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V-shaped domain wall probes for calibrated magnetic force microscopy

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Abstract— Magnetic force microscopy qualitatively resolves stray magnetic fields, but its fundamental flaws include limited quantitative analysis, and difficulties in measuring samples with heterogeneous magnetic areas. We propose a custom-made domain wall probe (DWP) with a V-shaped magnetic nanostructure on one face of a non-magnetic probe, which behaves as a low moment probe with high coercivity to reduce magnetic switching in the presence of strong stray fields. The performance of the DWP is compared against commercial standard and low moment probes with different approaches to quantify resolution from striped domain structures of a thin reference film. The reference sample is also used to calibrate the three probes by acquiring the tip-transfer function (TTF) from a Fourier transform approach. The calculated TTF is used to predict the MFM response from a test permalloy nanostructure and compared to experimental results.

Index Terms – domain wall probes, magnetic force microscopy, magnetic probe calibration, quantitative MFM, tip-transfer function

I. INTRODUCTION

Conducting reliable quantitative measurements of spatially resolved magnetic fields on the nanoscale is beneficial for a number of industries, ranging from automobile manufacture [1] to bio-medicine [2]. Although such measurements are available on a macroscopic scale, a universal calibration method for measuring nanomagnetic properties remains unresolved. A number of approaches have been reported in recent years such as superconducting quantum interference devices [3], or nano-Hall sensors [4]; however, they often require time-consuming sample preparation or specialized methods of operation [5].

For these reasons, magnetic force microscopy (MFM) is well placed to quantitatively determine nanoscale magnetic fields due to the technique's high spatial resolution (<10 nm) [6], ease of use in standard conditions, and high abundance in research and

industrial environments. Methods involving nanoscale coils [7], Hall sensors [8] and electron holography [9] are some of many proposed in literature to calibrate magnetic probes. However, MFM is still considered a qualitative technique as probe dimensions and properties wildly vary within and between commercial batches, and calibration values for the same probe can vary depending on the dimensions of samples being studied [7], [10].

Here we use a calibration technique based on imaging a reference film with a strong perpendicular magnetic anisotropy (PMA) and well known parameters such as domain shape, magnetization saturation and anisotropy constant. Using the reference film and a fast Fourier transform (FFT) approach, it is possible to extract the tip-transfer function (TTF), which describes the properties of an MFM probe without any assumptions of its geometry or magnetization [11], [12]. From a probe's TTF and a micromagnetic simulation of studied sample, quantitatively predicting the MFM becomes possible. Comparable output from results *in silico* and from experimental measurement is the ultimate test for a validated calibration technique.

The TTF model is assessed by calibrating different probes and simulating their responses to imaging a permalloy (Py) nanostructure. The probes studied are two commercially available probes, and a custom-made domain wall probe (DWP), which has a V-shaped magnetic nanostructure on a single face of the probe's pyramidal tip.

The DWP's nanostructure behaves similarly to other planar ferromagnetic architectures [13], [14], [15]. Thus, the probe is expected to behave as a low moment probe from its localized stray field at the probe apex, but with a higher coercivity due to its thicker magnetic coating and shape. The field distribution from a domain wall (DW) is expected to be different to the stray field of commercial probes, and thus represents a greater challenge for the TTF method.

The DWP's magnetic properties are compared against: (i) a Nanosensor PPP-MFMR standard moment probe (SMP), from which the custom probe was fabricated; (ii) an NT-MDT MFM_LM low moment probe (LMP).

Throughout the present work the quantification of spatial resolution and sensitivity is discussed from a number of methods. This includes introducing a simplistic method to quantify resolution for different probes and comparing it with established techniques (i.e. TTF and the edge spread function). These methods can all be performed from the same reference film images, thus providing useful insights of the probe's properties without the use of mathematically and experimentally complex methods [16], [17].

II. EXPERIMENTAL

A. Probe Fabrication

DWPs were fabricated using commercially available probes (Nanosensors™ PPP-MFMR AFM [18]) by Ga-etching the magnetic CoCr coating [19] (thickness (t) $\approx$ 30 nm) with a focused ion beam (FIB) system; forming a V-shape structure on one face of the pyramidal tip. Fig. 1a shows an SEM image of the DW probe with the lithographed V-shape alongside its schematic drawing (b). The nanostructure's arms are 7.78 μ m in length (l) by 400 nm in width (w), and they meet at $\sim 40^\circ$.

Figure 1. (a) SEM image of a DWP fabricated from a Nanosensor MFMR probe. (b) Schematic diagram of the magnetic nanostructure. MFM images from the same area of Co/Pt film with SMP (c), DWP (d) and LMP (e). (f) Single pixel-width line profiles in locations indicated for all probes – note the phase changes have been offset at zero for easy comparison. White profiles represent the areas analyzed by edge spread function in results.

B. Reference and Test sample

The reference sample is a sputtered [Co(0.4nm)/Pt(0.9nm)]_N multilayer where N=100 [20]. The interface anisotropy of the Co/Pt interfaces provides a PMA of $K = 0.52$ MJ/m³ which competes with the shape anisotropy of the thin film. Thus the magnetization collapses into a multi-domain state at remanence, forming the well-known labyrinth pattern with neighboring up and down domains. The average domain width is 180 nm but due to a domain transition width of ~ 20 nm and the non-regularity of the domain arrangement, the sample contains lateral features from about 20 nm up to the scan size of 5 μ m.

An L-shaped Py nanostructure, of $t = 25$ nm and $w = 100$ nm was used as a test sample to validate the TTF approach. It has areas with low/high stray field, with particularly intense field at the structure's apex[21].

C. Measurement Techniques

Probes were magnetized *ex situ* with a Nd magnet (~ 0.4 T) and standard two-pass tapping mode MFM was performed using the Dimension Icon scanning probe microscope (Bruker, Massachusetts, USA) to map the topography and magnetic distribution of the tested samples. The oscillation setpoint (A_s) for the first pass was 40 nm peak-to-peak, whereas the free oscillation amplitude (A_f) was 50 nm (defined at 3.5 μ m above the surface). For the second pass, the interleave amplitude (A_L) of 42 ± 1 nm was observed for all probes at a 25 nm lift height (h_L) above the surface. The 'true' lift height for MFM measurements was determined from the amplitude sensitivity by manually approaching the surface until signs of contact were seen from the scope. From this, $h_L = 0$ nm was defined and thus the total distance (d_{total}) and true lift height with respect to the surface was determined (i.e. $d_{\text{total}} = h_L + A_s$).

The spring constants of the probes, used in the TTF, were calculated from the tapping mode deflection sensitivity and thermal tuning. These were 4.001, 2.457 and 3.633 N/m for the SMP, LMP and DWP, respectively.

All MFM visualization and data analysis were conducted with a combination of the Gwyddion (CMI, Brno, Czech Republic), and Origin Pro 2016 (OriginLab, Massachusetts, USA) software alongside programs written in-house with MATLAB® (MathsWorks Inc., Massachusetts, USA) and SigMath (TU Dresden, Germany). The edge spread function was calculated from a custom plugin in the image processing software ImageJ (NIH, Maryland, US).

D. TTF and micromagnetic modelling

In the present implementation of the quantitative MFM code within SigMath the TTF describes the probe by quantifying its stray field derivative profile at d_{total} below the physical tip apex. The concept is based on the magnetostatic interaction between sample and probe, which can be expressed in Fourier space by multiplying the sample's effective magnetic surface charges with the above defined TTF according to:

$$\frac{\partial F_z}{\partial z}(k) = -\sigma_{\text{sample}}^*(k) \cdot \frac{\partial H_z^{\text{tip}}}{\partial z}(k). \quad (1)$$

For calculating the TTF, first the effective surface charge map of the reference sample is derived from the measured MFM image by considering the saturation magnetization value, domain transition width and film thickness. Then the TTF is computed in Fourier space by deconvolving the MFM image and surface charge map by means of the Wiener invert filter [20].

With a calibrated tip, the MFM signal of an arbitrary sample can be predicted quantitatively once the effective surface charge pattern is known. Here the Py nanostructure is used as a test sample as it possesses a completely different magnetization structure than the reference sample, and is still well defined. The spatial magnetization distribution was calculated with a parallelized micromagnetic solver [22], [23], which

time-integrates the Landau-Lifshitz-Gilbert (LLG) equation by means of a norm-conserving scheme based on Cayley transform, and the volume density of effective magnetic charge ($\rho = \text{div}M$) was subsequently computed. Due to the small film thickness (t_f) the surface charge density can be approximated as $\sigma = t_f \cdot \rho$. The surface charge map is then convolved with the previously obtained TTF to produce a simulated MFM image, which is quantitatively compared with experiment.

III. RESULTS

A. Reference film characterization

The same $2.5 \times 2.5 \mu\text{m}^2$ area of the Co/Pt film was imaged with the two commercial probes and the DWP (Fig. 1 c-e). The SMP showed great ability to resolve domain structures and resulted in $\sim 17^\circ$ range in phase change ($\Delta\phi$). This type of probe is used for measuring strong MFM signals because of its thick coating and design (i.e. optimized spring constant, coercivity and remanent magnetization) [18]. From qualitative comparison the LMP image sharpness and range is reduced compared to the SMP image, i.e. $\Delta\phi(\text{LMP}) = 0.19 \times \Delta\phi(\text{SMP})$ (as calculated from a full area histogram) demonstrating the LMP's smaller stray field.

Despite the reduced area of magnetic coating on the DWP, the quality of the image is comparable to the LMP, with miniscule reduction in sharpness. The line profiles in Fig. 1f appear near-identical despite the differences in magnitude seen on the color scale (Fig. 1 d and e), which indicates higher sensitivity for the DWP. This is because the line profiles are a localized region which are coincidentally similar, but DWP has a significantly greater phase contrast than LMP across the full scan area ($\Delta\phi(\text{DWP}) = 0.29 \times \Delta\phi(\text{SMP})$). Though this is an indirect comparison due to differences in k and Q between LMP and DWP.

The comparable sensitivity, despite less amount of magnetic material, can be attributed to its strongly localized stray field generated by the DW at the probe's apex. The thickness of the nanostructure accounts for the loss of coating area, when compared to the LMP, to give higher sensitivity.

LMPs can switch magnetic orientation when imaging hard magnetic samples such as the Co/Pt film. This can lead to distorted images (Fig. 2). All three images shown were from the same LMP but with different scanning parameters: the left hand image is as seen in Fig. 1e with “manually optimized” scanning parameters, i.e. no switching is observed; the central and right-hand images were obtained with “software optimized” parameters, at lift heights 75 and 25 nm respectively.

Thus it is possible to image a film with large PMA using a commercial LMP, however optimization of scanning parameters is required; this requires further proficiency and time compared to using software-optimized parameters. On the other hand, switching

artefacts were never seen for the DWPs, suggesting that it is more reliable and simpler than commercial LMPs in operation. All further comparisons of probes are from the manually optimized conditions, as seen in Fig. 1, including the non-switched LMP for an impartial quantification of magnetic properties.

Figure 2. Low moment probe in manually optimized conditions to prevent switching (left), and automated parameters at 75 nm (center) and 25 nm (right) lift heights above the surface.

B. Magnetic probe calibration

The TTFs for the three probes are summarized in Fig. 3. As expected, the LMP has stray field values reduced by about a factor of five compared to the SMP. But also the DWP has a similarly reduced TTF peak, classifying it as a low moment probe. The commercial probes are very symmetrical when comparing the x and y cross-sections, whereas the DWP is not. This is attributed to the presence of the DWP nanostructure on a single pyramidal face as opposed to the even four-sided coating of the commercial probes. As the TTF profile samples the magnetic charges on the sample surface during MFM measurement, the sharpness of the TTF peak is a qualitative measure of the probe's lateral resolution. When comparing the TTFs to their peak maximum, the LMP possesses the sharpest peak.

Figure 3. Cross-sectional plots of the real space TTFs for SMP (top), LMP (bottom solid) and DWP (bottom dashed) in x and y directions (burgundy and green respectively).

C. One-dimensional statistical analysis

The resolution quality factor (R_s) is derived from separation chromatography, where it defines the separation of adjacent peaks with respect to their peak widths [24]. R_s is defined as:

$$R_s = \frac{x_{c2} - x_{c1}}{\frac{1}{2}(w_{b1} + w_{b2})} = 1.18 \times \frac{x_{c2} - x_{c1}}{w_{0.5h1} + w_{0.5h2}} \quad (2)$$

where $x_{c2} - x_{c1}$ is the distance between peak maxima on the line profile, and w is the profile base full width (b) and full width at half maximum ($0.5h$) respectively. The two forms in equation (2) are equivalent, with the right hand form preferred for our purposes as the base width is less reliable.

Equation (2) is simple, thus useful as a coarse test for lateral resolution, but the following assumptions are made; the adjacent peaks are Gaussian shaped and have the same areas and standard deviations. [25]. These assumptions are considered oversimplified for asymmetric peaks; however, the striped domains of this reference film are approximately of the same widths and generate similar stray fields, thus the model can be considered relatively accurate. Although there is no physical background to consider profiles from MFM images Gaussian, we have found that Gaussian functions accurately fit the data-sets well for this statistical comparison.

Due to physical processes (e.g. signal attenuation) adjacent peaks are rarely fully resolved if they are near in proximity, leading to a reduction in maximum/minimum phase signals Fig. 4a).

In Fig. 4 (b-c), we demonstrate the R_s method against the edge spread function (ESF), a simplistic ISO method which quantifies image sharpness based on line profile steepness (D_{80-20}), see Ref. [26]. The first trough to peak for profiles in Fig. 1 (indicated in white) were used to calculate the ESF for all probes; the distance between points of defined intensities (20% to 80% is common) was used as a measure of sharpness (Fig. 4c). The results are displayed in Table I.

Table I. Quantification of resolution from the peak resolution equation (R_s) and edge spread function (D_{80-20}) with their expanded uncertainties ($2u$). Best calculated resolution from both methods are indicated in bold.

Probe	$R_s \pm 2u$ (arb)	ESF $\pm 2u$ (nm)
SMP	1.21 ± 0.04	70.8 ± 3.9
LMP	1.13 ± 0.12	70.1 ± 4.4
DWP	1.16 ± 0.06	65.8 ± 3.1

Qualitatively, it appeared that the SMP image was the most resolved, whereas the two other images were more similar. These observations were well reflected in the R_s value, but not in the ESF, which ranked the DWP as the sharpest profile and the SMP the shallowest, contradicting the TTF and qualitative results. The ESF approach uses the image files rather than the raw data, hence the arbitrary intensity units in Fig. 4c, however this should not affect the outcome providing a reasonable color scale was used in the visualization software. We can conclude the model is not a good statistical test for resolution from striped reference materials.

We demonstrate an example of both the qualities and limitations of the peak resolution method in Table I. SMP was quantified as the sharpest from its larger R_s value, also seen in qualitative and TTF results (when considering TTF sharpness and magnitude). However, R_s contradicts the TTF by calculating the LMP is lower resolution than the DWP. This arises from a worse fit for the LMP, as indicated by its expanded uncertainty ($2u$) in the table.

For a more meaningful demonstration of the fit we compared the adjacent R^2 values for the three line profiles; this quantifies the quality of the fit, with a perfect fit defined as $R^2 = 1$. The R^2 indicates a good fit

Figure 4. (a) Labelled schematic of the peak resolution method. (b) The data (black spheres) and fit (red line) of the line profiles from Fig. 1 for the DWP (left), SMP (center) and LMP (right). (c) Edge spread function data (black spheres) and fit (green line) from white line profiles in Fig. 1.

for SMP and DWP (both 0.997), however LMP was considerably worse ($R^2 = 0.978$).

We recommend for a coarse comparison of resolution to use the R_s method over the ESF as it is simple and reliable for probe and/or parameter comparisons on the striped domain reference film. However, caution is advised and the R_s value should be qualified with an $R^2 \geq 0.99$. If R^2 is significantly lower the results are less conclusive and characterized by higher uncertainty.

D. Simulated MFM response

Figure 5a is a color representation of one of many magnetization configurations for the Py structure with tail-to-tail magnetization and thus a DW at the apex. Hence a highly localized surface charge density is seen, as illustrated in Fig. 5b.

By convolving the density map with the three TTFs, phase shift responses expected from the MFM experiment are calculated (Fig. 5c). Due to the lateral extension of the TTF (tens of nanometers), the contrast outside of the Py nanostructure is also expected. The calculations predict a negative symmetrical phase shift signal at the corner of the L-shape; the absolute values are seen in the profiles (Fig 5e).

Figure 5d displays the measured MFM signal. Both LMP and DWP exhibit the expected negative peak in the localized region, and peak height and width compare remarkably well with the predicted equivalents (Fig. 5e right and left panels respectively). In the case of the DWP, the phase shift contrast appears physically “squeezed” to the outer boundary of the of the L-shape corner. This anomaly can arise from: (i) a probe asymmetry larger than derived from the reference measurement; (ii) the probe’s stray field interferes with the remanent domain state of the Py nanostructure.

The SMP contrast resembles an alternative stable remanent state without a DW at the L-shape’s corner (see Ref. [13]). It is likely that the large stray field from the probe moves the DW away from the apex and the signal arises from magnetic flux bending around the corner. The experimental profile from SMP was included in Fig. 5e (left) for comparison (blue dashed profile), and it is expectedly different to the simulated response. The profile shape is much more jagged due to the opposite contrasts in close proximity, with the second trough corresponding to nucleation around the inner corner of the L-shape opposed to the outer apex. This study is a prime example for the necessity of low moment probes for imaging magnetic samples with low coercivity.

IV. CONCLUSION

We experimentally observe that the custom-made domain wall probe performs similarly to the commercial equivalents, behaving as a low moment probe with lower chance of magnetic switching *in situ*. The DW is clearly induced in a highly localized area at the nanostructure’s apex; though initially predicted to improve resolution, we have demonstrated that this is

Figure 5. (a) Modelled magnetization configuration for Py L-shape nanostructure after field was applied at 45° . (b) Effective sample surface charge map with large negative field at the L-shape’s apex. (c) Simulated MFM images alongside experimental results (d). (e) Simulated (left) and experimental (right) line profiles for all probes from the dashed lines in (c) and (d).

not true for this current design. Altering the geometry/dimensions of the nanostructure can be the subject of further study to improve the resolution.

We have adopted a new method for simply and coarsely quantifying resolution with a reference film, and demonstrated that it is more accurate than the ESF method for quantifying image sharpness; and, unlike the TTF, does not require knowledge of the reference sample’s magnetic parameters. However, the success of the fit is imperative to the resolution quality factor. A failure to adhere can produce large uncertainty and reduces the reliability for comparison.

TTFs further confirmed the similarity in sensitivity between the DWP and the LMP and revealed a higher lateral resolution for LMP than DWP. The asymmetry in the TTF for DWP demonstrated its ability to quantify uncommonly shaped probes alongside probes with more typical magnetic coating.

The TTF method was successfully proved to be an appropriate calibration technique. For the LMP and DWP, the qualitative images of the L-shaped Py nanostructure with head-on domains were remarkably similar in both domain shape and magnetic contrast. On the other hand, modelling cannot yet predict the domain perturbation by the SMP’s strong stray field, or DW “squeezing” seen from DWP. However, for the non-perturbing probes, simulations successfully

predicted the MFM response as is evident from the comparative line profiles. Thus, we conclude the TTF method is a well suited calibration technique, providing the correct probe and/or reference material is selected for calibrated measurement of a test sample.

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