MAGNETIC FORCE MICROSCOPY WITH DUAL-TIPS

BACHELOR’S THESIS

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MAGNETIC FORCE MICROSCOPY WITH DUAL-TIPS

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ABSTRACT

High-resolution characterization of nanomagnetic elements was exploited by a novel method. Dual-Tip Magnetic Force Microscopy is a two-pass scanning technique that employs dual-tips - one magnetic and one non-magnetic - to independently explore topography and stray field of soft magnetic structures without disturbing its state. Silicon Nitride probes were tailored by Focused Ion Beam and coated with Permalloy by Magnetron Sputtering. Scanning Electron Microscopy, Vibrating Sample Magnetometry, and Atomic Force Microscopy were employed for morphological, magnetic and in-operand characterization of the tips.

Flawless silicon nitride probes generated high-quality tips of equal or superior performance than commercial MESP probes. Cobalt Nanohills were analyzed with a conventional tip and a dual tip. The first presented a higher spatial resolution than the Scanning Electron Microscopy image, whereas the second presented a mixed topographic and magnetic signals due to manufacturing defects. Permalloy nanorings were characterized with conventional and dual tips. The topographic image revealed a flat ring with rounded particles on the surface for all tips. The magnetic signal obtained with the conventional tips indicated an uneven magnetization between opposite sides of the ring, but revealed little information about the stray fields in it. On the other hand, the dual tips fully characterized a remanent vortex state, with uneven magnetization between opposite sides of the ring and with higher magnetization on the edges of the ring.

Further investigations include the fabrication of conventional and dual tips and the implementation of Magnetic Force Microscopy measurements with variable external magnetic field.

Keywords: Magnetic Force Microscopy. Dual-Tip. Nanostructures.
RESUMO

Caracterização de elementos nanomagnéticos em alta resolução foi explorado por um novo método. A Microscopia de Força Magnética de Dupla Ponta é uma técnica de varredura em duas passagens que emprega pontas duplas - uma magnética e outra não magnética - para explorar de forma independente a topografia e o campo desmagnetizante de estruturas magnéticas macias sem perturbar seu estado. Sondas de nítreto de silício foram adaptadas por Feixe de Ion Focalizado e revestidas com Permalloy por Magnetron Sputtering. Microscopia Eletrônica de Varredura (MEV), Magnetometria Vibracional e Microscopia de Força Atômica foram empregadas para a caracterização morfológica, magnética e in-operando das pontas.

Sondas de nítreto de silício sem falhas tornaram-se pontas de alta qualidade com desempenho igual ou superior ao das sondas comerciais MESP. Nanohills de cobalto foram analisados com pontas duais e convencionais. Essas apresentaram melhor resolução espacial que imagens de Microscopia Eletrônica de Varredura, enquanto aquelas apresentaram sinais magnéticos e topográficos misturados devido a defeitos de fabricação. Nanoanéis de permalloy foram analisados por pontas convencionais e duais. A imagem topográfica revelou que a superfície dos anéis é plana e recoberta com partículas arredondadas. O sinal magnético obtido pela pontas convencionais indicou uma magnetização desigual entre lados opostos do anel, mas não revelou informações quanto ao campo desmagnetizante da superfície do anel. Já as pontas duais evidenciaram que a amostra está no estado de vórtice remanente, com uma magnetização desigual entre lados opostos do anel e com uma maior magnetização nas bordas do anel.

As perspectivas desse trabalho incluem a fabricação de pontas convencionais e duais e a implementação de um sistema para realizar Microscopia de Força Magnética com campo magnético externo variável.

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1 Introduction

In recent years, nanotechnology has arisen as a source of innovative solutions to major global challenges. Such demanding task requires the ability to understand and control matter which at least one physical dimension is below 100 nm. At this scale, system components present new functionalities and properties, enabling novel applications across all fields of technology.

First, the size scale of nanomaterials allows full integration with biological structures, boosting the medical field. Nanomedicine has emerged bringing remarkable progress to diagnostic devices, contrast agents, analytical tools, physical therapy applications, and drug delivery vehicles (NNI, 2016; MOGHIMI; HUNTER; MURRAY, 2005).

Second, the high surface-to-volume ratio of nanostructures enhances chemical reactivity. This effect can leverage renewable energy production, contribute to sustainable energy supply, and increase efficiency on each part of the chain in the energy sector - from generation and distribution to storage and usage (LUTHER, 2008). Manufacturing highly affordable electric cars and solar cells is an ongoing trend of this sector.

Finally, quantum effects associated with nanomaterials also established a close relationship between nano and information technology. This long-term relation was silently introduced, such that, integrated circuits already reached a limiting critical length scale, analog electronics are currently being replaced by optoelectronic devices, memory storage technology is exploring spin-based phenomena, and quantum computing became a real possibility.

The tremendous development of nanotechnology has not just revolutionized our society, but has also profoundly impacted our economy. The U.S. National Nanotechnology Initiative’s member agencies have cumulatively spent nearly $24 billion since 2001 (NNI, 2016). The global revenue from nano-enabled products presents even more astonishing numbers and is estimated to reach U$4.4 trillion by 2018 (HARPER, 2011).

While it is evident that nanoscience is firmly entrenched in our society, the development of this field of research is relatively new. Individual atoms of electrically conductive surfaces were first manipulated only after the introduction of the Nobel-Prize winning scanning tunneling microscope (STM) by Gerd Binnig and Heinrich Rohrer in 1981 (NOBELPRIZE.ORG, 2016). Non-conductive materials just followed this trend five years later, after Binnig et al. devised the Atomic Force Microscope (AFM) (NNI, 2016). This landmark development allows
not only to manipulate, but also to characterize metals, polymers, and ceramics at a nanometer scale.

State-of-the-art AFM operates under a fundamentally similar mechanism as the original creation. A schematic view of its working principle is illustrated in Figure 1.1. The central system component is the AFM head, which consists of basically three elements: a cantilever, a laser beam, and a position sensitive photodetector (PSPD). A tip is located at the end of a cantilever, which raster scan the surface of a sample to analyze its topography. The laser beam is reflected by the back of the cantilever onto the photodetector, so that changes in the cantilever deflection corresponds to shifts in the laser position. The path length between cantilever and detector amplifies the cantilever oscillation seen by the PSPD, allowing for sub-angstrom detection of vertical movement (HOWLAND; BENATAR; INSTRUMENTS, 1997).

While the scan is performed, the probe interacts with the sample, conditioning the cantilever to bend according to the forces between the former and the surface of the sample. The cantilever behaves like a compressed spring with modified spring constant ($k$) given by (HART-MANN, 1999a; PORTHUN; ABELMANN; LODDER, 1998a):

$$k = k_0 - \frac{\partial F}{\partial z}$$  \hspace{1cm} (1.1)

where $k_0$ is the natural spring constant, $F$ is the interaction force, and $z$ is the coordinate perpendicular to the surface. An attractive interaction renders $\frac{\partial F}{\partial z}$ to be positive hence softening the modified spring.

This interaction force $F$ can be estimated by the Lennard-Jones potential ($V_{LJ}$), a simple model that describes the intermolecular potential between two non-bonding neutral atoms given by:

$$V_{LJ} = -4\epsilon \left[ \frac{\sigma^6}{r^6} - \frac{\sigma^{12}}{r^{12}} \right]$$  \hspace{1cm} (1.2)

where $\epsilon$ is the well depth, $\sigma$ is the distance at which the interaction becomes null and $r$ is the tip-to-sample distance.

The term $r^{-12}$ refers to short-range repulsive interactions that arise when an atom from the tip approaches an atom from the surface of the sample and their electronic wave functions start to overlap. According to the Pauli exclusion principle, this generates an increase in energy of one overlapping electron in order to maintain both wave functions with different quantum numbers. Besides, the proximity leads to an incomplete screening of the nuclear charge, creating Coulomb repulsion.
Figure 1.1 – Schematic diagram of basic AFM operating principles. The cantilever is bent according to interaction forces between tip and sample surface (inset) leading to shifts in the detected laser position.

The term $r^{-6}$ refers to long-range attractive interactions originated, basically, from van der Waals and capillary forces. The former derives from induced dipoles in atoms and molecules, whereas the latter derives from the condensation of water at the sample surface and on the tip during experiments under ambient conditions.

Figure 1.2 presents the interaction force with its main contributions and the distance regimes of contact, non-contact and tapping AFM modes. Contact AFM operates in the repulsive regime by gently tracing the tip a few angstroms from the sample surface. Cantilever deflection is detected by the PSPD and fed to the controller in constant-force or constant-height mode. In the first, the total force applied to the sample is kept constant by moving the scanner up or down in z-direction and the image is formed by registering the motion of the system. While this mode is robust and allows a precise tracking of the sample surface, it is limited by the response time of the feedback circuit. The constant-height scanning mode overcomes this difficulty by fixing the distance from the cantilever to the surface. In this configuration, the spatial variation of the laser reflection provides a profile of the forces on the tip, which demands high instrument stability and low surface roughness.

Non-contact AFM operates in the attractive regime by setting the cantilever to vibrate close to its resonant frequency and holding the tip tens to hundreds of angstroms from the sample surface. The attractive forces reduce the cantilever amplitude of oscillation and alter its spring constant as described by Equation 1.1, constraining these signals to reflect tip-to-
sample distance. Variations in oscillation amplitude are inferred from laser deflection; whereas variations in spring constant are indirectly measured by monitoring the resonant frequency \((w)\) and applying the square law:

\[
k_0 = mw_0^2
\]  

(1.3)

where \(m\) is the effective mass. Similarly to the constant-force mode, resonant frequency or vibrational amplitude of the cantilever is kept invariable and sample topography is revealed by the motion of the scanner.

Tapping AFM operates in a dynamic mode between repulsive and attractive regimes. Topography information is extracted as described in the non-contact mode, with the advantage of imaging surfaces with greater roughness. In addition, tapping mode does not damage the sample neither the tip like in the contact mode. The motion of the probe can be described by a steady-state solution (HARTMANN, 1999a):

\[
d(t) = d_0 + \delta \cos(wt + \phi)
\]  

(1.4)

where \(d_0\) is the probe-sample distance at zero oscillation amplitude, \(d(t)\) is the instantaneous separation. The amplitude of oscillation \((\delta)\) is given by:

\[
\delta = \frac{\delta_0 w_0^2}{\sqrt{(w^2 - w_0^2)^2 + 4\gamma^2 w^2}}
\]  

(1.5)

where \(\gamma\) is the damping factor. The cantilever’s resonant frequency can be expressed as:

\[
w = w_0 \sqrt{1 - \frac{1}{c} \frac{\delta F}{\delta z}}
\]  

(1.6)

where \(w_0\) is the natural resonant frequency.

Finally, the phase shift between the probe motion and the driven signal amounts to:

\[
\phi = \arctan \left( \frac{2\gamma w}{w^2 - w_0^2} \right)
\]  

(1.7)

Forefront AFM innovations allow discerning other components of the interaction force \(F\), correlating three-dimensional profiles of unprecedented resolution with several material properties. Local magnetic domains, static charge distribution, surface elastic moduli, and friction are just some of the features that can be investigated with slight or none adaptions of the microscope (WEBSTER; EREN, 2014).
Figure 1.2 – Interatomic potential between non-bonding neutral atoms from the tip and the samples as a function of their distance. Distance regimes of contact, non-contact and tapping AFM modes are indicated.

1.1 Magnetic Force Microscopy

Magnetic Force Microscopy (MFM) is the powerful AFM-based technique for magnetic imaging. The principles of this method were formulated in the year following the AFM invention and are based on employing a sharp tip coated with a ferromagnetic thin film instead of a conventional probe. In this case, cantilever’s deflection emerges essentially from Lennard-Jones and magnetic interactions between the tip and the surface under scrutiny, with a mixing ratio given by the tip-to-sample distance and by the intensity of the magnetostatic interaction. The intricate process of differentiating both contributions makes tapping mode unsuited for MFM applications.

Lift mode is a two-pass technique developed for dynamic magnetic imaging based on tapping and on constant-height modes. This approach benefits from the fact that, at short distances, Lennard-Jones forces are dominant and the image carries basically topographic information. As the distance is increased, only long-range magnetic coupling between probe and sample remains relevant and the probe shapes a map of this interatomic force. Thus, the two-pass method acquires data at two different distances, as illustrated in Figure 1.3. The first pass is done in tapping mode and records a height profile as shown in Figure 1.3 (a). At the end, the
Figure 1.3 – Schematic view of MFM lift mode principle. During the first scan (a), the tip traces the height profile in order to provide surface topography. In the second scan (b), the probe is lifted by a height $\Delta h$, so the tip maps the stray magnetic fields on the surface.

probe is lifted by a distance $\Delta h$ from the surface - usually 5 to 50 nanometers. The second pass is performed in non-contact mode at a constant height and without feedback, so the tip retraces the previously acquired height profile at the specified $\Delta h$ as pictured in Figure 1.3 (b).

Figure 1.4 (a) shows a diagram of how the AFM electronics module enhances the signal and provide feedback control, allowing for topographic and magnetic detection in Lift Mode. Although most microscopes use the same feedback control system, the explanation below refers to the one employed by the Bruker Multimode 8 Scanning Probe Microscope. The cantilever deflection is read by the PSPD and the AC signal is sent to an amplifier, which determines the difference in signal between the upper and lower quadrants of the photodetector. This signal is fed to a lock-in amplifier and used for topographic and magnetic detection by applying the following procedure:

- In the first pass, the RMS amplitude of such signal is used as "amplitude signal" and is fed to a Proportional-Integrator controller. The system acts on the scanner according to
Figure 1.4 – Block diagram of controller signal for MFM in phase measurement mode of the Multimode 8 Bruker System (a). Gradients in the magnetic force cause a shift ($\Delta F_0$) in the resonance frequency, which generates a phase shift ($\Delta \phi$) between the drive signal and the cantilever response.

- User-defined proportional and integral gains to decrease the difference between the actual amplitude value and the user-defined amplitude setpoint.

- In the second pass, the phase signal is defined by the phase shift ($\Delta \phi$) between the reference signal and the actual signal at the drive frequency as shown in Figure 1.4 (b).

The feedback control allows for the high-spatial resolution observed in the fine structure of individual domain boundaries - as Figure 1.3 (b) presents - and is just one of the assets that made MFM popular. This particular benefit became relevant in the 1990s as it met the criteria of the magnetic recording industry for developing high-density data storage devices. Fundamental research of magnetic materials also benefits from some other major advantages of this method (JILES, 2015):

1. Non-destructive;

2. Sample preparation is not required since long-range magnetic interactions are not sensitive to surface non-magnetic contamination;

3. Several environmental settings can be applied; from atmospheric pressure to ultra-high vacuum, low to room temperature, gaseous to under liquids conditions;

4. Electrical conductivity is not a prerequisite;

5. Sensitive to magnetic fields and forces down to $10 \mu T$ and $10^{-14} N$, respectively;
6. Spatial resolution of 40 nm.

Although MFM presents several benefits compared to other scanning techniques, it also has two major limitations. First, it is not possible to perform a quantitative analysis of the magnetic domain topology. Second, it leads to a mutual change of local magnetization of the tip-sample system.

The former shortcoming arises from two factors. The image acquired is strongly influenced by the shape and the magnetic configuration of the probe, which makes this information essential to solve the detected signal for the local magnetization of the specimen. However, it is impractical to magnetically characterize all tips with the required spatial resolution and, as a consequence, real modeling of domain configuration is not possible. Besides, tip-sample interaction is based on near-surface stray fields produced by ferromagnetic materials, not by the magnetization itself. Even if we find the exact stray field arrangement, it is not possible to reconstruct the domain topology from it.

The second drawback can be explained after careful analysis of the magnetic forces of the tip-sample system, where the probe acts as an external field on the magnetic distribution in the sample and vice-versa. The Landau free energy of a magnetic material is given by the sum of exchange, anisotropy, magnetostatic, and external field energies.

Exchange interaction is a quantum-mechanical phenomenon that gives the energy required to exchange two overlapping identical particles. For two fermions, the overall wave function must be antisymmetric, requiring the spatial component to be antisymmetric and the spin component to be symmetric or vice-versa. In the first case, the two electrons have separate orbits which lower Coulomb repulsion. As a result, the alignment of adjacent spins is energetically favorable.

Magnetocrystalline anisotropy energy arises from spin-orbit coupling of electron spins to anisotropic orbitals in the crystalline structure. The interaction is proportional to the product between the orbital and spin momenta. In the absence of magnetic field, the energetic minimum is obtained when both momenta are parallel, inducing the magnetization to lie on a specific axis.

Magnetostatic energy originates from the interaction of the magnetization with the magnetic field generated by the specimen itself and from the interaction between an applied external field and the magnetization. This external field energy can be expressed as the product of the applied field and the magnetization.

Even though the micromagnetic state of the probe-sample system cannot be defined,
contrast formation on MFM still remains valid if the domain configuration of the tip is assumed to be completely rigid, i.e. in the absence of the second limitation. This defines two conditions: a magnetostatic sample-probe regime, in which mutual change of local magnetization of the tip-sample system does not occur; and magnetodynamic sample-probe regime, in which it does.

1.1.1 Magnetostatic Sample-Probe Regime

The magnetic force (\( F_{\text{mag}} \)) acting on the tip during the second lift-mode pass is illustrated on figure 1.5 and can be estimated as the following:

\[
F_{\text{mag}} = \mu_0 \int \nabla (M_{\text{tip}} \cdot H_{\text{sample}}) dV_{\text{tip}}
\]

The integration is carried out over the magnetic volume of the tip (\( dV_{\text{tip}} \)), where \( \mu_0 \) is the vacuum permeability, \( M_{\text{tip}} \) is the tip magnetization and \( H_{\text{sample}} \) is the sample stray field. The interaction clearly depends on the entire magnetic sensitive part of the probe. Therefore, thicker coatings generate a stronger signal averaged over a larger area, worsening the spatial resolution. The sample stray field can be determined from its magnetostatic potential (\( \phi_s \)):

\[
H_{\text{sample}}(r) = -\nabla \phi_s(r)
\]

\[
\phi_s(r) = \frac{1}{4\pi} \left[ \int \frac{d^2n \cdot M_s(r')}{|r - r'|} - \int d^2r' \frac{\nabla M_s(r')}{|r - r'|} \right]
\]

where \( M_s \) is the sample magnetization, \( n \) is the outward normal vector from the sample surface.

The first integral determines the influence of surface charges due to magnetization components perpendicular to the surface, whereas the second relates to volume magnetic charges from interior divergences of the magnetization vector field.

Sample stray field can be extracted from relative variations in phase, frequency, or amplitude of cantilever oscillation driven by the magnetic interaction force. Even though phase and frequency detection require additional instrumentation, these modes are preferred for imaging since they present higher signal-to-noise ratio (PORTHUN; ABELMANN; LODDER, 1998b) (HARTMANN, 1999b).

Phase detection method monitors the phase lag between the cantilever drive signal and its output signal, which is linked to variations of local surface properties. For a 1-dimensional system, the phase shift (\( \Delta \phi \)) can be expressed as:

\[
\Delta \phi = \frac{Q}{k} \frac{\partial F}{\partial z}
\]
Figure 1.5 – MFM tip composed of a Silicon core and a magnetic coating. Variables required to estimate the sample magnetostatic potential (Equation 1.10) are indicated.

where $Q$ is the system quality factor. Typical images acquired with lift mode and phase method can be seen in Figure 1.3.

1.1.2 Magnetodynamic Sample-Probe Regime

Soft magnetic structures present low magnetocrystalline anisotropy and are characterized by requiring low external fields to null its macroscopic resultant magnetization. In these low coercivity materials, magnetostatic surpasses other contributions and becomes a key determinant of domain wall configurations. If a tip/sample presents low coercivity compared to one another, reversible or irreversible alterations are made on the magnetic arrangement of the system. Besides destroying the tip/sample magnetic distribution, this leads to an unstable fallacious magnetic image.

In order to determine the sample stray field, each dynamic component of the Landau free energy would have to be solved numerically. One less computationally expensive alternative is to use the free energy of a domain wall to help elucidate the configuration of the system. Modifications in the magnetic structure of both probe and specimen are usually associated with domain wall motion, which can be calculated using variational calculus or numerical micromagnetics methods. Nonetheless, such complex procedure was seldom explored and little is known about its accuracy.

The aim of this work is to address this limitation, implementing an alternative MFM method for imaging soft magnetic structures.
1.2 Soft Magnetic Structures

The enticing confluence of magnetism and nanoscience created its own field of research, called nanomagnetism. Emerging spin-electronics technologies, or simply spintronics, use electron spin to process and store information, rather than electron charge. Nanomagnetic elements can then be designed to operate as degenerate logical states in a thermodynamically reversible scheme. Devices based upon this architecture formed a new paradigm: computation can finally be performed close to the theoretical limit of thermodynamic efficiency, limited by the entropy of information (LAMBSON; CARLTON; BOKOR, 2011).

The first magnetic-based logic was introduced in 2002 by Allwood et. al. This all-metallic device performed logical NOT operations through domain wall propagation along wires (ALLWOOD et al., 2002). Exclusive-not-OR and not-AND gates were later introduced using spin wave interferometry to vary either phase or amplitude of spin waves (SCHNEIDER et al., 2008). Yet another concept was applied to produce magnetic quantum-dot cellular automata systems. This three-input majority logic gate was demonstrated by physically coupling induced anisotropy in arrays of nanomagnets (IMRE et al., 2006).

Such spin-based systems meet several information technologies and communications demands, including reproducibility, scalability, high-density circuitry, high performance, low-power consumption, and low cost. Although spintronics is not fully integrated with CMOS and other semiconducting technologies, these entropy-limited schemes indicate the potential of an all-magnetic processing system that transcends current technology.

Precise engineering of magnetic arrangements is essential to master these novel spintronic devices, where the key challenge is to find a high-resolution method to manipulate and control these magnetic configurations that does not perturb the system. Technological MFM innovations are a natural candidate for this task.

High-resolution MFM images can be obtained by operating sharp and high aspect ratio probes close to the surface. The ultimately achievable lateral resolution is bounded by the superparamagnetic limit of the tip, under which smaller ferromagnetic structures acquire random magnetization at room temperature. As a result, the finite size probes produce an average interaction with the stray fields in the sample, even when in contact mode.

The required tip-to-sample distance renders such method inaccurate for imaging magnetically soft structures, as spintronic devices. Figure 1.6 compares a sequence of 25 x 25
Figure 1.6 – A sequence of 25 x 25 $\mu$m$^2$ MFM images obtained on a YSmBiGaFe garnet film of 4.5 $\mu$m thickness at probe-sample separations of (a) 910 nm, (b) 520 nm, (c) 390 nm, and (d) 910 nm. The domain configuration of the film is perturbed at probe-sample separations of 520 nm and 390 nm leading the sample magnetization to a new remanent state (HARTMANN, 1999a).

The destructive influence of the probe is clearly seen in Figures (b) and (c) in which the specimen is led to an entirely new remanent state by decreasing probe-sample separation during the scanning process. Recent studies show that MFM can generate, drag, destroy or control the chirality of vortexes (MAGIERA et al., 2012; MIRONOV et al., 2007). Figure 1.7 illustrate this experiment within cobalt particles with dimensions of 400 $\times$ 600 nm. The initial state (S1) was imaged in constant height mode, presenting a clockwise vortex orientation. The scanning height was then reduced, registering a transition state (TS) - the exquisite contrast obtained between the indicated scan lines pinpoints a magnetic transition. Afterward, the tip was again lifted and a final state (S2) revealed a counterclockwise vortex orientation.

In 2015, exploring spintronic devices with high spatial resolution finally became a reality. Dual-tip MFM (DT-MFM) is a novel two-pass scanning method that preserves the configuration of soft magnetic nano-objects (PRECNER et al., 2015). This technique uses a dual-tip - a short magnetic and a long non-magnetic - with distinct resonant frequencies to independently probe and differentiate topographic from magnetic signals. During the first pass, the non-magnetic tip is set to vibrate and scans the surface in tapping mode, acquiring a height
Figure 1.7 – Manipulation of vortex chirality from an initial state (S1) to a final state (S2) through a transitional state (TS) (MIRONOV et al., 2007).

profile. During the second pass, the magnetic tip is set to vibrate and scans the specimen in non-contact mode, recording the magnetic state of the structure. Since this tip is shorter, it does not touch the sample surface and direct contact between the magnetic tip and the specimen is avoided.

Figure 1.8 compares the local magnetization of a vortex in a permalloy (Py) particle obtained by using lift-mode with commercial tip (Figure 1.8 (a)); lift-mode with dual-tip (Figure 1.8 (b)); and DT-MFM with dual-tip (Figure 1.8 (c)). In the first two cases, the vortex appears distorted probably due to close interaction between probe and sample. Whereas on the third case, the magnetic map matches the theoretical prediction.

Figure 1.8 – Vortex in a Py particle imaged by using lift-mode MFM with commercial tip (a); lift-mode MFM with dual-tip (b); and DT-MFM with dual-tip (c). The scanning height is 150 nm (PRECNER et al., 2015).
2 METHODOLOGY

The first MFM probes were made of etched iron or nickel wires (MARTIN; WICKRAMASINGHE, 1987). The high magnetic volume of these tips provided a spatial resolution of only 100 nm and significantly disturbed sample magnetization.

Current technologies allow far better resolution by employing sharper magnetic tips produced, basically, by three methods:

1. Magnetic coating sharp Si/Si$_3$N$_4$ cantilevers: magnetic thin films can be deposited on the probes through evaporation, sputtering or electrochemical deposition.

2. Placing magnetic nanowires on probe apex: carbon nanotubes with embedded magnetic nanowires, coated carbon nanotubes, cobalt/nickel nanowires or particles can be placed on a cantilever with a nanomanipulation systems or grown on the probe apex by dielectrophoresis;

3. Modifying a probe with Focused Ion Beam (FIB): magnetic particles are milled at the probe apex or deposited in it by Electron Beam Induced Deposition;

Figure 2.1 relates MFM resolution with developments of sharp magnetic tips for imaging soft and hard magnetic materials (FUTAMOTO et al., 2013). Si/Si$_3$N$_4$ tips coated by evaporation or sputtering techniques became the major trend of this business due to its excellent spatial resolution, reproducibility and mass-production technology.

In this work, MFM tips were produced following the method of magnetic coating Si$_3$N$_4$ cantilevers with permalloy (Py) through a sputtering technique. The experimental procedure comprises the determination of probe specifications, cutting and coating methods.

2.1 Probe Fabrication

2.1.1 Probe Specifications

The Si/Si$_3$N$_4$ tip model must be carefully chosen to match the experimental procedure. Cantilevers with medium spring constant - 1 to 5 N/m - corresponding to medium resonant frequency - 50 to 100 kHz - are usually applied for MFM imaging. Lower stiffness is recommended for mechanically soft materials as it improves sensitivity, but it also makes the tip
Figure 2.1 – MFM spatial resolution according to publication year for Si, Si-N base-tip (method 1); Carbon nanotube (method 2), and Focused ion beam (method 3) (FUTAMOTO et al., 2013).

more susceptible to damage. Higher resonance frequency is preferable due to its faster scan rate. Besides, polymeric surfaces become stiffer at a higher frequency, which reduces sample damage.

Material and thickness of the ferromagnetic coating are also crucial parameters on image contrast. High magnetic moment and coercivity probes are used on permanent magnets due to their robustness under high external magnetic fields. Low magnetic moment tips are employed on soft magnetic samples and provide a better resolution, but a lower signal. Such probes are typically coated with permalloy (Ni_{81}Fe_{19}) or nickel-cobalt alloys and report a coercivity of tens of Oe. High aspect ratio and low radius probes are desirable to optimize resolution; however, magnetic coating usually makes the tip blunt, increasing tip radius on standard MFM probes to 30 or 50 nm.

Table 2.1 list the characteristics of the probes used in this project according to the manufacturer. ScanAsyst-Air-Hr tip was selected for presenting higher scan rate, better sensitivity and superior resolution. Permalloy coating was preferred for its lower magnetic moment for imaging soft magnetic structures.
Table 2.1 – Specification of MFM tips used in this project.

<table>
<thead>
<tr>
<th>Model</th>
<th>Resonant Frequency (kHz)</th>
<th>Spring Constant (N.m$^{-1}$)</th>
<th>Tip Radius (nm)</th>
<th>Coating</th>
</tr>
</thead>
<tbody>
<tr>
<td>MESP</td>
<td>75</td>
<td>2.8</td>
<td>35</td>
<td>CoCr</td>
</tr>
<tr>
<td>ScanAsyst-Air-Hr$^1$</td>
<td>130</td>
<td>0.4</td>
<td>2</td>
<td>Ni$<em>{81}$Fe$</em>{19}$</td>
</tr>
</tbody>
</table>

Values before magnetic coating$^1$.

2.1.2 Probe Cutting Method

Micro- and nanoscale manufacturing technologies are vital for the development of material science. Lithography based micro-electrical-mechanical system is a widespread technique that enables batch fabrication of nanopatterns. An incredible sub-10 nm resolution can be achieved by electron-beam lithography, although ion induced surface diffusion prevents the creation of high aspect ratio structures.

Focused Ion Beam (FIB) is a technology for high-resolution imaging and nanofabrication that surmounts such difficulty. FIB nano-machining is based on four major concepts: milling, implantation, ion-induced deposition, and ion-assisted etching of materials.

Figure 2.2 presents a schematic representation of FIB milling. Commercially available systems employ liquid metal - typically gallium (Ga) - to produce a highly focused ion beam and sputter the surface of a specimen. In this scheme, liquid Ga wet a tungsten gun and are extracted from the apex of a Taylor-Gilbert cone by field emission. The beam is limited by an aperture, accelerated to 5-30 keV and focused by electrostatic lenses down to 10 nm feature size. Scanning coils deflect the beam in the X and Y directions so it raster scans over the surface of the sample. Quantitative aspects of sputtering depend on several particularities, including type and crystal orientation of the material milled; nature and incidence angle of the ion beam. Precise control of these parameters can minimize redeposition and amorphization problems, allowing to mill high aspect ratio structures.

An FEI Strata 235 dual beam Focused Ion Beam belonging to the Lawrence Berkeley National Laboratory was chosen to fabricate the DTs. The probe was placed on the sample holder with the tip pointing towards the beam to facilitate the alignment of the milling area with the center of the tip. The cut was done by creating a 0.4 $\mu$m wide by 10 $\mu$m long rectangle over the tip and several 0.4 $\mu$m wide by 10 to 30 $\mu$m long rectangles over the cantilever from tip to base.
2.1.3 Probe Coating Method

Physical Vapor Deposition is the most widely used technologies for thin film coatings. One of its earliest techniques is thermal evaporation, in which the film is formed by atoms evaporated from a source material and transported through vacuum. This technique is cheap and directional yet presents high impurity.

Sputtering technique emerged to overcome the poor coverage limitation exhibited by thermal evaporation. Figure 2.3 shows the principle of operation of magnetron sputtering technique. An ultra-high vacuum chamber is filled with an inert gas - usually Argon (Ar) - which is ionized by an electrical field generated between an anode and a cathode plate. The Ar⁺ ions accelerate towards the cathode colliding with a target material. If the incident particles inelastically transfer enough energy to surpass the surface binding energy, target atoms are brought into vacuum. The sputtered particles travel to the substrate in ballistic regime and grow a film over the surface of the substrate. Magnetron sputtering is the most common sputtering technology as it increases sputtering efficiency due to the introduction of a simple artifact: a magnetic field that is employed to confine the charged particles close to the target surface.

Evaluating strengths and weakness of both techniques, sputtering clearly prevails over thermal evaporation. The former grants better uniformity, larger coverage area, and more con-
Figure 2.3 – Schematic view of a Magnetron Sputtering. The Ar$^+$ plasma is represented in purple and the field line in black. The inset shows the collision of an Ar$^+$ ion with an atom from the target material.

trolled deposition rate - down to one atomic layer per second. On the other hand, this method presents higher surface damage and poorer directionality.

Ferromagnetic coating of ten ScanAsyst-Air-Hr probes - five conventional MFM tips (probe 1 - 5) and five Dual MFM tips (probe 6 - 10) - was executed by an AJA ATC Orion 8 UHV Magnetron Sputtering System belonging to the Nanometric Conformation Laboratory (UFRGS). Two different geometries were explored:

1. Probes were positioned at the center of the substrate plate with the tip pointing towards the target;

2. Probes were positioned at the edges of the substrate plate with the tip axis at an approximately 60° angle towards the substrate plane. The side of the probe that faced the target was exposed to the sputtered atoms and became magnetic; the other side remained shadowed receiving reduced or none magnetic coating.

Probes 1 and 2 followed the first deposition geometry to produce a uniformly coated tip alike commercially available ones. Probes 3 through 5 followed the second geometry, as to investigate the influence of unequal coating on magnetic imagery. Probes 6 through 10 followed the second geometry to generate DTs with one magnetic coated tip and one essentially uncoated tip. Mechanical stress between the 35 nm Py layer and the Si$_3$N$_4$ probe was reduced by sputtering a 5 nm intermediate layer of Gold (Au).
Table 2.2 – Py and Au Sputtering parameters.

<table>
<thead>
<tr>
<th></th>
<th>Pressure (mTorr)</th>
<th>Power (W)</th>
<th>DCV (V)</th>
<th>Time (s)</th>
<th>Rate (Å.s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Py Pre-Sputtering</td>
<td>2</td>
<td>150</td>
<td>377</td>
<td>180</td>
<td>-</td>
</tr>
<tr>
<td>Au Pre-Sputtering</td>
<td>2</td>
<td>30</td>
<td>350</td>
<td>180</td>
<td>-</td>
</tr>
<tr>
<td>Py Sputtering</td>
<td>2</td>
<td>150</td>
<td>379</td>
<td>350</td>
<td>1</td>
</tr>
<tr>
<td>Au Sputtering</td>
<td>2</td>
<td>30</td>
<td>55</td>
<td>55</td>
<td>0.906</td>
</tr>
</tbody>
</table>

Surface contamination of the targets was eliminated by pre-sputtering. The depositions were carried out in an initial chamber pressure of 8.5 × 10⁻⁸ Torr according to the parameters presented in table 2.2.

2.2 Probe Characterization

2.2.1 Morphological Characterization

As previously stated, the shape and the magnetic configuration of the probe dictate its spatial resolution, making it crucial to determine them. Morphological characterization was done by Scanning Electron Microscopy (SEM), a versatile non-destructive analytical technique for microstructure characterization. SEM was combined with FIB technique in order to real-time monitor the cutting step and to verify the shape of the tip at the end of the procedure.

Figure 2.2 presents a schematic representation of SEM. The field emission gun produces a very bright electron beam by applying a strong electrostatic field to a sharp tungsten tip. The beam is accelerated from 1 to 30 keV and focused through field and magnetic lenses. Scanning coils deflect the beam in the X and Y directions so it raster scans over the surface of the sample.

An FEI Strata 235 dual beam Focused Ion Beam belonging to the Lawrence Berkeley National Laboratory was chosen to perform morphological characterization of the probes throughout the cutting procedure.

2.2.2 Magnetic Characterization

Ferromagnetic materials exhibit small regions, or domains, with spontaneous magnetization in different directions. In the presence of an external field, the individual domains of such materials line up in the direction of the field in a state of saturation magnetization as illustrated in Figure 2.4 (a). When this field is removed, a remanence magnetization persists and a coercivity field must be applied to demagnetize the ferromagnet. The current state of null
magnetization can reach a negative saturation by increasing the external field in the negative direction. The sample retrieves the positive saturation state under the influence of a positive external field, passing by a state of zero magnetization. This last step does not retrace the previous path, evidencing a history dependence of the magnetic state in a hysteresis loop. Assessing the hysteretic behavior of the ferromagnetic coating of the manufactured tips is essential to characterize their response.

Vibrating Sample Magnetometer (VSM) is a popular method for extracting information on the averaged magnetic behavior of materials. If available, Superconducting Interference Device (SQUID) may be preferred for this step on account of its higher sensitivity. Figure 2.4 (b) gives a schematic representation of the working principle of a VSM. A sample is mounted on a glass rod and placed in a region of constant magnetic field ($B$) in between an electromagnet. The rod is set to vibrate along an axis perpendicular to $B$, so that a pair of pick-up coils sense the stray field from the sample as an alternating field. The change in magnetic flux causes an electric current in the pick-up coils with the same frequency of vibration and proportional to the magnetization of the sample - a phenomenon known as Faraday’s law of induction. The output of the pick-up coil is fed to a lock-in amplifier used to only select signals at the vibrating frequency.

The Microsense EZ9 Vibrating Sample Magnetometer belonging to the Magnetism Laboratory (UFRGS) was used to estimate the magnetization of the cantilever. This VSM presents a single signal noise of 0.4 $\mu$emu, moment accuracy of 1% and repeatability better than 0.5% at the conditions applied in this work.
In Operando Characterization

A Bruker Multimode 8 Atomic Force Microscope belonging to the Magnetism Laboratory (UFRGS) was employed to characterize probe performance with a reference magnetic sample and with soft magnetic nanohills and nanorings. The microscope was operated at room temperature under the electrical and magnetic lift modes for phase and frequency. The reference sample consists of a metal-evaporated videotape and was provided by Bruker together with a small magnet for probe magnetization.

Nanohills are self-organizing Cobalt (Co) nanostructures conformed on an aluminum (Al) surface that may help elucidate the influence of textured surfaces on magnetic interactions. A sample containing such structures was kindly lent by professor Sabrina Nicolodi. A 0.25mm thick Al foil was electropolished by a 25:100 volumetric mixture of HClO$_4$ and C$_2$H$_5$OH at 20 V for 2 minutes to reduce surface roughness. The foils were then anodized in 0.3M oxalic acid at 40 V for 1 hour to create a highly ordered hexagonal template. Metal pickling in a solution of 0.2M H$_2$Cr$_4$O$_4$ and 0.4M H$_3$PO$_4$ for 1 hour at 65°C temperature was performed in order to remove the Al$_2$O$_3$ cover. A 15 nm Copper layer and a 2.5 nm Cobalt layer was thermal evaporated at a base pressure of $10^{-7}$ Torr over the precursor matrix. Finally, the sample was submitted to a thermal treatment for 1 hour at 400°C to grow Co nanostructures over the nanohills. Magnetic characterization of the Co nanostructures was done by a VSM at room temperature. The system presented an in-plane coercivity of 70 ± 1 Oe and an out-of-plane coercivity of 490 ± 5 Oe.

Asymmetrical nanorings are potential contacts for spin injection in semiconductor devices. A structure consisting of two concentric asymmetric nanorings can have its vortex chirality switched from parallel to antiparallel, which can be used as a high-density and stable memory system. Single Py asymmetric nanorings were produced in the International Iberian Nanotechnology Laboratory and were kindly lent by Professor Alexandre Viegas (AVILA et al., 2015). The sample was created by sputtering the following films: 5 nm Ta, 10 nm permalloy, 5 nm Ta, 5 nm Ru, and 15 nm TiWN. The sample was covered by approximately 100 nm of resist and the nanorings were tailored by e-beam lithography. The nanorings were studied by micromagnetic calculations and are expected to present two characteristic states, one with a head-to-head and a tail-to-tail domain wall, a saturated state called the onion state and, another with a unique circular domain, a remanent state called the vortex state.
3 RESULTS

3.1 Probe Characterization

3.1.1 Scanning Electron Microscopy

Figure 3.1 shows probe 7 before (a) and after (b) the cutting procedure and probe 8 before (c) and after (d) cutting the tip. Both probes presented flawless well-centered tips producing high standard DT whose height difference are 280 nm and 715 nm respectively. Figure 3.1 (d) inset presents the cut near the cantilever base, evidencing the creation of two independent cantilevers.

Figure 3.2 shows probe 9 and 10 before (a and c) and after (b and d) the cutting procedure. Both probes presented off-centered blunt tips even before any usage, leading to a flagrant decline in the quality of the DTs. The height difference is of 1860 nm and 790 nm for probes 9 and 10, respectively.

Figure 3.1 – SEM of probe 7 before (a) and after (b) the cutting procedure and probe 8 before (c) and after (d) cutting the tip.
3.1.2 Vibrating Sample Magnetometry

Figure 3.3 presents the hysteresis curve of probe 10 with a parallel (a) and a perpendicular (b) magnetic fields. The probe was mounted on the glass rod with its long axis parallel to the vibration direction, i.e., perpendicular to the applied field. The Si$_3$N$_4$ probes are made of a diamagnetic material, therefore, the main contribution to the hysteresis curve comes from the ferromagnetic coating. The first measurement was executed preserving probe 10 intact. The second measurement was executed after the cantilever was removed from the probe.

Figure 3.3 (a) reveals that the hard axis of magnetization is parallel to the probe axis. This is expected since it takes more energy to reverse the magnetization along the probe axis. The hysteresis loop presents two phases, indicating that the top and the side of the probe were coated. Figure 3.3 (b) confirms that the easy axis is perpendicular to the probe axis. The measurements with and without cantilever are similar and present a coercivity of $10 \pm 1.5$ Oe and $5 \pm 1.5$ Oe for in-plane and out-of-plane fields. These coercivity values indicate the achievement of low moment coating, as intended.
3.1.3 Atomic Force Microscopy

Cantilever response to the drive in signal was studied before and after magnetic coating. This was done by loading the probes on the AFM and sweeping the drive signal frequencies from 50 kHz to 300 kHz. Manual tuning allows the user to select a range of frequencies - down to 10 kHz - within which the microscope will select the best drive frequency. Double gaussian whose centers are less 10 kHz apart are indistinguishable for this procedure.

The results revealed that both CTs and DTs presented single and double gaussian around their resonant frequencies and that the magnetic coating step did not significantly modify the cantilever response. Ideal CTs should present a single gaussian with large amplitude centered at the cantilever resonant frequency, while ideal DTs should reveal a double gaussian with large amplitude centered at the tips resonant frequencies. In a naive analysis, CTs that exhibit double gaussian around their resonant frequencies would be classified as damaged and DTs with single gaussian demonstrate that both tips possess the same frequency.

Magnetic coating slightly altered the separation distance between the double gaussian centers of the DTs. This can be easily understood by examining equation 1.3. In the case in which the separation distance decreased, the mass from the magnetic layer gained by the magnetic tip probably balanced the mass gained by the longer non-magnetic tip, matching their resonant frequency. In the case in which the separation distance increased, the mass of the magnetic layer gained by the magnetic tip greatly surpassed the difference in mass before the sputtering.

Figure 3.3 – VSM of probe 10 with a parallel (a) and a perpendicular (b) magnetic fields with and without cantilever. The lines are guides to the eyes.
Due to the low number of probes fabricated, no direct correlation could be established between CTs/DTs and single/double gaussian and all resonant frequencies were set close to the center of the largest gaussian regardless of whether the cantilever response presented single or double gaussian. Accordingly, MFM was operated only on lift-mode.

Figure 3.4 presents the topographic view of the reference magnetic sample (magnetic tape) taken with: (a) reference probe, (b) commercial probe; and the tailored DTs represented by (c) probe 7, (d) probe 8, (e) probe 9, and (f) probe 10. Images taken with the reference probe can be found in the Bruker Manual in grayscale and were false-colored in Adobe Photoshop to a "warm" colormap for comparison. Images taken with the commercial probe and probes 1 to 10 were analyzed with the open source software Gwyddion version 2.5 and are displayed in warm colormap.

Figure 3.4 (a) reveals that the surface of the tape is flat with grains less than 60 nm height. Figure 3.4 (b) shows an image with clear features, but inaccurate length scale. Figure 3.4 (c) and (d) shows images with sharp features and adequate length scale, similar to the reference image. This evidences that probes 7 and 8 have performances comparable to the ones from high standard Bruker probes. Figure 3.4 (e) and (f) shows images with adequate length scale, although with dull features. This artifact is typical of blunt tips, indicating that probes 9 and 10 are damaged.

Figure 3.5 exhibits the magnetic view of the reference magnetic sample taken with: (a) reference probe, (b) commercial probe; and the tailored DTs represented by (c) probe 7, (d) probe 8, (e) probe 9, and (f) probe 10. Figure 3.5 (a) displays the magnetic stray field in the reference sample inferred from variations in the frequency of cantilever oscillation given in hertz (Hz). Figure 3.5 (b) to (f) display the same information inferred from variations in phase of cantilever oscillation given in degrees (deg). Even though scales are not comparable, image qualitatively is: bright and dark tracks represent recorded bits in the magnetic tape. Ideally, the magnetic pattern would be composed of perfectly straight lines and the magnetic profile would be a square wave. However, limitations in the fabrication technology left some disorder in the domains between tracks and rounded the magnetic profile to a sinusoidal wave.

Figure 3.5 (a) depicts a region of random magnetization (center) between diagonal (top) and vertical (bottom) tracks of recorded bits. Figures 3.5 (b) and (e) shows diagonal and horizontal tracks with clear features, but not so distinct domains as in the reference image. Figure 3.5 (c) and (d) shows sharp features and conspicuous disorder in the domains between tracks.
Figure 3.4 – Topographic characterization of the reference magnetic sample taken with: (a) reference probe, (b) commercial probe; and the tailored DTs represented by (c) probe 7, (d) probe 8, (e) probe 9, and (f) probe 10.
Figure 3.5 – Magnetic characterization of the reference magnetic sample taken with: (a) reference probe, (b) commercial probe; and the tailored DTs represented by (c) probe 7, (d) probe 8, (e) probe 9, and (f) probe 10.
Figure 3.6 – Normalized magnetic profiles of the highlighted regions of Figures 3.5 (b) to (f).

Even though phase shift measurements are less sensitive to stray field variations than frequency measurements, the image obtained is similar to the reference one. This indicates that probes 7 and 8 hold tips with impressive sharpness and magnetization. Figure 3.5 (f) shows very dull features, indicating that probe 10 was not correctly magnetized.

Figure 3.6 (a) and (b) presents the magnetic profile of the region highlighted by a 20 pixels width white bar in Figures 3.5 (b) to (f), respectively. All profiles were normalized by their maximum and aligned by setting the first trough to zero. A valuable profile would present a periodic wave with a constant large amplitude, as seen in the profiles taken by probes 7, 8 and 9. A valueless profile would not present these characteristics due to flaws in the scanned region or in the probe. Such is the case of the profile taken by the commercial probe and probe 10.

3.2 Soft Magnetic Structures

3.2.1 Magnetic Nanohills

Figure 3.7 reveals the topographic view of nanohills taken with probe 3 (a and b) and probe 9 (d), and with SEM (c, e and f). Figure 3.7 (a) and (e) evidence that some regions of the sample surface are covered with undesired particles originated during the thermal treatment, clusters of anodized material or defects in the structure due to the metal pickling process. Figure 3.7 (b) depicts a region with undamaged surface. It can be seen that the grown structures present hexagonal symmetry with a diameter in the range from 160 to 180 nm and height from 30 to
Figure 3.7 – Topographic view of nanohills taken with probe 3 (a and b), and probe 9 (d). Scanning Electron Microscopy of the sample at different scales.
40 nm. It is also noticeable that each building block (nanohill) possess a small incision on its surface. This same pattern can be perceived on Figure 3.7 (d), although its dull contours suggest probe-based dilation, an image artifact caused by blunt tips. Figure 3.7 (c) depicts a SEM image of a region free from "debris" carefully chosen from Figure 3.7 (e). The hexagonal structures have a diameter from 150 to 160 nm, which is compatible with the AFM measures. Figure 3.7 (f) exhibits a superficial view of the sample in a scale similar to the scale from Figures (b) and (d). The hexagonal structures are conspicuous; though, its fine details are imperceptible.

Figure 3.8 exhibits the magnetic view of the nanohills taken with probe 3 (a and b) and with probe 9 (bottom c and d). Figure 3.8 (a) indicates that the hills, as well as the undesired particles seen in Figure 3.7, are magnetized. The structure no longer presents a hexagonal symmetry, as the magnetic domain of the hexagon center joined the domain of one of the hills. Nonetheless, the structure is uniformly magnetized, so each element possesses a single domain. Figures 3.8 (a) and (b) present a larger effective pixel size when compared to 3.7 (a) and (b), which is a typical effect of magnetic images taken from small scan areas - in this case, 330x330 nm². Each pixel depicts the interaction force between a finite volume of the sample surface and a finite volume of the magnetic sensitive part of the tip, decreasing the spatial resolution to the range of this interaction and creating a pixelation effect on the image (see Figure 1.5). Such was not observed in Figure 3.5 because its spatial-resolution is not limited by the number of lines per scan.

Figure 3.8 (d) was taken with the same parameters as Figure 3.8 (b). Along most of the scan, the pattern observed in Figure 3.7 (d) is concealed by noise, except on a narrow band where the acquire signal resembles the one from 3.7 (b). This was seldom detected exclusively on magnetic scans performed by probe 9. Figure 3.8 (c) compares the topographic view taken with probe 3 (top) with a noiseless magnetic view taken with probe 9 (bottom). Since such bands present an excellent spatial resolution and no pixelation effect, we can conclude that such signals originate from topographic or electrostatic interactions with the specimen. Thus, probe 9 presented mixed magnetic and topographic signals, which makes it inappropriate for the characterization of these structures.

3.2.2 Magnetic Nanorings

Soft magnetic nanorings were analyzed in lift-mode with probes 1, 3, 7 and 8. In all measurements, phase and amplitude signal from the first pass were employed to infer the to-
Figure 3.8 – Magnetic view of nanohills taken with probe 3 (a and b), and probe 9 (d). Images taken with probe 9 display a narrow band as shown in Figure 3.8 (d) and bottom of Figure 3.8 (c) which resembles the topographic view taken by probe 3 and presented at top of Figure 3.8 (c)).
pography of the sample and phase signal from the second pass was employed to infer the stray field. From each image, one ring was selected and two profiles were mapped in order to compare the measured signals.

Figure 3.9 (a and b) presents the topographic view of Py nanorings acquired with probes 1 and 8, and Figure 3.9 (c and d) shows topographic profiles 1 and 2 extracted from the lines indicated in Figures 3.9 (a and b). All profiles were traced from the top to the bottom of the image. Figure 3.9 (a) and (b) reveals that the surface of the ring is flat and is covered with rounded particles of 2 to 10 nm height. The height of the ring was measured as the distance from its base to its top as being 120 nm and 118 nm for profile 1 and 2 of Figure 3.9 (c) and 115 nm and 123 nm for profile 1 and 2 of Figure 3.9 (d). The internal diameter at half maximum was estimated for both profiles of Figure 3.9 (c) as 512 nm, for profile 1 and 2 of Figure 3.9 (d) as 543 nm and 555 nm, respectively. However, the rings are asymmetric, presenting widths of 288 nm, 313 nm (Profile 1), 354 nm and 271 nm (Profile 2) for the left and right walls of Figure 3.9 (c); and widths of 330 nm, 334 nm (Profile 1), 303 nm and 374 nm (Profile 2) for the left and right walls of Figure 3.9 (d).

Figure 3.10 (a and c) presents the topological view and the magnetic view of Py nanorings extracted by phase information and acquired with probe 1. Phase information encodes difference in heights or of materials into a contrast map, where high signal indicate a change of height or material. Figure 3.10 (a) shows dark and light approximately concentric rings. The light rings indicate a change of height, whereas the dark rings represent the surface of the rings. Small rounded particles can be perceived on the surface of the rings, which seems flat. Figure 3.10 (b and d) shows normalized phase and magnetic profiles 1 and 2 extracted from the lines indicated on Figures 3.10 (a and c). The dashed gray lines on Figure 3.10 (b and d) indicate the half maximum height of the rings estimated by the amplitude signal. Large peaks are observed in Figure 3.10 (b) due to the transition from the substrate to the top of the ring on profile 1, whereas small peaks are observed due to the ring-substrate transitions - the reverse can be noticed on profile 2. Figure 3.10 (c) reveals asymmetric rings with uneven height, where the down part of the ring (closer to the bottom of the image) returns a stronger signal. This observation is confirmed by Figure 3.10 (d). The left wall of profile 1 - upper part of the ring - presents a phase of only 0.7 of the highest signal from the right wall. Both signals drop to zero close to the dashed lines, i.e., close to sample-ring transitions and rise to a phase of 0.3 in the region corresponding to the substrate. The signal corresponding to the ring maintains a phase
Figure 3.9 – Topographic view obtained from amplitude signal of the nanorings taken with probes 1 (a) and 8 (b). Height profiles 1 (solid line) and 2 (dotted line) extracted from Figure 3.9 (a and b) are shown in Figure 3.9 (c and d), respectively.
Figure 3.10 – Topographic and magnetic view obtained from phase signal of the nanorings taken with probes 1 (a and c). Normalized phase profile 1 (solid line) and 2 (dotted line) extracted from Figure 3.10 (a and c) are shown in Figure 3.10 (b and d). The dashed gray lines indicate the half maximum height of the rings estimated by the amplitude signal of Figure 3.9 (c).

Figure 3.11 (a and c) presents the topological view and the magnetic view of Py nanorings extracted by phase information and acquired with probe 8. Figure 3.11 (a) shows dark and light approximately concentric rings with small rounded particles in agreement with the results from probe 1. Scar defects can be observed on the ring-substrate transition. These artifacts were also noticed on images acquired by another DT, suggesting that this may be caused by the magnetic tip. Figure 3.11 (b and d) shows normalized phase and magnetic profiles 1 and 2 extracted from the lines indicated on Figures 3.11 (a and c).
Figure 3.11 (c) reveals asymmetric rings with uneven height, where the down part of the ring (closer to the bottom of the image) returns a stronger signal. This observation is confirmed by Figure 3.11 (d): the right wall of profile 1 - bottom part of the ring - presents a phase of 0.76 of the highest signal from the left wall. Both signals drop to zero close to sample-ring transitions and rise to a phase of 0.54 and 0.71 in the region corresponding to the substrate of profiles 1 and 2. The signal corresponding to the ring maintains a phase larger than its neighboring regions, indicating that the magnetization of the ring differs from the substrate. Stray fields on the top of the ring appear larger on the edges of the ring, presenting a reduction of 0.11 to 0.2 on the interior of the ring when compared to the edge. The left side of profile 2 exhibits a rounded magnetization, evidencing that the ring magnetization is not uniform. This result was supported by the measurements with probe 7 and seems compatible to remanent vortex states in nanorings in the literature (DENNIS et al., 2002; YANG et al., 2011). Further investigation must be applied with variable in-plane and out-of-plane external magnetic field to determine the magnetic state of the nanorings in remanence and saturation regimes.
Figure 3.11 – Topographic and magnetic view obtained from phase signal of the nanorings taken with probes 8 (a and c). Normalized phase profile 1 (solid line) and 2 (dotted line) extracted from Figure 3.11 (a and c) are shown in Figure 3.11 (b and d). The dashed gray lines indicate the half maximum height of the rings estimated by the amplitude signal of Figure 3.9 (d).
4 CONCLUSIONS

Spintronics is a field that exploits overlooked spin properties for classical and quantum computations. This work implements a high-resolution method to accurately manipulate and characterize such soft magnetic devices - a technique called Dual-Tip Magnetic Force Microscopy.

Ten MFM probes were fabricated by coating Bruker AFM tips with a soft magnetic material (Py). According to VSM measurements, the probes presented a coercivity of $10 \pm 1.5$ Oe and $5 \pm 1.5$ Oe for in-plane and out-of-plane applied fields. This ensures that soft magnetic coating was achieved.

Five Dual-tips were manufactured by cutting Bruker AFM tips. Flawless well-centered probes produced high-quality DTs while off-centered blunt probes generated low-quality DTs. The height difference between tips was higher than intended, indicating that the cutting process needs to be improved on future works. In fact, only probe 7 attained an acceptable morphology with sharp tips 280 nm apart. The worse case observed was probe 9 which possessed serious manufacturing defects. The magnetic tip of this probe became, at least, 1860 nm distant from the sample surface. At this range, it was not expected to strongly interact with the sample and all the signal acquired from this probe can be presumed to originate from the other tip. Besides, with this height difference, the magnetic tip was not expected to shadow the topographic tip such that both tips were magnetically coated.

Flawless fabricated DTs and CTs presented an equally or superior performance than commercial MESP probes, producing topographic and magnetic images with sharp features and high contrast with reference and unknown samples. This indicates that MFM tips can be in-house fabricated at a considerably lower cost than commercially available MFM probes.

Co nanohills were analyzed with a CT, probe 3, and a damaged DT, probe 9. SEM and AFM measurements show a structure with hexagonal symmetry, whose surface is covered with undesired particles originated during the fabrication process. Probe 3 generated a topographic image with sharp contrast displaying fine details imperceptible in the SEM image. Magnetic information obtained with probe 3 revealed a pixelation effect. Probe 9 generated a topographic image with dull contrast and a magnetic image with mixed signals. This evidences that the factory defects of this probe invalidated the measurements performed by it.

Py nanorings were analyzed with CT and DT. Topographic information was successfully
extracted from amplitude and phase modes. The information obtained in phase mode at a 30 nm height distance differs significantly from the topographic information obtained in phase mode. This demonstrates that the signal acquired is indeed magnetic.

The measurements indicate that the surface of the ring is flat and is covered with rounded particles of 2 to 10 nm height. The height of the ring was measured as the distance from its base to its top as being between 115 nm and 120 nm. The measurements also reveal an internal diameter between 512 and 555 nm with asymmetric walls ranging with up to 83 nm of difference in the same profile. Magnetic phase information shows an uneven signal intensity from the nanorings with a drop of up to 0.3 in the normalized signal when comparing both sides of the ring. Little information about the stray fields on the top of the ring was detected by the CT. On the other hand, DTs measured a reduction of 0.11 to 0.2 on the normalized signal from the interior of the ring when compared to the edges. The results obtained by the DTs enable us to identify the nanorings as being in the remanent vortex state.

Fabrication of more probes is necessary to improve the characterization of both CTs and DTs. Long term perspectives on this work include developments in the AFM Multimode8 as, for example, equipping the microscope with a system to perform MFM measurements with variable external magnetic field. This would allow us to characterize samples not only in the remanent but also in saturation states.
REFERENCES


