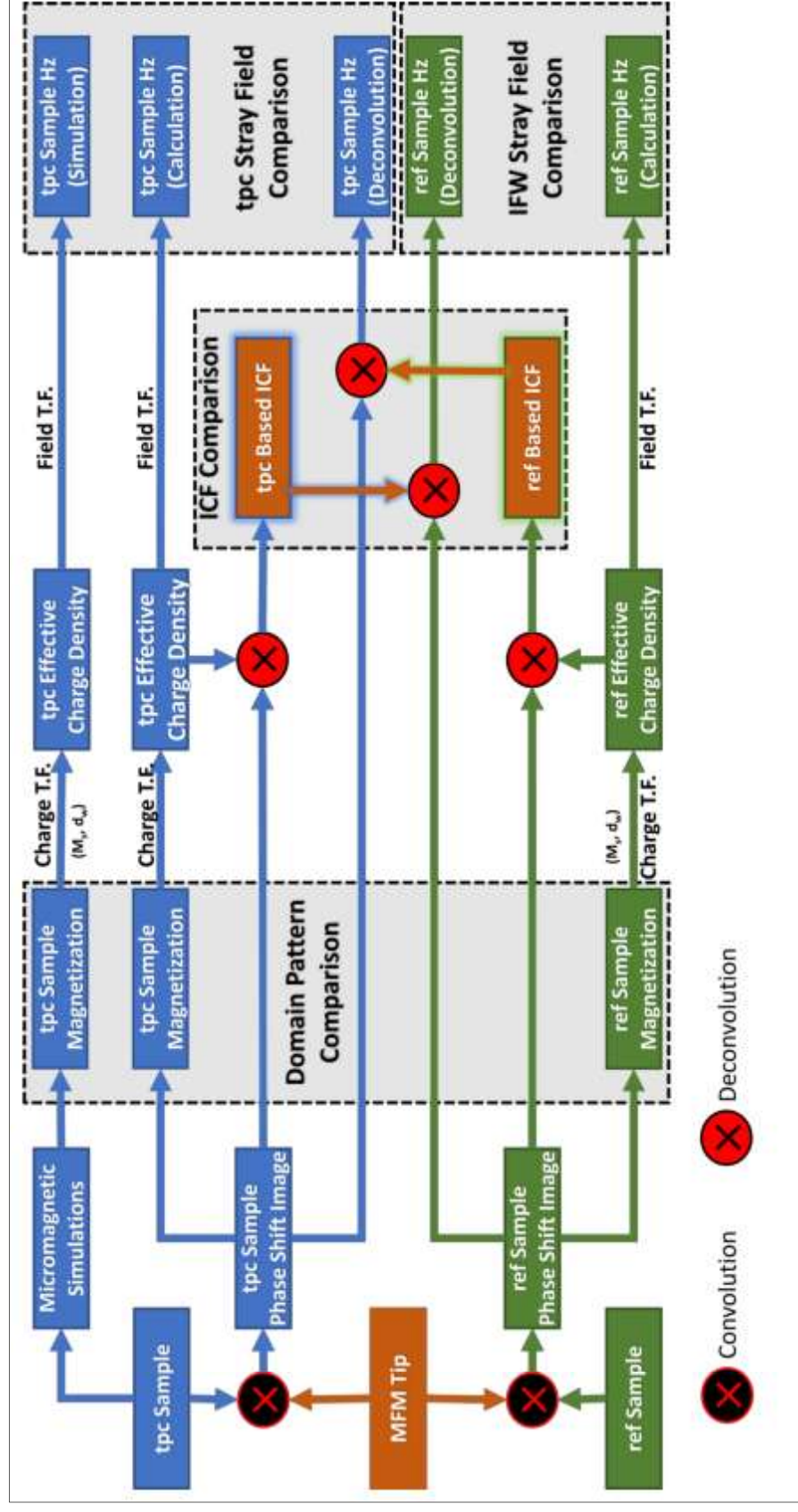


Figure 5.1. Flowchart of the validation process. The different steps of simulations and measurements used to validate the N5R20 (tpc) sample's micromagnetic parameters. The grey shaded boxes mark the comparisons for different approaches and samples.

5. Ti/Pt/Co MULTILAYER STACK as a REFERENCE SAMPLE

The number of reference samples for transfer function based quantitative magnetic force microscopy measurements are limited. The perpendicularly magnetized [Ti(0.3)/Pt(1.5)/Co(0.5)/Pt(1.5)]₂₀ stack fulfils the requirements of a reference sample by its calculability of the magnetization distribution, precise control of the fabrication. In this section, the agreement between the micromagnetic simulations will be emphasized by comparing stray fields obtained with three different approaches. Afterwards, the validity of the new reference sample will be checked by calibrating the tip with the N5R20 sample to be used to measure the stray field of the well-known pre-existing reference sample ([Pt/Co]₁₀₀ from IFW) . In another word, the roles of the two samples will be switched. Furthermore, the accessible magnetic structure sizes covered by calibration with N5R20 and with pre-existing reference samples will be discussed. The detailed flowchart of the validation for the new reference sample is given in Figure 5.1 where the N5R20 sample is named as “tpc” and the pre-existing reference sample from IFW as “ref”.

5.1. Stray Field Comparison for Three Different Approaches

The accurate quantitative stray field measurement is the main motivation of this dissertation. In the 3rd chapter, the qMFM calibrated stray field measurements of three different multilayer-stacks was demonstrated. In the 4th chapter, one of the samples, N5R20 is studied by micromagnetic simulations and the missing parameters required for a reference sample are defined. In this section, the measured stray field with two different theoretical approaches will be compared with the experimentally measured fields. The first theoretical approach is the simulation of the stray fields by using the magnetization pattern of each layer. The second theoretical approach will be the calculation of the stray field from the MFM data as it was described in chapter 2.3.3. The latter is the common approach used in the calibration of the tip.

5.1.1. Simulation of the Stray Field

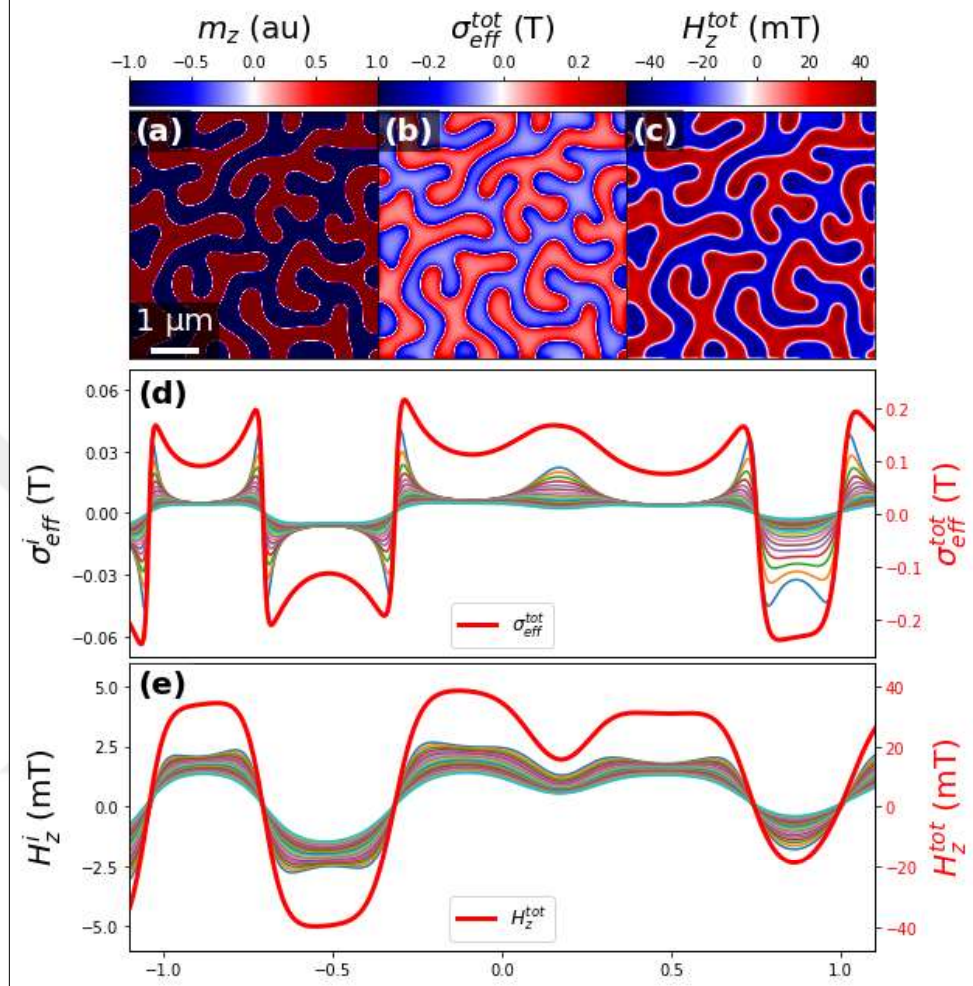


Figure 5.2. Effective charge and stray field simulations. **a.** The micromagnetic simulations of reduced magnetization m_z^{sim} by relaxing from the predefined magnetization calculated from experimental MFM data of the N5R20 sample. **b.** The total σ_{eff}^{tot} **c.** The total H_z^{tot} . **d.** The effective surface charge σ_{eff}^i and **e.** The stray field H_z^i at tip apex ($z = 64 \text{ nm}$) calculated by using the magnetization of each layer separately m_z^i . The different colours are related with the σ_{eff}^i or H_z^i of i^{th} layer. The red curve is the total σ_{eff}^{tot} and H_z^{tot} .

In chapter 4.2, the micromagnetic simulations of the N5R20 sample were discussed in detail. The simulation discussed in the summary of chapter 4.2 with the size of $(5.12 \times 5.12) \mu\text{m}^2$ relaxed from a predefined initial magnetization calculated by

experimental the MFM data is given in Figure 5.2a. The simulation has the same size as the MFM data but its resolution is twice higher ($\delta_A^{MFM} = (10 \times 10) \text{ nm}^2$, $\delta_A^{sim} = (5 \times 5) \text{ nm}^2$). Equations (2.17) and (2.20) are used to calculate the effective magnetic surface charge and the stray field of a magnetic body with homogeneous magnetization over thickness. Therefore, it can be also used to calculate the contributions of each layer separately. The micromagnetic simulations provide the reduced magnetization of each layer (m_z^i) thus the σ_{eff}^{sim} and H_z^{sim} can be calculated precisely by the combination of each layer separately. The σ_{eff}^{sim} given in Figure 5.2b, d and calculated by;

$$\sigma_{eff}^{sim} = \sum_i \sigma_{eff}^i = \sum_i m_z^i(\mathbf{k}) \cdot (1 - e^{-kd_{Co}}) \cdot e^{-kz_0^i} \cdot M_s^{Co} \quad (5.1)$$

where σ_{eff}^i is the effective magnetic charge on the surface due to i^{th} layer, $m_z^i(\mathbf{k})$ is the reduced magnetization of the i^{th} layer in Fourier space, $d_{Co} = 0.5 \text{ nm}$ is the thickness of Co layer, $M_s^{Co} = \frac{d_{tot}}{d_{Co}} \cdot M_s = \frac{3.8}{0.5} \cdot 201 \text{ kAm}^{-1}$ the saturation magnetization of the Co layer and z_0^i is the distance between the i^{th} layer and the sample surface.

Similarly, the H_z^{sim} given in the Figure 5.2c, e is calculated by;

$$H_z^{sim} = \frac{1}{2} \sum_i \sigma_{eff}^i \cdot e^{-kz} = \frac{1}{2} \sum_i m_z^i(\mathbf{k}) \cdot (1 - e^{-kd_{Co}}) \cdot e^{-k(z+z_0^i)} \cdot M_s^{Co} \quad (5.2)$$

where $z = 64 \text{ nm}$ is the distance where the stray field wanted to be calculated. The different coloured field distributions in the line plots (Figure 5.2d, e) are the contribution of each magnetic layer; σ_{eff}^i and H_z^i .

The deconvolved and simulated stray fields are available and the next step is the calculated stray field of the sample for the comparison.

5.1.2. Calculation of the Stray Field

The calculation of the σ_{eff} and H_z from the discriminated image was discussed in chapter 2.3.3 and performed for the reference sample in chapter 3.2.5.1. Briefly, the phase shift data of the MFM measurement is lever corrected then discriminated. Discriminated image is used to calculate the reduced magnetization of the whole sample. In the last step, the reduced magnetization is used to calculate the σ_{eff}^{cal} and/or H_z^{cal} of the sample. In Figure 5.3, the calculation of the effective charge density σ_{eff} and the stray field H_z of the N5R20 is given. The lever correction of the N5R20 MFM data was calculated in Figure 3.23.

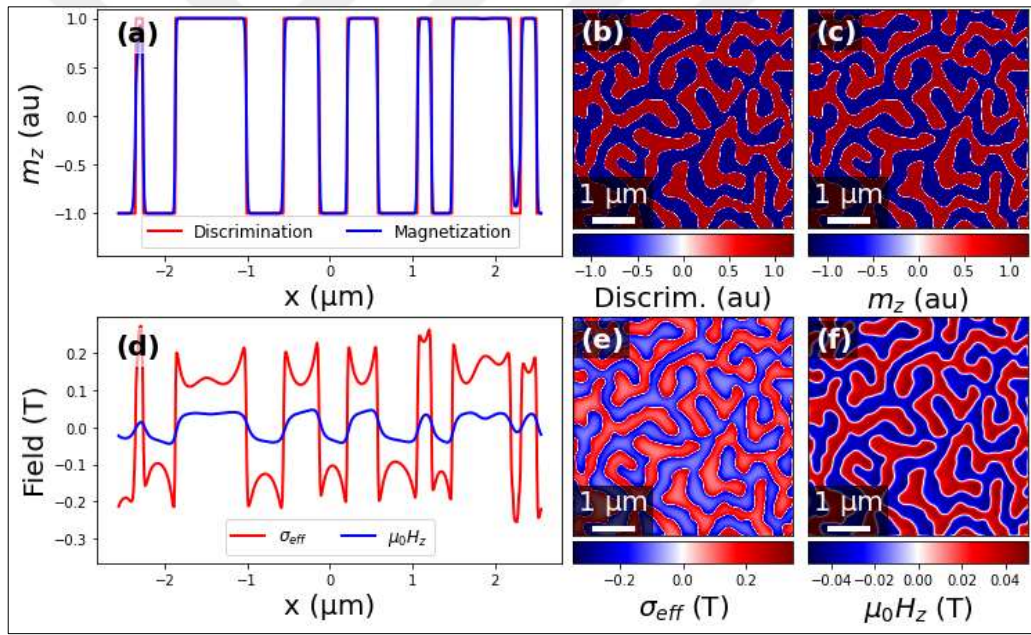


Figure 5.3. Charge density and stray field calculation of N5R20 sample. **b.** Discriminated image. **c.** Perpendicular component of reduced magnetization m_z . The z component of effective magnetic surface charge **(e)** and stray field at $z=64$ nm **(e)**. **a, d.** The plotlines through **(b,e)** - red and **(c, f)** - blue).

In Figure 5.3b the discriminated image of the MFM data is given. This binary matrix is convoluted with the domain wall kernel calculated by the equation (2.19) where the $\delta_{DW} = \delta_{DW}^{N5R20} = 27$ nm is the domain wall width obtained from the micromagnetic

simulations by using the Lilley formula. The reduced magnetization is used to calculate the effective surface charge of the sample than its stray field at the tip apex ($z = 64 \text{ nm}$).

5.1.3. Comparison of Fields

The stray field of the sample at 64 nm is obtained from three different approaches. The first one is qMFM deconvolution. The steps of the deconvolution is demonstrated in chapter 3.2.5; the tip is calibrated by a reference measurement over the well-defined [Pt/Co]₁₀₀ sample provided by Dr Volker Neu from IFW-Dresden. The instrumental calibration function ICF is used to deconvolve the MFM data of the N5R20 sample to obtain the σ_{eff}^{dec} where the *dec* is the abbreviation for the “deconvolved”. Afterwards the H_z^{dec} is calculated by using the equation (2.20).

The other method to obtain the standard calculation approach used widely in qMFM calibrations by discriminating the MFM data for calculating its reduced magnetization. The reduced magnetization is used to calculate the σ_{eff}^{cal} and/or H_z^{cal} of the sample where *cal* is the abbreviation of the “calculated”.

The last method was using the micromagnetic simulations results. The reduced magnetization of each layer was simulated by relaxing the initial magnetization defined from the experimental data to an equilibrium state. The equations used to calculate σ_{eff} has one requirement; thickness independent magnetization. Therefore, each magnetic layer of the sample can be treated as one magnetic sample. The contribution from each magnetic layer simulated separately and used to calculate the total σ_{eff}^{sim} and H_z^{sim} by using the equation (5.1) and (5.2) where the *sim* is the abbreviation of the “simulated”.

In Figure 5.4 the perpendicular stray fields components obtained from three different approaches are compared in detail. All of the stray fields are measured/calculated at $z = 64 \text{ nm}$ above from the surface on the plane at the tip apex. In Figure 5.4g the line plots of the fields are in good agreement.

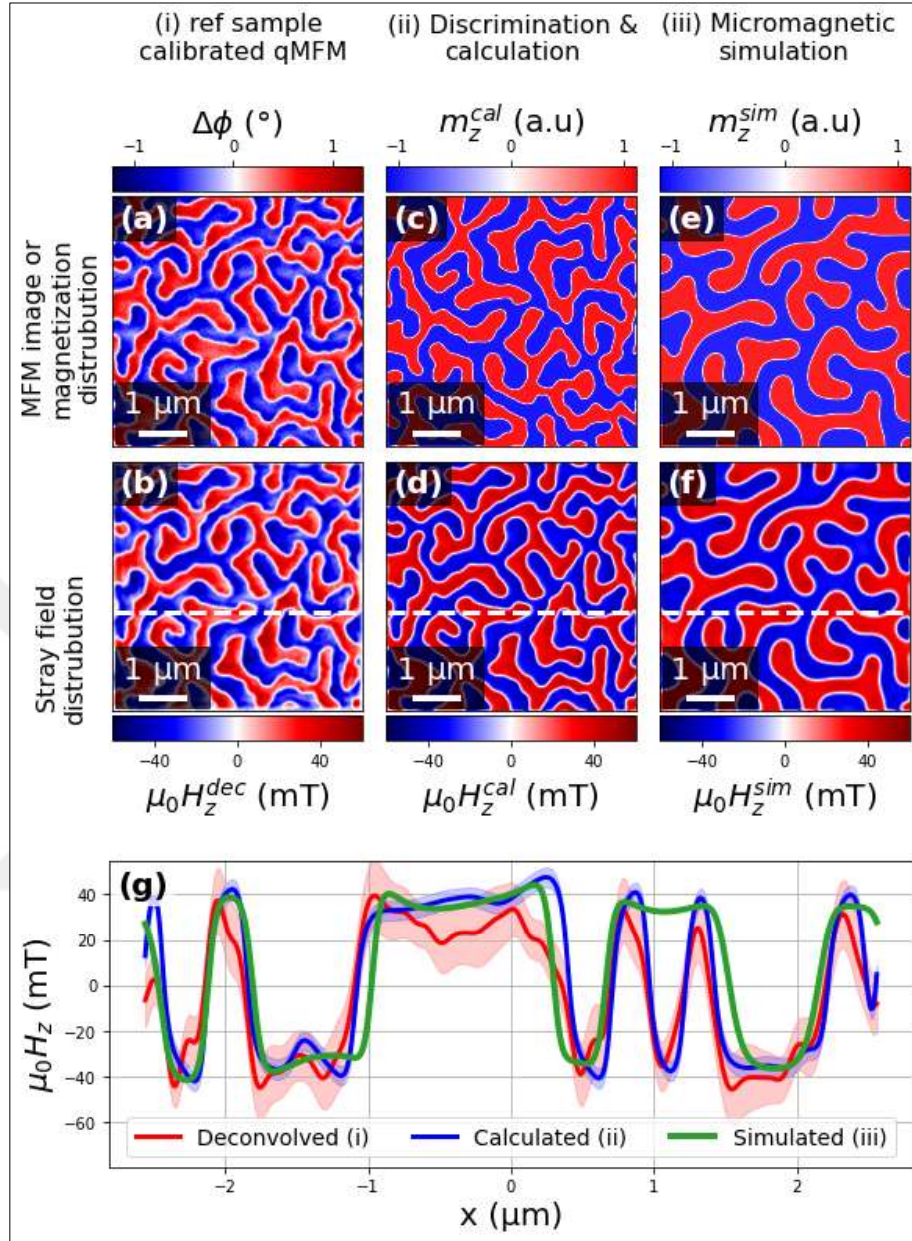


Figure 5.4. Comparison of simulated and experimental N5R20 sample data. a. Measured MFM phase shift data and b. perpendicular stray field components $\mu_0 H_z^{dec}$ data calculated using the [Co/Pt]₁₀₀ samples calibrated qMFM; c. The z component of the magnetization calculated from discrimination of the MFM phase shift data from (a); d. The thereof calculated perpendicular stray field components $\mu_0 H_z^{cal}$ data using the N5R20 micromagnetic material parameters; e. The z-component of the magnetization from the micromagnetic simulation and f. The perpendicular stray field components $\mu_0 H_z^{sim}$ data calculated thereof using a layer by layer approach; g. The stray field data with uncertainty bands from the data marked by the dashed lines in the stray field images in (b,d,f).

This result is further important as the $\mu_0 H_z^{cal}$ is calculated by using the effective medium approach where the magnetic layer is considered as the whole Ti/Pt/Co stack thus magnetic parameters are scaled; the M_s is calculated by using the thickness of the whole sample $d_{tot} = 3.8 \text{ nm} \times 20$. However, the $\mu_0 H_z^{sim}$ is calculated by using a layer-by-layer approach and by describing the magnetization of each layer as the magnetization of only Co layers. Therefore, the saturation magnetization M_s^{Co} is calculated by using the thickness of Co layers $d_{Co} = 0.5 \text{ nm} \times 20$. The agreement between the $\mu_0 H_z^{cal}$ and $\mu_0 H_z^{sim}$ verify the use of this approach to calculate the stray field of the sample for calibrations. Additionally, the agreement between the qMFM measurement further validates the accuracy of the transfer function-based field measurements.

The shaded bands which show the uncertainties are calculated by using the Guide to the expression of uncertainty in measurement GUM [97], [98]. The uncertainty calculations are explained in the following section.

5.2. Uncertainty Evaluation

To estimate the uncertainties of the calculated and deconvolved field distributions, the uncertainties of the measurement data and measurement parameters are propagated through the deconvolution following a GUM conform approach [97].

5.2.1. Uncertainty Propagating due to the Fourier Transform

In the qMFM measurements, there are multiple back-and-forths partial Fourier transforms. Even though the transformations are well defined, they contribute to the uncertainties at each transformation. The propagation of the uncertainty from the real to the Fourier domain for a 2D image of G_{mn} is described by;

$$u_{RG}^2 = \sum_{m=0}^{M-1} \sum_{n=0}^{N-1} \cos^2 \left(2\pi \frac{km}{M} + 2\pi \frac{ln}{N} \right) u_{Gmn}^2 \quad (5.3)$$

$$u_{SG}^2 = \sum_{m=0}^{M-1} \sum_{n=0}^{N-1} \sin^2 \left(2\pi \frac{km}{M} + 2\pi \frac{ln}{N} \right) u_{Gmn}^2 \quad (5.4)$$

where the u_{RG}^2 and u_{SG}^2 are the square of the real and imaginary part of the uncertainty for G_{mn} in Fourier space, u_{Gmn} is the uncertainty of the G_{mn} in real space and $k, l = 0, \dots, M-1, N-1$. The propagation of the uncertainty from the Fourier to the real domain for a 2D image of F in Fourier space is described by;

$$u_{Fmn}^2 = \frac{1}{(MN)^2} \left[\sum_{m=0}^{M-1} \sum_{n=0}^{N-1} \cos^2 \left(2\pi \frac{km}{M} + 2\pi \frac{ln}{N} \right) u_{RF}^2 + \sum_{m=0}^{M-1} \sum_{n=0}^{N-1} \sin^2 \left(2\pi \frac{km}{M} + 2\pi \frac{ln}{N} \right) u_{SF}^2 \right] \quad (5.5)$$

where u_{RF} and u_{SF} are the real and imaginary parts of the uncertainties of F in Fourier space. Using both equations (5.3), (5.4) and (5.5), the uncertainty due to the back-and-forth Fourier transforms can be calculated.

5.2.2. Uncertainty Propagation due to the Wiener Filter

The regularized Wiener filter was defined in equation (2.15). Based on this definition, each input is written with the real and imaginary parts;

$$R_T = \frac{R_G R_F + S_G S_F}{R_F^2 + S_F^2 + \alpha} \quad (5.6)$$

$$S_T = -\frac{R_G S_F + S_G R_F}{R_F^2 + S_F^2 + \alpha} \quad (5.7)$$

The sensitivity coefficients for each input and can be written. The real part of the is described by;

$$R_{RG} = \frac{\partial R_T}{\partial R_G} = \frac{R_F}{R_F^2 + S_F^2 + \alpha} \quad (5.8)$$

$$R_{SG} = \frac{\partial R_T}{\partial S_G} = \frac{S_F}{R_F^2 + S_F^2 + \alpha} \quad (5.9)$$

$$R_{RF} = \frac{\partial R_T}{\partial R_F} = \frac{R_G (S_F^2 - R_F^2 + \alpha) - 2S_G R_F S_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.10)$$

$$R_{SF} = \frac{\partial R_T}{\partial S_F} = \frac{S_G (R_F^2 - S_F^2 + \alpha) - 2R_G R_F S_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.11)$$

$$R_\alpha = \frac{\partial R_T}{\partial \alpha} = -\frac{R_G R_F + S_G S_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.12)$$

where the imaginary part is given by;

$$S_{RG} = \frac{\partial S_T}{\partial R_G} = \frac{S_F}{R_F^2 + S_F^2 + \alpha} \quad (5.13)$$

$$S_{SG} = \frac{\partial S_T}{\partial S_G} = \frac{R_F}{R_F^2 + S_F^2 + \alpha} \quad (5.14)$$

$$S_{RF} = \frac{\partial S_T}{\partial R_F} = \frac{S_G(S_F^2 - R_F^2 + \alpha) - 2R_G R_F S_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.15)$$

$$S_{SF} = \frac{\partial S_T}{\partial S_F} = \frac{R_G(R_F^2 - S_F^2 + \alpha) - 2S_G R_F S_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.16)$$

$$S_\alpha = \frac{\partial S_T}{\partial \alpha} = -\frac{R_G S_F + S_G R_F}{(R_F^2 + S_F^2 + \alpha)^2} \quad (5.17)$$

Using the coefficients given in the equation sets of (5.6) - (5.17), the uncertainty of the T in the Fourier domain becomes;

$$u_{R_T}^2 \equiv (R_{RG})^2 u_{RG}^2 + (R_{SG})^2 u_{SG}^2 + (R_{RF})^2 u_{RF}^2 + (R_{SF})^2 u_{SF}^2 + (R_\alpha)^2 u_\alpha^2 \quad (5.18)$$

$$u_{S_T}^2 \equiv (S_{RG})^2 u_{RG}^2 + (S_{SG})^2 u_{SG}^2 + (S_{RF})^2 u_{RF}^2 + (S_{SF})^2 u_{SF}^2 + (S_\alpha)^2 u_\alpha^2 \quad (5.19)$$

where the u_{R_T} and u_{S_T} are the real and imaginary parts of the uncertainty.

5.2.3. Uncertainty of Calculated Fields

The magnetic field of the reference sample $\mu_0 H_z^{cal}$ is calculated from the discriminated MFM phase shift image convoluted with the domain wall kernel $M_{0,dw} = M_s \cdot m_z$. It is assumed that there is no uncertainty here. The uncertainties of $\mu_0 H_z^{cal}$ are due to propagation of the uncertainties of the measurement height z , the sample thickness d and the sample saturation magnetization M_s , following the formula;

$$H_z^{cal} = RH_z^{cal} + iSH_z^{cal} = \frac{1}{2} \cdot e^{-kz} \cdot (R\sigma_{eff}^{cal} + iS\sigma_{eff}^{cal}) \quad (5.20)$$

Therefore, the uncertainty of the H_z in Fourier domain can be described as;

$$u(RH_z^{cal})^2 = \left(\frac{k}{2} \cdot e^{-kz} \cdot R\sigma_{eff} \right)^2 \cdot u(z)^2 + \left(\frac{1}{2} e^{-kz} \right)^2 \cdot u(R\sigma_{eff})^2 \quad (5.21)$$

$$u(SH_z^{cal})^2 = \left(\frac{k}{2} \cdot e^{-kz} \cdot S\sigma_{eff} \right)^2 \cdot u(z)^2 + \left(\frac{1}{2} e^{-kz} \right)^2 \cdot u(S\sigma_{eff})^2 \quad (5.22)$$

where the $u(z)$ is the uncertainty of the measurement height. The uncertainties of σ_{eff} in Fourier domain can be written as;

$$u(R\sigma_{eff}^{cal})^2 = \left(\frac{\mu_0}{d} Rm_z \cdot \frac{1}{2} (1 - e^{-dk}) \right)^2 \cdot u(M_s)^2 + \left(\frac{\mu_0 M_s}{d^2} Rm_z \cdot \frac{1}{2} (d \cdot k - 1 - e^{dk}) e^{-dk} \right)^2 \cdot u(d)^2 \quad (5.23)$$

$$u(S\sigma_{eff}^{cal})^2 = \left(\frac{\mu_0}{d} Sm_z \cdot \frac{1}{2} (1 - e^{-dk}) \right)^2 \cdot u(M_s)^2 + \left(\frac{\mu_0 M_s}{d^2} Sm_z \cdot \frac{1}{2} (d \cdot k - 1 - e^{dk}) e^{-dk} \right)^2 \cdot u(d)^2 \quad (5.24)$$

where the Rm_z and Sm_z are the real and imaginary parts of the reduced magnetization in Fourier space $m_z(\mathbf{k}) = m_z(k_x, k_y)$ calculated by domain wall convolution with the discriminated MFM data. $u(d)$ and $u(M_s)$ are the uncertainties of the thickness d and the saturation magnetization M_s .

5.2.4. Uncertainty Parameters

The uncertainty bands used throughout the study are calculated by using the equations derived in this section by using the parameters given in table 5.2

Table 5.2. Uncertainty parameters used in uncertainty band calculations

Parameter	Uncertainty
MFM Phase Shift, $\Delta\phi$	$u(\Delta\phi) = 0.2^\circ$
regularization parameter, α	$u(\alpha) : 1\%$
stack thickness of N5R20 sample, d^{N5R20}	$u(d) = 2 \text{ nm}$
stack thickness of ref sample, d^{ref}	$u(d) = 4 \text{ nm}$
saturation magnetization of N5R20 sample, M_s^{N5R20}	$u(M_s) : 6\%$
saturation magnetization of ref sample, M_s^{ref}	$u(M_s) : 6\%$
measurement height, z	$u(z) : 10\%$

5.3. Cross-Validation of Pt/Co Sample and Ti/Pt/Co sample

The perpendicular stray field of the N5R20 sample comparison verifies the validity of its micromagnetic parameters. Therefore, the sample could be used to calibrate the tip. As it was shown in Figure 5.1, the roles of the N5R20 (or tpc) as the target sample and the pre-existing reference (ref) sample as the calibration samples could be switched; Calibrate the tip by using N5R20, measure the field of the ref.

Each step before the deconvolution was already calculated for the N5R20 sample and demonstrated in Figure 5.4. The deconvolution of ICF was calculated by using the MFM data and the σ_{eff}^{cal} from the N5R20 (Figure 5.4a). The regularization parameter α is optimized by using the L-Curve criterion (see chapter 2.3.4). In optimization, α was scanned between 5.1×10^{-6} and 4.9×10^{10} . The optimum $\alpha = \alpha_{opt}$ was found at 5.6×10^1 . The cut-off frequency for α_{opt} shown in Figure 5.51, was around $140 \mu m^{-1}$.

The lower frequencies are not filtered out and this indicates a high accuracy on the optimization. The circular average of the ICF was calculated to suppress the artefacts in the image as discussed in chapter 3.2.5.1. According to Figure 5.5c, there is no significant information lost during the averaging process. The ICF^{N5R20} is used to deconvolve the effective charge density σ_{eff}^{dec} of the reference sample.

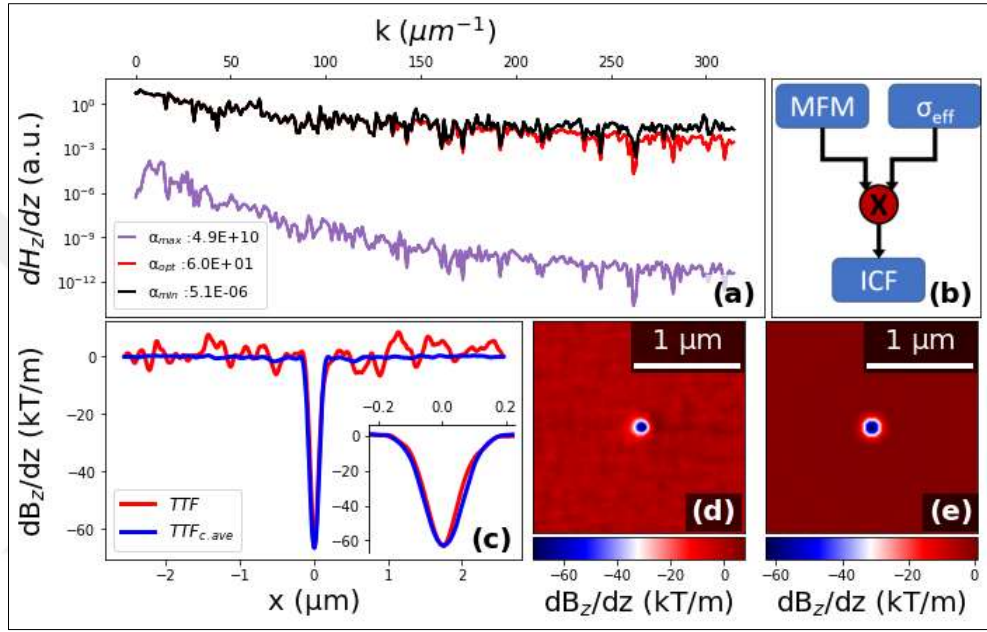


Figure 5.5. Deconvolution of ICF from the N5R20 sample. **a.** The Fourier spectra of the deconvolved ICF by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data measured and the σ_{eff}^{ref} calculated from N5R20 sample deconvoluted to obtain the ICF . **d.** The deconvoluted ICF^{N5R20} . **e.** The circular average of the ICF^{N5R20} . **c.** The plotlines through (d) and (e). The inset figure is the zoomed-in of the (c).

In Figure 5.6 the stray field measurement of the reference sample by deconvolution is given. The effective surface charge density σ_{eff}^{dec} is deconvoluted from the MFM data of the reference sample by using the ICF^{N5R20} calibrated from the N5R20 sample. The Fourier spectrum of the deconvolution shows slightly over filtering (Figure 5.6a). Even though the filtering level exceeds the acceptable range, the L-curve optimized parameter has been used to eliminate any discrepancy due to changes in the

deconvolution method between the two samples. The deconvoluted σ_{eff}^{dec} does not have artefacts as many as the other field deconvolutions. This is due to the over filtering described above.

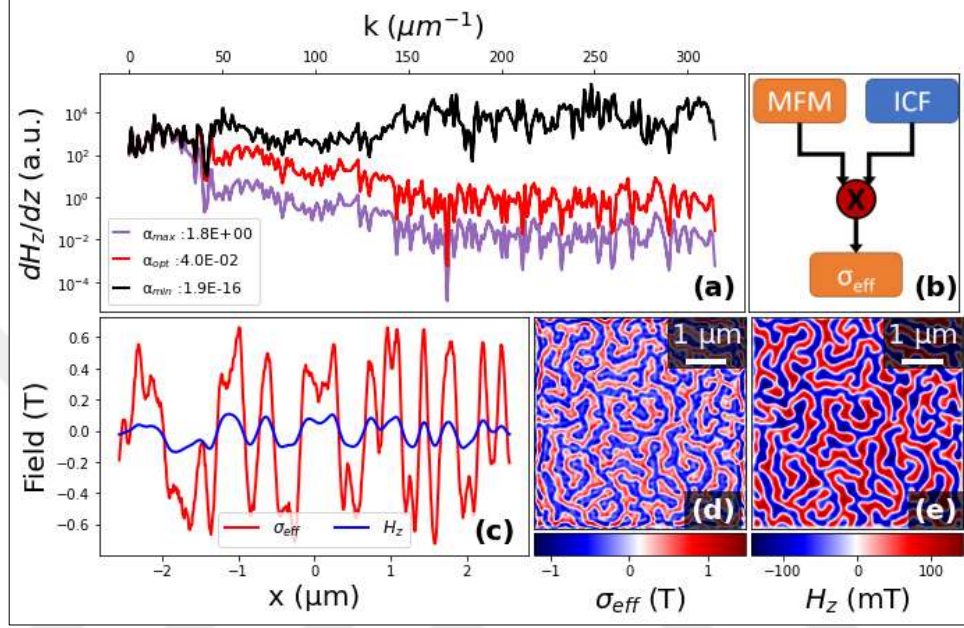


Figure 5.6. Field deconvolution of the ref sample. **a.** The Fourier spectra of the deconvoluted σ_{eff} by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data measured from ref sample and the ICF is calibrated from N5R20 sample deconvolved to obtain the σ_{eff}^{ref} . **d.** the deconvoluted σ_{eff}^{ref} . **e.** The stray field of the ref sample H_z^{ref} at tip apex ($z' = 64$ nm). **c.** the plotlines through (d) and (e).

The quantitative ref sample stray field H_z^{dec} measured from the deconvolution of MFM phase shift data and the ICF^{N5R20} is shown in Figure 5.7b. The calculation of H_z^{cal} was demonstrated in chapter 3.2.5.1 (Figure 3.19) where the field was forward simulated using the micromagnetic parameters of the ref sample. The line plots of both stray fields taken along the dashed lines plotted together with the uncertainty bands. The uncertainty bands are calculated from the uncertainties given in table 5.2 by using the equations described in chapter 5.3. The deconvoluted and calculated perpendicular stray fields of the ref sample is in excellent agreement (Figure 5.7e) with small differences

mostly within the uncertainty margins. This indicates that the N5R20 sample is suitable to calibrate the magnetic tips as the stray field of the $[\text{Pt}/\text{Co}]_{100}$ was measured quantitatively with high-accuracy.

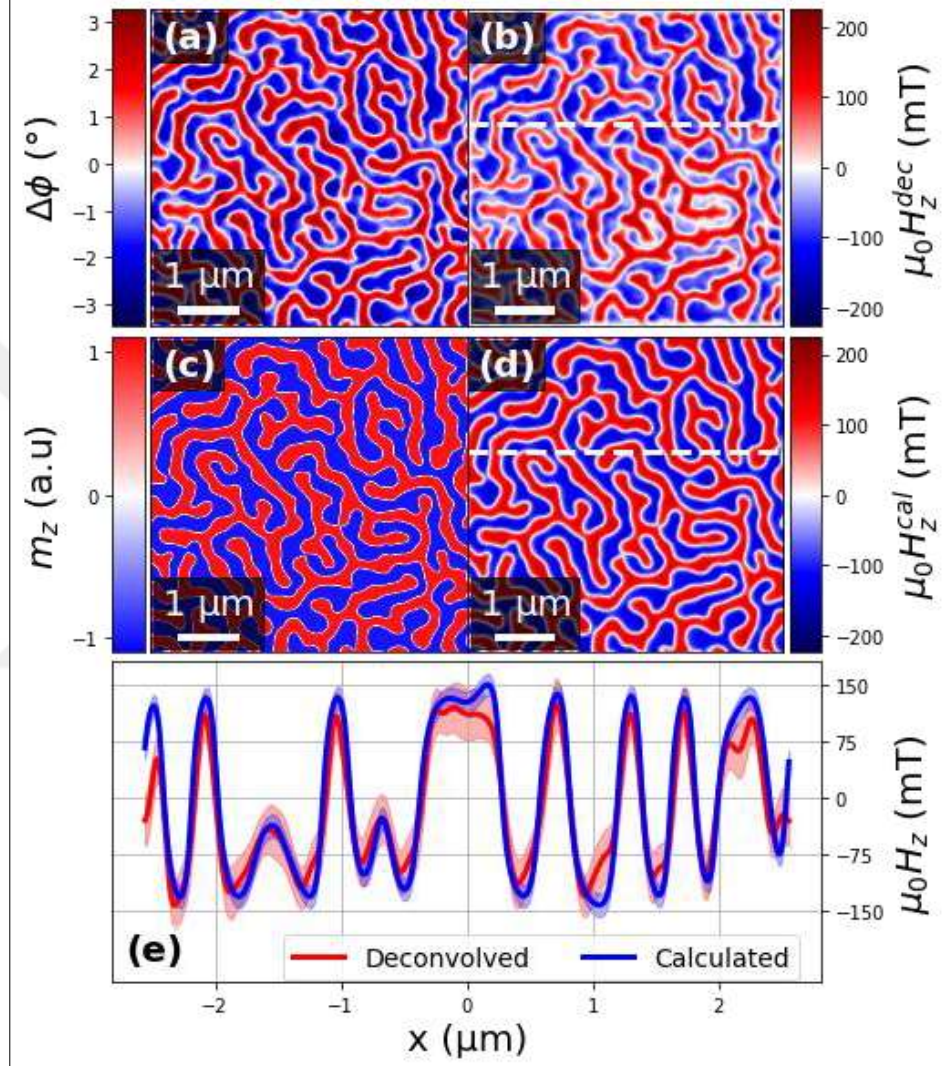


Figure 5.7. Validation of N5R20 calibration by deconvoluted and calculated field comparison. **a.** MFM data and **b.** The quantitative perpendicular field component $\mu_0 H_z^{dec}$ measured by N5R20 calibrated qMFM of the $[\text{Pt}/\text{Co}]_{100}$ (ref) sample. **c.** Reduced magnetization m_z^{ref} of the ref sample calculated from the discriminated phase shift data followed by a convolution with a domain wall kernel. **d.** Perpendicular field component $\mu_0 H_z^{cal}$ calculated thereof by forward simulations using the known micromagnetic material parameters. **e.** Plotlines of through the $\mu_0 H_z^{dec}$ (b) and $\mu_0 H_z^{cal}$ (d) along the dashed lines together with the uncertainty bands.

In Figure 5.8a and b, the ICF s calibrated from N5R20 (blue) and ref (red) samples which were already demonstrated in Figure 5.5 and Figure 3.20 respectively. To have the comparability, both are calculated at 64 nm away from the tip apex (on sample surface). The TTF s, reflecting the magnetic properties of the tip, thus ideally should be independent of the calibration samples. The comparison in Figure 5.8c shows very good agreement with small discrepancies. These discrepancies might be attributed with the differences found in the Fourier spectra of the N5R20 and ref samples.

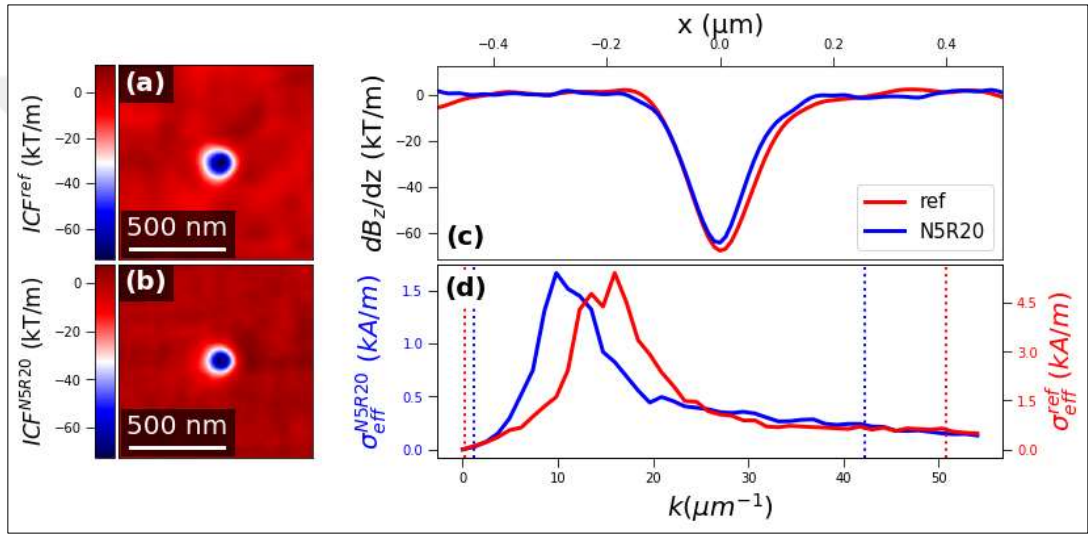


Figure 5.8. Comparison of ICFs and the calibration spectra. The ICF calibrated from ref (a) and N5R20 (b) sample measurements. c. The plotlines through the maxima of the TTFs for both TTF^{ref} (red) and TTF^{N5R20} (blue). The ICFs are calculated at 64 nm away from the tip apex, so on the sample surface ($z = 0$). d. The Fourier spectra of the calculated effective surface charge density σ_{eff}^{cal} of ref (red) and N5R20 (blue) samples. Dotted vertical lines mark the area of k-vectors accessible after the calibration with the respective calibration sample.

In Figure 5.8d the Fourier spectra of the N5R20 (blue) and ref (red) samples are shown. Both spectra show significant overlap. This overlap justifies the qMFM characterization of the N5R20 sample calibrated with ref sample (or visa-versa) even though the dominant components in the spectra are not identical. The amplitude of the N5R20 sample is significantly lower due to its lower saturation magnetization and lower thickness compared to the ref sample. The dotted vertical lines mark the area of k-

vectors accessible by the calibrations performed with the respective calibration sample for a measurement performed with a noise standard deviation of 0.2° . The details of the estimation for the accessible spatial frequency ranges will be discussed in the following chapter.

5.4. Estimation of Accessible Spatial Frequency Range

Above, the usability of the N5R20 sample was discussed from the standpoint of the calculability and field range. However, the application range of the N5R20 sample for qMFM based measurements of the target sample must also be discussed in terms of the accessible feature size spectrum. The magnetic features on length scales not covered by the calibration samples accessible spatial frequency range will be suppressed in the calibration measurements. This covered feature sizes can be quantified in the Fourier spectrum of the sample's effective charge distribution σ_{eff} as a function of the spatial frequency (Figure 5.8d). However, the accessible spectral range does not depend only on the Fourier spectrum of calibration samples effective charge density $\sigma_{eff}(\mathbf{k})$, but rather on its combination with contributions from the instrumentation; The SPM used in MFM measurements and the magnetic tip. The MFM detection system determines the noise floor of the measurement and the tip's magnetic field gradient, $\frac{dB_z}{dz}$ enters into the measurement sensitivity. This follows from the fact that the sensitivity of the MFM is determined by the tip's stray field gradient;

$$\Delta\phi(\mathbf{k}, z') = -\sigma_{eff}^* \cdot \frac{Q}{c} \cdot \frac{dB_z^{tip}}{dz} \quad (5.25)$$

where the lever correction is neglected. Therefore, the spectral spectrum accessible by the calibration should be investigated together with such instrumental contributions. The sensitivity of the system during the calibration measurement is determined by the noise level of the phase shift data which is determined by the MFM detection system. To estimate the accessible range of the spatial frequency, of the calibration, a generic tip's

stray field gradient was modelled by using a Gaussian function (Figure 5.9a, b inset). The amplitude of the function was 60 kTm^{-1} and the full width half maximum was $\Sigma_x = \Sigma_y = 50 \text{ nm}$. These values are derived from the typical values found in the real-world tips.

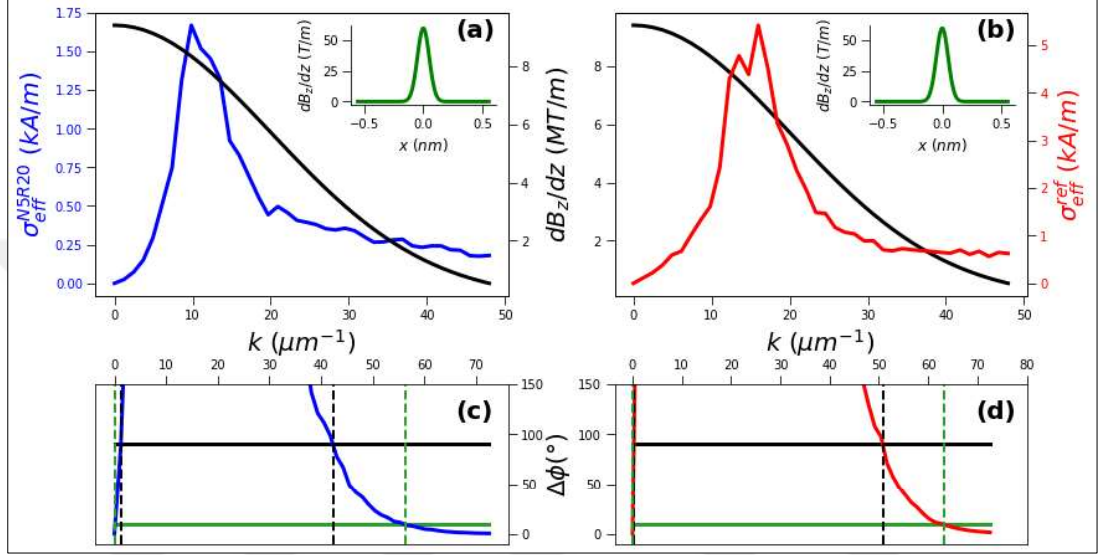


Figure 5.9. Estimation of accessible wave vector range after calibration. The tip mediated sensitivity in dB_z/dz and the circularly averaged Fourier spectra of σ_{eff}^{cal} for **a.** N5R20 and **b.** ref samples. The green inset figures show the generic tips, dB_z/dz in real space. The phase shift data $\Delta\phi$ of a simulated calibration measurement by using the generic tip from **c.** N5R20 sample and **d.** ref sample. The horizontal lines mark the noise floor for a Gaussian white noise with the standard deviations of 0.2° (black) and the 0.02° (green) respectively.

In Figure 5.9a, b, the tip mediated sensitivity $\frac{dB_z}{dz}$ is plotted with the circularly averaged effective charge densities σ_{eff}^{cal} of the N5R20 (a) and ref (b) sample calculated from the discriminated MFM data. Both spectra are plotted in Fourier space. The forward simulated phase shifts are calculated by using the tip mediated sensitivity and the effective charge density. The cut-outs of the cross-sections of these simulated phase shifts in Fourier space $\Delta\phi(k)$ were plotted in Figure 5.9c, d for the N5R20 and ref sample respectively. The simulations were calculated by using a quality factor of $Q = 250$ and a cantilever stiffness of $c = 3 \text{ Nm}^{-1}$.

Two typical noise levels are selected to define the wave-vector cut-off frequencies. The noises are Gaussian white noises with the standard deviations of 0.2° and 0.02° . The higher noise (0.2°) is typical for the ambient condition MFM systems and plotted with the black horizontal line in Figures 5.9c and d. The lower noise (0.02°) is typical for low noise setups such as vacuum MFM and plotted with the green horizontal line in the same figures. The interceptions of the noise levels (horizontal lines) with the phase shift $\Delta\phi(\mathbf{k})$ are marked with dotted vertical lines and defines the low (k_{low}) and high (k_{high}) wave-vector cut-off frequencies. In both cases for both of the samples, the low cut-off frequencies (k_{low}) are limited by the sample size. However, the high cut-off frequencies are sample and noise dependent. In table 5.9 the derived k -vectors and the corresponding wavelength data is given where the wavelength is calculated by $\lambda = \frac{2\pi}{k}$. Therefore, the table summarizes the range of detectable characteristic structure sizes by MFM after a calibration with the related sample.

Table 5.1. Cut-off wave vector and corresponding wavelength data for the N5R20 and the ref sample for two different noise levels.

$\Delta\phi$	Low Cut-Off		High Cut-Off	
	Frequency	Wavelength	Frequency	Wavelength
<i>Ti/Pt/Co multilayer stack (N5R20)</i>				
0.2°	$< 1.22 \mu m^{-1}$	$> 5.12 \mu m$	$42.256 \mu m^{-1}$	$149 nm$
0.02°	$< 1.22 \mu m^{-1}$	$> 5.12 \mu m$	$56.295 \mu m^{-1}$	$112 nm$
<i>Pt/Co stack (ref)</i>				
0.2°	$< 1.22 \mu m^{-1}$	$> 5.12 \mu m$	$50.726 \mu m^{-1}$	$124 nm$
0.02°	$< 1.22 \mu m^{-1}$	$> 5.12 \mu m$	$63.231 \mu m^{-1}$	$99 nm$

The accessible feature sizes after the calibrations, performed with typical noise floor in ambient condition measurements ($std_{ambient} = 0.2^\circ$), are $149 nm - 5.12 \mu m$ for the N5R20 sample and $124 nm - 5.12 \mu m$ for the ref sample. The calibrations with typical noise floor in vacuum measurements ($std_{vacuum} = 0.02^\circ$), would allow access to smaller structure sizes; $112 nm - 5.12 \mu m$ for the N5R20 sample and $99 nm - 5.12 \mu m$ for the ref sample.

5.5. Estimation of the Surface Roughness

The surface roughness of the N5R20 sample was studied with atomic force microscopy in a Park Nx-Hivac system using a PPP-NCHR tip. The sample was measured in AFM shows a sub-nm scale surface roughness (Figure 5.10). The RMS roughness of this sample was calculated $R_q = 0.6 \text{ nm}$.

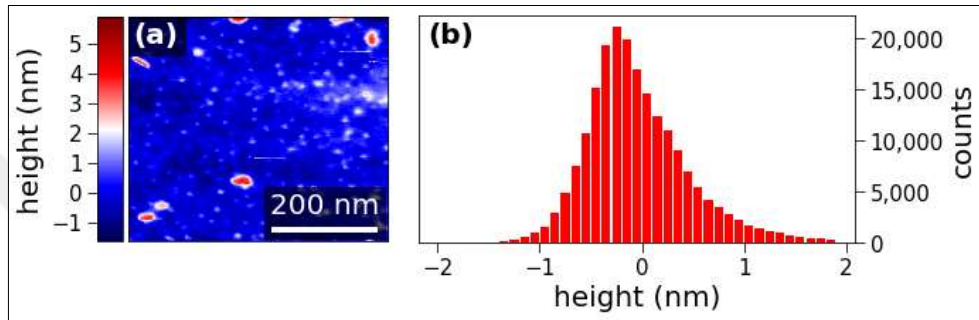


Figure 5.10. Surface roughness analysis. **a.** The topography of the N5R20 sample measured by AFM and **b.** The histogram plot of the

In parallel works performed by Melek Türksoy Öcal, a similar stack with one repetition was characterized by STM and the surface roughness was found 0.3 nm . This agreement is considered excellent as the STM measurements of the single stack tpcp multilayer measured in-situ even the thicknesses of both samples are different with a huge margin, 2.8 nm & 76 nm [83].

5.6. Discussion

In this section, the validity of the N5R20 sample was discussed in detail and consequently has been proven to be a suitable reference sample. The sample covers the feature size from the $10 \mu\text{m}$ to the 100 nm range. Therefore, it can be used in quantitative characterizations of the relevant micromagnetic structures. The N5R20 sample has well-defined interfaces and a very low (sub-nm) surface roughness. The magnetic properties of the sample were defined by both macroscopic magnetic

characterizations and micromagnetic simulations. The magnetic structures resulted from the micromagnetic simulations were validated by qMFM measurements. The N5R20 sample exhibits $\pm 60 \text{ mT}$ stray field 64 nm above the surface while the ref sample has the stray field maximum even higher than $\pm 130 \text{ mT}$. This low stray field with similar feature size spectra makes N5R20 sample a good candidate to be used as a reference sample for the calibration of low coercivity tips. The N5R20 sample was also successfully used as a “reference” sample for the qMFM stray field measurements of the “ref” sample from IFW ([Pt/Co]₁₀₀).



6. QUANTUM CALIBRATION of MFM (QuMFM)

In this section, we report the work was performed as a collaboration with Prof. Dr Fedor Jelezko's group where quantum sensing was introduced into classical field calibrations by exploiting the quantum effects of the NV centres. This section reports the first validation of the classical calibration techniques by an independent direct measurement technique, where the tip's stray field measured directly by an atom-sized quantum field sensor. Furthermore, a novel approach to calibrate the MFM tip was introduced by using the NV technology where the accuracy of the calibration is independent of the calibration sample, thus the open roads towards quantum traceable calibration of any MFM.

The measurements in this section were performed at Institute for Quantum Optics, Ulm University, Germany together with Dr Yan Liu at Prof. Jelezko's lab. Dr Liu build the setup, performed the NV measurements of the MFM tip and provided the ODMR data (in terms of $\mu_0 H_z$ and β of the tip). The author was responsible for the MFM measurements over the reference sample and the analysis of the data.

6.1. NV-Centers in Diamond and NV-Magnetometry

Nitrogen-Vacancy (NV) Centers, point-like defects in the diamond crystal are crucial physical systems for quantum information such as quantum computers and quantum sensing [99]. The NV centres are formed by the replacement of a Carbon atom next to a vacancy spot in the diamond (Figure 6.1a). Even though the colour centres in diamond are discovered long before [100, 101], the experimental demonstration of the optically detected magnetic resonance of a single NV centre was demonstrated by Gruber *et al.* in 1997 [102]. There are two types of NV centres; neutral (NV^0) and negatively charged (NV^-). In this study, a NV^- was used for the quantitative measurements.

The electronic structure of the NV^- centres are formed of a ground states triplet ($^3A_2$), an excited state triplet (^3E) and two intermediate singlets ($^1A_1$, ^1E) (Figure 6.1b). The ground states can be excited incoherently using a green light source ($\lambda_{exc} = 532\text{ nm}$). The excited states are connected to the ground states by an optical transition with $\lambda_{relax} = 637\text{ nm}$ zero-phonon-line (ZPL). Briefly, the NV centre excited by a green laser will result in red light radiation.

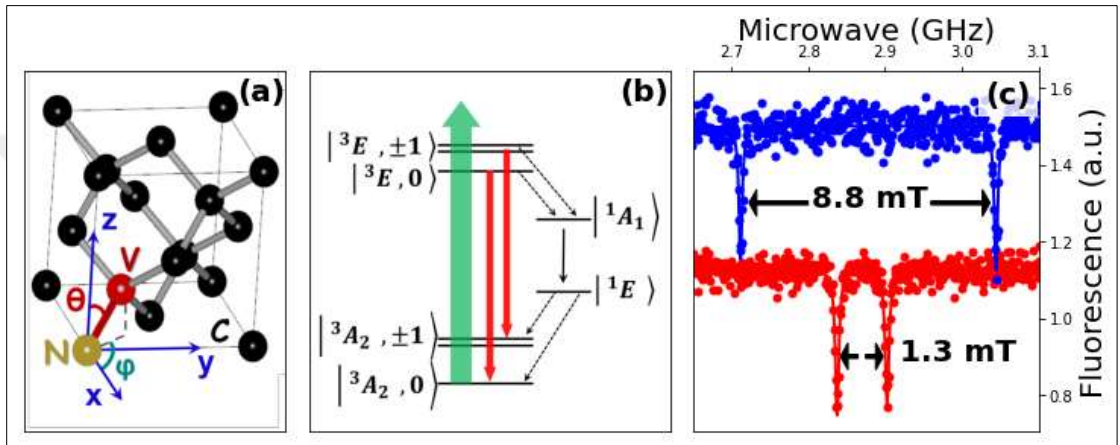


Figure 6.1. Nitrogen vacancy centre in diamond and ODMR signal. **a.** The schematic of a nitrogen-vacancy centre where the N (yellow) and V (red) mark the Nitrogen atom and the vacancy respectively. **b.** Energy level structure of the NV centre. $^3A_2 (\pm 1, 0)$ ground-state triplet and $^3E (\pm 1, 0)$ excited-state triplet and $^1A_1$, ^1E are the intermediate state singlets. The green arrow indicates the excitation of the ground states with $\lambda_{exc} = 532\text{ nm}$ green light and red arrows indicate the relaxation $\lambda_{relax} = 637\text{ nm}$ transitions. **c.** Typical ODMR signals for different external magnetic fields.

The triplet ground states of the NV^- are $m_s = \pm 1$ and $m_s = 0$. The split between the ± 1 and 0 states is 2.87 GHz due to the crystal field. This splitting can be tuned by an external magnetic field due to the Zeeman effect [103]. Thanks to the photoluminescence property of the NV centres described above, this splitting can be observed optically.

The NV^- is one of the many colour centres (> 100) [103] however, the only one whose spin resonance has been detected is the fluorescence. The $m_s = 0$ states is polarized by the optical excitation and the population transferred to the “dark”

($m_s = \pm 1$) states by a resonant microwave. When the resonance condition is met, the transfer of the population results in a measurable decrease in the fluorescence (Figure 6.1c). The resonance frequency read-out can be then used to derive the magnetic field strength and the angle between the field vector and the NV axis (Figure 6.2c). Therefore, if the fluorescence of a single NV⁻ can be measured, it can be used as a magnetic sensor with atomic resolution. This technique is called optically detected magnetic resonance (ODMR). The Hamiltonian of the NV centre can be described by;

$$\mathcal{H} = \mu_B g \mathbf{B} \cdot \mathbf{S} + \mathfrak{D} \left(\frac{S_z^2 - S(S+1)}{3} \right) + \mathcal{E} (S_x^2 - S_y^2) \quad (6.1)$$

where $S = 1$, μ_B is the Bohr magneton, g is the g-factor, $\mathfrak{D}$ and $\mathcal{E}$ are the zero-field splitting parameters and $\mathbf{B} = \mu_0 \mathbf{H}$ is the external magnetic field. This equation can be used to derive the resonance frequencies of the spin transitions $m_s = 0 \leftrightarrow \pm 1$. $\mathfrak{D}$ and $\mathcal{E}$ can be defined by the measurements performed in zero-field. The external magnetic field can be described by its magnitude, the polar (θ) and azimuthal (φ) angles relative to the NV axis. These components can be obtained from the ODMR data as a solution of equation (6.1)

$$x^3 - \left(\frac{\mathfrak{D}^2}{3} + \mathcal{E}^2 + b^2 \right) x - \frac{b^2}{2} (\mathfrak{D} \cos(2\theta) + 2\mathcal{E} \cos(2\varphi) \sin^2 \theta) - \frac{\mathfrak{D}}{6} (4\mathcal{E}^2 + b^2) + \frac{2\mathfrak{D}^3}{27} = 0 \quad (6.2)$$

where $b = \mu_B g B$. By definition, the energy $S_z = 0$ state is x_0 , thus the positions of $S_z = \pm 1$ states are given by $x_{\pm} = x_0 + v_{0\pm}$. The $v_{0\pm}$ are the frequencies of the $m_s = 0 \leftrightarrow \pm 1$ spin transitions measured experimentally [104].

6.2. Experimental Techniques

In this study, the magnetic field of an MFM tip was profiled by using a single NV centre. Afterwards, the same tip is used to record MFM data from the well-known [Pt/Co]₁₀₀ reference sample. The SPM head used in the measurements was a Bruker® JPK

NanoWizard. The MFM tip used in these measurements and calibrations, different from that used in the studies reported above and was a low moment MFM tip from TipsNano. All the experiments were performed without any external field and at room temperature.

Two consecutive cycles of NV and MFM measurements were performed and both delivered comparable results indicating good stability of the tip under the given measurement conditions.

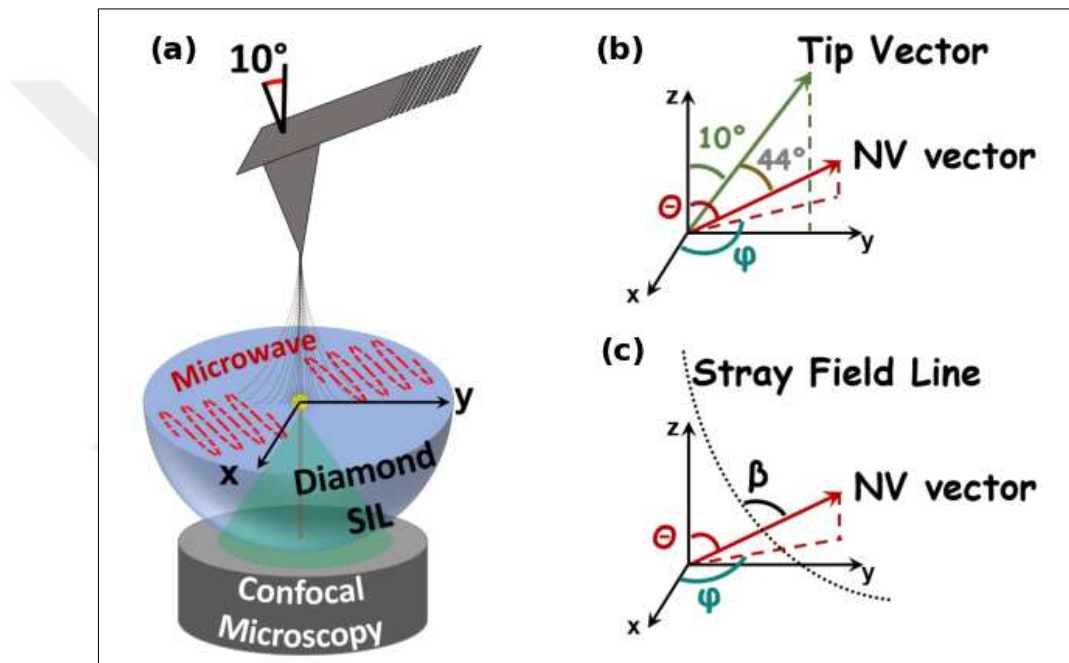


Figure 6.2. NV-Magnetometry Setup. **a.** Tip stray field measurement by single NV centre. The ODMR signal was measured by using confocal microscopy for every position of the tip over the NV centre. The SPM feedback channel was integrated with confocal microscopy to detect the NV position. **b.**, **c.** The coordinate system of the measurement where the NV vector relative to the tip vector (**b**) and relative to the stray field lines (**c**) was shown. The β is the angle between the stray field line and NV vector at a certain position. It is used to define θ , the angle between the tip vector and the NV vector.

NV measurements are performed on an SPM with a confocal microscope with bottom optical access (Figure 6.2a). The NV hosting diamond was a solid immersion lens (SIL) in a hemispherical structure. The planar surface of the SIL was (001). The SIL was an electronic grade type IIa bulk diamond provided by Element Six, Oxford, United

Kingdom. A thin ultra-pure diamond layer was grown on the planar surface of the SIL by chemical vapour deposition. Afterwards, NV centres were formed by implanting ^{15}N with 2.5 keV . The SPM's feedback channel was connected to the single-photon avalanche diode (SPAD) to finely position the MFM tip over the NV. While the NV was excited, thus it illuminates, the SPAD is used to count the photons for each position of the MFM tip. When the tip approaches the NV, the magnetic field splits the energy levels of NV and the photoluminescence decrease dramatically. These dark (or sometimes bright) spots are used to find the position of the NV centre for scanning. One of the various implanted NV centres was selected for the measurement. The selected NV centre had a photon rate of 1.2 Mcs^{-1} and implanted 3.6 nm below the surface. The depth was derived from the proton magnetic resonance [104, 105].

6.3. Stray Field Measurement of the Tip

The MFM tip scanned over a $2 \times 2 \mu\text{m}^2$ to measure the topography. Afterwards, the tip was scanned at 80 nm away from the surface over a 20×20 grid with $100 \times 100 \text{ nm}^2$ pixel size. The ODMR signal was measured at each pixel while the tip oscillated freely at the at pixel position with 3 nm oscillation amplitude and for a duration enough to collect data. The ODMR data of each pixel was used to derive the total field $\mu_0 H$ and the angle β by using the equation (6.2). This measurement will be called 2D horizontal mapping.

The $200 \times 200 \text{ nm}^2$ the peak-maximum area was defined initially from the $\mu_0 H$ data (figure 6.3). The ODMR signal was measured in this area with different tip surface distances over a 4×4 grid. This measurement will be called a vertical field scan. The tip-surface distance varied from $60 - 200 \text{ nm}$. The lower distances cannot be measured due to the measurement limitations of the NV magnetometry. The vertical data was measured without cantilever oscillation. The zero tip-surface distance was defined by a repulsive tip-surface force of 2.66 nN .

According to the equation (6.2), the φ can be derived from the ODMR data alongside the field strength $\mu_0 H^{tip}$ and the angle θ . However, for the strain-free samples, like the SIL used in this study, $\mathcal{E}$ is neglectable thus φ cannot be derived. Therefore, the polar component of the NV axis θ was be derived from the ODMR data while the azimuthal component φ was derived from the symmetry axis of 2D mapping of the β .

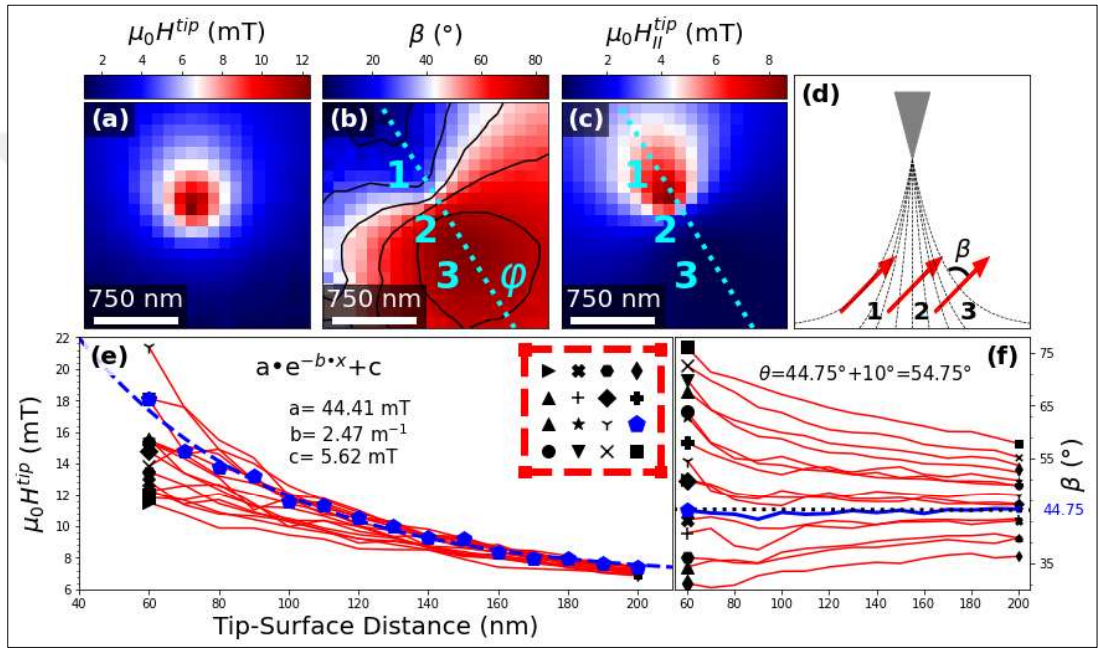


Figure 6.3. Characterization of MFM tip stray field by NV Magnetometry. 2D horizontal mapping of **a.** Tip stray field $\mu_0 H^{tip}$ and **b.** angle β of the tip stray field relative to the NV axis measured by NV centre at $z = 80 \text{ nm}$ away from the surface. The dashed line is the in-plane component of the NV axis. **c.** The stray field strength parallel to the NV axis $\mu_0 H_{\parallel}^{tip}$. **d.** Sketch of the relative angle β of the tip stray field. The numbers indicate the minimum, intermediate and maximum angles of the NV in **(b)**, **(c)** and **(d)**. the vertical scan of the stray field scan of the Tip stray field $\mu_0 H^{tip}$ **(e)** and angle β **(f)**. The vertical field scan used to derive the polar angle $\theta = 54.75^\circ$. From the interpolation (blue dashed line **(e)**) the maximum stray field at tip apex was derived 40 mT .

Figure 6.3a shows the 2D tip stray field distribution $\mu_0 H^{tip}$ with a rotation-symmetric maximum near to the NV centre and a sharp drop towards the edges. In Figure 6.3b, the 2D plot of the field angle β is shown. The dotted line marks the in-plane

projection of the NV axis. The azimuthal angle can be derived from this symmetry axis as $\varphi = 119^\circ$. As the tip stray field is rotation symmetric, the β is mirror-symmetric around φ . In Figure 6.3d, the expected variation of β along the projection axis is illustrated. The stray field lines of the point-like tip were sketched relative to the NV axis. The red arrows indicate the NV axis with a polar angle of $\theta = 54.75^\circ$. The three different positions of the tip and NV centres are marked by related numbers. At position (1), the NV axis is parallel to the stray field lines. Therefore, the minimum angle (dark blue region) in Figure 6.3b corresponds to (1). For the position (2), the tip is positioned above the NV centre. In this position, the stray field lines are perpendicular to the surface and near-linear. This result with distance independent β . Furthermore, the angle measured in this position can be used to calculate the polar angle $\theta = \beta + \theta_0$, where the $\theta_0 = 10^\circ$ is the canting angle of the tip. (3) is the position where the stray field line is nearly perpendicular to the NV axis $\beta \approx 90^\circ$. This region corresponds to the dark red area in Figure 6.3b.

The vertical scan ODMR data for each pixel and each distance was used to plot the total stray field strength of the tip $\mu_0 H^{tip}$ and the angle between the NV axis and the stray field lines β . As described above, the β is independent of tip-surface distance while the tip is positioned above the NV centre. Figure 6.3f plots the β as a function of tip-surface distance. The angle of the pixel marked with blue ($\beta = 44.75^\circ$), is nearly constant of distance indicating the position where the MFM tip is above the NV centre. The polar angle defined by this measurement as $\theta = \beta + \theta_0 = 44.75^\circ + 10^\circ = 54.75^\circ$. The polar $\theta = 54.75^\circ$ and the azimuthal $\varphi = 119^\circ$ defines the NV axis precisely. The values are in excellent agreement with the

This angle is also used to define the θ of the NV (Figure 6.2b). While the tip was aligned with the NV, $\beta \approx \theta$ will be equal to the tip vector and the NV axis. With the additive of 10° from the canting angle of the cantilever, the polar angle of the NV is found $\theta = 54.75^\circ$. These values are in excellent agreement with the angles derived from the crystal structure of the diamond.

In Figure 6.3e, the height dependence of the total stray field strength is plotted. The blue pixel which is marked for the NV position is fitted exponentially. The

extrapolation of the maximum tips stray field is derived from the fitting parameters and found 40 mT . This value is in good agreement with the estimations from qMFM calibrations for similar types of low moment MFM tips [106]. The extrapolation to $x \rightarrow \infty$ shows a background field of 6 mT . This background field can be due to the magnetic film coated on the cantilever. The scanning electron microscope (SEM) images and Energy-dispersive X-Ray (EDX) measurements of the cantilever that the whole tip-side of the cantilever was coated with the CoCr magnetic film (Figure 6.4). The manufacturer confirmed that the whole tip-side of the cantilever was coated by 20 nm CoCr film.

The Figure 6.3c shows the map of the stray field component parallel to the NV axis $\mu_0 H_{\parallel}^{tip}$. The parallel component is calculated its definition;

$$H_{\parallel}^{tip} = H^{tip} \cdot \cos \beta \quad (6.3)$$

where the H^{tip} and β is the 2D map profiles of the tip given in the Figure 6.3a and b. The same symmetry axis of the φ is also marked in this figure. The position (1) where the β goes to zero, $\cos \beta \rightarrow 1$ therefore, $\mu_0 H_{\parallel}^{tip}$ is large. The maximum value of the $\mu_0 H_{\parallel}^{tip}$ is between (1) and (2). The exact position is defined by the competition between the increase of $\mu_0 H^{tip}$ and decrease of $\cos \beta$. In the region between (2) and (3), $\mu_0 H^{tip}$, thus the $\mu_0 H_{\parallel}^{tip}$ decrease sharply.

The phase shift of the MFM data is known to be the convolution of the z components of the stray field. Therefore, the $\mu_0 H_z^{tip}$ has to be calculated from the projection of the magnetic field onto the NV axis $\mu_0 H_{\parallel}^{tip}$. For this calculation the fact that in the free space outside of the tip, where the $\nabla \times \mathbf{H} = 0$, there is a scalar potential Φ with $\mathbf{H} = -\nabla \Phi$ was exploited. Therefore, the z component of the magnetic field H_z determined all components of the field with $\mathbf{H} = (H_x, H_y, H_z)$. In partial Fourier space, $\mathbf{H}$ is described by [23]

$$H = \left(-\frac{ik_x}{k}, \frac{ik_y}{k}, 1 \right) \cdot H_z \quad (6.4)$$

Therefore, the field component $H_{\parallel}$ along NV axis described by its normalized axial-vector $\mathbf{n}$ is calculated as;

$$H_{\parallel} = \mathbf{n} \cdot \left(-\frac{ik_x}{k}, \frac{ik_y}{k}, 1 \right) \cdot H_z \quad (6.5)$$

The NV axial-vector $\mathbf{n}$ is defined by the polar ($\theta = 54.75^\circ$) and azimuthal ($\varphi = 119^\circ$) angles. Consequently, given $\mu_0 H_{\parallel}^{tip}$ and $\mathbf{n}$, the z-component of the tip stray field $\mu_0 H_z^{tip}$ can be calculated.

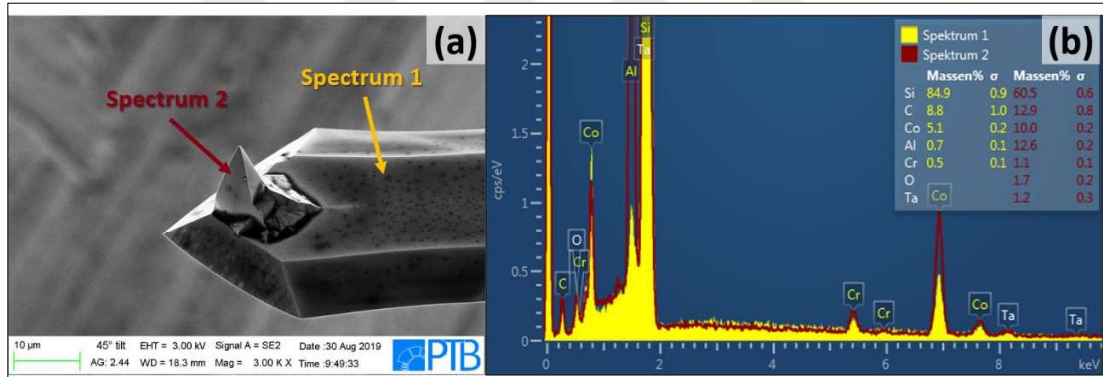


Figure 6.4. SEM images and the EDX spectrum of the calibrated tip. a. The SEM image of the low moment TipsNano tip. b. EDX spectra recorded from the tip (red) and the cantilever (yellow). The composition of both spectra is the same indicating the magnetic field covering the whole cantilever tip-side.

In Figure 6.5a the calculated z-component $\mu_0 H_z^{tip}$ of the tip stray field calculated from the NV measurement data at $z = 80 \text{ nm}$ away from the tip apex (on the diamond surface) is given. The stray field is maximum at the coordinate where the tip was situated above the NV centre and shows a rotational symmetry as expected. However, on the edges of the $\mu_0 H_z^{tip}$ there is a sharp decrease in the intensity. This effect is an artefact

due to the lack of information in the partial Fourier space such as lack of long-range components and low resolution. This effect is studied by forward simulating artificial NV data.

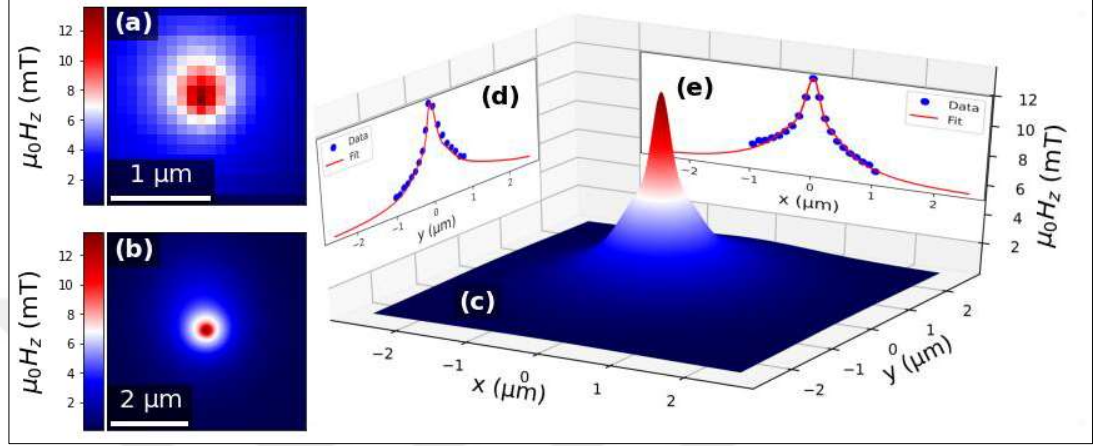


Figure 6.5. Quantum calibrated tip stray field distribution. **a.** 2D map of the z -component of the tip stray field $\mu_0 H_z^{tip}$ derived from the NV data. **b., c.** Quantum calibrated tip stray field $\mu_0 H_z^{tip,NV}$ extrapolated from the data fits of **(a)**. Data fits are performed along x - **(e)** and y -direction **(d)** through the maximum of the $\mu_0 H_z^{tip}$. **d, e** The fits are used to extrapolate the NV measured stray field to an area of $5.11 \times 5.11 \mu m^2$ to consider the longer-range magnetic interactions between the tip and sample.

Figure 6.5d, e shows the linear section of the $\mu_0 H_z^{tip}$ (blue dots) along x and y direction through the maximum of the distribution. The blue dots were fitted in both directions by using a Voigt + Gaussian distribution where the centres of the peaks are not in the same position $(x_0^G, y_0^G) \neq (x_1^G, y_1^G)$. The fit was used to extrapolate the stray field value outside the $2 \times 2 \mu m^2$ scanning window to consider the long-range magnetic interactions. Figure 6.5b, e shows a maximum of 14 mT and a slight distortion of the rotational symmetry. The distortion of the symmetry is mostly resulting from the tilt of the cantilever. The z -component of the tip stray field $\mu_0 H_z^{tip,NV}$ at $z = 80 \text{ nm}$ away from the tip apex (on the diamond surface) obtained from the NV measurements was compared with the one obtained from the MFM calibrations $\mu_0 H_z^{tip,MFM}$

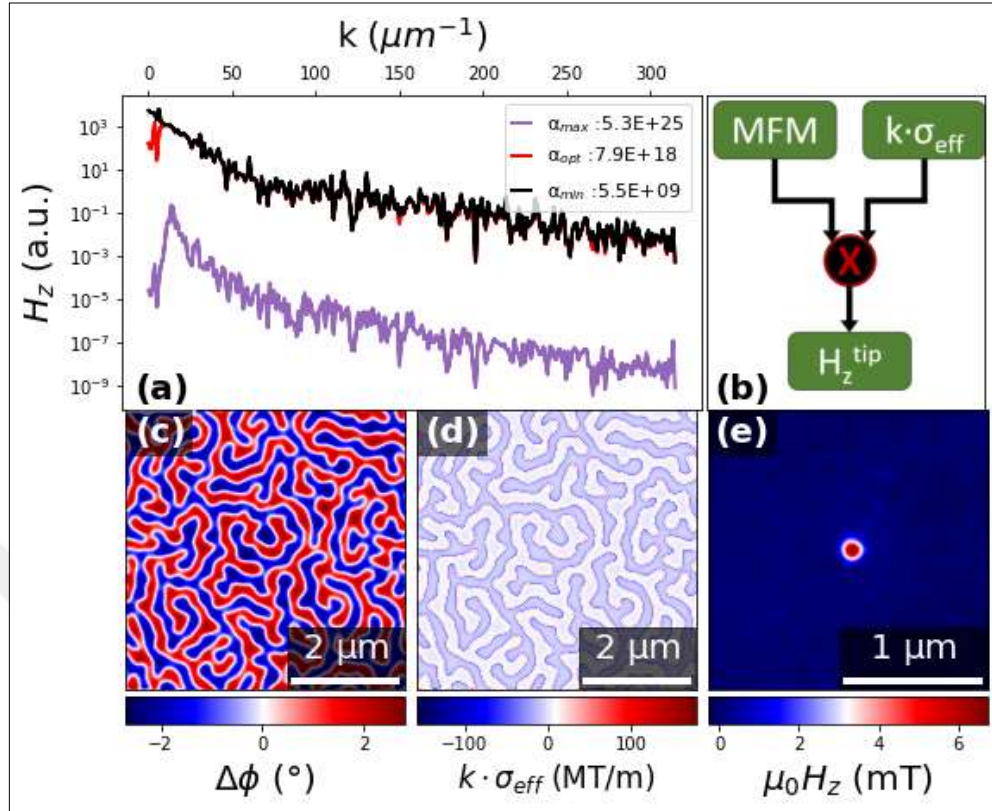


Figure 6.6. qMFM calibrated tip stray field distribution. **a.** The Fourier spectra of the deconvolved ICF by using different regularization parameters α . The α_{min} (black) and α_{max} (purple) are the limits of the L-Curve scan. α_{opt} is the optimized regularization parameter by using the L curve. **b.** The deconvolution scheme where the MFM data (**c**) and the $k \cdot \sigma_{eff}^{ref}$ (**d**) measured and calculated from the reference sample deconvolved to obtain the ICF in terms of $\mu_0 H_z^{tip,MFM}$ (**e**) at $z = 80 \text{ nm}$ away from the tip apex (on sample surface).

In Figure 6.6c The MFM measurement from the $[\text{Pt/Co}]_{100}$ samples performed from 65 nm lift height with the 15 nm setpoint amplitude is shown. The total distance between the tip and surface was $z = 65 \text{ nm} + 15 \text{ nm} = 80 \text{ nm}$ which is the same z value used in NV measurements. The free oscillation amplitude (drive amplitude dA) was 20 nm . The scan range of the MFM was $5.11 \times 5.11 \mu\text{m}^2$ with 512×512 resolution ($\delta_A = 10 \times 10 \text{ nm}^2$).

The cantilever stiffness (spring constant) was determined by using the thermal fluctuations. The free fluctuations of the tip are plotted as a function of the frequency.

The curve is fitted by using the harmonic oscillation function. The area of the fit can be used to calculate the total energy of the system in the resonance state. The equipartition theory can be used to derive the spring constant from the total energy;

$$E_{Ther} = \frac{1}{2} k_B T = \frac{1}{2} c \langle q^2 \rangle \quad (6.5)$$

The cantilever stiffness was measured $c = (3.159 \pm 0.442) \text{ Nm}^{-1}$. The quality factor was calculated from the resonance curve of the cantilever using the same approach described in the previous chapters and found ($Q = 160.7 \pm 5$).

The tip calibration, i.e., deconvolution of tip stray field was explained in chapter 2 and performed throughout the studies (chapter 3.2.5.1, chapter 5.3). In these chapters, the calibrations were performed by deconvolving the MFM phase shift data with the σ_{eff}^{cal} of the reference sample resulting in the calibration function related with the tip stray field gradient $ICF \approx \mu_0 \frac{dH_z^{tip}}{dz}$. However, the NV calibrations result in the tip stray field $\mu_0 H_z^{tip,NV}$. Therefore, in this chapter the deconvolution of the MFM data with the effective surface charge gradient of the reference sample $k \cdot \sigma_{eff}^{cal}$ (Figure 6.6d) was performed resulting in the perpendicular component of the tip stray field $\mu_0 H_z^{tip,MFM}$. The Fourier spectra of the regularized deconvolution show that the high-frequency regime is retained quite good. However, there is a loss of information in the low-frequency regime $k = 5 \mu\text{m}^{-1}$ (Figure 6.6a).

In Figure 6.7, the very first comparison out-of-plane component of the tip stray field obtained from two different approaches is shown; a direct quantitative measurement (NV) and a mathematical deconvolution (MFM). The $\mu_0 H_z^{tip,NV}$ was obtained by extrapolating the $\mu_0 H_z^{tip}$ measured by the NV centre. The $\mu_0 H_z^{tip,MFM}$ was obtained from the classical calibration based on the calculation of the magnetization of a well-known reference sample. The results show a significant difference in terms of shape and intensity. The NV measured tip stray field has twice larger maximum field strength compared to the MFM calculated tip stray field. Furthermore, the $\mu_0 H_z^{tip,NV}$

decays slowly while the $\mu_0 H_z^{tip,MFM}$ decays much faster, similar to a gaussian distribution. This indicates that the whole information of the MFM tip could not be extracted with the calibration. The information extracted dependence on the spatial frequencies of the sample will be discussed by forward simulations.

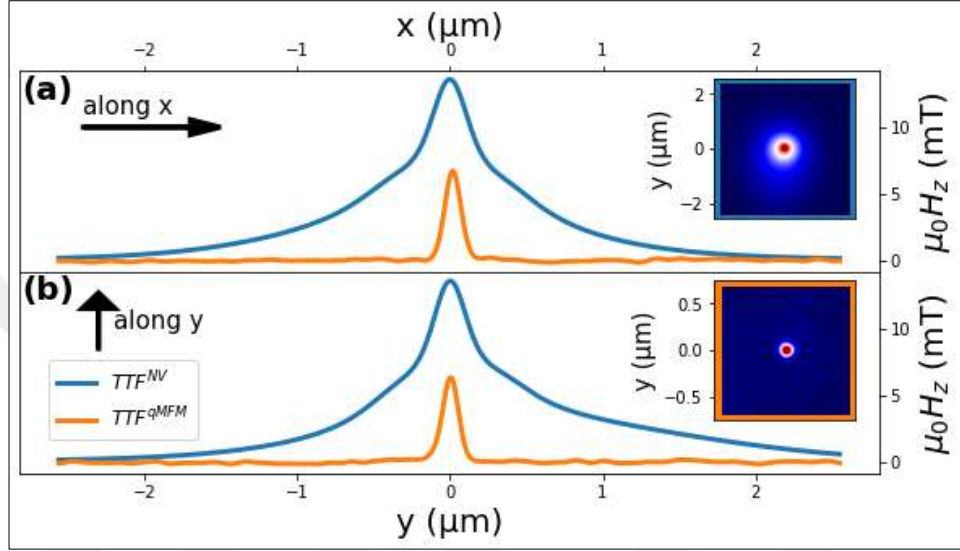


Figure 6.7. Comparison of the tip stray fields from NV and MFM. The plotlines of the NV measured tip stray field $\mu_0 H_z^{tip,NV}$ (blue) and MFM deconvolved tips stray field $\mu_0 H_z^{tip,MFM}$ (orange) at $z = 80 \text{ nm}$ away from the tip apex along x- (a) and y-directions (b). The inset figures of (a) and (b) are the 2D distribution of the $\mu_0 H_z^{tip,NV}$ and $\mu_0 H_z^{tip,MFM}$.

6.4. Validation of Quantitative MFM

Using the NV magnetometry perpendicular component of the MFM tips stray field was measured for the first time by an independent technique. The results and the discrepancies in MFM and NV measurements were discussed by forward simulating both MFM and NV data.

6.4.1. Forward Simulations of NV Data

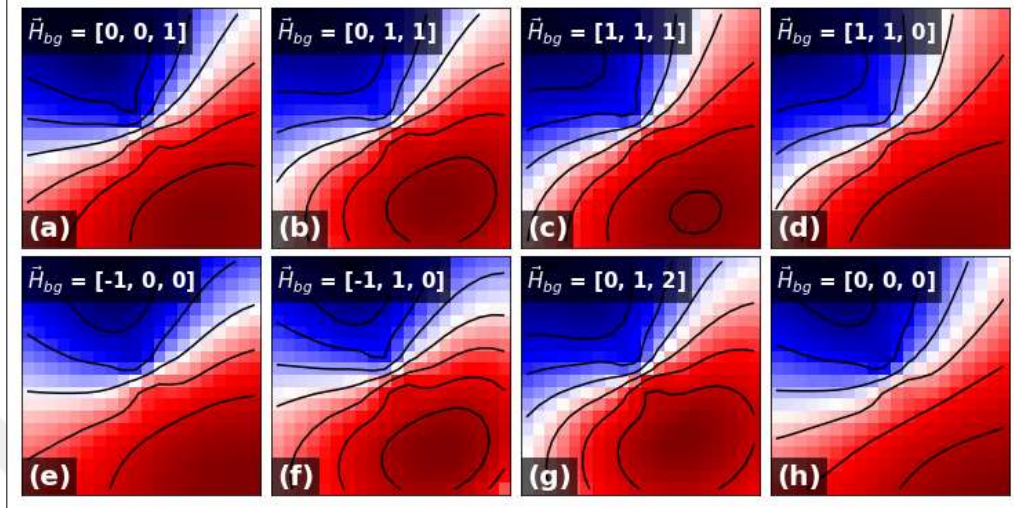


Figure 6.8. Forward simulations of NV-Field angle β for different background fields. The simulations run by using different background field vectors with different orientation $[0,0,1]$, $[0,1,1]$, $[1,1,1]$, $[1,1,0]$, $[-1,0,0]$, $[-1,1,0]$, $[0,1,2]$, $[0,0,0]$ for **a**, **b**, **c**, **d**, **e**, **f**, **g**, and **h** respectively. The closed loop was not observed when the background field was zero in **(h)**. The most similar background β distribution was for **(b)**, **(c)**, **(f)** and **(g)**. The simulations ran over the same scan range and resolution with the NV measurements ($2 \times 2 \mu m^2$, $\delta_{x,y} = 10 nm$).

The stray field profiles obtained from the NV measurements has unexpected artefacts in the edges. Furthermore, the β along the symmetry axis was increasing after the expected minima and maxima resulting in a closed-loop in the contour image. The edge artefacts are believed to be a result of the low resolution and lack of long-range components in the Fourier spectrum. Additionally, the closed contour in the β map is thought to be caused by the background field detected in the extrapolation of exponential decay of the field (see Figure 6.3e). Considering these two effects, the NV data was forward simulated for various conditions.



the background fields vectors of $[0,1,1]$, $[1,1,1]$, $[-1,1,0]$, $[0,1,2]$. These simulations show that the closed contours are definitely caused by the background magnetic field.

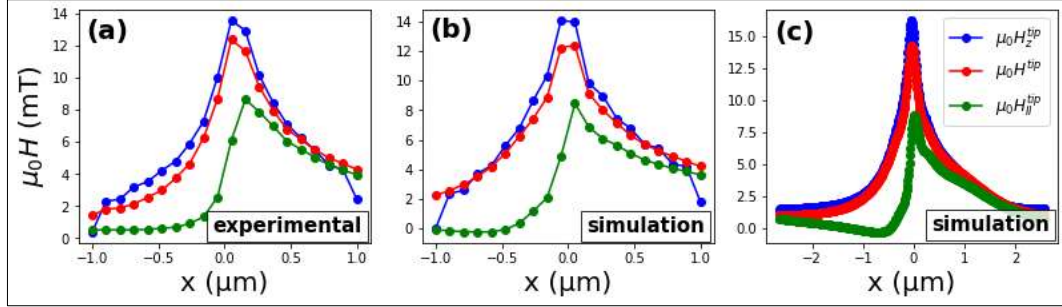


Figure 6.10. Comparison of the forward NV simulations with experimental data. The perpendicular stray field component of the tip stray field $\mu_0 H_z^{tip}$ (blue), the tip stray field $\mu_0 H^{tip}$ (red) and the projection of the stray field on the NV axis $\mu_0 H_{||}^{tip}$ (green) obtained from the experimental data (a), from the simulation with scan size and resolution of $(2 \times 2 \mu m^2 - \delta_{x,y} = 100 \text{ nm})$ (b) and from the simulation with scan size and resolution of $(5.12 \times 5.12 \mu m^2 - \delta_{x,y} = 10 \text{ nm})$. The simulation (b) and the experimental (a) data are in excellent agreement.

The next simulations were aimed at different experimental parameters such as resolution and scan size and on finding their relation with the edge artefacts observed in calculated H_z^{tip} . In Figure 6.9, the simulations with two different scan sizes; $5.12 \times 5.12 \mu m^2$ and $2 \times 2 \mu m^2$, and three different resolutions $\delta_{x,y} = 10 \text{ nm}$, 50 nm and 100 nm were presented. The background field used in the simulations was selected to be $[0,1,2]$. The scan range ($2 \times 2 \mu m^2$) and resolution of $\delta_{x,y} = 100 \text{ nm}$ is the same as the experimental parameters. The simulations show that the sharp decrease in the edges is definitely due to the scan size. A similar artefact is observed at all three ($2 \times 2 \mu m^2$) with different resolutions. In Figure 6.10, the experimentally measured and simulated $\mu_0 H^{tip}$ (red) $\mu_0 H_z^{tip}$ (blue) and $\mu_0 H_{||}^{tip}$ (green) are given. The $\mu_0 H_z^{tip}$ was calculated by using precisely the same steps described in the previous chapter. This figure emphasizes further the effect of the scan size on the accuracy of the perpendicular component of the tip stray field measured by the NV magnetometry. Furthermore, the simulations with similar conditions to the experiments are also in excellent agreement.

The forward simulations of the NV showed that the calculations performed from the measured data were accurate and can be used as the “Real Tip Stray Field Distribution”. These calculated fields will be used to validate the qMFM and furthermore, develop a new tip calibration approach.

6.4.2. Forward Simulations of MFM Data

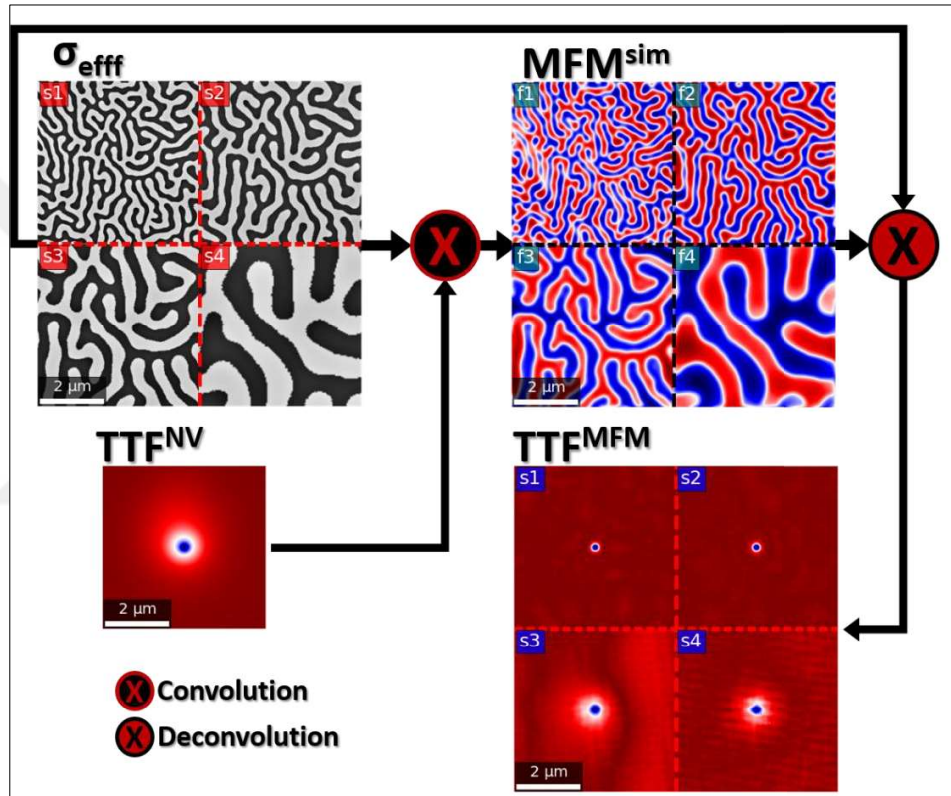


Figure 6.11. Forward simulations of MFM calibrations. The forward simulations of MFM by convolving the $k \cdot \sigma_{eff}$ with different sized domain structures and the TTF^{NV} which is the perpendicular component of the tip stray field. The simulated MFM data deconvolved back by using the $k \cdot \sigma_{eff}$ used to simulate the data to obtain back TTF^{MFM} .

In chapters 3.3 and 5.4, the accessible spatial frequencies dependence on the accuracy of the qMFM was discussed. This dependency is due to the information of the MFM tip that can be extracted from the data in calibration. Briefly, the stray field of the tip calculated by using the qMFM calibration approach will depend strongly on the spatial

frequency spectra of the calibration sample. Therefore, the difference observed in Figure 6.7 is due to the lack of necessary long-range spatial components in the reference sample. In this section, two different claims will be checked by MFM forward simulations;

- The information that could be extracted from the tip calibration depends on the calibration sample's accessible spatial frequency range.
- Even though the TTF does not have all the information related to the tip, it would be sufficient to measure the field of a target sample if calibrated from a reference sample with a similar spatial frequency.

The first claim can be checked by forward simulating MFM data by using a domain structure with a different Fourier spectrum. The direct measurement of the MFM tip by NV centre magnetometry allowed to construct the “real z-component of the tip stray field”, TTF^{NV} profile at $z = 80 \text{ nm}$ away from the tip apex. Therefore, it can be used to forward simulate MFM phase shift data.

In Figure 6.11 the forward simulation scheme is given. The MFM image from the reference measurement was discriminated and used to generate 4 different magnetization distributions with different sizes by a zooming-in central area of the image. The same domain pattern, with the increased size, is used to keep the domain shapes the same, therefore only varying parameters will be the average width of the domains. The MFM data used in the simulations is recorded from the same reference sample but as a separate measurement with the same tip. The magnetization distributions are used to calculate the $k \cdot \sigma_{eff}$ then convolved with the TTF^{NV} to generate forward simulated MFM data with a noise addition. The noise spectrum added to the convolution is the phase shift data recorded far away from the surface of a non-magnetic substrate so it is a fairly realistic. The simulated MFM phase shift was treated as a calibration measurement. The TTF^{MFM} are deconvolved from the simulated MFM data with using the $k \cdot \sigma_{eff}$.

In Figure 6.12, the deconvoluted TTF^{MFM} from different domain sizes are given with the Fourier spectra of the two inputs of each deconvolution; MFM data (red), σ_{eff} (green). The blue and the orange field distributions are the experimental results; NV

measured $\mu_0 H_z^{tip,NV}$ and MFM deconvolved (experimentally) $\mu_0 H_z^{tip,MFM}$. The (s1) simulation was the control group. The average domain width of the magnetization used in the (s1) forward simulation is the same size as the data from which the $\mu_0 H_z^{tip,MFM}$ was deconvolved. Therefore, it is expected that the TTF obtained from the experimental data and the TTF obtained from the simulated data will be similar. The TTF obtained from the (s1) simulation is in excellent agreement with the experimental TTF . This step further validates the difference observed

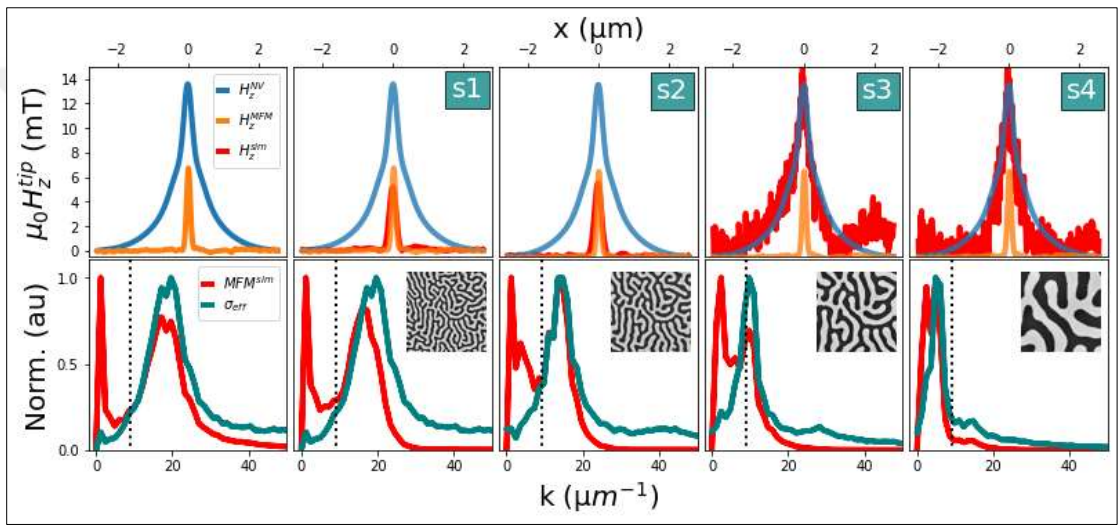


Figure 6.12. TTF dependency of spatial frequency spectra of the calibration sample. The top row is the transfer functions deconvolved from the forward simulated data (s1-4). The $\mu_0 H_z^{tip,NV}$ (blue) and $\mu_0 H_z^{tip,MFM}$ (orange) are plotted together to compare with the deconvolution outputs (red) of (s1-4). The bottom row is the Fourier spectra of the deconvolution inputs; MFM data (red) and σ_{eff} (green). The inset figures are the σ_{eff} used to simulate the corresponding MFM data. The dotted line marks the required k-vectors of the reference sample to reconstruct the tip stray field.

in Figure 6.7 where the NV measured and MFM deconvolved $\mu_0 H_z^{tip}$ were compared. The (s2) simulation results are nearly the same as the (s1). This is an expected result considering that the Fourier spectra of the domain structures are also quite similar. The TTF deconvolved from the (s3) simulation is near-perfectly fits the TTF^{NV} . This marks the spatial k-vectors (in Fourier space) required to extract all the information of the tip stray field from the calibration measurement. The deconvolution of the (s4)

simulation is also similar to the TTF^{NV} however not good as the (s3). The Fourier spectra comparison of the simulations clarifies the differences. The k-vector with the frequency of $\sim 10 \mu m^{-1}$ is the required spatial size of the reference sample to reconstruct the tip stray field profile much closer to the real distribution. The (s1) and (s2) which has similar Fourier spectra with the experimental data does not have enough component in this frequency to reconstruct accurate tip stray field. The (s4) have a significant contribution of k-vectors in this frequency, however, the dominant contribution was at lower frequency k-vectors, thus the accuracy of the field profile started to decrease.

These forward simulations prove the 1st claim; *The information that could be extracted from the tip calibration depends on the calibration sample's accessible spatial frequency range.* Furthermore, for the tip used in the experiments, the required spatial frequency was also derived for the first time quantitatively to reconstruct an accurate tip stray field profile.

The 2nd claim was that the TTF could be used for quantitative field measurements, even if it did not have all the information of the MFM tip. The prerequisite for this was that the calibration sample and the target sample had similar Fourier spectra.

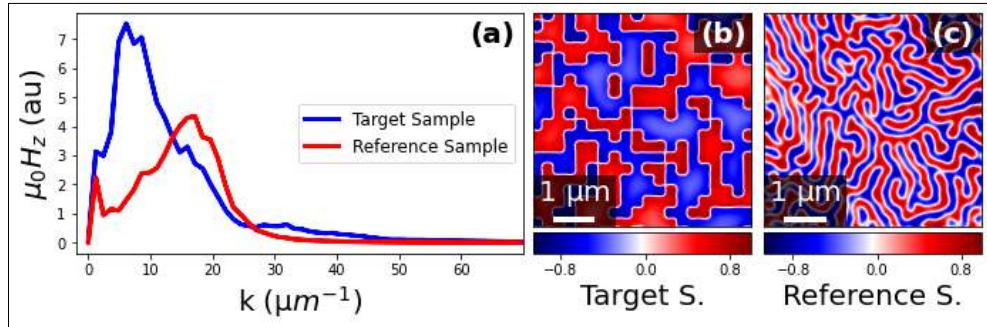


Figure 6.13. Fourier spectra comparison of the reference and target sample. a. The Fourier transform of the stray field profile $\mu_0 H_z$ of the target (b) and the reference (c) sample with normalized amplitudes.

Additional forward MFM simulations were calculated to check this claim. The target sample simulation was designed as a pattern sample measured in the saturated state. The pattern of the sample was a QR code structure and was previously used in the

literature for MFM calibrations [36]. In Figure 6.13 the Fourier spectra of the calibration sample and the target sample was compared. The image (b) and (c) used to calculate the Fourier spectra are calculated in terms of $\mu_0 H_z^{sample}$ with arbitrary sample parameters. Even though the spectra of both samples do not overlap, the dominant components were in similar frequencies. Using the QR structure given in Figure 6.14, the σ_{eff} was calculated, then the $k \cdot \sigma_{eff}$ was convolved with the $TTF^{NV} = \mu_0 H_z^{tip,NV}$ resulting in the forward simulated MFM phase shift data of the target sample.

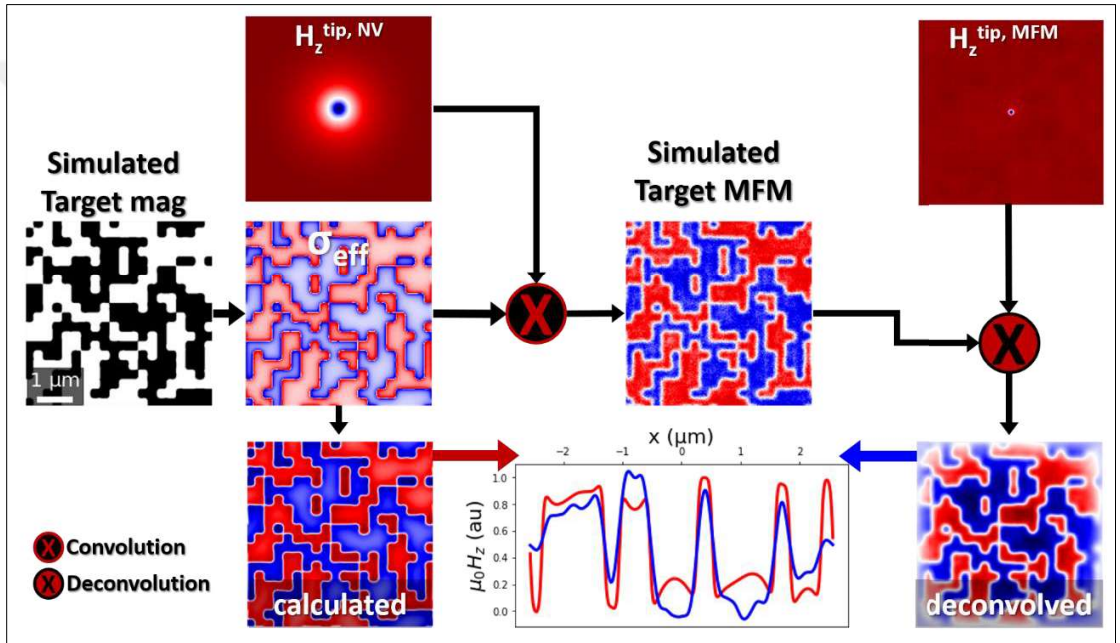


Figure 6.14. Validation of qMFM by forward simulation of the target sample. A QR-code structured patterned sample was forward simulated in the saturated state. The MFM simulations were calculated by using the NV measured tip stray field $\mu_0 H_z^{tip,NV}$. The stray field of the target sample was deconvolved from the simulated MFM data by using the transfer function obtained from the experimental calibration measurement $\mu_0 H_z^{tip,MFM}$. The deconvolved stray field and the calculated stray field are in good agreement validating the qMFM. The field values are changing from 0 to 1 indicating the saturated state of a patterned sample.

The simulated MFM data is deconvolved by using the $TTF^{MFM} = \mu_0 H_z^{tip,MFM}$. Briefly, a target sample's MFM data was simulated by using the $\mu_0 H_z^{tip,NV}$ then deconvolved back to obtain the field distribution back by using the $\mu_0 H_z^{tip,MFM}$ where

the $\mu_0 H_z^{tip,MFM}$ does not have all the information of the MFM (see Figure 6.7). The field deconvolution of the simulated target sample is in good agreement with the calculated stray field. The fade-out area around the deconvolved field is due to the extra windowing function. Especially for this deconvolution, the inputs are deconvolved with the Welch function discussed in chapter 2.3.4. The use of function was due to the sharp edges of the patterned sample image.

The result of the simulation proves the 2nd claim. The $\mu_0 H_z^{target}$ of the target, sample could be measured even though the transfer function used in the deconvolution (TTF^{MFM}) does not have all the information of tip. However, the extracted information from the calibration was sufficient to measure the stray field of the target sample due to the similar frequency spectrum of the calibration and target samples. This study was also the very first validation of the qMFM by using an independent and direct field measurement technique. In the next section, a new method to calibrate the tip therefore to measure the quantitative stray field of a sample with perpendicular magnetization will be introduced.

6.5. Quantum Calibrated MFM

The classical tip calibration described throughout the dissertation relies on simplifying the assumptions on the magnetic tip and stray field distribution of the calibration sample. The NV-centers can also be used for quantum-based quantitative stray field measurements of the sample directly by using NV-implanted SPM tips [17, 107, 108]. However, these techniques are comparably slow and do not allow quantitative measurement in the relevant field range of many nanomagnetic materials of few tens to a few hundred mT. In this section, a new approach to calibrating the tip is proposed where the transfer function is not derived from an MFM measurement, instead of measured directly by the NV-Magnetometry. Therefore, the TTF measured by NV magnetometry will open the road towards quantum calibration of virtually any MFM. The new approach is called “Quantum calibrated Magnetic Force Microscopy -QuMFM”

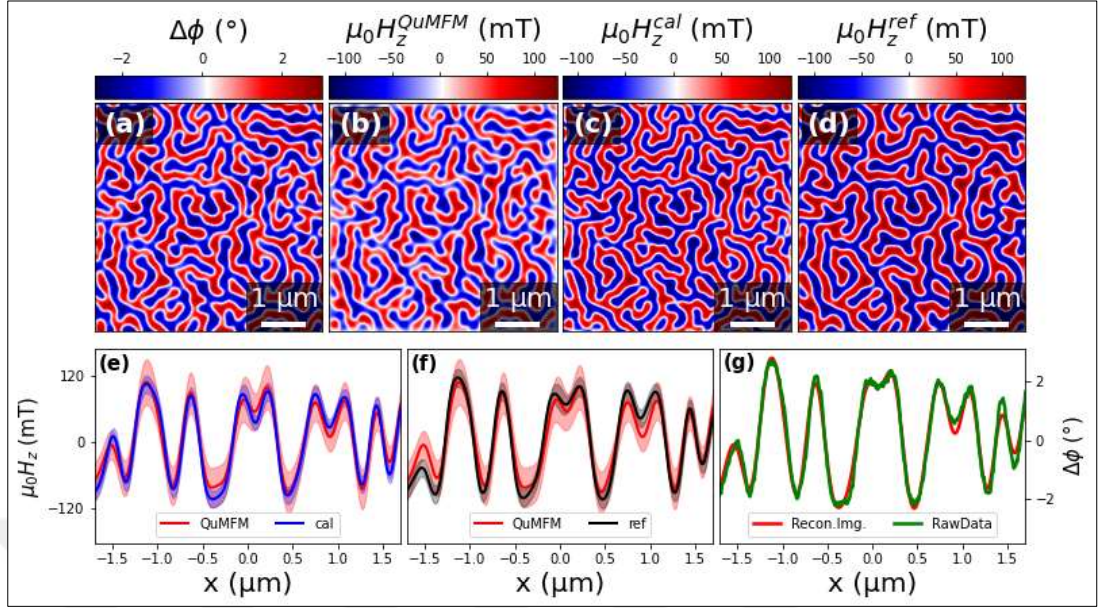


Figure 6.15. Quantum calibrated MFM. **a.** MFM data of the $[\text{Pt/Co}]_{100}$ reference sample. **b.** Quantum calibrated $\mu_0 H_z^{\text{QuMFM}}$ $z = 80 \text{ nm}$ above the surface, obtained from deconvolution of the MFM data with the instrument calibration function based on the quantum calibrated $\mu_0 H_z^{\text{tip,NV}}$. **c.** Calculated $\mu_0 H_z^{\text{cal}}$ based on the discriminated domain pattern after QuMFM deconvolution and the measured macroscopic material parameters. **d.** Classically calculated $\mu_0 H_z^{\text{ref}}$ of the reference sample based on discrimination of the MFM data of **(a)** and the measured macroscopic material parameters. **e.** Comparison of $\mu_0 H_z^{\text{QuMFM}}$ (**b**, red) and $\mu_0 H_z^{\text{cal}}$ (**c**, blue). The data agree well within the uncertainty. **f.** Comparison of $\mu_0 H_z^{\text{QuMFM}}$ (**b**, red) and $\mu_0 H_z^{\text{ref}}$ (**d**, black). The data again agree within the uncertainty bands. **g.** Test of data reconstruction errors due to noise filtering in the deconvolution process. The raw data and the reconstructed filtered data show a good agreement.

The sample used in QuMFM measurement was the $[\text{Pt/Co}]_{100}$ reference sample. All the data used to validate this approach was the same data used in the previous chapter (6.3-4). The QuMFM measured stray field was basically calculated by deconvolution of the phase shift data (Figure 6.15a) with the $TTF^{\text{NV}} = \mu_0 H_z^{\text{tip,NV}}$, measured in chapter 6.3, resulting in the σ_{eff} of the sample. The σ_{eff} was used to calculate the stray field of the sample $z = 80 \text{ nm}$ above the surface. The deconvolution of the very first quantum calibrated stray field distribution $\mu_0 H_z^{\text{QuMFM}}$ (Figure 6.15b) was regularized by checking the Fourier spectrum so that the noise was filtered without cutting-off contributions

from the stray field modulations. Furthermore, the regularization parameter and the deconvolution were controlled by re-convolving back the deconvolved image. Figure 6.15g shows the re-convolved Wiener filtered MFM data (red) with the raw MFM data (green). Both data agree well but a small information loss due to the filtering.

Figure 6.15c shows the expected calculated stray field $\mu_0 H_z^{cal}$ at $z = 80 \text{ nm}$. The calculation of the field was performed by using the same steps described in “chapter 2.3.3. Calculation of effective magnetic surface charge density”. However, there was an important difference. The reduced magnetization was not obtained from discriminating the MFM data, instead of discriminating the QuMFM deconvolution output. The $\mu_0 H_z^{ref}$ in Figure 6.15d was calculated by using the standard field calculation described in chapter 2.3.3 used in the classical calibration of the MFM tip.

Figure 6.15e compares the line plots through b and c. The $\mu_0 H_z^{QuMFM}$ and $\mu_0 H_z^{cal}$ agrees well within the uncertainty bands thereby validating the QuMFM. The uncertainty of the $\mu_0 H_z^{QuMFM}$ is calculated around $u_{tot} \approx 20\%$ for the fields around the 100 mT. The uncertainties are calculated by using the same algorithm described in chapter 5.2. One of the downsides of this technique is that the instrumental factors do not cancel out in the field measurements as long as only one MFM measurement is performed. $u_{tot} \approx 20\%$ is dominated by the uncertainty of the cantilever stiffness ($u_c = 14\%$) and Q quality factor ($u_Q = 3\%$). In Figure 6.15f, the $\mu_0 H_z^{QuMFM}$ (red) was compared with $\mu_0 H_z^{ref}$ (blue) where the $\mu_0 H_z^{ref}$ is the input for classical qMFM calibrations. The agreement of the data is within the uncertainty bands. This comparison is the first independent validation of the calculation of the reference sample’s stray field.

The qMFM reference magnetization pattern is derived from the MFM image without considering the unknown broadening of the magnetic structures by the tip [36]. This results in the suppression of narrow stray field features thus an overestimation of the field amplitude of wider ones. The examples of these systematic errors can be seen in the 2D field profiles are given in Figure 6.15b, c and d. Furthermore, the overestimation become apparent in Figure 6.15f, where the absolute field values of the H_z^{ref} generally exceed the ones measured by H_z^{QuMFM} .

6.6. Discussion

The quantum-based quantitative magnetic field measurements were used to measure to 2D profile to the tip's stray field which led to reconstruction of its z-component. The calculations of the $\mu_0 H_z^{tip,NV}$ were performed in the partial Fourier space by using the field reflected of the NV-axis and the NV-vector. Due to the lack of the insufficient scanning range which cause a non-saturating field, the calculated $\mu_0 H_z^{tip,NV}$ has sharp intensity losses in the image borders. Furthermore, the background field in the measurements result in a closed contour in the β profile due. These situations were studied by forward simulating the field of an MFM tip on a NV center where the orientation of the NV and the field strength and the width of the MFM tip's stray field was derived from the experimental data.

The $\mu_0 H_z^{tip,NV}$ was compared with the distribution calculated by the deconvolution of the MFM data of the well-known reference sample ($\mu_0 H_z^{tip,MFM}$). In the 2D stray field comparison, it was observed that the tip's stray field obtained from NV measurements was much more different in terms of amplitude and width than that obtained from MFM measurements. These differences were studied by forward MFM simulations calculated using $\mu_0 H_z^{tip,NV}$. The results of the simulations showed that these differences were strongly related to the intensity of the low frequency k-vectors of the calibration sample. The spatial k-vectors required in order to measure the stray field distribution of the MFM Tip used in the measurement with the transfer function approach were determined quantitatively for the first time. Further forward simulations show that quantitative field measurements are valid if the calibration and target samples have similar spatial Fourier components, even when the transfer function differs from the actual stray field distribution of the tip.

The NV measurements were also used as calibration measurements with calculating an instrumental transfer function (*ICF*) from $\mu_0 H_z^{tip,NV}$. This calibration was independent of the Fourier spectra of any reference sample and assumption on tips. However, the dependence on instrumental parameters such as cantilever stiffness c ,

quality factor Q is much higher. The deconvolution of this first ever Quantum calibrated tip transfer function with the MFM data is used to measure the stray field of the well-known Pt/Co sample. The measurement result was compared by the simulated stray field profiles calculated by using the surface effective magnetic charges of the sample. This comparison showed that the quantum calibrated magnetic force microscopy (QuMFM) is also sensitive to the narrow magnetic features in the sample which were overestimated due to the tip's stray field. The overestimated domains were reconstructed by calculating the stray field from the discrimination of the QuMFM output instead of the discriminated image of the MFM data. This result led to an alternative approach to calculating the stray area of the reference field. The uncertainty bands in the QuMFM calibrated stray field were slightly higher than the classical approach. This was due to the relatively high uncertainties in the instrumental parameters which can be improved dramatically.

7. CONCLUSION

In this dissertation, it was aimed to improve the capability of quantitative magnetic field measurements of nanostructures. In order to do so, a new reference sample was introduced to the literature. In addition, the validity of the quantitative MFM approach was studied by using an independent measurement technique. Furthermore, a novel technique for MFM tip calibration with quantum traceability was introduced for the first time.

The reference sample proposed was a Ti/Pt/Co/Pt multilayer stack prepared in 20 repetition on a naturally oxidized Si(111) substrate. The structural effect of Ti was investigated in single-stack (1 repetition) and found that the Ti layer promotes the fibre textured growth of Pt in (111) direction. The thickness of the Ti seed layer was 0.3 nm allowing to prepare the whole multilayer stack onto substrate directly without any additional underlayers. This thickness is one of the thinnest seed layers proposed in order to promote PMA in Pt/Co multilayers. The repetition of Ti layer at each stack allowed the magnetic properties of the multilayer to be controlled. Thus, a multi-stack could be prepared with desired domain structure and stray field strength for the MFM tip calibrations.

The new reference sample, N5R20; [Ti (0.3 nm) / Pt (1.5 nm) / Co (0.5 nm) / Pt (1.5 nm)]₂₀ was discussed in details and validated to be a suitable reference sample for transfer function-based tip calibrations. It's accessible spatial frequency range between $> 5.12 \mu m$ and 100 nm and its stray field strength $\pm 60 mT$, 64 nm above from the surface makes this sample a good candidate from the calibration of the low coercivity tips. This new reference sample is one of the very few reference samples in the literature used to calibrate the magnetic tip for quantitative field measurements with the transfer function approach. The high control over the fabrication process guarantees the availability of the sample as a reference sample. Furthermore, the inherent strip domain structure of the sample provides a broad Fourier spectrum with sufficient intensity. Even though the artificially patterned samples have excellent control over the spectral range,

the defects and artefacts attributed to the patterning make the control over the magnetization and topography much harder.

The quantitative magnetic force microscopy is a well-known approach for quantitative field measurements and has been validated by round-robin comparison of four different laboratories measurements. The measurements were performed from the same reference and target samples [36]. However, an independent validation of qMFM has been never done. Quantum-based quantitative magnetic field measurements using NV-centers were used to validate qMFM. This was the first independent validation of qMFM in terms of reference field calculation (section 6.5) and target field measurement by calibrated deconvolution (section 6.4). Furthermore, using the NV-center based quantitative magnetic field measurements, the required spatial k-vectors in the reference sample for measuring the stray field of the tip by qMFM calibration was defined quantitatively for the first time. Most importantly, NV-Center measurements of the MFM tip for the first time delivered a quantum calibrated $\mu_0 H_z^{tip,NV}$. The transfer function based quantitative field measurement calibrated by NV-center magnetometry is called quantum calibrated magnetic force microscopy (QuMFM).

This novel approach – QuMFM has several advantages over the classical qMFM. It is quantum traceable and it is not restricted by the reference sample properties as the classical calibrations are only reliable in the range of spatial frequencies of the reference sample's magnetic features. Larger and smaller structures cannot be measured quantitatively. However, in QuMFM, NV measurements of the tip stray field can be performed over high-density spatial grids of over large areas below the tip apex for quantifying the large-k and low-k contributions of the $\mu_0 H_z^{tip,NV}$. Thus, QuMFM calibrated tip can be used to measure magnetic stray field landscapes showing any length scale, also combining nanometer and micrometre feature sizes. The smoothness of the tip's $\mu_0 H_z^{tip}$ profile even allows using an adapted mesh in NV measurements, which renders such multiscale calibrations feasible. This will in the future enable quantitative studies of magnetic multiscale phenomena, e.g. interaction domains in nanocrystalline permanent magnet samples [109], which cannot be analyzed quantitatively, so far. The

uncertainties of the QuMFM can be improved dramatically. Especially, the cantilever stiffness can be measured by an uncertainty $< 1\%$ by proper calibration [110]. The vacuum MFM will have orders of magnitude higher Q factor which will result in a significantly reduced noise. Therefore, 1% or fewer uncertainties are predicted for the future.

Another advantage of QuMFM is the calibration of the reference samples. The NV measurements of the MFM tip demonstrated in this chapter is time-consuming and requires high-end expensive setups. Here one of the most promising routes to quantum traceable MFM measurement is to provide the quantum traceably calibrated transfer standards. Figure 6.15b also represents the first quantum traceable calibration of the stray field of a Pt/Co reference sample for qMFM calibrations. This will turn the reference sample based qMFM calibrations into quantum traceably calibrated stray field measurements. Furthermore, QuMFM will allow to development of new types of reference samples with different properties to specific applications and field ranges since the calibration is no longer dictated by the hard requirement of the calculable reference sample.

In conclusion, the reference sample presented as a result of the study is suitable for commercialization by obtaining patents and will probably be a valuable tool to the research and technology for quantitative field measurements of nanostructures. Furthermore, the validation of qMFM is critical for reliable metrological field measurements and fills an important gap in the literature. The novel calibration approach, which combines quantum-based field measurements with classical calibration, both increases the sensitivity of qMFM and provides the opportunity for quantum traceable field measurements for any MFM system.

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